Microstructure dependent damage mechanisms studies and modeling on high silicon spheroidal graphite iron

Sujakhu, Surendra

2018

Sujakhu, S. (2018). Microstructure dependent damage mechanisms studies and modeling on high silicon spheroidal graphite iron. Doctoral thesis, Nanyang Technological University, Singapore.

<http://hdl.handle.net/10356/73877>

<https://doi.org/10.32657/10356/73877>

Microstructure Dependent Damage Mechanisms Studies and Modeling on High Silicon Spheroidal Graphite Iron

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A thesis submitted to the Nanyang Technological University
in partial fulfillment of the requirement for the degree of
Doctor of Philosophy

Statement of Originality

I hereby certify that the work embodied in this thesis is the result of original research and has not been submitted to higher degree to any other University or Institution.

I certify that the intellectual content of this thesis is the product of my own work and that all the assistance received in preparing this thesis and sources have been acknowledged.

Date

SURENDRA SUJAKHU

Acknowledgement

I would like to express my sincere gratitude to my supervisors, Prof. Sridhar Idapalapati and Prof. Sylvie Castagne for supervising my PhD research. Their constant guidance, constructive suggestions, motivation and encouragement has always inspired me to step forward in my PhD journey. The research traits that I learned from them during the period of my PhD are invaluable.

I am very grateful to my co-supervisor Dr. Wang Wei for his support and guidance over the years. I would like to thanks him for providing additional exposure to ARTC facilities and resources.

I would like to express my gratitude to my TiROP exchange supervisor Prof. Motoki SAKAGUCHI for his motivation and supervision during the exchange period in Tokyo Tech. I am thankful for letting me work in his lab.

I would like to express my heartfelt appreciation to thesis advisory committee members Prof. Anders Jarfors and Prof. David Butler for their valuable feedbacks and comments to improve my work.

I am immensely pleased to work in collaboration with the research team from Jonkoping University, Sweden lead by Prof. Anders Jarfors. I am deeply indebted to Prof. Kent Salomonsson for his guidance in modeling part. I would like to express special thanks to Dr. Ehsan Ghassemali and Dr. Keivan Kasvayee for collaborating in this research.

I would like to thank oral examination chair Prof. Xiao Zhongmin, and Prof. Fan Hui and Prof. Matteo Seita for providing their sound feedbacks regarding this thesis.

I am thankful to NTU, ARTC and A*STAR for providing me the SINGA scholarship. I appreciate all the help provided by the technical staff in MAE, Ms. Mei Yoke, Mr. Leong Kwok Phui, Ms. Sandy, Mr. Lew Sui Leung and Mr. Tan Ngee Kwan for their support in research matters and friendly environment in Laboratory.

I extend my gratitude to Dr. Bala, Mr. Zhang Jing, Dr. Taureza and Ms. Indira for their encouragement and help during these years.

My sincere regards to all my friends, naming each of them could flood the pages, for motivating me all the way through my PhD journey.

Finally, I am grateful to my parents, Indra and Saraswoti, my sister Indira, my brother Binod, and my wife Yogita for their unconditional love and support.

Abstract

Spheroidal Graphite Irons (SGIs) have graphite particle inclusions in an iron matrix. The matrix structure controls overall mechanical properties, and the graphite morphology plays a vital role in crack initiation and propagation behavior. High silicon Solution Strengthened Ferritic (SSF) SGIs are developed to provide higher strength with excellent ductility. In SSF SGIs, graphite nodules shape has a key role in damage micromechanisms. The graphite nodule growth morphology can go through transitions to form degenerated graphite particles other than spheroidal graphite nodules in SGI microstructure. Additional thermal and mechanical processes influence SGI microstructure affecting mechanical properties and damage micromechanisms. Most of the damage mechanism studies on SGI were focused on the role of spheroidal graphite nodules on the stable crack propagation region. In this work, microstructure and properties of different SGI grades were compared, and EN-GJS-500-14 SSF SGI was further deep cold rolled and thermal cycled to study the effect of these processes on the material microstructure. Tensile and fatigue damage mechanisms were studied in detail to understand the role of different forms of graphite particles in SGI microstructure. The microstructure characterization result and damage mechanisms were formulated in a Representative Volume Element (RVE) model, and then to multiscale SGI material microstructure model.

Microstructure studies and nanoindentation test results showed that the general microstructure of as-cast, deep cold rolled (DCR) and thermal cycled SGI can be characterized by graphite morphology, matrix composition and its phase properties. In DCR process, plastic hardening of the ferrite matrix was obvious. The large plastic flow of the ferrite matrix caused subsurface graphite particles to appear as a surface crack, which must be considered in graphite particles characterization. In thermal cycling process, the graphite-ferrite interface state was the most susceptible region in the microstructure, which needs to be included in microstructure characterization.

In the tensile test, the matrix-nodule interface decohesion and plastic deformation of the ferrite matrix were the dominant damage mechanisms. Less influenced by nodule shape, graphite particles showed decohesion from the ferrite matrix at the overall stress of 400 MPa to 420 MPa, which is close to the yield stress of the material. In a separately performed Fatigue Crack Initiation (FCI) and Fatigue Crack Propagation (FCP) tests, the graphite particle shape plays a decisive role in crack initiation and propagation. In the crack initiation region, degenerated

graphite particles dominated cracks initiation, and in the crack propagation region, the spheroidal graphite-matrix decohesion inducing a crack tip blunting effect was the most frequent damage mechanism in the SGI microstructure. FCI tests exhibited that cracks initiation were either by internal graphite cracking or by decohesion or by a combination of decohesion and internal cracking. A quantitative study of graphite nodules damage revealed that internal cracking and combine damage of most of the graphite nodules with Roundness Shape Factor (RSF) less than 0.5, whereas the spheroidal nodules with RSF higher than 0.9 showed the matrix-nodules interface decohesion. In FCP test, the degenerated graphite particles were mostly fractured, and crack branching was often observed either by main crack kinking towards nearby graphite nodules or by secondary cracks growth toward the main crack. The presence of shrinkage cavities of a size comparable to graphite particles size behaved similar to degenerated graphite particles, initiating microcracks in the ferrite matrix.

Graphite particles were modeled as voids, unbound elastic particles and surface based cohesive interface bound elastic particles in the RVE model. The RVE model with surface based cohesive interface showed a better representation of the SGI microstructure. It enabled graphite particles to be modeled as bound particles until critical stress was reached, which was later modeled as partially debonded graphite particles. X-FEM crack initiation and propagation in the RVE model illustrated requirement of defining multiple enrichment regions in ABAQUS to allow initiation and growth of multiple cracks. Further, a multiscale SGI material microstructure modeling approach was developed for miniature Compact Tension (CT) specimen. The microstructure submodel generated by FE representation of real SGI micrograph well represented the stress and strain inhomogeneity in the microstructure. The inhomogeneous strain in the microstructure model was validated by DIC result, which showed slightly higher strain magnitude at the similar higher strain locations. X-FEM crack initiation and propagation simulation in the complex microstructure model showed the influence of the graphite particles in the crack initiation and propagation. However, the simulation could not be completely converged mostly due to limitations in available X-FEM formulation in ABAQUS. So, it is suggested to use ABAQUS user subroutines to model X-FEM crack growth in complex models.

Table of Contents

Acknowledgement	i
Abstract	iii
List of Figures	xiv
List of Tables	xv
Abbreviations and Symbols	xvii
1 Introduction	1
1.1 Background	1
1.1.1 Cast irons	1
1.1.2 Spheroidal graphite cast iron	3
1.1.3 Microstructure influence on SGI component failure	5
1.2 Research Objectives	6
1.3 Scope	6
1.4 Motivation	7
1.5 Thesis Outline	8
2 Literature Review	9
2.1 Ductile Cast Iron	9
2.1.1 Brief history of ductile cast iron	11
2.1.2 SGI microstructure	12
2.1.3 Graphite nucleation and growth in SGI casting	14
2.1.4 Factors affecting mechanical properties	15
2.1.5 Solution Strengthened Ferritic SGI	18
2.1.6 Fracture of SGI	22
2.1.7 Review of SGI damage mechanism studies	23
2.2 Deep Cold Rolling Process and its Effect on SGI	28
2.2.1 Introduction to DCR process and parameters	29
2.2.2 DCR fundamentals	31
2.2.3 Effects of DCR process on fatigue life improvement	32
2.2.4 DCR challenges in SGI material	34
2.3 Heat Treatment and Thermal Processing of SGI	35
2.3.1 Heat treatment of SGI and its influence on microstructure	36

2.3.2	Effect of high Silicon on heat treatment	36
2.3.3	Thermal cycling of SGI	37
2.4	Extended Finite Element Method (X-FEM)	40
2.4.1	Basic Review	41
2.4.2	Enrichment functions	42
2.4.3	Level set method	44
2.4.4	Numerical integration and convergence	45
2.4.5	X-FEM implementation in ABAQUS	46
2.5	Microstructural Damage Modeling on SGI Material	49
2.5.1	Cell model	49
2.5.2	Real microstructure based model	51
2.6	Summary	51
3	SGI Microstructure Characterization: Effects of Thermal and Mechanical Processes	55
3.1	As-cast SGI Material	55
3.1.1	Mechanical properties	57
3.1.2	Microstructure	57
3.1.3	Nanoindentation phase hardness and elastic modulus	60
3.1.4	Elastic-plastic phase properties	64
3.2	Effect of DCR process on SGI	76
3.2.1	Experimental design and setup	76
3.2.2	Microstructure	78
3.2.3	Nanoindentation phase properties	89
3.2.4	Effective deformation zone on DCR process	91
3.3	Effect of Thermal Cycling on SGI	93
3.3.1	Microstructure study	95
3.3.2	Nanoindentation phase properties	97
3.4	Summary	98
4	Tensile and Fatigue Damage Mechanisms in SGI	101
4.1	Test Specimens and Methods	102
4.1.1	Tensile test	103
4.1.2	Fatigue Crack Initiation (FCI) test	104
4.1.3	Fatigue Crack Propagation (FCP) test	104
4.1.4	Fatigue test for S-N curve	106
4.2	Tensile Damage Mechanisms	108
4.2.1	Crack initiation and propagation	108
4.2.2	Fracture behavior	115
4.3	Fatigue Damage Mechanisms	116
4.3.1	Fatigue crack initiation test	117
4.3.2	Fatigue crack propagation test	131
4.3.3	Fracture toughness	142
4.3.4	Stress-life (S-N) curve for EN-GJS-500-14	142
4.3.5	Effect of thermal cycling on FCGR	144
4.4	Damage Criteria	146

4.4.1	Tensile damage criteria	146
4.4.2	Fatigue damage criteria	148
4.5	Summary	150
5	SGI Microstructure Modeling and X-FEM Crack Simulation	153
5.1	Background: ABAQUS X-FEM	155
5.2	Homogenized RVE Approach	155
5.2.1	Model geometry and boundary conditions	156
5.2.2	Material properties and constitutive equations	158
5.2.3	Stress and strain evolution	163
5.2.4	Interface properties and decohesion	168
5.2.5	Crack initiation and propagation	172
5.3	Microstructure Submodeling of SGI Material	178
5.3.1	SGI microstructure model using OOF2	180
5.3.2	Material properties, constitutive equations and boundary conditions	182
5.3.3	Stress and strain evolution	183
5.3.4	DIC validation of strain distribution in the microstructure model	185
5.3.5	X-FEM crack growth simulation	187
5.4	2D Approximation of 3D Microstructure: Possible Misinterpretation	195
5.5	Summary	196
6	Conclusions and Future Works	199
6.1	Conclusions	199
6.2	Major Contributions	203
6.3	Future Works	205
	Author's Publications	207
	Bibliography	209

List of Figures

1.1	Microstructure of different types of cast irons a) Gray cast iron/Flake graphite cast iron [1] b) Compacted graphite cast iron [2] c) Ductile cast iron/Spheroidal graphite cast iron [2].	2
1.2	Deep-etched scanning electron micrographs showing three-dimensional shape of the graphites [3] a) Flake graphite b) Compacted graphite c) Spheroidal graphite.	2
1.3	Influence of graphite morphology on tensile behavior of cast irons [4].	3
1.4	Spheroidal graphite cast iron microstructure a) EN-GJS-500-7 (Ferritic-pearlitic) b) EN-GJS-500-14 (SSF SGI grade).	4
2.1	Iron-carbon equivalent phase diagram and the types of cast irons obtained by controlling cooling rate [5].	10
2.2	Typical microstructure of SGI with graphite nodule embedded in ferrite and pearlite matrix (2 % nital solution etched), graphite nodules in black, white ferrite outside graphite and pearlite in light brown.	12
2.3	Roundness shape factor definition for graphite nodularity.	13
2.4	Effect of nodularity on yield stress and tensile strength of ferritic SGI [6].	16
2.5	Effect of silicon content on the mechanical properties of SSF grades a) tensile strength b) yield stress [7].	19
2.6	Deviation in graphite shape in SSF a) chain of small graphite nodules and b) SEM image of non-nodular graphite [8].	21
2.7	Onion-like internal damage mechanism of spheroidal graphite nodules in ferritic SGI [9].	24
2.8	Fatigue damage micromechanism in different ductile iron grades a) graphite nodule decohesion in the ferritic SGI [10], b) crack branching in the ferritic SGI [11], c) crack propagation in the pearlitic SGI [11], d) crack branching in the pearlitic SGI [12], e) crack propagation by graphite decohesion in the ferritic-pearlitic SGI [11], and f) crack branching in the ferritic-pearlitic SGI [11].	25
2.9	Schematic diagram of the DCR process.	29
2.10	DCR process mechanism base on FE simulation study [13].	31
2.11	Influence of deep rolling force on the distribution of residual stress a) axial and b) tangential direction for normalized SAE 1045 [14]. .	33

2.12	Influence of rolling pressure on roughness for nominal SAE 1045 [14].	34
2.13	Variation of modulus of elasticity of cast irons with thermal cycling; ADI-austempered ductile iron; DI-ductile iron; CGI-compacted graphite iron; GI-gray cast iron [15] a) 120-600 °C b) 300-750 °C.	38
2.14	Variation of hardness of cast irons with thermal cycling; ADI-austempered ductile iron; DI-ductile iron; CGI-compacted graphite iron; GI-gray cast iron [15] a) 120-600 °C b) 300-750 °C.	39
2.15	Graphite nodule separation in SGI after decohesion and oxidation during thermal cycling (120 - 500 °C).	40
2.16	a) Enriched nodes in the X-FEM formulation (Heaviside enrichment represented by blue circles and crack tip enrichment by green squares) [16] b) illustration of normal and tangential coordinates for a crack [17].	43
2.17	Definition of level set function for crack modeling in X-FEM [18]. .	45
2.18	Integration scheme in X-FEM a) sub-division of elements cut by the crack b) phantom node approach.	46
3.1	Specimen casting pattern geometry (reprinted with permission from project collaborator Anders E. W. Jarfors, Jonkoping University). .	56
3.2	Microstructure of cast EN-GJS-500-7 containing spheroidal graphite embedded in ferrite and pearlite (2 % Nital solution etched) (graphite nodules in black, the ferrite in white and pearlite in brown).	58
3.3	Microstructure of high silicon cast SGI with complete ferrite matrix (2 % Nital solution etched) a) EN-GJS-500-14 and b) EN-GJS-600-10 (graphite nodules in black and ferrite in white).	58
3.4	Casting defects observed on the SGI microstructure a) flake graphite on the casting surface b) shrinkage cavity.	60
3.5	Schematic diagram of nanoindentation process with the corresponding load-displacement response [19].	61
3.6	Load-displacement curve for different phases of cast SGI materials.	62
3.7	Nanoindentation hardness and elastic modulus for different phases of cast SGI materials (95 % confidence interval).	63
3.8	Stress-strain curve of ferrite and pearlite in EN-GJS-500-7 by optimizing bulk material response.	66
3.9	Three-dimensional nanoindentation simulation model.	68
3.10	Nanoindentation simulation results and effect of plastic material parameters on the P-h curve for pearlite ($E = 240$ GPa) a) deformation of the sample due to nanoindentation ($\sigma_Y = 2200$ MPa, $K = 500$ MPa, $n = 0.25$), b) effect of yield stress ($K = 500$ MPa, $n = 0.25$), c) effect of hardening coefficient ($\sigma_Y = 2200$ MPa, $n = 0.25$) and d) effect of exponent ($\sigma_Y = 2200$ MPa, $K = 500$ MPa).	70
3.11	Comparison of P-h response from nanoindentation test and simulation to optimize pearlite phase properties ($K = 500$ MPa and $n = 0.5$).	71

3.12	Comparison of P-h response from nanoindentation test and simulation to optimize ferrite phase properties ($K = 500$ MPa and $n = 0.35$).	72
3.13	Comparison of residual indentation mark in nanoindentation test and simulation a) residual mark size b) residual depth.	73
3.14	Residual nanoindentation marks within the ferrite grain (300 nm indentation process) a) optical microstructure image–nanoindentation marks inside red circles b) SEM image of single indentation mark within a ferrite grain.	74
3.15	As-cast 500-14 block used for DCR experiment a) sample block after machining and grinding b) sample block after DCR process illustrating rectangular DCR regions, RD: rolling direction & CD: cross direction.	78
3.16	Microstructure images after DCR process (HG6 tool, 100 % overlap, 1000 mm/min feed rate) a) 100 bar rolling pressure b) 300 bar rolling pressure.	78
3.17	SEM image of SGI microstructure after DCR (400 % overlap and 1000 mm/min feed rate) a), c) for HG6 at 100 bar rolling pressure; b), d) for HG13 at 200 bar rolling pressure.	80
3.18	SEM images of DCR sample after polishing a), c), e), g) for HG6 at 100 bar rolling pressure; b), d), f), h) for HG13 at 200 bar rolling pressure.	81
3.19	SEM images of DCR sample after fine grinding and polishing a), c), e) for HG6 at 100 bar rolling pressure; b), d), f) for HG13 at 200 bar rolling pressure.	83
3.20	Inverse pole figure maps a) Unrolled EN-GJS-500-14 sample, b) Deep cold rolled sample surface (HG13 at 200 bar), c) Closer view on DCR sample surface (Black regions are graphite particles).	85
3.21	EBSD study on the cross-section of DCR sample (HG13 at 200 bar) a) Inverse pole figure map and b) Grain orientation spread.	87
3.22	EBSD study on the cross-section of DCR sample (HG6 at 100 bar) a) Inverse pole figure map and b) Grain orientation spread.	88
3.23	Effect of DCR process and parameters on graphite nodules and ferrite hardness (95 % confidence interval).	90
3.24	Comparison of P-h response of the cold rolled ferrite matrix to the as-cast ferrite matrix.	91
3.25	Estimation of effective DCR influenced zone based on EBSD Grain Orientation Spread map over the distance from cold rolled edge.	92
3.26	Thermal expansion behavior of the as-cast SGI during thermal cycling measured at 55 °C (RT-600°C) a) ferritic 500-14 b) ferritic-pearlitic 500-7 [2].	94
3.27	Schematic diagram of thermal cycling from RT to 600 °C.	95
3.28	Microstructure study after 45 thermal cycles, a), c) and d) partially debonded graphite-ferrite interface; b) Magnified image of image a) at the interface.	96

3.29	Effect of the thermal cycling on the nanoindentation hardness of the ferrite and graphite nodule in SGI microstructure (95 % confidence interval).	97
4.1	Miniature tensile test specimen design (all dimensions are in mm) a) Tensile test specimen design (thickness = 1 mm) b) Specimen loading and its fixture assembly.	103
4.2	Miniature crack propagation test specimen design (all dimensions are in mm) a) CT specimen design (thickness = 1 mm) b) Fatigue crack propagation fixtures with front and back microscopes to measure crack length.	105
4.3	Fatigue test method for S-N curve and the specimen used a) design and dimension of standard half size fatigue test specimen (all dimensions in mm and thickness = 2 mm) b) polished specimen before the test c) JSME S002 standard procedure to obtain S-N curve using minimum test specimens (numbers represent test sequence). .	106
4.4	Microstructure damage mechanisms at different parts of the tensile test.	109
4.5	SEM images of TDM-2 specimen microstructure after different loading levels a) after 420 MPa, b) after 450 MPa and c) after fracture at 580 MPa.	110
4.6	SEM images of spheroidal graphite nodules damage in tensile test of TDM-2 a) after 420 MPa, b) after 450 MPa and c) after fracture at 580 MPa.	110
4.7	SEM images of compacted and irregular graphite nodules damage in tensile test of TDM-2 a) after 420 MPa, b) after 450 MPa and c) after fracture at 580 MPa.	111
4.8	SEM images of degenerated graphite particles damage in tensile test of TDM-2 a) after 450 MPa, b) and c) after fracture at 580 MPa.	113
4.9	SEM images of the ferrite matrix cracking in tensile test of TDM-2 a) after 420 MPa, b) after 450 MPa and c) after fracture at 580 MPa.	114
4.10	SEM images of the fracture surface of TDM-2, a) overall fracture surface (ellipse indicating the ferrite fracture regions); b) microvoids on the ferrite fracture region; c) graphite nodules void growth due to large plastic deformation of the ferrite matrix.	116
4.11	Images showing fatigue crack initiation in elongated graphite nodules on specimen FCI-as-cast-4 (bold arrows indicates graphite cracks and line arrows indicates ferrite cracks) a) and b) after 200,000 cycles, c) and d) after 250,000 cycles, e) and f) 310,000 cycles, g) and h) after 390,000 cycles.	119
4.12	Images showing damage of spheroidal graphite nodule under cyclic load on specimen FCI-as-cast-4 a) after 250,000 cycles b) after 310,000 cycles c) after 390,000 cycles.	120
4.13	SEM images of fatigue crack initiation and growth from shrinkage cavity on specimen FCI-as-cast-7 a) after 200,000 cycles b) after 330,000 cycles.	121

4.14	SEM images of fatigue crack initiation from the ferrite matrix on specimen FCI-as-cast-7 a) and b) after 330,000 cycles.	122
4.15	SEM images of fatigue cracks initiation on face 1 of the specimen FCI-as-cast-4 after 390,000 cycles.	123
4.16	SEM images of fatigue cracks initiation on face 2 of the specimen FCI-as-cast-4 after 390,000 cycles.	124
4.17	SEM images of degenerated graphite particles and initiated cracks after final failure of the specimen FCI-as-cast-4.	126
4.18	SEM images of spheroidal graphite nodules after final failure of the specimen FCI-as-cast-4.	127
4.19	SEM imaged of large degenerated graphite particles on the fracture surface of the specimen FCI-as-cast-4 a) stable region fracture surface, b) unstable region fracture surface, c) and d) degenerated graphite nodules on the fracture plane.	128
4.20	SEM images of the fatigue crack initiation and graphite nodules damage observed in specimen FCI-as-cast-14 after 250,000 cycles ($R = 0.4$) a)–d): crack initiation from degenerated graphites, e)–g): spheroidal graphite debonding and internal cracking, and h)–i): crack initiation in the ferrite matrix and from the shrinkage porosities.	129
4.21	Fatigue crack propagation by partial decohesion of the spheroidal graphite nodules a) $R = 0.4$ (FCP-as-cast-5) b) $R = 0.1$ (FCP-as-cast-8).	133
4.22	Fatigue crack propagation through degenerated graphite particles a) $R = 0.4$ (FCP-as-cast-5) b) $R = 0.1$ (FCP-as-cast-8).	133
4.23	Fatigue crack branching observed around graphite nodules a) $R = 0.4$ (FCP-as-cast-2) b) $R = 0.1$ (FCP-as-cast-8).	134
4.24	SEM images of short fatigue crack growth in specimen FCP-as-cast-6 ($R = 0.1$, $\Delta K_{Start} = 13 \text{ MPa}\sqrt{m}$, $a = 0.75 \text{ mm}$, $\Delta K = 27 \text{ MPa}\sqrt{m}$).	135
4.25	SEM images of long fatigue crack growth in specimen FCP-as-cast-7 ($R = 0.1$, $\Delta K_{Start} = 13 \text{ MPa}\sqrt{m}$, $a = 1.67 \text{ mm}$, $\Delta K = 35 \text{ MPa}\sqrt{m}$).	136
4.26	SEM studies of graphite particles along the crack path of completely fractured FCP-as-cast-1 ($R = 0.1$, $\Delta K_{Start} = 13 \text{ MPa}\sqrt{m}$).	138
4.27	Fracture surface and magnified SEM images of fracture surface of FCP-as-cast-8 ($R = 0.1$, $\Delta K_{Start} = 13 \text{ MPa}\sqrt{m}$).	139
4.28	Fatigue crack growth rate for EN-GJS-500-14 with Paris' law fit.	141
4.29	Stress-life (S-N) curve obtained from 14 fatigue tests according to JSME S 002 standard.	143
4.30	Comparison of fatigue crack growth rates of thermal cycled SGI with as cast material ($R=0.1$).	145
4.31	Cyclic force vs. deformation curve in fatigue crack initiation test.	149
5.1	Three graphite RVE model and the model dimensions (all dimensions are in μm).	157
5.2	X-FEM based cohesive traction-separation behavior in ABAQUS a) linear and b) nonlinear [17].	161

5.3	Stress evolution in the RVE model at different displacement load (top edge) a) graphite particles modeled as void, b) elastic graphite particle unbound to the ferrite matrix, c) elastic graphite particles bonded to by surface-based cohesive behavior.	164
5.4	Stain evolution in the RVE model a) graphite particles modeled as void, b) elastic graphite particle unbound to the ferrite matrix, c) elastic graphite particles bonded to by surface-based cohesive behavior.	166
5.5	Strain concentration at the edge of the decohesed interface after degradation of the cohesive behavior ($\varepsilon_{yy} = 0.368\%$) (deformation scale = 1).	167
5.6	Stress and strain evolution at different cohesive interface stiffness a) default contact enforcement, b) stiffness = 1×10^5 , c) stiffness = 1×10^8 , d) stiffness = 1×10^{10}	169
5.7	Overall stress-strain plot for the RVE model from FE simulation at different cohesive contact stiffness.	170
5.8	X-FEM crack initiation and propagation simulation in the RVE model implemented in ABAQUS a) $\varepsilon_{yy} = 0.37\%$, b) $\varepsilon_{yy} = 0.74\%$ and c) $\varepsilon_{yy} = 1.11\%$	174
5.9	Definition of multiple X-FEM enrichment regions in the RVE model a) three enrichment regions and b) six enrichment regions.	177
5.10	Illustration of microstructure submodel in CT specimen.	179
5.11	Microstructure submodel from the real SGI microstructure using OOF2 a) SGI micrograph, b) image processed micrograph, c) FE mesh generated from OOF2.	181
5.12	Stress evolution on the CT sample model and the microstructure submodel at the notch a) $K = 7.5 \text{ MPa}\sqrt{m}$, b) $K = 15 \text{ MPa}\sqrt{m}$, c) $K = 22.5 \text{ MPa}\sqrt{m}$ and d) $K = 30 \text{ MPa}\sqrt{m}$	184
5.13	Validation of strain evolution on the microstructure submodel for CT sample 1 a) DIC strain map and b) FEA strain distribution.	185
5.14	Validation of strain evolution on the microstructure submodel for CT sample 2 a) DIC strain map and b) FEA strain distribution.	186
5.15	Crack initiation result in the multiscale model with single enrichment X-FEM region (displacement applied = 0.1 mm, time step completed = 0.163).	188
5.16	Illustration of multiple X-FEM enrichment regions definition in the microstructure model.	189
5.17	Crack initiation result for multiple enrichment X-FEM model (red arrows indicating cracks initiated) (displacement applied = 0.1 mm, time step completed = 0.163).	191
5.18	Single enrichment X-FEM crack propagation in SGI microstructure model.	192
5.19	Multiple enrichments XFEM crack propagation in SGI microstructure model.	192

List of Tables

2.1	EN 1563 specified standardized SSF grades [20].	20
2.2	Literature summary of damage mechanisms studies in different SGI materials.	26
3.1	Chemical composition of cast SGI metals (% wt.)	56
3.2	Tensile properties of as-cast SGI materials.	57
3.3	Graphite morphology characterization and matrix composition of as-cast SGI grades.	59
3.4	Optimized Ramberg-Osgood model parameters for ferrite and pearlite in EN-GJS-500-7 [21].	66
3.5	Comparison of phase properties estimation methods using $\sigma - \varepsilon$ optimization and P-h optimization	75
3.6	DCR process parameters and experimental design.	77
3.7	Material removal during sample preparation steps.	82
4.1	Details of the specimens used in tensile damage mechanism studies.	108
4.2	Summary of the FCI specimens and tests.	117
4.3	Quantitative analysis of damage mechanisms for different graphite forms in specimen FCI-as-cast-15.	130
4.4	Summary of FCP specimens and tests.	132
5.1	Summary of the effect of model, material and control parameters on the X-FEM crack growth and simulation convergence.	193

Abbreviations and Symbols

Abbreviations

ADI	Austempered Ductile Iron
CGI	Compacted Graphite iron
CT	Compact Tension
CTE	Coefficient of Thermal Expansion
DCR	Deep Cold Rolling
DIC	Digital Image Correlation
DOFs	Degrees Of Freedom
EBS	Electron Backscatter Diffraction
EDS	Energy Dispersive Spectrometer
FE-SEM	Field Emission SEM
FEM	Finite Element Method
FOD	Foreign Object Damage
GCI	Gray Cast Iron
GOS	Grain Orientation Spread
GTN	Gurson-Tvergaard-Needleman
HCF	High Cycle Fatigue
HG tools	Hydrostatic tools
IIT	Instrumented Indentation Test
IPF	Inverse Pole Figure
LCF	Low Cycle Fatigue
LEFM	Linear Elastic Fracture Mechanics
LPB	Low Plasticity Burnishing
LSM	Level Set Method

LSP	Laser Shock Peening
MAXE	Maximum Nominal Strain
MAXPE	Maximum Principal Strain
MAXPS	Maximum Principal Stress
MAXS	Maximum Nominal Stress
MTS	Maximum Tangential Stress
NND	Nearest Neighbor Distance
OM	Optical Microscope
OOF	Object Oriented Finite element
PBC	Periodic Boundary Conditions
QUADE	Quadratic Separation-interaction
QUADS	Quadratic Traction-interaction
RE	Rare Earth
RSF	Roundness Shape Factor
RVE	Representative Volume Element
S–N	Stress-Life
SEM	Scanning Electron Microscopy
SGI	Spheroidal Graphite Iron
SGIs	Spheroidal Graphite Irons
SIF	Stress Intensity Factor
SSF	Solution Strengthened Ferritic
TMF	Thermo-Mechanical Fatigue
UTS	Ultimate Tensile Strength
VCCT	Virtual Crack Closure Technique
X-FEM	eXtended Finite Element Method

Symbols

A	Projected indentation contact area
A_g	actual area of the graphite particle
A_{gm}	area of circumscribe circle to the graphite particle

C_{eq}	Carbon Equivalent
c_1, c_2	Material constants in fatigue crack initiation
c_3, c_4	Material constants in fatigue crack propagation
f	Damage parameter
E	Young's modulus
E_I	Elastic modulus of indenter
E^*	Reduced modulus in nanoindentation
E_S	Elastic modulus of sample
$F(x)$	Asymptotic near-tip field in X-FEM
da/dN	Fatigue crack growth rate
G	Strain energy release rate
ΔG	Relative fracture energy release rate
G_c	Critical strain energy release rate
h	Nanoindentation depth
$H(x)$	Heaviside enrichment function in X-FEM
h_c	Total residual nanoindentation depth
h_{max}	Maximum indentation depth at P_{max}
Δw	Inelastic hysteresis energy per fatigue load cycle
K_{Ic}	Fracture toughness
l_{gm}	diameter of the circumscribe circle
ε_{max}^0	Maximum allowable principal strain
ν	Poisson's ratio
P	load or force applied
P_{max}	Maximum applied indentation load
$\phi(x)$	Level set function normal to the crack face
ν_I	Poisson ratio of indenter
ν_S	Poisson ratio of sample
$\psi(x)$	Level set function tangential to the crack tip
R	Fatigue load ratio
σ	Stress

ε	Strain
ΔK	Stress intensity factor range
ΔK_{start}	SIF at the beginning of FCP test
ε_{pl}	Plastic strain
σ_Y	Yield stress

Chapter 1

Introduction

This chapter provides background information and motivation for this research. The objectives and scope of the present work are explained, followed by a brief outline of the thesis.

1.1 Background

In today's rapidly developing world, the demand for cheap engineering materials with relatively higher strength is high. Constantly evolving design and functional optimizations deem the requirement of an efficient manufacturing process to fabricate complex parts to their final form. Casting as a production process offers easy fabrication of complex parts close to their final shape and dimension. Cast iron parts are fabricated by the casting process and the material itself being cheap, cast iron parts are widely used in many industries. Excellent castability, machinability and economy have made cast irons common choice of material for many applications. In general, cast irons are considered brittle iron-carbon alloy but all types of cast irons are not brittle. Ductile cast iron in specific is ductile and its toughness and ductility are comparable to those of carbon steel [22].

1.1.1 Cast irons

In cast irons, carbon is precipitated out as graphite phase inclusions in the iron matrix. These graphite particles are weak, soft and brittle, and act as a form of void or defect resulting into lower strength and toughness of cast iron compared

Figure 1.1: Microstructure of different types of cast irons a) Gray cast iron/Flake graphite cast iron [1] b) Compacted graphite cast iron [2] c) Ductile cast iron/Spheroidal graphite cast iron [2].

Figure 1.2: Deep-etched scanning electron micrographs showing three-dimensional shape of the graphites [3] a) Flake graphite b) Compacted graphite c) Spheroidal graphite.

to similar grade steel. The shape of graphite inclusions differentiate the types of cast irons, and not only give them a name but also provide different properties. Figure 1.1 and Figure 1.2 show microstructure and deep etched Scanning Electron Microscopy (SEM) images of three commonly used cast irons. Gray Cast Iron (GCI) contains flake graphite particles that provide higher vibration damping properties but lower strength and impact properties [1, 23]. Spheroidal Graphite Iron (SGI) has graphite particles in the form of spheroidal nodules that provide added benefit of higher fatigue resistance, toughness and ductility in cast iron [22, 24]. These unique properties of ductile iron are attributed to the nodular graphite form [22, 25, 26]. The nodular form of graphite helps to minimize the stress intensification effect that occurs at the edge of the graphite nodule which improves its mechanical and fatigue properties. In Compacted Graphite iron (CGI), the shape of graphite particles is in between flake and spheroidal hav-

ing a rounded edged and worm-like shape, also referred to as vermicular graphite. CGI has properties in between SGI and GCI. As shown in Figure 1.2 b), compacted graphite in 3D are the complex network of irregular graphite particles that result in strong adhesion between the graphite particles and the iron matrix. The plain flake graphite particles in gray iron promote crack initiation and growth, thus rendering the material relatively weak and brittle [3].

Microstructure variation in cast irons contributes to different material properties. The graphite form in specific has a dominant effect on mechanical properties of cast iron as shown in Figure 1.3 [4, 25]. Spheroidal graphite in SGIs exhibited higher strength and ductility resulting to improved properties. Graphite nodules not only influence mechanical properties but also play a vital role in fatigue crack initiation and propagation [10, 27, 28]. Due to their superior mechanical and fatigue behavior, SGIs have found applications in various forms which include, ductile iron pipes for transportation of water, sewage, slurries and process chemicals, wind turbine parts [29, 30] and automotive parts [31, 32].

Figure 1.3: Influence of graphite morphology on tensile behavior of cast irons [4].

1.1.2 Spheroidal graphite cast iron

With superior mechanical and fatigue properties, SGIs are undoubtedly better cast iron type for components considering fracture and fatigue. Even if the graphite shape plays a significant role in determining the overall behavior of SGIs; the iron

matrix also plays an important role as most of the applied load is distributed into the matrix. For the as-cast SGI, the matrix can be either ferrite or pearlite or ferrite-pearlite. The matrix can be transformed into aus-ferrite in Austempered Ductile Iron (ADI) or martensite in martensitic ductile iron by post-processing treatment. The ferrite matrix is soft having good ductility and toughness, whereas the pearlite matrix is harder having higher strength and wear resistance. So, usually, ferritic-pearlitic grades are preferred to achieve an adequate balance of strength and toughness. One of the commonly used ferritic-pearlitic grades is EN-GJS-500-7 in many automobile parts. In such ferritic-pearlitic grades, one common problem is a large variation in hardness and properties. In recent years, new SGI grades are casted with higher silicon content to reduce properties variation within the cast component. Silicon is a ferrite promoter and graphitizer. The addition of a higher amount of silicon creates a completely ferritic matrix and also solution strengthen the ferrite matrix, significantly reducing properties variation [7, 33]. Such higher silicon grades are known as high-silicon cast irons and sometimes referred to as Solution Strengthened Ferritic (SSF) SGI. The arrival of SSF grades has got positive reception and enormous market penetration. Predominantly, two grades EN-GJS-500-14 and EN-GJS-600-10 have been in extensive use and research.

Figure 1.4: Spheroidal graphite cast iron microstructure a) EN-GJS-500-7 (Ferritic-pearlitic) b) EN-GJS-500-14 (SSF SGI grade).

The microstructure of SSF EN-GJS-500-14 is shown in Figure 1.4 b) with complete ferrite matrix as compared to the ferritic-pearlitic matrix in EN-GJS-500-7 (Figure 1.4 a)). Both of the SGI grades have similar strength, EN-GJS-500-14 having much higher ductility. Such SSF grades are capable of replacing many SGI grades, and even some steels. EN-GJS-500-14 can adequately replace EN-GJS-500-7 in automobile parts providing much higher ductility. Similarly, the same grade can

replace EN-GJS-400-18 in wind turbine parts providing higher strength at slightly less ductility.

SGIs used in automobile and wind turbine parts are exposed to repeated mechanical and thermal loads. These applied loads separately or in combination make SGI components prone to failure. To increase failure resistance, many post-processing treatments like heat treatment [31, 34] and surface peening processes are applied [14, 32, 35–37]. Heat treatment processes change the matrix structure to achieve desired properties [34], and surface enhancement processes like Deep Cold Rolling (DCR) usually introduce compressive residual stress by surface plastic deformation [32, 36, 37]. Whether it is a complete change of matrix in heat treatment process or changing properties of the same matrix in DCR process, one common point in both processes is that the microstructural properties change. These processes help in some extent to improve strength and fatigue life at the expense of additional post processing cost. Possible cost effective alternative to this is to achieve higher strength and ductile grade by controlling chemical composition as achieved for SSF SGIs. However, there exists a limit to this as the strengthening effect drastically deteriorates at over 4.3 wt% silicon addition [7]. With the current trend towards remanufacturing, it is more desirable to design an optimized component, estimate safe operating load and life, and to replace the component after its service life. This approach is usually valid for a wide range of industries and provides safety with an economy. However, the groundwork for this approach is the estimation of component life at the applied load with reasonable safety factor. For all sorts of component failures, whether it is rapid fracture or steady fatigue crack growth, they all initiate from some microstructure features and influence further crack growth [38].

1.1.3 Microstructure influence on SGI component failure

Over the past years, many works [10, 11, 27, 28, 30, 39–44] have been reported to investigate damage mechanisms in SGI grades. Most of the SGI parts under repeated load fail due to fatigue crack initiation and propagation, so the focus is mostly in fatigue damage micromechanisms. Even though the studies were conducted on different SGI grades by various researchers, all of them agreed that the peculiar SGI microstructure with spheroidal graphite nodules demonstrated higher toughness and fatigue resistance [10, 11, 27, 30, 39, 41, 43, 44]. The matrix controls mechanical properties [22, 45] and spheroidal graphite nodules reduce stress inten-

sification around graphite nodules [10–12]. In the presence of casting defects like shrinkage cavities, porosities and nonmetallic inclusions, cracks usually initiate from these defects [29, 46–48]. To some extent, these defects can be controlled by optimizing the casting process. Other common defects like degenerated graphite particles (Flake, compacted, irregular and exploded graphites) cannot be completely controlled as some graphite particles deviate from the spheroidal shape. Such degenerated graphite particles and spheroidal graphites behave like defects in the iron matrix, potentially initiating microcracks. Propagation of the crack depends on distribution, size and form of the graphite nodules. The overall crack path along the fracture surface is due to graphite nodules distribution and can be predicted by simulating inhomogeneous stress and strain distribution in the microstructure. With all this in mind, it is worth to develop a proper understanding of damage mechanisms in SGIs considering microstructure and implement the knowledge to estimate microstructure dependent failure of SGI components.

1.2 Research Objectives

The objectives of this research work are as follows:

- To characterize microstructure of high silicon SGI casting, and study the effects of thermal and mechanical processes on the SGI microstructure.
- To investigate microstructure dependent tensile and fatigue damage mechanisms in solution strengthened ferritic SGI.
- To develop real micrograph based SGI microstructure modeling approach and eXtended Finite Element Method (X-FEM) crack growth prediction on the microstructure model.

1.3 Scope

The main contributions of this research work are microstructure study of SGI with higher silicon content and the effect of DCR and thermal cycling on the microstructure; comprehensive understanding of microstructural damage micromechanisms in tensile and fatigue loading; and microstructure modeling of real SGI microstructure to perform microstructure dependent analysis on SGI components. However, the scopes of this research work are limited to the followings.

- Microstructure characterization of as-cast SGI, after the deep cold rolling process and after thermal cycling for microstructure evolution study. Different grades of as-cast SGIs studied to differentiate SSF SGI with usual grade, and SSF grade EN-GJS-500-14 used for further DCR and thermal cycling effect study.
- Tensile and fatigue damage mechanisms study to generalize damage micromechanisms in SGI material. Fatigue damage mechanism studied in two separate fatigue crack initiation and propagation test experiments to clearly understand the effect of graphite nodules on crack initiation and propagation stage. Thermal cycled condition is considered to estimate the effect of the thermal process on SGI damage mechanisms.
- SGI microstructure modeling using OOF2 for FE representation of SGI microstructure and damage criteria derived from damage mechanism study. Digital Image Correlation (DIC) results from collaborators used to validate strain distribution. X-FEM formulation available in ABAQUS used to simulate crack growth in Compact Tension (CT) sample microstructure model.

1.4 Motivation

Usually, steels are the common material choice for many structural applications, but they have a major drawback of poor castability. The motivation of using SGIs is that it has combined properties of cast irons (i.e. easy castability, machinability and economy) and steels (i.e. higher fatigue resistance and toughness). Different from other cast irons, SSF SGIs have spheroidal graphite nodules in the ferrite matrix which contributes to higher mechanical and fracture properties. Most of the SGI parts fail due to microcrack initiation and propagation, where graphite morphology plays a significant role. So, it is necessary to clearly understand the roles of graphite particles on crack initiation and propagation at different loading condition. Most importantly it is crucial to note that all the graphite particles are not spheroidal in SGI. Thus, it is motivating to understand the roles of graphite morphologies on SGI damage mechanisms, which will not only help to predict failure but will also provide guidance on graphite morphologies and defects to avoid in the casting process to develop better material grades.

1.5 Thesis Outline

This chapter provides background information to this research work with clearly stated research objectives and scope. Chapter 2 presents a review of SGI materials, their properties, damage mechanism studies and effect of thermal and deep cold rolling process on SGI microstructure. Chapter 2 also discusses basic theory and capabilities of X-FEM crack simulation and critically reviews X-FEM damage modeling for SGI material. In chapter 3, as-cast SGIs microstructures are characterized, and further the effects of thermal cycling and deep cold rolling process on SGI microstructure is investigated. Chapter 4 primarily discusses SGI damage mechanism based on the separately performed tensile test, fatigue crack initiation tests and fatigue crack propagation test. The SGI damage study is further extended to the thermal cycled case in chapter 4. Then, in chapter 5, the damage mechanisms and criteria obtained are used to formulate crack propagation simulation in X-FEM capabilities available in ABAQUS. Finally, the major conclusions of this research work along with the suggestions for the further research are presented in Chapter 6.

Chapter 2

Literature Review

This chapter reviews the relevant literature on Spheroidal Graphite Iron (SGI) materials starting from the history to the recent damage mechanism studies. The effect of Deep Cold Rolling(DCR) process and thermal process on SGI material is critically reviewed. Further, the theory of eXtended Finite Element Method (X-FEM) is discussed, and different modeling approaches used to model SGI material are reviewed.

2.1 Ductile Cast Iron

Ductile cast iron also known as nodular cast iron and SGI is a type of soft cast iron. Cast iron is a ferrous alloy with the addition of other alloying elements on pig iron melt. Carbon is the major alloying element with an amount ranging from 2.4 - 4.3 % wt., moreover, silicon is an important alloyant as it forces carbon out of the iron solution to form graphite nodules [5]. The microstructure of cast irons largely dependent on chemical composition which in turn determines mechanical properties and general characteristics of the cast grade. Figure 2.1 shows the iron-carbon phase diagram and illustrates the formation of different cast irons by controlling the cooling rate. Inoculation of the melt and Mg treatment are vital to form spheroidal graphite nodules in ductile iron. During the solidification process, the first solid to crystalize from the liquid is the graphite phase. Graphite particles prefer to nucleate in low energy interface of inclusions like MgS, CaS and MgO.SiO₂. With reducing temperature down to the eutectic line, graphite particles gradually grow by depleting carbon from the melt [22].

Figure 2.1: Iron-carbon equivalent phase diagram and the types of cast irons obtained by controlling cooling rate [5].

Cast irons can have a wide range of properties depending on alloying composition, heat treatment and final microstructure of the alloy. Major advantages of cast irons are excellent castability, good machinability, excellent anti-vibration properties and cheap cost. Along with these benefits, it comes with the main drawback of some brittleness [6, 49]. In that prospect, SGIs stand out as their peculiar microstructure offer better ductility with cast iron benefits. These unique properties of ductile iron are attributed to the nodular graphite form [22, 25, 26]. Graphite morphology and its interaction with the matrix play a vital role in the overall properties of ductile iron [25, 50, 51]. Matrix composition and properties control

mechanical properties of ductile irons, and these matrices are used to identify different grades of ductile irons.

Different standards use a different label to designate different SGI grades. Some of the commonly used nomenclature systems are ASTM A536, EN 1563, ISO 1083, SAE specification No. J434, BS 2789 and Chinese standard GB 1348. Even there are many types of standards to label different grades of SGIs, almost all of them are somehow based on the matrix structure and composition, and are comparable to each other. For as-cast SGI, the matrix can be ferrite, pearlite, or a combination of them. Out of these, ferritic-pearlitic SGI is considered to have a wide range of mechanical property. Ferrite matrix contributes for good ductility and toughness; pearlite matrix contributes to higher strength, wear resistance with moderate ductility and austenite contributes to good ductility and toughness at all temperature [6]. So, a major part of the load is carried by the iron matrix, but the overall properties of SGIs are also highly affected by the form of graphite particles present.

2.1.1 Brief history of ductile cast iron

Archeologists have stated that cast iron was developed in China in the 5th century to make weapons and pots. Because of its cheap cost and easy availability, its use continued for centuries. In the western countries, iron casting started from the 15th century to replace bronze cannons. In the early age before the invention of the microscope, white and gray irons were the only types of irons known based on their fracture surface. The first paper on cast iron was published in 1896 defining shrinkage, strength, deflection, set, chill, grain and hardness as primary properties of cast iron. During that time cast irons were highly used material accounting for about 70 % of the total casting products [52]. Scientists and foundry men continued to search for better iron with superior properties. Incidentally, in 1943, Keith Dwight Mills added Magnesium (as copper magnesium alloy) to cast iron melt trying to transform all the carbon to carbide [6]. But, carbon in cast iron changed to nearly spheroidal graphite, giving birth to ductile cast iron. After five years, in 1948 AFS convention, Henton Morrogh reported the successful production of spheroidal graphite by the addition of cerium as a spheroidizing agent, to announce the official birth of present ductile iron.

With the successful invention of ductile iron, foundry men were still looking to improve its properties. Ductile irons were applied with various heat treatment pro-

cesses to further refine microstructure and grain size. In 1972 Tecumseh Products successfully applied austempering process on SGI compressor shaft. The austempering process was carried out by heating to a temperature range of 840 °C to 950 °C, then quenching the alloy to a temperature range of 250 °C to 450 °C and maintaining at that temperature followed by air cooling [53]. Through this process, the matrix is transformed to aus-ferric matrix forming Austempered Ductile Iron (ADI) with higher yield strength and better ductility.

2.1.2 SGI microstructure

Figure 2.2: Typical microstructure of SGI with graphite nodule embedded in ferrite and pearlite matrix (2 % nital solution etched), graphite nodules in black, white ferrite outside graphite and pearlite in light brown.

Figure 2.2 shows a typical SGI microstructure, which contains round black graphite nodules, ferrite phase in white and pearlite structure in light brown. This type of microstructure with white ferrite phase around black graphite nodules is known as “*bull’s eyes*” microstructure. The matrix may be different depending on alloying composition, casting parameters like temperature, cooling rate and heat treatment process, producing different types and grades of SGI. Graphite morphology is the major difference between SGIs and other cast irons, which plays a crucial role to improve its mechanical and fatigue properties [25]. Effect of ferrite matrix [46, 54], pearlite matrix [45, 55], aus-ferric matrix [31, 56], retained austenite [57] and martensite [58] on mechanical properties of ductile irons have been illustrated in previous research. Graphite particles in cast iron microstructure are considered as a form of defect where failure might initiate [48]. Discontinuities caused by graphite defects in the continuous matrix are the reason for SGI to be more vulnerable than similar grade steel. However, the same spheroidal graphite

differentiates it from other forms of cast irons to give it higher strength, toughness and ductility.

Graphite morphology (size, shape and nodule count) has a direct effect on mechanical properties and fracture behavior of SGI. Large graphite nodules would act as a large defect on the iron matrix, weakening the bulk material. Similarly, more graphite nodules mean more defects then more chances of failure by crack initiation on these defects. The size of graphite nodules varies depending on cooling time, and count depends on graphite formers concentration. Nodularity, defining degree of roundness or closeness to a circular periphery of a graphite particle, is another important graphite parameter. Higher nodularity means more reduction in stress concentration effect and nodularity depends on nodularizers concentration (e.g. magnesium and cerium) [6]. In some work, graphite nodules were also used to estimate the strain field based on a change in nodularity by the process as exemplified in [59]. Graphite nodules count and size are inter-connected for the same carbon content in the alloy. Large nucleation sites as a result of the adequate concentration of silicon would give higher nodule count but smaller nodules, whereas larger graphite nodules would be linked to lower nodule count. Nodularity being one of the important aspects of graphite nodule, it needs to be well estimated following standards. ASTM E2567 [60] provides a comprehensive explanation for determining nodularity and nodule count of SGI. Eq. 2.1 can be used to evaluate average graphite nodularity in SGI microstructure. The minimum required Roundness Shape Factor (RSF) value chosen to qualify a particle as being a nodule is suggested to be 0.60. RSF for each graphite particle can be obtained from Eq. 2.2 with the parameters illustrated in Figure 2.3.

Figure 2.3: Roundness shape factor definition for graphite nodularity.

$$\% \text{ nodularity by count} = 100 \times \left(\frac{\text{Number of all graphite particles above acceptance criteria}}{\text{Number of all graphite particles which meet min. area requirement}} \right) \quad (2.1)$$

$$RSF = \frac{A_g}{A_{gm}} = \frac{4A_g}{\pi l_{gm}^2} \quad (2.2)$$

where, A_g is the actual area of the graphite particle

A_{gm} is the area of circumscribe circle to the graphite particle

l_{gm} is the diameter of the circumscribe circle

2.1.3 Graphite nucleation and growth in SGI casting

Graphite nodules shape has a significant effect on fatigue crack initiation and propagation behavior, as it is the major graphite parameter that differentiates SGIs from other types of cast irons. Even though SGI materials were treated to form spheroidal graphite nodules, other forms of graphite particles could grow due to the effect of other trace elements or insufficient addition of inoculant or nodulizer during the solidification process [61–63]. Chunky, compacted, vermicular and irregular graphite particles are commonly observed degenerated graphite particles due to poor treatment of melt. Other elements like S, F, O, P, N, B were found in trace quantity in cast irons. These trace elements concentration in the melt affected graphite particles nucleation and their growth morphology [62]. The effect of these trace elements was divided into three parts; S and B as flake graphite stabilizer, O and F stabilizing compacted or vermicular graphite growth, P and N mostly neutral. It was reported that the graphite particle growth morphology was related to the impurities in the Fe-based liquid [64]. To stabilize these elements and to favor graphite nodule nucleation and spheroidal graphite growth, inoculant and nodulizer were added to the iron melt. There were many commercially available inoculants which were the primary alloy of Fe-Si with inoculating elements such as Al, Ca, Ba, Sr and Zr. These elements had strong affinities towards oxygen and sulphur in the melt. The addition of inoculant introduced certain types of micro-particles, mostly oxides and sulfides, that provided nucleation sites for graphite

nodules. They also controlled the concentration of S and O in the melt to support nodular graphite growth morphology. In SGI casting, the melt was treated with nodulizer. Usually, Mg-based treatment was carried out. The addition of Mg in the melt formed MgO and MgS by dissolving O and S. Skaland [65] had reported that oxide and sulfide particles would have at least one lattice spacing matching the graphite lattice spacing that creates the possibility of a favored substrate for graphite growth. It was also reported that MgO particles were found at the center of a graphite nodules [61,66]. Thus, MgO in the melt assist on graphite nucleation, and MgS control the concentration of S to favor nodular graphite growth. The study of the effect of S and O on the graphite morphology reported the easy addition of atoms in the a-direction (basal plane) but with the lower probability of attaching in the c-direction normal to the graphene monolayer [64]. The growth of graphite nodule occurred mainly in the a-direction and infrequently in the c-direction that only occur under certain circumstances. The dominant growth direction of flake graphite was along a-direction, while spheroidal graphite usually grew along c-direction. The compacted or vermicular graphite particles grew in a complex way and did not have one preferred growth direction. Muhmond et al. [61] had shown that the graphite nodules growth were along the circumference in SGIs and the tentative size of different growth region was reported by Di Cocco et al. [10]. As pointed out by Di Cocco et al. [67], the graphite growth morphologies had a significant effect on graphite properties and resulting damage mechanisms.

2.1.4 Factors affecting mechanical properties

The mechanical properties of SGI materials are directly influenced by the final microstructure. Factors that affect microstructure and mechanical properties of the SGI materials are discussed below.

Effect of graphite morphology

The morphology of graphite particles (size, shape, amount and distribution) and their interaction with matrix structure significantly influence the mechanical properties attained by cast irons. The nodularity of spheroidal graphite has been determined to be an effective indicator of SGI mechanical properties [6, 68, 69]. However, the effect of graphite morphology on mechanical properties must be considered together with matrix structure because the exact nature of crack initiation

Figure 2.4: Effect of nodularity on yield stress and tensile strength of ferritic SGI [6].

and propagation due to graphite morphology will also depend on the matrix microstructure. Figure 2.4 illustrates the effect of graphite nodularity on yield stress and tensile strength of ferritic SGI. The observed effect is significant on tensile strength, which decreases with a decrease in percentage nodularity. Some debated that the effect of nodularity above 40 % was not significant in heavy section ferritic casting [70]. The graphite nodularity has a significant effect on the elongation of the ferritic SGI, while the pearlitic irons exhibited a lesser decrease in elongation with a decrease in nodularity. Not only nodularity, some other work [71] highlighted that the spacing between nodules, also termed as the Nearest Neighbor Distance (NND), has a detrimental effect on the mechanical properties. Regarding the dynamic properties, it was reported that the presence of exploded and chunky graphite particles resulted in a drastic reduction of elongation and impact properties [69]. Spherical nodules and higher spacing promoted higher fracture toughness while elongated nodules and lower spacing resulted in lower fracture toughness values. Javaid et al. [71] have shown that a small nodule size, good nodularity and higher nodule spacing are very important to obtain good fracture toughness and fatigue limit in SGI.

There exist many prospective regarding the effect of nodule count on the mechanical properties of SGIs. However, in general, higher nodule count is preferred in SGI castings since high nodule count reduces intercellular structure, improves

nodularity and increases the amount of ferrite by higher graphitization of carbon. Conversely, higher nodule count will decrease the inter-nodular distance, so during the crack propagation, the crack has to travel shorter matrix distance to reach the nearest graphite particle, leading to a final brittle fracture with very less plastic deformation of the matrix. However, spheroidal graphite nodules are believed to arrest crack resulting into improved fracture behavior [69]. It appears that there may be an optimum value of nodule count where best static and dynamic properties can be achieved. The tensile strength of SGIs decreased with an increase in nodule size, whereas the effect in the yield strength depended on the matrix structure. For the ferritic grade, yield strength did not change much by variation in nodule size, but for the pearlitic matrix, yield stress decreased with increasing nodule size [69, 70].

Effect of matrix structure

The matrix in SGI microstructure is the main constituent to carry applied load. The matrix structure is responsible for strength, which largely depends on the matrix composition (ferrite-pearlite ratio), fineness of the pearlite structure and solid solution strengthening of the matrix. The matrix composition and matrix properties depend on the cooling rate, the chemical composition of the melt and partly on the nodules count. In general, the pearlite structure is a phase with higher strength and lower elongation, whereas the ferrite matrix is a ductile phase with moderate strength. So, increased pearlite structure results into higher strength grade with a compromise in elongation. Ferritic SGI with minimum pearlite structure has good fracture behavior. Thus a solution strengthened ferritic SGI is better in terms of higher strength and fracture toughness.

Effect of chemical composition

The chemical composition of the melt has a direct influence on the SGI microstructure and the mechanical properties. Gonzaga et al. [45, 55] have shown that different matrix structures and mechanical properties can be achieved by careful control of the chemical composition. As far as melt composition is concerned, an important factor is carbon equivalent (C_{eq}). The carbon equivalent value represents the equivalent percentage of carbon considering the effect of other alloying elements. Carbon equivalent mostly depends on C, Si and P content and can

be calculated using Eq. 2.3. The desired carbon equivalent for SGIs is around eutectic (slightly hyper-eutectic) in order to minimize the problems of graphite flotation and extensive dendritic structure formation.

$$C_{eq} = C\% + 0.33Si\% + 0.33P\% - 0.029Mn\% + 0.41S\% \quad (2.3)$$

Silicon is another important element in the SGI cast. Silicon is a graphitizer and, as such, promotes graphite nodules formation during solidification and matrix formation. It accelerates the diffusion of carbon and lowers carbon solubility in austenite. In cases when strength is preferred, an increase in Si content increases proof stress, hardness and tensile strength. However, increase in the mechanical properties is obtained up to the silicon content of 4.3 %. Excessive addition of Si results into exploded and chunky graphite particles formation and high impact transition temperature mostly in ferritic ductile irons. Other alloyants are also added to the iron melt, manganese as strong pearlite promoter and to neutralize sulfur present in the melt, sulfur to increase hardness, nickel to refine pearlite and graphite structure, a small amount of chromium, copper, molybdenum and vanadium to stabilize carbide and refine graphite [4].

Effect of section modulus

The effect of section modulus on SGI casting, also known as size effect is usually related to the microstructure and casting defects. Many studies [29, 68–72] have reported that the mechanical properties decrease with increasing the size of the casting. However, the yield strength in annealed-ferritic irons was found relatively unaffected by changes in section size whereas, the tensile strength showed a moderate decrease with increase in section size [70]. Detail study of size effect on SGI casting by Shirani and co-workers [29, 72] showed a drastic reduction of fatigue life with either larger casting size (technological size effect) or larger component machined from the cast plate (geometrical size effect).

2.1.5 Solution Strengthened Ferritic SGI

Solution Strengthened Ferritic (SSF) SGIs were first reported in the early 90s after extensive research to improve ductility and strength of cast irons. Before the development of SSF, SGIs were structure strengthened by forming pearlite

Figure 2.5: Effect of silicon content on the mechanical properties of SSF grades a) tensile strength b) yield stress [7].

in the matrix. Silicon is the major element that blends intermediate strength with excellent ductility in SGI by solid solution strengthening of the ferrite matrix [7, 73]. Figures 2.5 a) and b) show the effect of silicon content in tensile strength and yield stress of two commonly used SSF SGI grades. Both properties increase with increasing silicon content. With the improved strength and ductility, lightweight castings of SGI materials are possible offering energy and material savings with better dimension control and geometric tolerance. Furthermore, higher silicon content increases the eutectoid temperature of casting which improves high-temperature properties of SGI castings. Due to their uniform ferrite matrix, they have smaller hardness variations than the conventional grades that contain both ferrite and pearlite. SSF SGIs are also reported to have better weldability and machinability with a potential to replace some steel grades. Up to 20 % reduction in machining cost has been reported for SSF SGIs as compared to ferritic-pearlitic grade [20]. SSFs also demonstrated better stability at elevated temperature and thermal cycles also reported by Larker [74]. Regarding fracture behavior, fracture toughness determined by instrumented Charpy testing indicated that SSF SGIs are indeed slightly tougher than equivalent ferritic-pearlitic grade [75]. Although SSF has attractive mechanical properties compared to usual SGI grades, brittleness and lower impact energy have posed some challenges in using SSF grades in some applications [75]. The strengthening effect of silicon is peaked at 4.3 %, and properties start to deteriorate afterward. The decline in mechanical properties of SSF SGIs with higher silicon is argued due to formation of embrittlement regions created by the segregation of excess silicon and graphite particles degeneration in the microstructure [73]. European standard has specified three standardized SSF grades having better mechanical properties which are listed in Table 2.1 with the

required casting properties.

Table 2.1: EN 1563 specified standardized SSF grades [20].

Material designation		Relevant wall thickness (t) mm	0.2 % proof strength (Rp0.2) MPa	Tensile strength (Rm) MPa	Elongation (A) %
Symbol	Number				
EN-GJS-450-18C	5.3108	$t \leq 30$	350	440	16
		$30 < t \leq 60$	340	420	12
		$60 < t \leq 200$	Guidence value to be provided by the marnufacturer		
EN-GJS-500-14C	5.3109	$t \leq 30$	400	480	12
		$30 < t \leq 60$	390	460	10
		$60 < t \leq 200$	Guidence value to be provided by the marnufacturer		
EN-GJS-600-10C	5.3110	$t \leq 30$	450	580	8
		$30 < t \leq 60$	430	560	6
		$60 < t \leq 200$	Guidence value to be provided by the marnufacturer		

Strengthening mechanism

Strengthening mechanism means restricting easy movement of dislocation across the crystal structure by introducing some form of crystal defects to resist dislocation movement. Four conventional methods exist to strengthen polycrystalline metals by grain size reduction, solid solution strengthening, strain hardening and precipitation hardening. In high Si SGIs, the ferrite matrix is strengthened by solid solution strengthening mechanism, which introduces impurity atoms (Si) into the crystal structure either by substitution or interstitially [5]. Higher silicon content is not solely responsible for enhanced mechanical properties in SSF grades as studies have shown that the properties can also be influenced by the presence of other elements in the chemical composition. Selection of elements in the chemical composition is also important to achieve higher strength grades as reported by Serrallach et al. [76].

Effect of high Si on the microstructure

EN 1563 standard accepts SSF SGIs with up to 20 % non-spherical graphite in the matrix [20], nodular graphite being defined as form V and VI in EN-ISO 945-1 standard. Graphite degeneracy is a major problem in SGI microstructures with higher silicon addition affecting SGI quality and properties. The most damaging of these degenerate graphite particles are the branched and interconnected degenerated graphite particles often occurring in the thermal center of SGI castings [77]. The occurrence of chunky graphite particles in the microstructure is

unclear and one of the least understood phenomena in SGI casting. Different research works based on their prospective studies have discussed possible reasons and proposed preventive actions to reduce such degenerated graphite particles. Kallbom et al. [78] reported on the solidification sequence in SGI casting to attribute the presence of degenerate graphite particles in the microstructure used for individual castings. It was argued that chunky graphite particles form early during the eutectic solidification since spherical graphite nodules were present in the last solidified areas around the cast surface. It was further proposed that the volume percentage of chunky graphite particles present in the microstructure results in a decline of ultimate tensile strength and a fracture elongation of the cast component [77]. Chunky graphite particles are a critical problem in heavy section SGI castings with large section thickness (typically larger than 60 mm) requiring long solidification time. The presence of this form of graphite particles in these types of castings, especially with SSF irons, has proven detrimental to properties such as ductility and Ultimate Tensile Strength (UTS) although with a lesser effect on Brinell hardness and yield strength [76].

Figure 2.6: Deviation in graphite shape in SSF a) chain of small graphite nodules and b) SEM image of non-nodular graphite [8].

Figure 2.6 shows deep etched optical microscope and Scanning Electron Microscope (SEM) images of SSF cast samples containing degenerated graphite particles. They macroscopically resemble a cluster of degenerated graphite particles in the microstructure. It was suggested that such graphite degeneration is due to inoculation process relating to the presence of oxygen and sulfur in the cast changing melt conditions during solidification to micro-segregation of alloying elements in the composition. The hypothesis relating to shortage of oxygen and sulfur to inoculation seems credible because tests on reducing chunky graphite

through the supplemental increase of oxygen and sulfur in the melt while inoculating have recorded sizeable reduction in chunky graphite particles [74]. Change in melt conditions during solidification might prove to be more challenging to control especially for thicker cast sections since chunky graphite particles typically form at the thermal center. So, oxygen shortage can also be attributed to the presence of strong oxide formers like Aluminum, Calcium, Cerium and other Rare Earth (RE) metal in the melt.

2.1.6 Fracture of SGI

The fracture behavior is related to initiation and propagation of cracks in the SGI microstructure, and it is highly influenced by microstructural parameters such as graphite morphology, matrix composition, solute elements in solid solution and temperature. Past studies [10–12, 33, 43, 69] on different ductile iron grades have shown most of the cracks to initiate at the nodule-matrix interface, within the nodules, and in the matrix region. The grain boundaries were also reported to influence crack initiation, propagation and fracture behavior of SGI. Similar to the mechanical properties of ductile irons, the fracture behavior is also mainly influenced by graphite nodules and matrix structure.

Role of graphite nodules in the fracture behavior of SGI

The role of graphite nodules in SGI is complex as it has dual role to play in the microstructure and fracture. The presence of graphite nodules was reported as stress raiser in the matrix [9, 12, 48]; on the other side, the predominant role of spheroidal graphite nodules as possible crack arrester improved its fracture toughness [11, 12, 29]. The role of the graphite on fracture depends on nodule size, count and mainly on the shape. The presence of graphite nodules itself acts as defect or void in the matrix structure creating stress concentration. The resulting stress concentration not only initiated cracks but also assisted in propagation and crack branching. However, spheroidal form of graphite nodules showed a considerable reduction in stress concentration than other graphite forms. The crack arrest properties of the spheroidal graphite nodules could play a significant role in the propagation of the initiated crack and could delay rapid fracture. So, it is believed that graphite shape, expressed in terms of nodularity, in the SGI microstructure will have an important role to define its fracture behavior.

Role of the matrix structure in the fracture behavior of SGI

The role of the matrix structure is to sustain applied load. Different matrix behaves differently to adjust to the applied load. The ferritic matrix known for its good ductility undergoes large plastic deformation causing graphite nodule voids to elongate before fracture. In the fully pearlitic matrix, there is less plastic deformation of the matrix, and the pearlite itself has carbide precipitates that could lead to brittle fracture of the matrix. It was reported that the pearlite matrix is more affected by the stress concentration effects of the graphite nodules than a ferritic matrix [69]. In the SGI with mixed matrix structure, the ferrite ring around the graphite nodules will not deform as in the fully ferritic matrix due to the constraining effect of the pearlite structure connecting all the ferrite rings. Because of the combined effect of plastic deformation of the ferrite around graphite nodules and less plastic deformation of the remaining pearlite matrix, the fracture behavior in the mixed matrix is of interesting in many studies. The plastic deformation of the ferrite matrix around is understood to reduce stress concentration caused by graphite nodules and the resulting voids. However, cracks have been shown to initiate in pearlite colonies more than frequently [69]. In general, the ferritic matrix is considered to have ductile fracture due to large plastic deformation before fracture, whereas pearlite structure is expected to show brittle nature of fracture due to its inefficiency to undergo large plastic deformation.

2.1.7 Review of SGI damage mechanism studies

For a long time, it was understood that graphite nodules - matrix debonding was the main or, at least the more frequent damaging micro-mechanism in ferritic SGI [79]. Based on the understanding, it was believed that graphite nodules and matrix debond under low stress, and then plastic deformation occurs in the matrix around graphite that causes microcrack initiation in the deformed matrix. Propagation of microcracks link graphites and further linkage of cracks to form a macrocrack leading to final failure of SGI component. Some other common belief on SGI damage mechanism was to consider graphite as rigid sphere acting like a void or crack arresters that minimize stress concentration effects [27, 72]. Over the past years, damage mechanisms in different ductile iron grades had been investigated on many occasions [9–12, 28, 39, 41–43, 79–84]. These works are thoroughly reviewed to have state-of-art knowledge of the damage mechanisms in SGIs.

Figure 2.7: Onion-like internal damage mechanism of spheroidal graphite nodules in ferritic SGI [9].

Significant works were done by an Italian group of researchers led by Professor Francesco Iacoviello and Vittorio Di Cocco from the University of Cassino and Southern Lazio, Italy. They had studied damage mechanisms in different as-cast SGI grades. One of the major contributions they had reported was the role of graphite nodules and damage mechanisms in SGI to be more complex due to the presence of mechanical property gradient within graphite nodules, most probably due to different stages of graphite solidification [10]. Graphite nodule core was characterized to be of lower hardness with respect to graphite shield. Softer nodule core corresponds to graphite nucleation and growth from melt, and harder nodule shield corresponds to nodule growth due to carbon diffusion from austenitic shield around nucleated graphite. In an in-situ tensile test of ferritic ductile iron, “onion-like” damage mechanism of spheroidal graphite nodules into graphite core and shield was disclosed [9]. The observed internal damage of spheroidal graphite nodules is illustrated in Figure 2.7. Most of the fully grown graphite nodules corresponded to graphite nucleation and growth from the melt (C_M), growth from

Figure 2.8: Fatigue damage micromechanism in different ductile iron grades a) graphite nodule decohesion in the ferritic SGI [10], b) crack branching in the ferritic SGI [11], c) crack propagation in the pearlitic SGI [11], d) crack branching in the pearlitic SGI [12], e) crack propagation by graphite decohesion in the ferritic-pearlitic SGI [11], and f) crack branching in the ferritic-pearlitic SGI [11].

eutectic solidification (C_E), growth due to reduced C solubility in austenite grain (C_A) and growth by eutectoid transformation into ferrite (C_F), which caused property variation within those graphite nodules. It was reported that the lower resistance corresponded to the interface ($C_M + C_E$) versus ($C_A + C_F$), where the internal damage of the graphite nodules was usually observed [9, 10, 67]. They

had also reported graphite nodules decohesion, crack passing through the matrix, crack branching mechanisms in different cast iron grades.

The fatigue damage mechanisms reported by the team of Professor Francesco Iacoviello and Vittorio Di Cocco are summarized for different SGIs. For the ferritic SGI, the matrix-nodules interfaces were not necessary a preferential crack path as the crack also propagated nearby graphite nodules. Decohesion of graphite nodules were characterized by the presence of residual graphite nodules on the ferritic fracture surface (Figure 2.8 a)). Secondary cracks initiated were stated to occur at the matrix-nodules interfaces and in the ferritic matrix (Figure 2.8 b)). Some secondary damage of graphite nodules with short cracks around and inside nodules was also reported [10,11]. For the fully pearlitic SGI, pearlite matrix-nodules decohesion was the most frequent damage mechanism without residual graphite nodules on the pearlite fracture surface (Figure 2.8 c)). As pearlite itself is lamellar combination of ferrite and cementite, the crack was reported to propagate along ferrite lamellae or trans-granular in the pearlite matrix [12]. Secondary cracks were less frequent around graphite; however, crack branching and bifurcations were observed in the pearlite matrix (Figure 2.8 d)) [11,12,40]. For the ferritic-pearlitic SGI, crack propagation was characterized by the presence of many secondary cracks initiated from the main crack (Figure 2.8 e)), and the matrix-nodules decohesion characterized by clear partial decohesion (Figure 2.8 f)) [27,28,85]. Table 2.2 summarizes the list of references that have studied damage micromechanisms in different SGI materials.

Table 2.2: Literature summary of damage mechanisms studies in different SGI materials.

SGI grades	References
Ferritic SGI	[9] [10] [11] [27] [39] [79] [81] [84]
Pearlitic SGI	[11] [12] [27] [40]
Ferritic-pearlitic SGI	[11] [27] [40] [82] [85]
Austempered ductile iron	[27] [41] [42] [43] [83]

In mid 1980s, Clement et al. [81] studied short crack growth behavior in ductile iron, reporting short cracks to propagate faster than long crack and the crack closure being responsible for reducing long crack growth. Later, in the early 1990s, Tokaji et al. [44] studied fatigue crack growth resistance and crack closure in different SGIs with different matrix structures. They concluded that the dual-phase microstructure showed better Fatigue Crack Propagation (FCP) resistance because of their excellent crack closure, but the intrinsic FCP resistance in SGIs was

reported independent of the change in the matrix structure. With the development of ADI, the interest was shifted to this high strength grade. Many works on fatigue study of the ADI were reported, Greno et al. [43] and Marrow et al. [41,42] studied fatigue crack nucleation and growth in ADI. Greno et al. stated that the effect of the matrix structure in different crack closure and reported greater effective stress intensity threshold in ADI than for steels. They also specified an irregular graphite-matrix interface, potentially initiating microcracks from the interface [43]. A similar study by Marrow et al. concluded fatigue cracks to nucleate at the matrix-nodule interface and shrinkage micro-porosities [42]. Further, the study of short fatigue crack showed crack arrest and retardation in ADI was controlled by the prior austenite grain size and graphite nodule size [41]. At the same time, Berdin et al. [80] studied damage and fracture in as-cast SGIs with different silicon contents. It was reported that SGI with higher silicon content showed higher mechanical properties, but lower toughness. With the matrix-nodules interfaces identified as the weak point in the ductile iron microstructure, Guillermer-Neel et al. [84] studied damage mechanisms and hardening behavior in ductile iron, and proposed graphite nodules decohesion and voids growth to be a continuous process. In a study by Stokes et al. [83], the main propagating crack was proposed to mostly initiate from casting defects like pores, and other cracks at the graphite-matrix interface to initiate in those graphite nodules in front of the main crack. Additionally, they also reported deflection of the main crack as it approaches microcracks and coalescence. Xue et al. [82] studied damage mechanism in very high cycle range for the ferritic-pearlitic SGI. Fractography study on the work reported fatigue cracks to predominantly initiate from the subsurface microstructure defects and also stated non-existence of endurance limit in cast irons in contradictory to conventional belief of endurance limit in cast irons. In addition to graphite nodule defects, casting defects like shrinkage cavities and porosities are also important microstructural features that affect crack initiation and propagation behavior. Shirani and Harkegard [29,47] had reported a comprehensive study on the effect of casting defects on the fatigue behavior of ductile irons. Another important contribution by them is the size effect on the fatigue life of SGI components [72]. In another study by Endo et al. [48], they studied the effect of defects and matrix on SGI fatigue and proposed a different way to estimate defect size for fatigue analysis of the SGI components. Recently, in addition to the studies by Professor Francesco Iacoviello and Vittorio Di Cocco, Fernandino et al. [39] studied fracture of ferritic ductile iron. They compared the fracture surface at different loading conditions and concluded that in fatigue tests,

the fracture to be a combination of cleavage facets and ductile striations on the fracture surface. However, as in many previous studies, they reported unclear fatigue striations. With the introduction of high silicon and molybdenum grades to achieve higher strength in SGIs, the damage mechanism studies on these were studied. Very recently, Norman et al. [86,87] have studied damage mechanisms in silicon-molybdenum compacted graphite iron subjected to thermo-mechanical fatigue load similar to engine exhaust. It was reported that the fatigue damage was linked with unloading modulus, which linearly declined with the number of cycles. They also studied the effect of thermo-mechanical and superimposed high-cycle fatigue interaction in the unloading modulus response [88]. Most of the studies reported focus on the spheroidal graphite nodules and matrix. But, some fraction of non-spheroidal graphite always exists in the microstructure which should be discussed along with damage mechanism as it causes higher stress concentration than spheroidal graphite nodules. Also, damage mechanism studies reported miss a comprehensive study to clearly explain damage mechanisms and the role of different forms of graphite particles at different stages of loading.

2.2 Deep Cold Rolling Process and its Effect on SGI

Different mechanical processes are applied on as-cast materials to enhance their properties. Most of the mechanical post-processing processes are kind of surface treatment that improves surface properties. Deep Cold Rolling (DCR) is one of the types. DCR process is similar to Low Plasticity Burnishing (LPB) in terms of working principles and setup, but slightly different regarding related plastic work. DCR process induces compressive residual stress and work hardening on surface and subsurface as most of the surface treatment processes [37, 89]. In addition, DCR also improves surface finish compared to other processes. Shot peening is a widely used inexpensive method, but the associated cold work is high (up to 50 %) which accelerates relaxation of compressive stress layer at higher temperature [90]. Laser Shock Peening (LSP) on the other hand produces a better compression layer with a low level of cold work as compared to shot peening, but the process is expensive to use [91, 92]. Thus, the DCR process has evolved as an inexpensive surface enhancement process that produces a higher depth of compressive case with relatively low cold work. For high-temperature applications, LSP with even

less cold work is applied, which helps to retain compressive case for a longer time even at high temperature, and also it enhanced Foreign Object Damage (FOD) resistance [90]. One major disadvantage of the DCR process over other processes is that it requires a direct tool to specimen contact, so for the application of this process, specific tool type is required based on the component to be cold rolled. In this work, DCR process is selected as a representative mechanical process to study the effect of such processes on SGI microstructure.

2.2.1 Introduction to DCR process and parameters

The name “*Deep Cold Rolling*” is based on the fact that, it is a cold rolling process with a deeper case of an effect than similar other surface enhancement process. Figure 2.9 shows a schematic diagram of the DCR process. A pressurized roller ball is pressed against the surface of the metallic part. This contact force causes plastic deformation on the surface and near surface material layer to achieve three DCR effects of burnishing (smoothing), work hardening and compressive residual stress. The DCR process improves fatigue, fretting fatigue and stress corrosion performance even at elevated temperature [93]. At present, most well-known application of DCR is deep rolled crankshafts, but the application of the DCR process is extending its boundaries into other critical components like surgical implants, turbine blades in power plant and many other aircraft components. During the

Figure 2.9: Schematic diagram of the DCR process.

DCR process many aspects need to be considered for example roller and workpiece material properties, surface topography, geometry of tool and workpiece, rolling force, feed rate, friction between tool and workpiece, process environment, etc. [21]. Out of these parameters, four major process parameters are explained below.

Rolling ball size

DCR tools used are case specific to workpiece/component geometry and function of the component. DCR tools are normally hydraulically pressurized to apply a rolling force. Size and form of the tool do affect results on the workpiece. As illustrated by Prabhu et al. [94] ball diameter has an inverse relation with surface roughness and direct proportional relation with hardness.

Rolling pressure/force

In the hydrostatic tool, fluid pressure is applied to the roller. Rolling pressure has a higher influence on the DCR effects. Roughness and hardness both were stated to increase with rolling force [94]. In regards to residual stress, higher pressure results in higher case of depth and also higher magnitude of compressive residual stress along the crosswise direction [95].

Percentage overlap

Percentage overlap is a measure of how much % of rolling area is re-rolled or in simple form how many times the same area is rolled. % overlap can be calculated by using Eq. 2.4. Main effect analysis by Prabhu et al. shows a smoother surface with a higher number of the pass, but the work hardening level is reduced [94]. No significant change was noted on the residual stress profile [95].

$$\%overlap = \frac{w - a}{a} \times 100\% \quad (2.4)$$

where,

w is the track width of single rolled track line

a is the step over between two rolling track lines

Feed rate

Feed rate is the speed of the DCR tool with respect to the workpiece. Higher feed rate means short processing time but with some compromise in continuities of surface state and slower feed rate favors a smoother surface. Feed rate has no significant effect on the compressive residual stress profile [95].

2.2.2 DCR fundamentals

The DCR process was simulated by Mader et al. [13] to understand the process fundamentals and undergoing material deformation mechanism. Figure 2.10 illustrates the result of that study. As observed in Figure 2.10 a), the maximum equivalent stress extends on both sides of the contact normal (c.n.) towards the surface and ends directly next to the outer contact area (c.a.) between the ball

Figure 2.10: DCR process mechanism base on FE simulation study [13].

and the workpiece. Further increase of rolling force would increase effective stress to the yield point, causing plastic deformation of material beneath the ball and stretching material laterally up to the workpiece surface [13]. Figure 2.10 b) and c) shows in detail the vertical and horizontal material deformations. As observed in figure 2.10 b) large vertical compressive stress acts directly below the ball, causing vertical plastic compression to form a surface depression on the workpiece. Due to vertical compression, the horizontal extension of material in contact is also observed. The horizontal extension causes lateral displacement of the contact zone and this lateral displacement successively causes horizontal compression of the neighboring area surrounding the contact zone as seen in figure 2.10 c). The horizontal compression near the contact zone comes together with vertical extension, resulting into the elevation of the area surrounding the contact zone. The deformation of material directly beneath the rolling ball and elevation of the area surrounding contact zone result into the formation of a crater geometry as indicated in figure 2.10 d) and a compressive residual stress layer is developed just below the contact zone [13].

2.2.3 Effects of DCR process on fatigue life improvement

Three major DCR effects, compressive residual stress, work hardening and surface finish have been known and proven to have a positive influence on fatigue strength and life [32, 37, 89–91, 96, 97]. These effects are discussed in details below.

Compressive residual stress

As most of the surface enhancement processes, DCR is also aimed to induce a layer of compressive residual stress. It has already been well understood that compressive residual stress favors higher fatigue life and tensile residual stress quicken a fatigue failure. Compressive residual stress acts as a pre-applied compressive load to close any existing cracks and defects, stopping them from further growth. It also makes crack initiation difficult as the applied load must overcome compression before reaching its failure strength. The role of residual stress on fatigue strength was explored in [98]. Another important aspect of compressive residual stress is its relaxation during cyclic loading and even at a faster rate for high-temperature application. The relaxation of stress in cycling load highlights the importance of studying cyclic loading, which was studied previously in [99, 100]. Investigation

of thermomechanical residual stress and work hardening relaxation on deep rolled specimens demonstrated that residual stress relaxation occurs by two mechanisms: mechanical and thermal relaxation. If instability of work hardening occurs especially in the region controlled by the mechanical relaxation process, deep rolling has no beneficial effect on fatigue lifetime for cyclic loading at elevated temperature [100]. Effect of DCR parameters on compressive residual stress is explored in detail elsewhere [95]. Among the four DCR parameters, rolling pressure has a predominant effect on compressive residual stress. Figure 2.11 shows that higher rolling pressure will increase the depth of compressive layer in steel components.

Figure 2.11: Influence of deep rolling force on the distribution of residual stress a) axial and b) tangential direction for normalized SAE 1045 [14].

Work hardening

Cold work hardening induced by the DCR process in combination with compressive residual stress and surface improvement has already been understood to improve fatigue behavior. The specific effect of prior cold work in fatigue behavior was investigated in stainless steel by Ganesh et al [101]. It was reported that prior cold work increases total strain fatigue resistance at strain amplitude less than 50 %. Increase in cold work % shows a positive effect on strain fatigue resistance. Work hardening occurs because of dislocation movement and generation within the crystal structure due to plastic deformation. Hardening strengthens metal and makes them more resistant to crack initiation and propagation improving fatigue strength. Prabhu et al. [94] studied the main effects of DCR parameters on hardness; the results indicated that ball size and rolling pressure have a propor-

tional relation, and initial roughness and number of pass have an inverse relation with the work hardening effect in the DCR process.

Surface finish

Effect of surface finish on fatigue behavior has also been well understood long ago. Smoother surface implies fewer surface defects as fatigue failure frequently initiates from such surface defects if any are present. Many authors suggest that surface finish improvement also takes some account for fatigue improvement on deep cold rolled components. Previous work by Prabhu et al. [94] also explored the effect of individual DCR parameters on surface finish. Another work compiled by Schulze [14] suggests that surface improvement is significant to a nominal rolling pressure of around 90 bar for SAE 1045 and surface roughness slightly increases instead with increasing rolling pressure as illustrated in Figure 2.12.

Figure 2.12: Influence of rolling pressure on roughness for nominal SAE 1045 [14].

2.2.4 DCR challenges in SGI material

There is no doubt on DCR process being established as a promising surface enhancement process. It has already been implemented on steels and lightweight metals like titanium and nickel-based alloys. DCR process has also been successfully applied in SGI crankshafts mostly pearlitic, to improve fatigue life [32, 96]. But SGI microstructure is different from other metals due to the presence of soft and brittle graphite nodules in the iron matrix. The DCR process response on graphite nodules is predicted to be different from the metal matrix as they cannot

undergo plastic deformation. Also because of the brittle nature and low strength, it is most probable that breakage of these graphite particles close to the surface might occur during the DCR process. Such case of graphite breakage or any changes on graphite morphology might have a severe influence on fatigue behavior of cold rolled components, as fatigue behavior of SGI is highly dependent on graphite morphology. Although the DCR process has been applied on SGI crankshaft in the form of fillet rolling, no literature is discussing such effect. Therefore, it is very important and challenging aspect that need to be explored, which the author intends to investigate.

2.3 Heat Treatment and Thermal Processing of SGI

The as-cast SGI comprises of ferrite or pearlite or ferrite-pearlite matrix at the end of solidification depending on the chemical composition and cooling parameters. SGI microstructure and properties could be controlled in as-cast grades; however, heat treatment is a versatile post-processing technique for extending the range of microstructure and properties in all kinds of cast irons. One good example of post-heat treatment resulting into a change in the matrix structure to improve properties is ADI achieved after austempering heat treatment on as-cast SGI. The most common heat treatment process and their objectives as listed by Chakrabarty [34] are as follows:

- Stress relieving at sub-critical temperature range to relieve the residual stresses in the castings.
- Annealing to increase the ductility, toughness, and machinability, to decrease hardness and eliminate carbides.
- Normalizing to improve strength with adequate ductility.
- Through hardening and tempering to impart strength and hardness.
- Austempering to develop a microstructure providing high strength and wear resistance and some ductility.
- Surface hardening to impart high surface hardness and wear resistance in some selected areas of the casting.

2.3.1 Heat treatment of SGI and its influence on microstructure

Heat treatment processes are aimed at changing matrix microstructure in SGI. In general, heat treatment processes involve austenitization of the iron matrix and subsequent controlled cooling or isothermal soaking or combination of them. As a consequence, the matrix structure at the end of the cooling can be categorized into thermodynamically stable ferrite matrix or metastable austenitic matrix. Thus, matrix alteration is the major microstructural change that modifies the mechanical properties of the ductile iron. Heat treatment process is directly related to the iron-carbon phase diagram and the final microstructure can be predicted. High carbon content in SGI benefits to harden the matrix without the necessity for expensive alloying additions. On the other hand, higher carbon can cause quench cracking due to the formation of high carbon martensite. Graphite particles play an important role in heat treatment. In the process of austenitization, carbon from the graphite particles diffuses into the austenite matrix to fulfill higher carbon solubility. Similarly, during slow cooling, carbon atoms rejected out from the austenite return to the graphite particles. Such transfer of excess carbon from nodules to the matrix and vice-versa favors efficient heat treatment in SGI [102]. However, the treatment temperature has to be higher than austenitization temperature for the major change in phases. With such a long history of heat treatment of cast irons and steels, heat treatment is well understood and the effect on the resulting microstructure is also well established. Details of different heat treatment processes can be easily found in many text books.

2.3.2 Effect of high Silicon on heat treatment

High silicon ductile irons have stable ferritic matrix at room temperature. Usually, the ferrite phase is stable up to a temperature of 900 °C; however, the presence of higher silicon causes the ferrite to austenite transformation to rise. Another improvement in SGI with higher silicon is the rate of oxidation at the elevated temperature, which drastically reduces due to the formation of a dense silicate layer at the surface. Silicon being ferrite stabilizer, it prevents the formation of carbides and pearlite in the thermal process. Further increase in silicon content from 4 to 6 % improves high-temperature properties and oxidation resistance, but the resulting SGI becomes very brittle as the impact transition temperature

increases to above the room temperature. So, even higher silicon content provided better high-temperature behavior, they are not quite often used beyond 4 to 5 % as the ductility and toughness are significantly reduced [34].

2.3.3 Thermal cycling of SGI

Heat treatment processes are mostly one cycle thermal process to achieve change in microstructure and properties. Thermal cycling is a thermal process rather than treatment process, which is applied repeatedly. However, the maximum process temperature is usually lower than the austenitization temperature so that there would be no major change in the matrix structure. Unlike heat treatment process, thermal cycling is an operation condition that applies additional thermal stresses to the component in service. Such additional thermal stress if applied with repeated mechanical load is termed as Thermo-Mechanical Fatigue (TMF). The components under thermo-mechanical loads fail earlier than thermal or mechanical load alone. Thermo-mechanical fatigue loading is the main cause of a component failure in many systems like automobile engines, aircraft engines, wind turbine parts, bridge and structures in extreme temperature change. So, both the mechanical load and the thermal load should be well studied in the components undergoing thermo-mechanical load.

Thermal cycling experiments had been performed on ductile irons to study thermal fatigue behavior. Roehrig [103] in his study has summarized four major types of failure that the component under thermal cycling can undergo. Type 1 was explained as the cracks first appearing on the hot zone of the component; Type 2 was described as a severe distortion of the component; Type 3 was designated as gross cracking of the entire section of the component; and Type 4 was referred to lowering of mechanical properties due to microstructural changes. These four failure type describes most of the failure in thermal cycling, however, in many cases the applied thermal cycling might not cause direct failure. Instead the material properties might degrade as referred in Type 4. The type of failure also depends on whether the component is constrained or unconstrained. In most experimental study of thermal fatigue, specimens were constrained during thermal cycling stopping it from free expansion at a higher temperature. But, in applications like exhaust manifold of the engine, it may be unconstrained allowing it to expand freely. In such unconstrained condition, thermal cycling was reported to mostly degrade the mechanical properties at the beginning before the component fails.

Thus, for the unconstrained thermal cycling applications, the effect of thermal cycling on the material microstructure and the mechanical properties should be studied before it can be used under such condition.

The thermal cycling or fatigue could be categorized into two types based on the frequency of the thermal cycle; low-frequency thermal cycling and high-frequency thermal cycling. In an automobile engine, both types of thermal cycle exist. The engine startup and shutdown account for the low-frequency thermal cycle, which occurs only one cycle in each startup. During the normal engine operation, the temperature change at different strokes of engine operation is a high frequency thermal cycle.

Effect of thermal cycling on the mechanical properties

A detail studies of low-frequency thermal cycling was reported by Buni et al. [15]. The thermal cycling studies at different temperature ranges showed a slight decrease in Modulus of elasticity in most of the cast irons as shown in Figure 2.13. The reported change in elastic modulus was higher for gray cast iron at both the temperature ranges (a) 120-600 °C and b) 300-750 °C). Comparing the reduction of modulus of elasticity at two temperature ranges, the reduction is higher at higher temperature range. It was proposed that the reduction of modulus of elasticity

Figure 2.13: Variation of modulus of elasticity of cast irons with thermal cycling; ADI-austempered ductile iron; DI-ductile iron; CGI-compacted graphite iron; GI-gray cast iron [15] a) 120-600 °C b) 300-750 °C.

Figure 2.14: Variation of hardness of cast irons with thermal cycling; ADI-austempered ductile iron; DI-ductile iron; CGI-compacted graphite iron; GI-gray cast iron [15] a) 120-600 °C b) 300-750 °C.

at higher temperature range was due to oxidation of graphite phase resulting into graphite holes. And the higher reduction in gray cast iron is predicted due to larger graphite oxidation of flake graphite.

Similarly, the effect of low-frequency thermal cycling on cast iron hardness was also studied by Buni et al. [15] and the result shown in Figure 2.14. Rapid drop in hardness was observed for all the cast irons up to 1000 thermal cycles and remained almost constant afterward. The Higher decrease of hardness was reported for ductile irons as compared to CGI and GCI. The superior thermal conductivity and thermal diffusivity of CGI and GCI were referred to the lower hardness reduction in these cast irons. The different hardness result indicated the role of graphite morphology in thermal cycling as the compacted and flake graphite shapes in CGI and GCI respectively were responsible better thermal conductivity and thermal diffusivity.

Effect of thermal cycling on the microstructure

The detailed study by Buni et al. [15] also reported microstructural changed on the thermal cycling of SGI. The changes in microstructure were reported in three points. Firstly, the matrix decomposition of pearlite and bainite into ferrite matrix

Figure 2.15: Graphite nodule separation in SGI after decohesion and oxidation during thermal cycling (120 - 500 °C).

was reported. Secondly, graphite nodules separation was reported at a maximum temperature higher than 500 °C. Due to the continuous changing differential thermal expansion of graphite nodules and the matrix, decohesion of graphite nodules was reported that followed oxidation of those graphite nodules to show complete separation of graphite nodules from the matrix as illustrated in Figure 2.15. Finally, grain growth and grain boundary separation were reported. In another study by Pan et al. [104] on thermal fatigue of thin-section ductile iron up to 800 °C, localized phase transformation was reported in the microstructure as the maximum cycle temperature was closer to the critical temperature. As the cooling step in the experimental study was aid by water quenching, martensite was observed in the microstructure after very first thermal cycle.

2.4 Extended Finite Element Method (X-FEM)

The Finite Element Method (FEM) is widely used method in solving fracture mechanics problems. Many different commercial software packages have been developed to help solve fracture problems based on the FEM theory. Although FEM has been proved to be well suited for the study of fracture mechanics problems, modeling crack propagation turns out to be difficult due to the modification of the mesh topology. To accurately model discontinuities with FEM, the crack tip stress singularity need to be accurately represented, which required FEM discretization

to conform with the discontinuity. The mesh has to be regenerated at each time step, and the solution has to be re-projected on the updated mesh at each time step. Re-meshing and re-projection are not only costly but also may have a troublesome impact on the quality of the analysis result. To overcome these limitations in conventional FEM, several approaches have been investigated to model crack problems. Re-meshing being the major drawback, many approaches were attempted to avoid re-meshing in the crack modeling, one of them which hype its popularity is the enrichment technique based on the partition-of-unity extended finite element method (X-FEM).

After development of X-FEM in 1999, it has emerged as a powerful numerical technique to solve most of the fracture mechanics problems. It has been broadly recognized as a easy method to model crack growth under the assumption of Linear Elastic Fracture Mechanics (LEFM). Comparing X-FEM with the conventional FEM, many improvements are reported, major being the flexibility in the mesh independent crack growth. In FEM, the crack and its growth required to follow element edges, where as in X-FEM, the crack definition and its growth is independent of element edges, allowing it to grow on mechanism driven arbitrary path. The essential idea in X-FEM is to add discontinuous enrichment functions to the conventional FEM approximation facilitating extra Degrees Of Freedom (DOFs) to the fractured elements.

2.4.1 Basic Review

Belytschko and Black [105] in 1999 first proposed a method for enriching finite element approximations to facilitate solving crack problems with minimal re-meshing. Later Dolbow et al. [105] and Moes et al. [106] introduced much more sophisticated method by introducing enrichment functions in the form of asymptotic near-tip field $F(x)$ and a Heaviside function $H(x)$. The Heaviside jump function was used to define discontinuous function across the crack surface. Based on the methods developed by Belytschko and Dolbow, Daux et al. [107] proposed a method to account for multiple crack branches by adding new discontinuous function $J(x)$ for the branching crack. This technique was used to model multiple crack branching, voids and cracks initiating from holes. The two-dimensional X-FEM enrichment technique was extended and implemented into three-dimensional crack by Sukumar et al. [108]. The accuracy of the X-FEM enrichment was demonstrated in three-dimensional crack model, where the interior crack surface was presented by

discontinuous function, and the crack front was defined by the two-dimensional asymptotic crack tip field. Belytschko et al. [109] later unified and extended the X-FEM modeling approach to generalize crack modeling with arbitrary discontinuities they have previously proposed. Level Set Method (LSM) was used in conjugation with extended finite element method to model two-dimensional crack growth for the first time by Stolarska et al. [110]. They used LSM to represent the crack location with efficient crack tip identification. Because of easy crack definition with the LSM, this approach was implemented in three-dimensional crack growth [111,112]. The extended finite element method and level set method have been studied and implemented in many occasions due to its capabilities to simplify crack problems. A review of the X-FEM was reported by Abdelaziz and Hamouine [113] from the theory to the specific applications of X-FEM. With the development of the theoretical and computation framework for fracture mechanics problems using X-FEM, it has been implemented in many commercial finite element softwares. It was implemented in ABAQUS in 2009 for the first time, where after there was minimal improvement in the X-FEM formulation in ABAQUS.

2.4.2 Enrichment functions

The extended finite element method is based on the concept of partition of unity. The partition of unity method is an enrichment procedure to improve finite element approximation by including the analytical solution of the problem in the finite element formulation. In case of fracture mechanics problems, the analytical crack tip field could be added to the finite element discretization. The X-FEM includes two overlapping sub-domain enrichment function to the finite element discretization as illustrated in Figure 2.16 a) and explained in following sections.

Heaviside enrichment function

In Figure 2.16 a), two types of enrichments are shown in four-node bilinear elements. The circled nodes represent the elements completely cut by the crack. Fracture of these elements results into a jump in displacement field, resulting into two additional DOFs (total of four DOFs per node for 2D). The Heaviside function provides a simple mathematical approach to model the displacement jump along the crack surface. Eq. 2.5 express the mathematical form of the Heaviside function that represents discontinuous displacement jump in X-FEM. The normal

Figure 2.16: a) Enriched nodes in the X-FEM formulation (Heaviside enrichment represented by blue circles and crack tip enrichment by green squares) [16] b) illustration of normal and tangential coordinates for a crack [17].

and tangential coordinates are illustrated in Figure 2.16 b).

$$H(x) = \begin{cases} 1, & \text{if } (x - x^*) \cdot \mathbf{n} \geq 0 \\ -1, & \text{otherwise} \end{cases} \quad (2.5)$$

where,

x is a sample (Gauss) point

x^* is the point on the crack closest to x

$\mathbf{n}$ is the unit outward normal to the crack at x^*

Crack tip enrichment function

Another type of enrichment in Figure 2.16 a) is for those elements that are not completely fractured but contains the crack tip. The Heaviside function is not valid at those elements; instead, additional enrichment functions were used to describe the asymptotic crack tip displacement fields. The crack tip displacement fields are expressed by Eq. 2.6 below.

$$F_\alpha(r, \theta) = \left[\sqrt{r} \sin \frac{\theta}{2}, \sqrt{r} \cos \frac{\theta}{2}, \sqrt{r} \sin \frac{\theta}{2} \sin \theta, \sqrt{r} \cos \frac{\theta}{2} \sin \theta \right] \quad (2.6)$$

where,

(r, θ) is a polar coordinate system with its origin at the crack tip

$\theta = 0$ is tangent to the crack at the tip

The above enrichment function at the crack tip can reproduce the asymptotic mode I and mode II displacement fields in LEFM, and establish the near-tip stress and strain singularities observed in analytical solution. With the consideration of these near-tip behavior, the accuracy of Stress Intensity Factor (SIF) calculation in mode I and mode II is reported to improve significantly [16, 106].

Considering the above explained enrichment functions, the displacement approximation for modeling crack using X-FEM transforms into following form (Eq. 2.7).

$$u^h(x) = \sum_{i=1}^n N_i(x) \left[u_i + \underbrace{H(x)a_i}_{\text{only Heaviside nodes}} + \underbrace{\sum_{\alpha=1}^4 F_{\alpha}(r, \theta)b_{i\alpha}}_{\text{only cracktip nodes}} \right] \quad (2.7)$$

where, $N_i(x)$ is the nodal shape function and u_i is the standard DOF of node i (u_i represents the physical nodal displacement for non-enriched nodes only). $H(x)$ is the Heaviside function and $F(x)$ is the crack-tip functions, and $a_i, b_{i\alpha}$ are the corresponding DOFs. In the absence of the enrichment functions, the above equation reduces to the classical finite element approximation $u_{fe}(x) = \sum_i N_i(x)u_i$. Thus, X-FEM retains many of the advantages of the finite element method.

2.4.3 Level set method

Numerical simulation involving moving boundaries requires efficient modeling and tracking of the moving boundaries. The Level Set Method (LSM) developed by Osher and Sethian is a numerical technique to model the motion of interfaces. In this method, the moving interface is represented by defining a zero level set function. In case of fracture mechanics problems, the crack tip may grow with loading, so the crack tip and its location is defined using level set functions in X-FEM. Two level set functions are required to completely define the crack tip. A level set function normal to the crack face $\phi(x)$ and another function tangential to the crack tip $\psi(x)$ as shown in Figure 2.17. As shown in the figure, the level set function $\phi(x)$ has zero value at the crack face, negative value below the crack face, and positive value above the crack face. The function $\psi(x)$ has zero value at the plane normal to the crack tip, negative value on the cracked side, and positive value on the un-cracked region. In this way, by the combination of these two level

set functions, the location of the crack tip and those elements along the crack surface could be tracked to implement Heaviside function and crack tip function in X-FEM.

Figure 2.17: Definition of level set function for crack modeling in X-FEM [18].

2.4.4 Numerical integration and convergence

In crack modeling, fracture of the finite element changes displacement and stress into non-linear field, which cannot be accurately integrated by the standard Gauss quadrature. To solve this problem, these elements must be subdivided into sub-domains, considering crack as the subdomain boundary, shown in Figure 2.18. In Figure 2.18 a), the elements cut by the crack are sub-divided into tri and quad elements, and the numerical integration is carried out on these elements. The sub-triangulation method was first proposed by Dolbow [114]. Another versatile and easy method to subdivide the fractured element in X-FEM is phantom node approach illustrated in Figure 2.18 b). Phantom nodes are imaginary nodes, which are superposed on the original real nodes to simulate discontinuity of the cracked elements. Before fracture of elements, the phantom nodes are completely constrained to its corresponding real nodes. As the element is fractured, the two sub-domains are formed by combination of some real and phantom nodes as shown in Figure 2.18 b). With these two sub-domains the corresponding real and phantom nodes can move freely to represent discontinuous field on the enriched nodes. X-FEM method has better numerical accuracy than conventional FEM, in modeling discontinuities. However, the rate of convergence is not improved due to the presence of additional enrichment functions. So the end result is lower conver-

Figure 2.18: Integration scheme in X-FEM a) sub-division of elements cut by the crack b) phantom node approach.

gence rate than the FEM model. Some methods have been proposed to obtain optimal numerical accuracy with better convergence for X-FEM problems. Fixed enrichment area, higher order X-FEM and construction of blending elements are some of the proposed techniques to improve convergence.

2.4.5 X-FEM implementation in ABAQUS

The discontinuities modeling capabilities using the extended finite element method was introduced in ABAQUS 6.9 release in 2009 for the first time. Discontinuities are modeled by including enrichment function and additional degrees of freedom to the FEM formulation as explained in section 2.4.2. The enrichment functions consist of the near-tip asymptotic function to represent singularity around the crack tip, and a jump function to represent displacement function across the fracture surface. The location of the discontinuities and the crack tip is defined by numerical procedure based on the level set method. In the discretized mesh, in addition to the global coordinate system, each node of the FE model is characterized by two additional coordinate systems with their origin at the crack tip. The coordinate parameters are defined as PHILSM and PSILSM, which corresponds to $\phi(x)$ and $\psi(x)$ functions defined in section 2.4.3. These parameters are non-zero only for the enriched nodes, are also used to visualize the crack in by requesting them as the output parameters. The X-FEM implementation in ABAQUS/Standard is based on the phantom nodes approach, where phantom nodes are superposed to the real nodes to represent discontinuities. X-FEM formulation in ABAQUS provides an

attractive and effective way to simulate crack initiation and propagation along an arbitrary, solution-dependent path without the requirement of re-meshing. The formulation can be implemented in static analysis and limited implicit dynamic procedure. Other specific X-FEM analysis capabilities in ABAQUS are low-cycle fatigue analysis using direct cyclic approach, geostatic stress field approach and coupled pore fluid diffusion/stress analysis [17]. Other features of the X-FEM in ABAQUS as of v6.14 are listed below:

- It does not require the mesh to match the geometry of the discontinuities.
- It can also be used to perform contour integral evaluations for an arbitrary stationary crack without mesh refinement at the crack tip area.
- It allows contact interaction of cracked element surfaces based on a small-sliding formulation; however, finite sliding is not enabled in its capabilities.
- In fluid pressure fracture mechanics, the discontinuities can be modelled with fluid pressure field allowing fluid to flow within the fractured element surfaces.
- It also allows the application of distributed pressure loads to the cracked elements.
- It allows the output of some of surface variables on the cracked element surfaces.
- It allows both material and geometric nonlinearity in the analysis.
- Currently, X-FEM formulation is available only for first-order stress/displacement solid continuum elements, first-order displacement/pore pressure solid continuum elements and second-order stress/displacement tetrahedron elements.

There are two modeling approaches that can be used to model crack growth using X-FEM in ABAQUS. One of them is developed based on damage mechanics concept and uses cohesive zone formulation and other one is based on the fracture mechanics concept with the combination of the energy release rate used to predict crack growth. Both of the modeling approaches used phantom nodes to represent discontinuous jump function on the enriched nodes and are briefly described below.

Cohesive approach

Usually, in cohesive modeling approach, the crack propagates along a known path within the cohesive material region. But, using the X-FEM based cohesive approach enables the crack initiation and propagation to be modelled independent of the mesh and the crack can grow in any arbitrary solution dependent path within the enriched domain. Both cohesive element approach and surface based cohesive behavior capabilities in ABAQUS can be used in X-FEM analysis. The material response and the initiation and evolution criteria are defined based on the traction-separation behavior. In this approach, only the Heaviside jump function is defined along with the phantom nodes to the enriched domains. The main aim is to model growing crack, so the near-tip asymptotic singularity function is not included. The fracture process has to propagate across an entire element at a time to avoid the need to model the crack tip enrichment. Both brittle and ductile fracture process could be modeled using this approach.

Linear elastic fracture mechanics approach

Similar to the cohesive approach for crack growth, linear elastic fracture mechanics approach also include Heaviside jump function without modeling the crack tip enrichment, and the crack has to fracture entire element at a time. In this approach, the strain energy release rate (G) at the crack tip is calculated based on modified Virtual Crack Closure Technique (VCCT), and the crack will advance when the combine energy release rate equals or exceeds the critical value (G_c). VCCT approach is based on the assumption that in the process of small crack opening, the strain energy release rate is equal to the amount of the work required to close the crack. LEFM based X-FEM provides several empirical criteria to consider mix-mode behavior. Power law being one of the most commonly used relation to model mix-mode. Benzeggagh and Kenane (B-K) criterion is another law for mode mixity and is commonly used for delamination problems of the composite materials.

2.5 Microstructural Damage Modeling on SGI Material

The inhomogeneous stress and strain distribution in SGI microstructure leads to necessity of modeling damage in representative microstructure model. The SGI microstructure contains randomly distributed graphite nodules, which plays important role in the inhomogeneous stress-strain distribution and also in the failure mechanisms. To represent the overall behavior of the SGI microstructure, a smaller region representative of the microstructure is used, known as Representative Volume Element (RVE). Many definition of RVE have been stated, but the definition provided by Rodney Hill [115] is comprehensive, which states "*RVE is entirely typical of the whole mixture on average and contains a sufficient number of inclusions for the apparent properties to be independent of the surface values of traction and displacement, so long as these values are macroscopically uniform.*" In case of perfectly periodic materials, one periodic unit can represent whole microstructure, but in random microstructure, the RVE implementation is complex. So there are many methods that have been used to model microstructure of both inhomogeneous and homogeneous material. Hutter et al. [116] have provided a comprehensive review of micromechanism modeling on ductile iron. They have chronologically reviewed, from the experimental damage mechanism studies to the different modeling approaches used to model SGI material. In SGI microstructure, it consists of graphite nodules inclusions in the ferrite matrix. These nodules are periodic over the microstructure, however they are not identical. So, the RVE is created by considering it as periodic inclusions and in other approach sample microstructure model is used to represent random distribution, size, shape and orientation. Various approaches used to model SGI microstructure could reviewed into two broad categories in following sections.

2.5.1 Cell model

In cell model, spherical or ellipsoidal graphite particles arranged within a spherical, cylindrical or polyhedral matrix cell is used as RVE model. Base on the homogenization concept, one representative cell could be used as a RVE if the macroscopic fields did not change significantly over distance comparable to the spacing of the graphite nodules. In case, where one cell could not perfectly represent the overall macroscopic fields, a RVE with few graphite particles were modeled instead. Con-

sidering RVE shape, cubic cell could arrange to fill a room completely, whereas spherical or cylindrical cells could not fill all the space. However, due to macroscopically isotropic behavior, spherical cell was most extensively used in analytical solutions. Gurson model was a pioneering work that had been used to model SGI with graphite particles as void. Analytical solutions are not available for all the mechanisms like void coalescence, which required numerical method to model. In numerical method like FEM, cubic and cylindrical cells were implemented in RVE as the implementation of boundary conditions was easier than in spheroidal cell. In the context of numerical method, the Gurson-Tvergaard-Needleman (GTN) model proposed by Tvergaard and Needleman, considerably improved ductile fracture mechanism modeling. By adopting Gurson model, Monchiet et al. [117] have proposed plasticity damage based micromechanical modeling, which allowed taking into account plasticity and damage mechanisms occurred at the scale of persistent slip bands. Further, it was shown that the modeling criteria accounts for the mean stress effect [118].

Cell model was first implemented with prior existing voids for the graphite nodules of total volume fraction nearly equal to 10 % [79,119]. So, it was essential to represent the elastic-plastic properties of the iron matrix. The material response in these models was used based on the tensile tests for the ferritic bulk material as it has completely ferrite matrix as load bearing phase. Dong et al. [79] used the bulk properties for the ferritic SGI and have verified by the micro hardness test. Dahlberg [119] on the other side assumed almost identical plastic behavior of the matrix. Nicoletto et al. [120] simulate cell model with two voids of different size and reported that the macroscopic yield stress decreases with increasing difference of the size of the voids.

To address the difference between the cell models with and without graphite particle, Brocks and Steglich [121] simulated graphite nodule as a rigid unbonded particle. Comparison with void cell model hardly reported any effect initially; however, as the void coalescence started, they reported considerable effect of the graphite particle in the cell model. Dong et al. [79] modeled graphite particle as bonded elastic, which reported better agreement of the experimental result with the void model. Later Collini and Nicoletto [122] modeled graphite as unbonded elastic particle, which hardly reported any difference with the cell model without graphite particle. So, it is clear that graphite particles are important microstructural features in SGI, and modeling completely bonded or unbonded over the time will be the two extreme behaviors as the interface is usually bonded at the beginning but debonded at around yield stress.

2.5.2 Real microstructure based model

In contrast to homogenization approach in cell model, some studies [123–127] have attempted to develop RVE model based on the real material microstructure. Langer et al. [124] developed an image based finite element analysis method that was given the name Object Oriented Finite element (OOF). The image based FEM method has been updated in few occasions and details of the OOF2 program could be found in website [124]. In OOF2, the microstructure image can be inputted, in which finite element model can be developed by discretizing the micrograph based on the color maps and constitutive models could be incorporated. Wei et al. [123] have used OOF to model dual phase steel 600 for micromechanical modelling of bending under tensile forming. Ljustina et al. [125] used the OOF approach to model cast iron microstructure with graphite nodules for FE simulation of the machining process. These works reported efficient capabilities of OOF to represent real microstructure image to generate RVE model and FE simulation on the model developed. Fukumasu et al. [127] analyzed the cylinder rolling process on compacted graphite iron microstructure. The study of the numerical analysis reported efficient microstructural RVE model that well represented the stress concentration around the graphite flakes in the iron matrix. However, no particular implementation of OOF software could be found in SGI material for the damage modeling, which would be quite interesting to see.

2.6 Summary

Ductile cast iron is a type of cast iron with spheroidal form of graphite particles that allowed it to have cast iron properties of excellent castability, good machinability, excellent anti-vibration properties and cheap cost in combination with higher fatigue resistance, toughness and ductility. Due to spheroidal shape of graphite particles, it is also known as Spheroidal Graphite cast Iron (SGI). After its development in 1943, its application has increased because of its cheap cost offering wide range of properties. The underlying microstructure is the major reason for its superior properties. SGI microstructure consists of spheroidal graphite nodules in the iron matrix, which can be ferritic, pearlitic or combination based on the chemical composition and solidification parameters. Graphite morphology influence mechanical properties and also plays vital role in the crack initiation and propagation behavior. SGI materials are treated to form spheroidal

form of the graphite particles; however, the amount of spheroidal graphite nodules and its nodularity is highly dependable on the graphite nucleation and its growth morphology. The graphite growth can go through transition to form degenerated graphite particles like chunky, compacted, irregular and exploded graphite particles due to lack of certain elements locally in the melt. So, even if the chemical composition and casting process are well controlled, some fraction of degenerated graphite particles is inevitable. The role of such degenerated graphite particles and the matrix structure should be studied in combination to clearly understand crack initiation and propagation behavior in SGI material. The iron matrix in the SGI microstructure is the major load carrying phase on which SGI mechanical properties rely on. On the as-cast grade, the ferritic matrix provided excellent ductility with moderate strength, the pearlitic matrix provides better strength with reduced ductility and the ferritic-pearlitic has properties in between based on the matrix composition. Heat treatment processes have been in use for long time as a post process to alter the matrix structure. Another inexpensive method to improve mechanical properties in as-cast SGI grade is by adding higher silicon content in the melt to form high silicon SSF SGI. Silicon is an important element in the SGI cast. Silicon is a graphitizer, and as such promotes graphite nodules formation during solidification produce ferritic matrix structure. Silicon also helps to solid solution strengthen the ferrite matrix. Thus, the SSF SGI provided dual advantage of higher mechanical strength of the ferrite matrix and better ductility due to the formation of complete ferrite matrix. However, the improvement in properties is reported up to silicon content of 4.3 %. The crack initiation and propagation behavior in SGI microstructure is highly influenced by graphite particles and its size and shape. The role of graphite nodules in SGI is complex as it has dual role to play in the microstructure and fracture process. The presence of graphite nodules is reported as stress raiser in the matrix; on the other side, the predominant role of spheroidal graphite nodules as possible crack arrester improved its fracture toughness. So, it is essential to understand how the presence of degenerated graphite particles influences the crack initiation and propagation behavior and its comparison with the role of spheroidal graphite nodules. Such study on high silicon SGI will bear additional interest as it is not reported in literature.

Use of SGI material as cheap material alternative in many automobile and wind turbine components surged to improve the mechanical and fatigue properties. Surface treatment processes are one of the well explored alternatives to improve crack

initiation and growth resistance. Heat treatment processes have been in use for long. These mechanical and thermal processes may cause changes in microstructure and fatigue properties. Deep Cold Rolling (DCR) process is a surface enhancement process that is applied specifically to improve fatigue resistance by three effects of compressive residual stress, strain hardening and surface smoothening. In SGI material, DCR process could be challenging due to the presence of the non-metallic graphite inclusions in the iron matrix. The DCR process may influence graphite particles and the area around them, which demands detail investigation. Similarly, for the thermal process also some changes in the material microstructure could be expected that need to be studied in relation to the crack behavior.

In recent years, use of FEM simulation for crack problems to solve fracture mechanics problems and to predict failure have drastically increased with computational advances and development of advance FEM techniques. X-FEM clearly stands out in context of efficient and accurate crack simulation. The main advantage of X-FEM is mesh independent crack analysis and crack growth, which eliminates rigorous re-meshing to confirm mesh faces with the crack surface on each crack growth step. Many works have used X-FEM technique to simulate crack growth and to solve fracture mechanics problems. Use of Heaviside enrichment function to define discontinuities and crack tip enrichment function to explicitly define the crack tip stress singularity have improved crack analysis. Many commercial FEM softwares have attempted to include X-FEM analysis technique. X-FEM in ABAQUS is commonly used and reported formulation. In ABAQUS, X-FEM can be implemented in two basic approaches; cohesive approach and linear elastic fracture mechanics based VCCT approach. VCCT approach is suitable for brittle fracture case where the material is primarily within the elastic region. The cohesive approach provides more flexibility in terms of fracture process modeling. Damage modeling in microstructure model is growing with the development and implementation of advance techniques like X-FEM in commercial code. Literature review showed extensive use of cell model as a RVE model to study damage mechanisms. In SGI material, cell models were implemented with graphite particles as primary voids, graphite particles as elastic unbound phases or graphite particles as elastic particles constrained to the matrix by different researchers. Another alternative to cell model used is modeling of the real microstructure using image based FEM analysis approach, where real micrograph is represented into FEM model, and the constitutive and boundary conditions implemented on the FE rep-

resentation of the micrograph. With all the above reviews in mind, it would be interesting to study and connect microstructure study with the damage study and modeling to properly understand microstructure dependent damage mechanisms.

The literature review showed that damage mechanism studies in SGI as a hot research topic and further research works trending toward FE prediction of tensile and fatigue damage in SGI microstructure. However, a comprehensive micromechanism understanding is not very clear, specifically for the newly casted SSF SGI grades. This research work aims to establish microstructure characterization method and a better understanding of microstructure damage micromechanisms to develop a foundation for FE simulation of SGI micromechanism. The microstructure characterization and damage mechanisms will be implemented on RVE model (cell model) to check the effectiveness of the modeling approach and also to estimate some of the model parameters that cannot be obtained experimentally. Finally, the modeling approach will be attempted to formulate in real microstructure representative FE model to simulate crack propagation in SGI microstructure.

With the comprehensive literature review conducted, this research work will attempt to contribute on, a) microstructure and material characterization of SGI material and investigate the effects of thermal and mechanical processes on SGI microstructure, b) detailed damage mechanisms studies on high silicon SGI grade with comprehensive study of the effects of graphite particles in such damage micromechanisms, and c) develop new microstructure modeling approach to represent real SGI microstructure for microstructure dependent analysis like crack growth analysis using advance XFEM techniques.

Chapter 3

SGI Microstructure Characterization: Effects of Thermal and Mechanical Processes

This chapter discusses microstructure characterization of as-cast Spheroidal Graphite Irons (SGIs), their mechanical properties and phase properties. Two methods to estimate phase properties of SGI microstructure using tensile test and nanoindentation test are compared. Further, the effects of Deep Cold Rolling (DCR) and thermal cycling on as-cast SGI microstructure and phase properties are investigated. Based on the microstructure study in different SGI grades and after the thermal and mechanical process, representative microstructure parameters are identified.

3.1 As-cast SGI Material

The aim of this work is to study as-cast SGI materials that are strengthened by solid solution strengthening mechanism without incurring additional heat treatment expenses. Three as-cast SGI grades are studied in this work; one of them (EN-GJS-500-7) is an ordinary ferritic-pearlitic grade, and the other two are silicon Solution Strengthened Ferritic (SSF) grades. From now on in this thesis, the as-cast SGI grades will be denoted by their short forms 500-7, 500-14 and

600-10 for EN-GJS-500-7, EN-GJS-500-14 and EN-GJS-600-10 respectively. The wt. % chemical compositions of all the investigated grades are presented in Table 3.1. Silicon is an important element, which increases from 2.36 % in 500-7 to 3.71 % in 500-14 and 4.25 % in 600-10 grades. Higher Mn content in 500-7 promotes pearlite in the matrix to form a ferritic-pearlitic matrix. Magnesium is a nodulizer element to form graphite in spheroidal form. Carbon equivalent (C_{eq}) representing the equivalent percentage of carbon considering the effect of other alloying elements was evaluated using Eq. 2.3. The carbon equivalent indicated in iron-carbon phase diagram plays an important role in solidification of ductile irons from the melt, and for SGIs, C_{eq} is desired around the eutectic region (slightly hyper-eutectic). All the investigated SGIs have C_{eq} in the eutectic region and adequate nodulizer content to form spheroidal morphology in most graphite particles.

Table 3.1: Chemical composition of cast SGI metals (% wt.)

Grade	C	Si	Mn	S	P	Mg	Ceq	Fe
EN-GJS-500-7	3.51	2.36	0.408	0.004	0.006	0.036	4.11	balance
EN-GJS-500-14	3.2	3.71	0.182	0.006	0.009	0.042	4.15	balance
EN-GJS-600-10	3.13	4.25	0.169	0.007	0.0095	0.036	4.2	balance

Figure 3.1: Specimen casting pattern geometry (reprinted with permission from project collaborator Anders E. W. Jarfors, Jonkoping University).

The sample material was casted as a 50 mm thick plate (casting configuration is shown in 3.1). To increase homogeneous graphite nucleation sites, inoculation process was performed both in a ladle and in the stream using commercially available inoculant Foundrisil 67 (64–70 % Si, < 1.25 % Al, 0.17–1.25 % Ca, 0.75–1.25 % Ba and Fe balance). To favor spheroidal graphite growth morphology, Mg treatment was performed using commercially available FeSiMg nodulizer Ceriumfritt

3.1. AS-CAST SGI MATERIAL

(44–48 % Si, 5.5–6.5 % Mg, < 0.1 % RE, 0.3–0.5 % Ca, < 0.7 % Al, < 0.05 % Ce and Fe balance). The combination of inoculation and Mg treatment process was adequate to yield most of the spheroidal graphite nodules, but some fraction of graphite particles were irregular and compacted.

3.1.1 Mechanical properties

The mechanical properties of investigated SGI grades were determined from the standard tensile test. The mechanical properties of all the grades were higher than the minimum specified by European standard indicating that the casting process is adequate to achieve standard SGI grades (Table 3.2). 500-7 and 500-14 have equivalent Ultimate Tensile Strength (UTS), 500-7 strengthened by promoting pearlite structure in the matrix and 500-14 solution strengthened by higher silicon content. Comparison of the mechanical properties showed similar elastic modulus values, higher yield strength of 500-14, slightly higher UTS of 500-7 and much higher elongation of the 500-14 grade. The elongation rapidly increased in 500-14 with 3.71 % Si, however, further increase of silicon to 4.25 % in 600-10 started to show a decline in elongation. Comparison of the mechanical properties followed the understanding of solids solution strengthened ferritic SGI grade with higher UTS, yield strength and elongation.

Table 3.2: Tensile properties of as-cast SGI materials.

Grade	Elasticity modulus (GPa)	Yield strength (MPa)	Ultimate tensile strength (MPa)	Elongation to fracture (%)
EN-GJS-500-7	165±5.5	315±2.5	539±2.5	10.4±0.6
EN-GJS-500-14	170±1	410±9	511±6	17±6
EN-GJS-600-10	170	444	555	9.9

3.1.2 Microstructure

Microstructure characterizations of as-cast SGIs were carried out to explore microstructure constituents. As-cast SGIs were ground by SiC paper and polished by diamond suspension to 1 μm , and finally etched with 2 % nital solution to reveal underlying microstructure. 500-7 exhibited ferritic-pearlitic matrix around

Figure 3.2: Microstructure of cast EN-GJS-500-7 containing spheroidal graphite embedded in ferrite and pearlite (2 % Nital solution etched) (graphite nodules in black, the ferrite in white and pearlite in brown).

Figure 3.3: Microstructure of high silicon cast SGI with complete ferrite matrix (2 % Nital solution etched) a) EN-GJS-500-14 and b) EN-GJS-600-10 (graphite nodules in black and ferrite in white).

spheroidal graphite nodules as illustrated in Figure 3.2. In the optical microstructure image, black graphite nodules are surrounded by white ferrite phase and pearlite structure in connecting ferrite that resulted into “*bull’s eye*” microstructure. Figure 3.3 a) and b) shows optical micrographs for 500-14 and 600-10 grades respectively. Here, both the micrographs showed graphite nodules in all white ferrite matrix. As the nital solution has no etching effect on the ferrite phase, the only effect of etching was on grain boundaries to expose grains. By comparing the micrographs, it was difficult to sight any significant difference other than matrix structure. However, the graphite nodule shape showed some differences; for the 600-10 grade micrograph, which contains a larger number of non-nodular graphite particles compared to 500-14 and 500-7.

For the quantitative estimation of microstructure parameters, around ten micrographs were studied for each sample. The micrographs were image processed to give clear and distinct color to each phase, and were then read by *Image pro plus* software to identify the matrix composition. For the accurate determination of graphite morphology, the images were represented as a binary image in Matlab, where all the matrix and graphite nodules were given opposite values. In the binary image, each of the graphite nodules was characterized for its area and diameter, based on which nodularity of the whole micrograph was estimated. The average microstructure characterization results for all the as-cast grades are presented in Table 3.3. 500-7 has around 40 % ferrite and 60 % pearlite matrix composition. Around 170 graphite nodules were observed in 500-7 grade having an average diameter of 21.5 μm . The nodularity by area was 82.5 % for 500-7, which was higher than for 500-14 and 600-10 grades. For the SSF grades, the matrix was completely ferritic with a tiny patch of pearlite here and there. The average nodule diameter for SSFs was slightly higher than for 500-7, showing the reverse effect of lower nodule counts of 122 and 139 respectively in 500-14 and 600-10. Nodularity is another important measure that was observed to be affected by higher silicon in SSFs. In the literature, similar phenomena were reported [66, 69, 70], stating formation of degenerated graphite particles like chunky and irregular forms. As observed in the result the reduction in % nodularity is even higher for 600-10.

Table 3.3: Graphite morphology characterization and matrix composition of as-cast SGI grades.

Grade	Graphite morphology			Matrix composition	
	Avg. dia (μm)	Nodularity by area (%)	Nodule count (per mm^2)	Ferrite (%)	Pearlite (%)
EN-GJS-500-7	21.46 $\pm$ 7.5	82.5 $\pm$ 7.2	170 $\pm$ 32	41.06 $\pm$ 6.5	58.94 $\pm$ 7.8
EN-GJS-500-14	27 $\pm$ 8	74 $\pm$ 3	122 $\pm$ 36	98 $\pm$ 1	1 $\pm$ 1
EN-GJS-600-10	26 $\pm$ 11	68.8 $\pm$ 8.65	139 $\pm$ 12	98 $\pm$ 1	1 $\pm$ 1

In addition to graphite morphology and matrix structure, casting defects like degenerated graphite particles, shrinkage cavities, porosities and other inclusions have an adverse effect on the static and dynamic behavior of SGIs. In the investigated SGI materials, flake graphite particles were observed on the casting surface as shown in Figure 3.4 a). Such thin layer of flake graphite adjacent to the mold is due to higher sulfur in the molding sand. The sulfur in the sand reacts with the magnesium in the iron melt to form magnesium sulphides and effectively

Figure 3.4: Casting defects observed on the SGI microstructure a) flake graphite on the casting surface b) shrinkage cavity.

de-nodulise the iron. Thus, the casted surface was machined, and only middle section with optimized microstructure was used for all the experiments in this work. Some shrinkage cavities as shown in Figure 3.4 b) were also observed in the as-cast grades; however, it should be noted that the size of such cavities was similar to the graphite nodule size and no larger shrinkage cavities were observed. Optimized casting process and medium section thickness might be the reasons for larger defect free casting. Other common defects observed in the as-cast microstructure were degenerated graphite particles in the form of chunky graphite, compacted graphite and irregular graphite, and micro-porosities. These casting defects were randomly distributed and were not easy to detect under an optical microscope, so the casting defects on the different SGI grades under study could not be precisely compared.

3.1.3 Nanoindentation phase hardness and elastic modulus

SGI microstructure comprises graphite nodules and the matrix structure. The properties of graphite nodules are very different from the matrix properties, and the matrix itself may have different properties depending on the matrix composition. Phase properties along with microstructure characterization information could help to understand the effect of various material processing and loading on the SGI material. These properties cannot be directly determined from conventional material testing. Nanoindentation test also known as Instrumented Indentation Test (IIT) is a new material testing facility that can measure loading response on a very confine region. In nanoindentation test, the load and the in-

denter displacement were recorded during indentation process, and the material properties were estimated from the load-displacement (P-h) response [19]. Figure 3.5 shows a schematic diagram of the indenter in contact with the specimen and the corresponding load-displacement curve. Many works [128–135] have been published demonstrating nanoindentation test as a potential material testing method for testing microscopic level properties. At the current state of the art, nanoindentation test has been successfully established to measure hardness and elastic properties of different kind of materials. In most of the nanoindentation setups, the pioneer method developed by Oliver and Pharr [136] for Berkovich indenter are used to determine elastic modulus (Eq. 3.1) and hardness (Eq. 3.4).

Figure 3.5: Schematic diagram of nanoindentation process with the corresponding load-displacement response [19].

$$E^* = \frac{dP}{dh} \frac{1}{2} \frac{\sqrt{\pi}}{\sqrt{A}} \quad (3.1a)$$

$$\frac{1}{E^*} = \frac{(1 - \nu_I^2)}{E_I} \frac{(1 - \nu_S^2)}{E_s} \quad (3.1b)$$

$$A = 3\sqrt{3}h_c^2 \tan^2 \theta = 24.5h_c^2 \quad (3.2)$$

$$h_c = h_{max} - \varepsilon \frac{P_{max}}{dP/dh} \quad (3.3)$$

$$H = \frac{P}{A} \quad (3.4)$$

where, E^* , E_I and E_S are reduced modulus, elastic modulus of indenter and elastic modulus of sample respectively. ν_I and ν_S are Poisson ratios of indenter and sample respectively. P and P_{max} are the load and maximum load in the nanoindentation test respectively. h , h_c and h_{max} are the depth, total residual depth and maximum depth respectively, A is the projected contact area, θ is the face angle for the Berkovich indenter, $\varepsilon = 0.75$ for Berkovich indenter.

The nanoindentation tests were performed on the polished samples and etched afterward to expose the matrix in ferritic-pearlitic grade, as etching before the nanoindentation test had affected the microstructure integrity and the nanoindentation results. *Agilent Nano indenter G200* having Berkovich indenter was used in this work. For the uniformity of the test method, maximum indentation depth was fixed to 300 nm, considering the possibility that the surface exposed graphite particles and the matrix layer could be just few micrometers due to microstructural inhomogeneity.

Figure 3.6: Load–displacement curve for different phases of cast SGI materials.

For each phase in each of the samples, one representative P-h curve matching its E and hardness values to the average (in Figure 3.7) is presented in Figure 3.6. From the P-h curve, a clear difference in the load distribution among graphite nodules, ferrite and pearlite could be seen. Graphite nodules have the minimum load-bearing capacity, and their response is nearly elastic as there is less difference

in loading and unloading curves. The ferrite matrix has significant load capacity, which is even higher for the pearlite structure, illustrating matrix as major load carrying element in the SGI microstructure. Comparison of the P-h response for the ferrite matrix in different grades clearly showed higher load capacity in 500-14 and 600-10. The load-displacement response is in accordance with the fact that the SGIs with higher silicon produce ferritic grade with higher strength. For the graphite phase no significant change in load capacity was observed, other than the variation in the result. It should be noted that the variation in nanoindentation test is very small for the matrix, whereas for the graphite nodules large variation was observed as indicated by larger error bands. Such a large variation of graphite properties was reported by other studies as well [137,138].

Figure 3.7: Nanoindentation hardness and elastic modulus for different phases of cast SGI materials (95 % confidence interval).

At least 20 indentations were made on each phase for each material to evaluate average nanoindentation hardness. For the graphite phase, only one indentation was made per graphite particle. Relatively larger graphite particles were randomly selected in the microstructure for indentation. For the matrix hardness, series of indentations were made and etched afterwards to reveal the indentations on ferrite and pearlite phase. The average elastic modulus and hardness of each of the phases in all the studied materials are plotted in Figure 3.7 with 95 % confidence interval. For 500-7, pearlite was the hardest phase (6.17 GPa), graphite was the softest (0.95 GPa) and ferrite moderately hard (3.87 GPa). The evaluated average elastic moduli are slightly different for ferrite and pearlite (229 GPa and 240 GPa

respectively), however, the 95 % confidence interval suggest the difference to be less significant as the ranges are overlapping. Similarly, for 500-14 and 600-10 grades, the hardness of the ferrite matrix increases to 4.87 GPa and 5.21 GPa respectively, however, no significant difference in elastic modulus was observed. For graphite nodules, hardness and elastic modulus were observed to decrease, but no conclusion could be made as nanoindentation results had large variations. In the literature, it was reported that the elastic modulus of graphite particles have considerable variations ranging from 4 GPa to 300 GPa [137, 138]. Nanoindentation tests showed the elastic modulus values to vary from 18 GPa to 26 GPa, which is quite close to the value reported by Pradhan et al. [128]. One of the reported improvements in SSF SGIs is less variation of microstructure properties, but no clear improvement was observed as the standard deviation reduction is not significant. Standard deviations in hardness measurement for ferrite phase in 500-7, 500-14 and 600-10 were 0.31 GPa, 0.23 GPa and 0.26 GPa.

3.1.4 Elastic-plastic phase properties

Experimental nanoindentation test provided elastic modulus and hardness, but most of the metals go through large plastic deformation before final failure. In SGI microstructure, graphite particles could be assumed elastic as less plastic deformation was observed in the load-displacement response, whereas the matrix went through significant plastic deformation when load was applied beyond its yield stress. So, it was essential to characterize complete elastic-plastic behavior for the matrix structure. In this work two methods for estimating elastic-plastic phase properties of matrix constituents were studied. These two methods are explained and compared in the following sections.

Phase properties optimization from tensile test

In the conventional tensile test, stress vs strain response for the bulk material is plotted to determine the tensile properties. In this approach, the bulk stress-strain curve from tensile test was used to estimate elastic-plastic material law for the matrix phases. This approach was studied in detail by the project collaborator team led by Professor Anders Jarfors in Jkoping University and has been published [21]. In the case of ferritic-pearlitic grade (500-7), Ramberg-Osgood relation as represented by Eq. 3.5 was used to approximate ferrite and pearlite

material model.

$$E\varepsilon = \sigma + \alpha \left(\frac{|\sigma|}{\sigma_Y} \right)^{n-1} \sigma \quad (3.5)$$

where, σ is the stress, σ_Y is the yield stress, E is the Young's modulus, ε is the total strain, and α and n are material specific constants.

The Ramberg-Osgood relation is one of the commonly used relations to represent non-linear material behavior. The material parameters for the matrix phase (ferrite and pearlite) were determined by an optimization process, where the objective was to minimize the difference between the optimized bulk stress and tensile stress from the experiment. In Eq. 3.5, there are four material parameters (E , σ_Y , α , n) for each phase present in the matrix structure, which corresponds to four parameters for the ferritic grades and eight parameters for ferritic-pearlitic grades. The overall objective function of the optimization is defined in Eq. 3.6.

$$f(E_j^P, \sigma_j^P, \alpha_j^P, n_j^P) = \sum_{i=1}^k W_i \left\| \sigma_i^{sim} - \sigma_i^{exp} \right\| \quad (3.6)$$

where, P denotes the particular phase, k is the number of points in the test data, σ_i^{sim} is the simulated homogenized stress, and σ_i^{exp} is the corresponding average experimental stress.

The solution to the problem was obtained by the Hooke and Jeeves pattern search method [139]. Starting points were chosen based on an initial simulation of the stress-strain curve. It should be noted that the Hooke and Jeeves pattern search method always finds a local or global minimum of the objective function irrespective of the starting point. A re-produced image of the micrograph (before deformation) was used as a basis for the FE-model. The size of the area was such that it represented the same average volume fraction of the different phases irrespective of where it was taken along the specimen, thus it could be considered as a RVE. A second order continuum FE model was implemented in ABAQUS to simulate stress-strain response on the RVE model. The simulated homogenized stress response of the overall RVE was compared to the actual tensile curve of the bulk material. In the RVE, graphite particles were assumed as a linear-elastic phase and matrix as elastic-plastic phase. The FE model was not able to capture fracture and decohesion of the graphite particles. So, relatively lower effective modulus of 6 GPa was used for the graphite particles to counter react decohesion, which gave good correlation with the experimental result.

Table 3.4: Optimized Ramberg-Osgood model parameters for ferrite and pearlite in EN-GJS-500-7 [21].

Phase	E [GPa]	σ_Y [MPa]	α [-]	n [-]
Pearlite	206	385	5.5	6
Ferrite	197	370	13	5

Figure 3.8: Stress-strain curve of ferrite and pearlite in EN-GJS-500-7 by optimizing bulk material response.

The set of material parameters in Eq. 3.6 that showed minimum deviation of bulk behavior from experimental tensile curve were estimated as the phase properties. The optimized material parameters for 500-7 grade are presented in Table 3.4, and the estimated stress-strain curve for ferrite and pearlite phase are shown in Figure 3.8 obtained by Kasvayee et al. [21]. Figure 3.8 also shows the comparison of the experimental curve and the optimized bulk response, and the results were in good match. The values were calculated by true stress-strain curve fitting of the tensile data up to 10% strain in order to simplify the optimization process. The estimated ferrite and pearlite response were respectively lower and higher than the bulk properties. Comparison of the estimated phase properties with the bulk tensile properties illustrated higher elastic modulus and yield stress for both the matrix phases in 500-7 grade. From these results, it can be assumed

that the presence of graphite particles in ferrite and pearlite phase might have reduced these properties in the bulk material. Overall, this approach provided a good approximation of bulk phase elastic-plastic properties considering all the microstructural features within. However, it was desired to have a good estimate of one of the phase properties to estimate phase properties in multi-phase matrix SGI grades accurately.

Nanoindentation P-h curve optimization for phase properties

Nanoindentation test is being widely used to measure elastic properties, and many studies have been published in an attempt to extend nanoindentation test to estimate elastic-plastic properties [130, 132–134, 140–146]. The methods proposed by Giannakopoulos and Suresh [140] to compute elastic-plastic properties was one of the initial attempts. Dao et al. [141] later established forward and reverse analysis algorithms; forward algorithm allowing to calculate a unique indentation response for a given set of elastic-plastic properties, and reverse algorithm extracting elastic-plastic properties from a given set of indentation data. Further, Bucaille et al. [142] extended Dao's method by using different indenters. Bouzakis and Michailidis [132] claimed to develop continuous FEM simulation algorithm to obtain stress-strain curve based on nanoindentation and later developing software for fast and accurate determination of stress-strain curve, which they named FANOS (Fast Approach of stress-strain curves base on naNOindentationS). A different approach was used by Luo et al. [144] to determine mechanical properties from the indentation load-displacement curve. They established set of equations to relate P-h curve with the elastic-plastic material parameters in the power law relation, and an optimization method was proposed to obtain a unique set of parameters using two indenters [145]. Another approach was to use a spherical indenter that provided a smooth transition from elastic to plastic behavior. Pathak and Kalidindi [134] has provided a comprehensive review of these spherical indentation stress-strain approaches.

In this work, nanoindentation simulation along with nanoindentation tests were attempted to extract the elastic-plastic material parameters in the Ludwik form (Eq. 3.7) of the power law material.

$$\sigma = \begin{cases} E\varepsilon & \text{Elastic} \\ \sigma_Y + K (\varepsilon_{pl})^n & \text{Plastic} \end{cases} \quad (3.7)$$

where, σ is the stress, E is the Young's modulus, ε is the strain, ε_{pl} is the plastic strain, σ_Y is the yield stress, K and n are the material strain hardening coefficient and exponent index.

Experimental nanoindentation tests were used to obtain the complete load displacement curve, Young's modulus and hardness of the individual phases in the SGI microstructure. Young's modulus and hardness were evaluated by Oliver and Pharr method [136] assuming purely elastic unloading. For plastic parameters, Tabor factor [147], being a well-known empirical relation between hardness and yield stress, was used to have an initial estimate of the yield stress. At the beginning, different sets of material parameters ($\sigma_Y = 300\text{--}2000$ MPa, $K = 200\text{--}1000$ MPa and $n = 0\text{--}0.5$) and their combinations were used to define different material models. These material models were used to define a sample material in the nanoindentation simulation model (illustrated in Figure 3.9). The loading section of the P-h curve from the simulation was then compared to the experimental P-h curve to have an estimate of the best set of material parameters. The accuracy of the prediction could be improved by using an optimization algorithm.

Figure 3.9: Three-dimensional nanoindentation simulation model.

A three-dimensional FE model of nanoindentation test was developed in ABAQUS v6.14 as shown in Figure 3.9. A Berkovich indenter of $5\ \mu\text{m}$ diameter was constructed by using three faces with face angles of 65.27° and indenter corner radius of $150\ \text{nm}$ as recommended by Karimzadeh et al. [148]. Berkovich indenter was modeled as a diamond material with Young's modulus $1140\ \text{GPa}$ and Poisson's ratio 0.07 . For the sample, $5\ \mu\text{m}$ thick cylinder of diameter $10\ \mu\text{m}$ was used with

an elastic-plastic material model of one phase at a time. The friction effect at the contact interface in nanoindentation was studied in literatures [142, 148], which showed little dependence on the value of friction coefficient. In this simulation, friction coefficient value of 0.12 as suggested in literature was implemented [130]. The sample was meshed with linear tetrahedral 3D stress elements (C3D4), and a total of 171544 elements were present. The sample mesh was refined towards the center of the cylinder. For the indenter, linear tetrahedral 3D stress elements were refined at the face edges, and a total of 35438 elements were present. The sample was constrained in the vertical direction at the bottom surface. The top surface of the indenter was uniformly displaced to a total of 300 nm into the sample and retracted to its initial position to unload. Indentation load on the sample was estimated by summing vertical reaction forces over the bottom surface of the sample. Then, the total vertical reaction force was plotted against displacement of the indenter to obtain P-h curve from the nanoindentation simulation.

In simulation model, deformation zone, stress and strain during the indentation process was checked. In Figure 3.10 a), deformation of the material in the process zone of the sample is shown, which is small compared to the specimen size. The sample geometry and size used was adequate as deformation, stress and strain contours caused by the indentation process did not reach the sample boundaries. The effect of material model parameters on the P-h response of the nanoindentation process was studied. In Eq 3.7, there are four material parameters (E , σ_Y , K , n) which are required to define the elastic-plastic behavior of each phase completely. Among the required parameters, Young's modulus (E) was evaluated in nanoindentation test for each individual phase. For the plastic material parameters, σ_Y was initially estimated from the nanoindentation hardness using the extreme values (2.7 and 3.3) and the middle value (3) in the Tabor factor range (2.7 to 3.3). The plastic material parameters were varied in the range ($\sigma_Y = 300$ –2200 MPa, $K = 200$ –1000 MPa and $n = 0$ –0.5) suitable for most of the cast irons and steels, to understand the effect of these parameters on the P-h response of the nanoindentation process. As the elastic material parameter had already been evaluated from the unloading curve, only the loading curve was plotted and compared. Figure 3.10 b), c) and d), the P-h curves for two extreme cases were compared for yield stress, hardening coefficient and exponent index respectively. The yield stress showed dominant influence on the P-h response, higher yield stress resulting into steeper loading curve and higher load for the same indentation depth (Figure 3.10 b)). Hardening coefficient showed some effect, higher coefficient resulting into

Figure 3.10: Nanoindentation simulation results and effect of plastic material parameters on the P-h curve for pearlite ($E = 240$ GPa) a) deformation of the sample due to nanoindentation ($\sigma_Y = 2200$ MPa, $K = 500$ MPa, $n = 0.25$), b) effect of yield stress ($K = 500$ MPa, $n = 0.25$), c) effect of hardening coefficient ($\sigma_Y = 2200$ MPa, $n = 0.25$) and d) effect of exponent ($\sigma_Y = 2200$ MPa, $K = 500$ MPa).

slightly steeper loading curve (Figure 3.10 c)). The effect of the exponent index of the Ludwik model on the nanoindentation P-h response was observed to be small (Figure 3.10 d)). From these parametric understanding, it could be suggested that the yield stress is the major plastic parameter in Eq. 3.7 affecting the maximum load and slope of the nanoindentation P-h curve.

Graphite particles were assumed purely elastic phase in this study and their material properties were characterized by nanoindentation elastic modulus and Poisson ratio from the literature. The matrix in SGI microstructure is elastic-plastic and is responsible for carrying most of the applied load. Here 500-7 SGI with ferrite and pearlite matrix is considered to demonstrate nanoindentation simulation for estimating elastic-plastic material parameters of each matrix phase. As explained

Figure 3.11: Comparison of P-h response from nanoindentation test and simulation to optimize pearlite phase properties ($K = 500$ MPa and $n = 0.5$).

in section 3.1.3, nanoindentation test was performed on each matrix phase, and the corresponding load-displacement curve was plotted. It has been understood that the yield stress has a major influence on the P-h response of the nanoindentation process, so at the beginning, yield stress was approximated from the nanoindentation hardness measure. The measured nanoindentation hardness for pearlite and ferrite in 500-7 were 6.17 GPa and 3.87 GPa respectively. Thus, using Tabor factors (2.7, 3.0 and 3.3), three sets of the yield stress values for pearlite (1850 MPa, 2100 MPa and 2300 MPa) and ferrite (1300 MPa, 1450 MPa and 1600 MPa) were made as initial estimates. Hardening coefficient and exponent values were varied in the ranges demonstrated in Figure 3.10, and each set of these material parameters were used as sample material definition in the nanoindentation simulation to plot a P-h response. The P-h response from the simulation was compared to the P-h curve from the nanoindentation test. Comparison of the P-h curve from many sets of material parameters varying K and n did not show much change. However, the slightest change in the yield stress showed a change in P-h response. Figure 3.11 and Figure 3.12 show a comparison of these P-h curves for pearlite and ferrite respectively. From the figures, it can be observed that the simulated results were in a fair match with experimental results, but not perfect match to predict material parameters. R-square values were evaluated for each of the simulation

results and the result obtained for pearlite1, 2 and 3 were 0.98, 0.981, and 0.958 respectively. Even though the experimental simulation response showed a good match with the experimental result, one important thing to note here is the value of the yield stress itself, which is very large (around 1900 MPa) as compared to the bulk material yield stress (315 MPa). Simulation using the set of material parameters with yield stress equal to 300 MPa (around bulk yield stress) resulted in significantly less load on the sample as shown in Figure 3.10 b). To check whether similar higher yield stress was predicted by nanoindentation simulation for the ferrite phase, a similar combination of material parameters obtained using the Tabor factor was simulated and the P-h response compared to experimental results (Figure 3.12). For the ferrite phase also it predicted high yield stress values (in the range of 1500 MPa). With these higher yield stresses prediction for both the matrix phases, it was confirmed that a detailed study of nanoindentation process and microstructural property measured in the nanoindentation test was needed.

Figure 3.12: Comparison of P-h response from nanoindentation test and simulation to optimize ferrite phase properties ($K = 500$ MPa and $n = 0.35$).

Indentation marks were studied under OM and SEM to investigate the nanoindentation process in the matrix microstructure. The residual indentation mark size (circumscribe circle) and residual depth in the simulation were compared to the average residual indentation size and depth in the experiment. Figure 3.13 a) and b) respectively shows a comparison of residual size and depth. For the

Figure 3.13: Comparison of residual indentation mark in nanoindentation test and simulation a) residual mark size b) residual depth.

residual mark size, the % error evaluated were 14 % and 4 % for 300 nm and 500 nm nanoindentation respectively. Similar error calculation for the residual depth were 6 % and 4 % for 300 nm and 500 nm nanoindentation respectively. Material pile-up and its influence on the elastic recovery in the nanoindentation experiment was predicted to have contributed on the observed difference. The residual mark size and depth from simulation showed a good match to the experimental residual mark, which illustrated the adequate modeling of the nanoindentation process and the material model.

Figure 3.14 illustrates nanoindentation marks in the ferritic SGI microstructure. These nanoindentation marks when compared with the matrix grain size (around 50 μm) were very small. In OM image (Figure 3.14 a)), series of nanoindentation marks are shown, and it can be visualized that most of the indentation marks were within a ferrite grain and much smaller compared to the ferrite grain size. This result elucidated that the measured properties and response from the nanoindentation tests were representative of very local material behavior without considering many of other microstructural features like grain boundaries and micro-pores. It has been understood that the grain boundaries are a barrier to dislocation movement, resulting in higher yield stress with finer grains and micro-pores. However, it could not be clearly understood what caused such a high yield stress in nanoindentation test. Usually, properties of the matrix in SGIs mean bulk phase properties inclusive of the effects of most of the matrix defects (e.g. Grain boundaries, porosities) The bulk phase properties (estimated from tensile

Figure 3.14: Residual nanoindentation marks within the ferrite grain (300 nm indentation process) a) optical microstructure image–nanoindentation marks inside red circles b) SEM image of single indentation mark within a ferrite grain.

test optimization) simplifies the elastic-plastic properties estimation and its implementation to represent bulk phase in microstructure simulation model. In the event of using the matrix phase properties estimated from nanoindentation, additional challenges of modeling the effects of grain boundaries and micro-pores have to be considered separately. From this study, it seems that the estimation of elastic-plastic properties from nanoindentation P-h curve is an accurate method to estimate localized properties within a grain. Use of these properties should be carefully implemented to account for other microstructural defects.

Comparison of the methods

The purpose of this elastic-plastic material properties study is to implement them for representing phase behavior in microstructure simulation. Tensile test optimization method and nanoindentation P-h response optimization were studied to estimate phase properties. The two methods studied were compared based on the properties they measured in the SGI microstructure. Table 3.5 shows detail comparison of the two studied elastic-plastic phase properties optimization methods. Illustrated in the schematic diagram, $\sigma - \epsilon$ optimization of the tensile test estimated bulk phase properties considering effects of other microstructure defects. Stress-strain curve for each matrix phase in 500-7 was optimized in FE simulation of representative microstructure model to minimize error in the bulk stress-strain response of the model with the tensile test result. While in nanoindentation test, the measured properties were very local to the matrix grain without consideration

Table 3.5: Comparison of phase properties estimation methods using $\sigma - \varepsilon$ optimization and P-h optimization

	$\sigma - \varepsilon$ Optimization	P - h Optimization
Schematic illustration
Estimated properties	Bulk phase properties considering effects of other microstructure defects e.g. porosities, grain boundaries.	Very local phase properties within a grain without consideration of other defects.
Advantage	Direct method to estimate bulk phase properties from conventional tensile test result.	Accurate prediction of individual phase in microstructure without effect of other phase and defects
Limitation	Properties estimation interdependent to phase. Good prediction of one phase required in multiphase.	Implementation of the estimated properties requires additional concern of grain boundaries and other microstructure defects.

of other defects. In this method, elastic properties were determined from nanoindentation test, and for the elastic-plastic response, nanoindentation process was simulated to obtain a P-h response at different material parameters which were compared with the experimental P-h response to estimate the plastic material parameters. $\sigma - \varepsilon$ optimization is a direct method where the material parameters in stress-strain relation were estimated by optimizing $\sigma - \varepsilon$ curve from the conventional tensile test result. However, the method is interdependent to the phases present in the matrix, so the proper estimate of one of the properties is desirable to accurately predict each phase. P-h optimization is an indirect method, the plastic material parameters in stress-strain relations were estimated by optimizing P-h curve. The method accurately estimated elastic properties of individual phases, but for the plastic properties, it predicted very high yield stress which could not be clearly understood. However, further study on the nanoindentation marks revealed that the measured material response in the nanoindentation test were local grain properties without the effects of grain boundaries and micro-pores as illustrated in the schematic illustration in Table 3.5. Thus, it was concluded that implementation of the phase properties for the nanoindentation test would require

modeling of grain boundaries and porosities, which would make any model more complex. For the simplicity of the properties from the tensile test optimization, it was decided to use the elastic-plastic phase properties estimation from tensile test optimization in this work.

3.2 Effect of DCR process on SGI

In the previous section, it has been illustrated that graphite morphology and matrix structure along with the phase properties could completely represent as-cast SGIs microstructure. The mechanical post processes like Deep Cold Rolling (DCR) process, shot peening and laser shock peening could be applied to enhance strength and surface behavior of the as-cast materials. These mechanical processes improve surface properties by plastic deformation of the surface layer. In SGIs, the matrix continuity is broken by the presence of graphite particles. The spheroidal form of graphite particles exhibited better properties in SGIs. However, the presence of graphite particles itself posed a challenge for the mechanical post process treatment. Unlike the iron matrix, graphite particles are brittle and could not undergo plastic deformation causing discontinuous surface matrix. Such discontinuities surface layer can not only influence residual stress but could also result in unusual behavior at the interface. In this section, it is attempted to use DCR process as representative mechanical post-treatment process to study the effect of the mechanical process on graphite particles integrity and matrix properties. Si-solution strengthened grade having higher strength with excellent ductility (500-14) was used to study DCR process. The 500-14 grade is attractive to use in fatigue applications, where the effect of a process like DCR is more applicable to improve fatigue behavior of the component.

3.2.1 Experimental design and setup

Different types of DCR tools are available from ECOROLL Corp for different applications. In this study, Hydrostatic tools (HG tools) were used to roll on flat machined SGI specimens. The HG tool was mounted on a 3-axis CNC machined to guide the tool along the rolling path, and the pressure force was applied using hydraulic pump by ECOROLL designed for the DCR process. HG6 and HG13 with effective ball diameters of 6.35 mm and 12.7 mm were used in this study.

The continuous flow of coolant not only provided pressure force but also helped to cool the sample and tool during the whole DCR process. The SGI sample blocks were machined and ground to make the surface uniform and smooth before DCR process.

At first, track widths were measured for HG6 and HG13 tools by rolling single tracks at varying rolling pressures (100 bar, 200 bar and 300 bar). The measured track widths for HG6 were 0.61 mm, 0.79 mm and 0.95 mm respectively at 100 bar, 200 bar and 300 bar pressure. Similarly, for HG13 the measured track widths were 1.27 mm and 1.60 mm respectively at 100 and 200 bar. These measured track widths were used to calculate the step over (Eq. 2.4) required for a desired percentage overlap in the DCR experiment. For the preliminary study, HG6 was used at rolling pressures of 100 bar and 300 bar. At 100 % overlap, the rolling track was visible due to insufficient overlap. For 300 bar pressure, SGI microstructure deformed excessively initiating multiple microcracks. Thus, maximum rolling pressure of 200 bar, percent overlap of 400 % and federate of 1000 mm/min were used for the detailed study of the DCR process on the 500-14 material. Out of the four DCR parameters, roller size and rolling pressure have shown major influence on DCR effects, so two levels of each of these parameters were used to study the effect of these parameters on the SGI material. Table 3.6 presents the experimental design of DCR indicating all the process parameters for each case. The as-cast sample was machined and ground before DCR experiment. It helped in observation of sample material after DCR process without additional grinding and minimum polishing. The As-cast 500-14 cast block before and after DCR process is shown in Figure 3.15. For each experimental case three trials were repeated, one of each samples were used for microstructure study, nanoindentation study and residual stress measurement.

Table 3.6: DCR process parameters and experimental design.

Trial	Deep rolling parameters					
	Ball size (mm)	Rolling pressure (bar)	% (overlap)	Track width (mm)	Step-over (mm)	Feedrate (mm/min)
1	6.35	100	400	0.61	0.122	1000
2	6.35	200	400	0.79	0.158	1000
3	12.7	100	400	1.27	0.254	1000
4	12.7	200	400	1.60	0.32	1000

Figure 3.15: As-cast 500-14 block used for DCR experiment a) sample block after machining and grinding b) sample block after DCR process illustrating rectangular DCR regions, RD: rolling direction & CD: cross direction.

3.2.2 Microstructure

In the preliminary DCR trials, HG6 tool was used at rolling pressure of 100 bar and 300 bar (100 % overlap and 1000 mm/min feed rate). Samples were polished and observed under optical microscope. The microstructure images are shown in Figure 3.16. At 100 % overlap, each area was rolled twice, except for the beginning and end tracks. Samples when observed under the microscope showed a clear sign of the rolling tracks. The width of the observed tracks was around half of the single track width measured for the corresponding rolling pressure. Distortion and deformation were higher at 300 bar pressure with large graphite particles damage and cracks, whereas at 100 bar pressure graphite particles were integrated into the matrix with less damage.

Figure 3.16: Microstructure images after DCR process (HG6 tool, 100 % overlap, 1000 mm/min feed rate) a) 100 bar rolling pressure b) 300 bar rolling pressure.

DCR experiments were performed based on the experimental design presented in Table 3.6. The main effect analysis of the roller size and rolling pressure on phase

hardness and graphite nodularity were studied as a final year project by Su [149]. It was shown that the matrix hardness increases with increase in rolling pressure and ball size, but no clear conclusion could be made for graphite nodule hardness as the hardness depended on the extent of graphite nodule damage. Changes in graphite nodularity on the rolling direction surface and cross section were also studied in that work. Overall, DCR process showed a slight decrease in nodularity, which was observed to improve for larger roller size. The microstructure study was not conclusive because of the presence of semi-exposed graphite particles on the surface microstructure misleading actual graphite form and for the cross section it was difficult to focus on the region within the deformed layer (around 1 mm from the surface).

Scanning electron microscopy

Detailed study of SGI microstructure after DCR process is presented here. To get a detailed understanding, SEM studies were performed after DCR process, after polishing the deep cold rolled samples and after additional grinding and polishing. Figure 3.17 shows a comparison of SEM micrographs at two extreme case in the experimental design. The SEM observations were performed after DCR process without grinding and polishing. For both cases, the ferrite matrix deforms around graphite particles. Even if higher % nodularity (74 %) was reported for the as-cast 500-14, the surface microstructure after DCR process mostly appeared as elongated shapes. From the surface microstructure study, it was difficult to state on graphite nodularity because the surface microstructure has many graphite particles that could be partially exposed misleading the graphite shape. However, for all the DCR cases, cracks were observed in the ferrite matrix around the graphite particles. Comparing the micrographs for HG6-100 bar (Figure 3.17 a)) and HG13-200 bar (Figure 3.17 b)), earlier micrograph showed a higher number of degenerated graphite particles, this might be due to the smaller roller size. Comparison of Figure 3.17 c) with d) showed larger deformation of the ferrite matrix most probably due to higher rolling pressure. Overall, the surface microstructure for HG6-100 bar rolled and HG13-200 bar rolled did not show much differences. However, the microstructure after DCR showed the noticeable difference of ferrite cracks and uniform plastic deformation around the ferrite matrix.

The cold rolled samples were subsequently polished using 3 μm and 1 μm diamond suspensions for around 5 minutes in each stage. Samples were again studied under

Figure 3.17: SEM image of SGI microstructure after DCR (400 % overlap and 1000 mm/min feed rate) a), c) for HG6 at 100 bar rolling pressure; b), d) for HG13 at 200 bar rolling pressure.

SEM, and the images are presented in Figure 3.18. The general microstructure is shown in Figure 3.18 a) and b) respectively for HG6-100 bar and HG13-200 bar. The microstructure looked similar at this stage with the combination of nodular and elongated alike graphite particles. Comparison of Figure 3.17 and 3.18 showed that the metallographic polishing cleaned the sample allowing clear microstructure. It was observed that some material removal took place during polishing that exposed underlying graphite particles. However, there still exists many elongated graphite-like structures along with cracks in the ferrite matrix (Figure 3.18 c)–h)). Some of the observed cracks were long and connected multiple graphite particles (Figure 3.18 c)–f)). In other cases, the cracks were circular or semi-circular as shown in Figure 3.18 g) and h)). The tentative size of such circular cracks was measured quite close to the graphite nodule diameter. So, from the observed microstructure and size of the cracks, it was predicted that the submerged graphite particles were influencing material deformation on the surface. To confirm the presence of the graphite particles below such crack, En-

Figure 3.18: SEM images of DCR sample after polishing a), c), e), g) for HG6 at 100 bar rolling pressure; b), d), f), h) for HG13 at 200 bar rolling pressure.

ergy Dispersive Spectrometer (EDS) analysis was performed. The EDS analysis showed the presence of carbon (more than 70 %), which confirmed the presence of

graphite particles beneath the cracks and it could be proposed that the submerged graphite particles play a significant role in surface matrix cracking of SGI during DCR process. Comparison of the two deep cold rolled cases did not show significant difference; however, comparison with the unrolled as-cast SGI microstructure showed a clear difference in the microstructure.

Table 3.7: Material removal during sample preparation steps.

Steps	Load and Time	Speed (RPM)	Material removal (μm)
P1000 grinding	25N, 5 mins	300	21
P2000 grinding	25N, 3 mins	300	9
$3\mu\text{m}$ polishing	25N, 5 mins	150	4
$1\mu\text{m}$ polishing	20N, 5 mins	150	<2

In an attempt to expose the submerged graphite particles below the cracks and to check the depth of the cracks formed, samples were further ground for around 5 minutes and 3 minutes using SiC paper P1000 and P2000 respectively and polished (Table 3.7 presents the tentative thickness of material removed). Figure 3.19 illustrates the microstructure of the cold rolled samples after additional grinding and polishing. The overall microstructure shown in Figure 3.19 a) and b) resembled the as-cast microstructure. The surface cracks observed on the rolled samples were mostly limited on the surface, and no sign of deep matrix cracks was found. The DCR process not only affected surface graphite particles, but also submerged graphite particles. The presence of graphite particles close to the cold rolled surfaces affected plastic deformation behavior of the ferrite matrix during the rolling process. If the graphite particles were very close to the surface, surface cracks were formed around the graphite particles. In the case of graphite particles slightly inside the surface, still in the plastic deformation region, the graphite particle was deformed and compressed, but, no cracks or damage appeared in the ferrite matrix. However, when the samples were ground and polished, such subsurface graphite particles were partially exposed as in Figure 3.19 c) and d), and the ferrite matrix intruded around the graphite particles due to graphite particles shrinkage were observed as shown in Figure 3.19 e) and f). Comparison of microstructure after grinding and polishing for HG6-100 bar and HG13-200 bar rolled cases did not show clear differences. Thus, from this study it could be stated that the DCR process showed major influence on surface microstructure and subsurface graphite particles; however, different cases of DCR did not show the quantifiable effect on the microstructure.

Figure 3.19: SEM images of DCR sample after fine grinding and polishing a), c), e) for HG6 at 100 bar rolling pressure; b), d), f) for HG13 at 200 bar rolling pressure.

Electron backscatter diffraction analysis

The surface microcracks observed on the DCR sample surface were limited to surface graphite particles. However, SEM studies indicated large plastic deformation of the ferrite matrix around graphite particles, which misled to graphite particles characterization. To further study plastic deformation behavior after the DCR process, Electron Backscatter Diffraction (EBSD) analyses were performed. EBSD technique is a SEM based method to quantitatively characterize the crystal lattice orientation, grain boundary and grain deformation. Briefly stating work-

ing principle of EBSD, well-polished sample is tilted to the incident electron beam (70°) that penetrates to a narrow distance of 5 - 50 nm from the surface. When an electron beam is incident on the tilted crystalline specimen, the electrons are diffracted from the crystal planes which consist of Kikuchi bands. These Kikuchi bands are characteristic of the sample crystal structure and orientation which are detected by the EBSD detector. The identified bands match certain crystal orientation to identify crystal orientation at each measurement point.

The samples for EBSD study were prepared similar to that for the SEM study (Table 3.7) with additional colloidal silica polishing for 2 - 3 mins. In this analysis, an EBSD system (Oxford Instruments HKL) attached to JEOL 7600F Field Emission SEM (FE-SEM) system was used. Channel 5 acquisition package was used to collect and analyze the EBSD data. Inverse Pole Figure (IPF) maps were studied to identify the grains and their orientation by interpreting the crystallographic orientations with the sample coordinate system. Grain Orientation Spread (GOS) maps measures the degree of orientation change between every pixel in the grain and the grain's average misorientation is plotted for the whole grain. GOS is a primary strain analysis tool revealing grains which showed the most deformation and illustrating the spatial distribution.

The IPF maps for EN-GJS-500-14 sample before DCR and after DCR are compared in Figure 3.20. Figure 3.20 a) plots the IPF map for as-cast material, which illustrates random orientation of the grains suggesting isotropic material behavior. Black regions in the map are graphite particles which could not be indexed even if hexagonal closed pack graphite phase was also selected. Most of the grains were larger than graphite particle size. Two similar EBSD maps were used to estimate average grain diameter of the as-cast EN-GJS-500-14. The boundaries of the crystal grains are defined using misorientation angle, which is the difference in orientation between grains. The grain boundary (high-angle boundary) greater than 15° and the sub-grain boundary (low-angle boundary) greater than 2° is used in this analysis. Individual grain size was estimated from the channel 5 acquisition package, and smaller noises were filtered during the average grain size estimation. The average grain diameter was observed to be $33.56 \mu\text{m}$ for the as-cast grade. Figure 3.20 b) and c) plot similar IPF maps for cold rolled (HG13 at 200 bar) EN-GJS-500-14 sample surface. The cold rolled surface was metallographically prepared as explained above. In these IPF maps, gradient of the crystal orientation could be clearly visualized within a grain. The local crystal misorientation within grains was due to the severe plastic deformation of the ferrite matrix during

Figure 3.20: Inverse pole figure maps a) Unrolled EN-GJS-500-14 sample, b) Deep cold rolled sample surface (HG13 at 200 bar), c) Closer view on DCR sample surface (Black regions are graphite particles).

DCR process. Comparing Figure 3.20 a) with 3.20 b), the difference in misorientation caused by the DCR process on the surface microstructure could be seen. Further, it could be noticed that the gradient of crystal orientation is more significant around the graphite particles. SEM studies of DCR sample in the previous section showed the presence of surface and subsurface cracks in the microstructure, which were suggested due to the presence of non-metallic and weak graphite particles. These graphite particles could not deform plastically along with the ferrite phase, and as they were comparatively soft phase, it allows the ferrite matrix to deform highly around the graphite particles. Figure 3.20 c) presents a closer look at the IPF map demonstrating significant deformation around the so-called irregular graphite particles. While plotting the grain boundaries on the IPF maps for the DCR samples, prominent number of low-angle grain boundaries ($>2^\circ$) were noticed to surround around the graphite particles. So, in Figure 3.20, only high-angle grain boundaries ($>15^\circ$) were plotted as the low-angle grain boundaries appeared all around the graphite particles and frequently within the plastically deformed grains. These EBSD study on the surface microstructure supported the SEM study results that the ferrite matrix around graphite particles undergo severe plastic deformation. However, the presence of any subsurface cracks could not be confirmed. Regarding the average grain size, even if the ferrite matrix undergoes serious plastic deformation, no change in average grain diameter ($33.73\text{ }\mu\text{m}$) was observed.

Mechanical surface treatment process, like DCR, create a plastically deformed surface region. This strain hardened region represents the surface layer where microstructure change is significant. Microstructure characterization of the surface hardened zone will help to understand the influence of sub-surface graphite particles on the DCR process application, and to estimate the strain hardened region in DCR process. EBSD analysis was performed on the cross-section of DCR samples. Larger roller at high pressure (HG13 at 200 bar) and smaller roller at low pressure (HG6 at 100 bar) samples were used to study cross-section microstructure. Recent studies [150–153] have highlighted the ability of EBSD data like GOS maps to study and quantify strain hardening levels in surface treated samples. The EBSD grain misorientation parameters provide a more reliable method of assessing grain damage due to induced strain hardening [151]. IPF maps plot grain orientations that help to identify grain orientations and local orientation difference within a grain. The local grain orientation difference is averaged for the whole grain and plotted as GOS maps. IPF and GOS maps at the cross-section of HG13-200 bar

Figure 3.21: EBSD study on the cross-section of DCR sample (HG13 at 200 bar)
a) Inverse pole figure map and b) Grain orientation spread.

rolled sample are presented in Figure 3.21. IPF maps (Figure 3.21 a)) shows a high level of local orientation difference in surface and sub-surface grains. It was observed that the presence of graphite particles on the rolled surface and sub-surface caused additional in-grain misorientation around the graphite particles. The local in-grain misorientation extends to certain depth, and gradually at far depth grains are of uniform orientation. Figure 3.21 b) illustrates GOS map for the same sample. The grain's average misorientation are plotted on the scale of 0 to 8, 8 being grains with highest average misorientation. The surface and sub-surface grains showed higher GOS values indicating higher strain hardening level on the cold rolled region. It can be clearly noticed that the strain hardened region

extends deeper into the sample at higher pressure and larger roller size. Specifically, grain around larger and irregular graphite particles showed higher average misorientation. Thus, it can be said from the EBSD analysis that the graphite particles in the SGI microstructure affect the continuous plastic flow of the ferrite matrix by letting additional matrix deformation around graphite particles, most probably due to compression and breakage of soft graphite particles.

Figure 3.22: EBSD study on the cross-section of DCR sample (HG6 at 100 bar)
a) Inverse pole figure map and b) Grain orientation spread.

Similar IPF and GOS maps for HG6 at 100 bar are plotted in Figure 3.22. The local in-grain misorientation presented in IPF map (Figure 3.22 a)), and GOS map (Figure 3.22 b)) illustrate strain hardened surface and sub-surface region. However, it is immediately observable that the strain hardened region extends

progressively deeper into the sample for increased rolling pressure and larger ball size. For both the maps in Figure 3.21 and 3.22, local orientation differences are observed within deformed grains subjected to strain hardening. Away from the rolled surface, most of the grains showed minimum average misorientations which were also observed in the as-cast unrolled sample. Such random misorientations might be due to residual strain caused by over growth of surrounding grains during solidification process or due to some influence of sample preparation method. However, it is to note that all the samples were prepared identically using automatic machine, and minimum force applied at final polishing step (10 N for colloidal silica polishing). GOS maps as explained above have been successfully used to estimate strain hardened region on surface treated samples. Similar method prescribed in the literature [151] has been used to estimate DCR influence region, explained in section 3.2.4.

3.2.3 Nanoindentation phase properties

Study of deep cold rolled sample microstructure elucidated the evolution of surface cracks and the effect of graphite particles on plastic deformation and cracking of the ferrite matrix. For metallic components without inclusions like steels, titanium and nickel, it has been reported that the DCR process strain hardens the surface layer to increase its strength and hardness. In SGI material, the presence of graphite particles inclusions has shown its influence on the matrix deformation by inducing surface crack. So, it is crucial to understand how the phase properties were affected by the DCR process. Nanoindentation test was performed on cold rolled samples. As nanoindentation test results are critical to the surface roughness and require surface preparation below 1 μm , the cold rolled samples were ground and polished. Twenty-five graphite particles were selected for nanoindentation test, and only one test was performed on each graphite particle. For the ferrite matrix also 25 indentation points were randomly selected. Nanoindentation tests were performed on all the four deep cold rolling cases, and the average results were compared to the as-cast properties.

The average nanoindentation hardness of the graphite particles and the Si-solution strengthened ferrite matrix is plotted in Figure 3.23. The average hardness of graphite particles did not show significant change other than the variation within the 95 % confidence interval. It should be noted that the nanoindentation test results on the graphite particles showed a large variation. For all the deep cold

Figure 3.23: Effect of DCR process and parameters on graphite nodules and ferrite hardness (95 % confidence interval).

rolled cases, the ferrite matrix hardness was observed to be higher than the ferrite matrix in the as-cast microstructure. Strain hardening due to plastic deformation of the ferrite matrix is the reason for such increase in hardness after DCR process. Prabhu et al. [94] in their study reported a positive effect of larger roller ball and higher rolling pressure. Similar result could be noticed in this study as well for the ferrite matrix in deep cold rolled SGI. For both tools, if the hardness was compared at different rolling pressure, higher pressure showed higher hardness illustrating positive effect of rolling pressure. Similarly, comparing hardness at same rolling pressure for two ball sizes (tools), higher hardness was observed for a tool with larger roller ball illustrating positive effect of ball size as well.

In addition to comparing the nanoindentation hardness, representative P-h responses were also compared in Figure 3.24. The representative P-h curves plotted have similar hardness values to the average hardness. In the P-h response also the ferrite matrix in as-cast SGI clearly showed less load bearing capacity at similar indentation depth. The elastic-plastic loading curves showed a major difference, but the elastic unloading curves were very similar with almost identical slope. The very similar nature of unloading curves suggests no change in Young's modulus of the ferrite matrix with DCR process. The evaluated average Young's modulus during the unloading test also verified no change of Young's modulus of the ferrite after DCR process. The effect of roller size and rolling pressure could also be

Figure 3.24: Comparison of P-h response of the cold rolled ferrite matrix to the as-cast ferrite matrix.

noticed in the P-h response. Larger roller size and higher rolling pressure showed higher load bearing capacity in the loading curve. Comparison of average results in Figure 3.19 and individual P-h response in Figure 3.24 show some discrepancy. The average result showed higher hardness for HG13-100 case than for HG6-200 case, but in the representative P-h response, HG6-200 showed higher load capacity than HG13-100 case. Reasons for such variation are a) use of single experimental test data in P-h curve, b) nanoindentation hardness also depending on the maximum depth (slightly different for each experimental test) and c) local variation in material and test conditions. Even if the DCR process-induced surface cracks and graphite particles damage in SGI material, the working hardening effect on the ferrite matrix still persisted that improved the load capacity of the material.

3.2.4 Effective deformation zone on DCR process

The effects of DCR process extends to subsurface in the rolled sample. Generally, residual stress measurement is used to access DCR influence region [37,91,95–97]. Compressive residual stress is one of the important effects of DCR process, which

helps to improve the fatigue resistance of a component. However, one problem with residuals stress is that at high temperature it rapidly relaxes [100, 154]. Evans et al. [154] have demonstrated that one high-temperature isothermal fatigue cycle could reduce more than 50 % of the residual stress in Ni-based superalloys. Another study by Guechichi and Castex [155] reported that strain hardening is the primary contributor to fatigue resistance with additional contributions from the residual stress. So, it is more reliable to estimate DCR effect zone based on the strain hardened region. Number of articles [150–153] have already attempted to estimate strain hardening level using EBSD grain misorientation parameters. The results highlighted reliable method of assessing induced strain hardening level to estimate the influence of shot peening [151], hammer peening [152] and laser shock peening [153]. Similar method reported in [151] was used in this work to estimate effective DCR influence region. EBSD analysis was performed at the cross-section of the cold rolled sample (EBSD maps in Figure 3.21 and Figure 3.22). A grain-by-grain data was extracted from the EBSD maps, containing details of each individual grains (x & y coordinates of the grain center, grain area, grain diameter, GOS, etc.). Two maps were used for each DCR case. The extracted spreadsheet data was first sorted along y -coordinate (i.e. distance from the rolled surface), beginning with the grain farthest from the cold rolled edge. Then, a simple *Matlab*

Figure 3.25: Estimation of effective DCR influenced zone based on EBSD Grain Orientation Spread map over the distance from cold rolled edge.

program was used to calculate moving average of GOS over 150 grains. The moving average GOS for two DCR cases are plotted in Figure 3.25. In addition to the moving average GOS, a horizontal line corresponding to the sum of average GOS and standard deviation for the unrolled sample is plotted to establish threshold GOS value. The moving average GOS value above that threshold was considered significant, and indicative of DCR deformation region. Plotting from the far end, moving average GOS exceeding the horizontal line indicates the beginning of the DCR deformation zone. In Figure 3.25, strain hardened regions or DCR deformation zones are indicated in the average GOS vs. distance plot. It was identified that the deformation zone for HG6-100 bar and HG13-200 bar were respectively $150.74 \mu\text{m}$ and $384.17 \mu\text{m}$. These deformation zone sizes could be compared with the GOS maps for each case in Figure 3.21 (b) and Figure 3.22 (b). The larger deformation zone is due to higher rolling pressure and larger ball size, resulting into the larger strain hardened region. The deformation zone estimated could be used to state the depth over where microstructure changes occurred. So, the DCR deformation zone could be stated along microstructure characterization result to characterize deep cold rolled samples.

3.3 Effect of Thermal Cycling on SGI

Heat treatment is a versatile post treatment process to change matrix microstructure and mechanical properties. No graphite can nucleate in heat treatment or thermal treatment, but a slight change in graphite size might occur depending on the temperature and cooling parameters. Graphite particles and the ferrite matrix in SGIs have different thermal properties. Graphite particles have lower Coefficient of Thermal Expansion (CTE) of $7.8 \times 10^{-6} \text{ (m/(m K))}$, and cast iron usually have CTE in the range of $10 \times 10^{-6} - 12 \times 10^{-6} \text{ (m/(m K))}$ [156]. Due to the mismatch of the thermal expansion behavior, thermal stresses are generated on the graphite-ferrite interface. The interface stressed might affect the interface properties without major phase change in the microstructure. Moreover, the effect on the interface might accumulate to cause failure.

Overall thermal expansion behavior of the investigated material was studied after series of thermal cycling on the material in our collaborating institute, and the result was reprinted in Figure 3.26. Considering the maximum operating temperature of a diesel engine (600°C), the maximum temperature of thermal cycling

Figure 3.26: Thermal expansion behavior of the as-cast SGI during thermal cycling measured at 55 °C (RT-600°C) a) ferritic 500-14 b) ferritic-pearlitic 500-7 [2].

was selected as 600 °C. As the maximum temperature of the thermal process was below the austenitization temperature of 727 °C, no change in the matrix phase was anticipated. However, the decomposition of retained cementite into graphite particles and ferrite might occur in the vicinity of the maximum temperature, since the samples contained relatively high silicon. The dL/L_0 (dL and L_0 are change in specimen length and original length, refer to [2] for detail) values were measured at 55 °C after each thermal cycling. As seen in Figure 3.26 a) for the ferritic SGI, the dL/L_0 decreased at the beginning and become constant. It was reported that the initial drop in volume was related to decomposed carbon filling the already existing vacancies and pores in the graphite phase [2]. For the ferritic-pearlitic SGI, the volume was reported to increase in the first cycle, which was implied due to an increase in volume by graphitizing and fewer vacancies and pores in the microstructure to compensate decomposed carbon. However, the volume was reported to decrease until around 16 thermal cycles and increased afterward (Figure 3.26 b)). Continuous graphitizing of the carbon from the cementite enriched pearlite structure in the microstructure was stated as the main reason for the continuous increase of volume after 16 thermal cycles [2].

From the thermal expansion test, change in specimen length (dL/L_0) after thermal cycling was studied. In this work, similar thermal cycling was done on the cast 500-14 block, to further study effects of thermal cycling on the microstructure, phase properties and damage behavior. Figure 3.27 illustrates the step details of the thermal cycle performed for the study. The sample block was heated from room

Figure 3.27: Schematic diagram of thermal cycling from RT to 600 °C.

temperature to 600 °C at 10 °C/min, held for 20 minutes at the maximum temperature and cooled inside the furnace to minimize oxidation and residual stresses. Following the volume change results in Figure 3.26, microstructure study, nanoin-indentation phase properties study and damage mechanisms studies were performed on the samples after 16 and 45 thermal cycles. However, as no major changes were observed after 45 thermal cycles, the sample was further thermal cycled to 100 cycles.

3.3.1 Microstructure study

At the maximum thermal cycling temperature of 600 °C, ferrite is the stable phase of iron, so no change in the matrix structure is expected in this thermal cycling experiment. Moreover, the graphite phase itself is the stable form of the carbon precipitated out during slow cooling of the cast metal. The most susceptible area in the microstructure was the graphite-ferrite interface due to the mismatch in their thermal expansion behavior and weak nature of the interface bond. The sample after 16 thermal cycles (TC-16) and 45 thermal cycles (TC-45) was metallographically polished and observed under the SEM to investigate microstructure change, specifically at the graphite-ferrite interface. The microstructure results after 16 thermal cycles did not show any change in the interface state. The graphite particles were intact in the matrix as in the as-cast SGI. It seemed that the interface strength was not affected during the initial 16 thermal cycles. The only change that might have occurred during the initial 16 thermal cycles was graphitizing of the carbon to fill the existing vacancies and pores in the amorphous graphite particles. The sample after 45 thermal cycles was studied under the SEM and the results are shown in Figure 3.28. Decohesion of some of the graphite nodules

Figure 3.28: Microstructure study after 45 thermal cycles, a), c) and d) partially debonded graphite-ferrite interface; b) Magnified image of image a) at the interface.

from the ferrite matrix could be seen in the SEM images (arrows indicating areas of decohesion). Due to the continuous changing differential thermal expansion, thermal stresses were accumulated at the interface ultimately resulting into decohesion of the graphite particles. However, it should be noted that decohesion did not cause complete disintegration of the graphite nodules, only partial decohesion was observed. Most of the graphite particles were still fully integrated into the ferrite matrix, less than 5 % of the graphite nodules showed partial decohesion. The graphite particles after decohesion did not show any surface oxidation of the graphite particles as reported by Buni et al. [15] in their study after few hundreds

of thermal cycling on ductile iron. From this microstructure study and with reference to the thermal expansion studies, it could be estimated that during the initial thermal cycling the graphite-ferrite interface bond was not affecting. This allowed efficient diffusion of carbon into porous graphite particles to show an overall decrease in volume of SGI material. However, with additional accumulation of thermal stresses at the interface, the interface started to show partial decohesion of graphite particles, which not only affect efficient diffusion of carbon but will also affect the mechanical properties of the ductile iron.

3.3.2 Nanoindentation phase properties

The thermal cycling in this work can be compared with the thermal cycling experienced by the diesel engine during startup and shutdown. The rapid heating and slow cooling of the engine can be compared to the fast heating and cooling inside the furnace. With some effect of thermal cycling observed on bulk expansion behavior and microstructure, the nanoindentation properties of both the ferrite and graphite phases were also studied. As explained in previous sections, nanoindentation tests were performed on both the phases in the thermal cycled SGI microstructure. The nanoindentation plots are presented in Figure 3.29 with 95 % confidence level error bar. The average graphite phase nanoindentation hardness

Figure 3.29: Effect of the thermal cycling on the nanoindentation hardness of the ferrite and graphite nodule in SGI microstructure (95 % confidence interval).

did not show any change after 16 cycles or 45 cycles or 100 cycles. The reported average graphite hardness were 0.62 GPa, 0.67 GPa and 0.68 GPa for samples after 16, 45 and 100 thermal cycles as compared to 0.64 GPa for the as-cast material. The variation on the results could be considered as variation in the measurement data and dispersion of graphite nodule hardness. Even though diffusion of the carbon was reported in thermal expansion studies; the diffusion process did not show change in overall graphite nodule properties. For the graphite phase also, no major change in nanoindentation hardness was observed as shown in Figure 3.29. The reported average ferrite nanoindentation hardness were respectively 4.84 GPa, 4.71 GPa and 4.85 GPa for 16, 45 and 100 thermal cycled samples as compared to 4.87 GPa for the as-cast sample. Hardness reduction with thermal cycling was also reported for cast irons [15], where the tests were conducted to 15,000 cycles. The reason for such decrease in ferrite hardness is predicted to be diffusion of solid solution carbon atoms to the graphite nodule vacancies due to higher atomic mobility at high temperature. After 45 thermal cycling, a sign of graphite nodule decohesion was observed, and such decohesion will break the diffusion bridge reducing free mobility of carbon atom between graphite particles and the ferrite matrix. However, a slight increase in the hardness on the 100 thermal cycled sample could not be justified. Comparison of the nanoindentation hardness of the ferrite and graphite particles shows wide gap illustrating very different mechanical properties of the two phases. From this nanoindentation studies on the thermal cycled samples, it could be justified that even some changes in thermal expansion and microstructure are observed, no major change in phase properties occurred in the studied initial thermal cycling of the SGI material. So, it could be considered that the initial thermal cycling due to engine startup and shutdown affected the material and its properties to 100 thermal cycles.

3.4 Summary

In this chapter, microstructure and properties of as-cast SGIs with different silicon content were studied. Out of the three studied SGI grades, two of them were high silicon SSF grades that showed complete ferrite matrix in their microstructure. All three SGIs have a basic microstructure consisting of graphite nodules in an iron matrix, and it was shown that the as-cast SGI microstructure could be completely characterized by evaluating graphite morphology (size, shape, nodules count and distribution), matrix composition and the phase properties. For SGI

3.4. SUMMARY

grades with different silicon content, the main differences were in the matrix structure and slight changes were also observed in graphite morphology. Higher silicon content on SGIs yields higher mechanical properties. Comparing the mechanical properties of as-cast SGIs, EN-GJS-500-14 with 3.71 % silicon stands out with an adequate balance of strength and elongation. Higher strength and excellent elongation of EN-GJS-500-14 showed its potential to replace some of the steels and higher strength pearlitic grades. So, the 500-14 grade was further deep cold rolled and thermal cycled to investigate the influence of these processes on SGI microstructure and properties.

DCR process parameters used for cold rolling on the as-cast EN-GJS-500-14 block were discussed in this chapter. The effect of DCR process on SGI material and microstructure was explained in details. The major challenge observed for DCR process on SGI material was the presence of subsurface graphite particles that caused the ferrite matrix cracking on the rolled surface. However, the cracks were only surface cracks and did not extend much deeper on the rolled section. It was also pointed out that graphite characterization on the deep cold rolled surface might lead to errors as many partially exposed spheroidal graphite nodules appeared irregular or elongated due to partial surface exposure after the matrix deformation during DCR process. So, the microstructure characterization of deep cold rolled SGI should be done with adequate grinding and polishing to expose real graphite form. IPF maps and GOS maps from EBSD analysis were used to investigate grain orientations, and GOS maps further used to estimate strain hardened zone in the DCR process. Also, special consideration should be given to residual stress as it is an important effect in DCR process. Thus, the microstructure characterization result after DCR process should be stated with effective depth of strain hardening. To represent thermal process on the SGI material, thermal cycling and its effect on microstructure and phase properties were studied in this chapter. Although the initial thermal cycling showed decrease in thermal expansion behavior, no significant change in material microstructure and phase properties was noticed. However, the major microstructure change was decohesion of the graphite-ferrite interface due to dissimilar coefficients of thermal expansion. So, additional parameter illustrating the graphite-ferrite interface state must be included with graphite morphology, the matrix structure and its properties, to completely characterize SGI microstructure after thermal processes.

Overall, the SGI microstructure can be characterized by graphite morphology, matrix composition and phase properties. But, additional parameters must be

considered to completely characterize the microstructure after additional mechanical and thermal processes. For the deep cold rolling process, careful graphite characterization and effective depth of strain hardening must be stated. For thermal cycling case, the graphite-ferrite interface must be given importance. For the elastic-plastic properties of the matrix, two methods were explored based on tensile test and nanoindentation test. The phase properties estimated from tensile test better approximated the bulk matrix used in microstructure characterization. So in this work, the bulk phase properties will be used to define the matrix phase properties.

The microstructure studies of different grades of SGI contributed comparison of SGI materials with different silicon content. Microstructure investigation of SGI after thermal and mechanical processes evaluated microstructure changes in such processes. It was pointed out that the mechanical surface processes like DCR could severely deform surface microstructure requiring additional precaution in graphite morphology characterization. For the thermal cycling, it was identified that the graphite-matrix interface state should be included in microstructure characterization. It was stated that with the additional characterization of interface state for thermal processes and residual stress for mechanical processes, the SGI microstructure could be completely characterized by graphite morphology, matrix composition and phase properties.

Chapter 4

Tensile and Fatigue Damage Mechanisms in SGI

Damage in Spheroidal Graphite Iron (SGI) components originate as microcracks from the microstructure features and further propagation of the initiated microcracks depends on the stress-strain inhomogeneity at the crack tip due to microstructure features. Graphite nodules and cast defects in the iron matrix cause inhomogeneous stress-strain distribution in the microstructure. Review of damage mechanisms studies in different SGI grades showed graphite nodules as common microstructure features influencing damage initiation and propagation mechanisms. Even though different works have suggested different roles and mechanisms of graphite damage, most of them agreed to the point that matrix-nodule decohesion is one of the most frequent mechanisms. From the literature, it was noticed that most of the tensile damage mechanisms were studied based on the in-situ tensile tests, but the fatigue damage mechanisms were investigated based on crack propagation tests without much consideration to fatigue crack initiation. So, in this work, fatigue damage mechanisms in SGI material are investigated based on separately performed Fatigue Crack Initiation (FCI) and Fatigue Crack Propagation (FCP) tests.

The microstructure and material characterization of as-cast SGIs with different silicon contents showed an adequate balance of strength and ductility with stable ferrite matrix in EN-GJS-500-14 grade. So, it is more desirable to study damage mechanism in this SGI grade that has potential to replace ferritic grades to have better strength and ferritic-pearlitic grades to have better ductility. In chapter 3, the as-cast SGI material after Deep Cold Rolling (DCR) and thermal

cycling process were also studied. Microstructure characterization results at different conditions exhibited that graphite morphology, matrix composition and phase properties completely characterize basic SGI microstructure. However, additional microstructural parameter must be considered to account for additional mechanical and thermal processes. For DCR process, above-mentioned microstructure parameters alongside residuals stress state and careful graphite characterization completely described deep cold rolled SGI microstructure. Similarly, for thermal cycled SGI, above mentioned microstructure parameters alongside interface state described thermal cycled microstructure. Thus, in this work, damage mechanisms studies were focused on graphite nodules and matrix cracking on EN-GJS-500-14 SGI.

In this chapter, damage mechanisms on EN-GJS-500-14 were investigated by tensile and fatigue tests. Fatigue damage in specific was studied by fatigue crack initiation and propagation tests. This chapter starts with the details of specimens and test methods used for each experimental study. The tensile damage mechanisms are described in section 4.2 and section 4.3 followed by detail fatigue damage mechanisms. Finally, in section 4.4, damage criteria based on an experimental study of tensile and fatigue tests are established.

4.1 Test Specimens and Methods

A dynamic material testing machine by *Shimadzu ADT-AV10K1S5* air-servo system was used for all the experimental tests. The machine has a working range of ± 10 kN load (precision = $\pm 0.2\%$ of maximum test force) and ± 25 mm stroke (precision = $\pm 0.1\%$ of rated stroke) with the maximum operating frequency of 20 Hz. Due to the low capacity of the load cell, force measurement was accurate and suitable for miniature specimen testing; however, the stroke measurement by the machine was not very accurate. So, the tests were conducted as force controlled tests. The microstructural damage was studied in an Optical Microscope (OM) and Scanning Electron Microscope (SEM), which required the samples to be small enough to fit on the sample table. As the tests were repeated, each test requiring OM and SEM studies in between the tests, the whole test specimen should fit inside the SEM chamber and the sample table. Thus, miniature specimen designs were adopted for all the damage mechanisms studies in this work as explained in following sections.

4.1.1 Tensile test

A miniature pin loaded tensile test specimen was designed considering ASTM E8 for tensile test [157] as shown in Figure 4.1 a). The pin-loaded design was chosen as it was easier to align the specimen and avoid biaxial stresses in the test specimen. The specimen loaded in the machine together with the additional clamping fixtures are shown in Figure 4.1 b). The specimen was first loaded with the pin, and a small load was applied to self-align the specimen before it was clamped by the plates. The clamping fixture presents stress concentration at the pin to ensure failure at the narrow gauge section. The specimens were metallographically ground and polished before the test to enable efficient observation of the specimen microstructure without additional preparation. However, the specimens were not etched as the etched phase boundaries and grain boundaries will make crack detection difficult. Load and deformation in the test was measured using the in-built load cell and displacement sensor in the Shimadzu tester. It was desirable to measure deformation of the gauge section of the specimen, however, due to very narrow gauge section no suitable extensometer could be found. Thus, all the results were plotted and labeled with respect to applied stress. For each specimen, the test was stopped at different regions of the stress-strain curve, completely removed, studied under OM and SEM and reloaded to higher stress region. With reference to the average yield stress of 410 MPa, the tests were stopped at three regions before final failure; 400 MPa (just before yielding), 420 MPa (just after yielding) and 450 MPa (with large plastic deformation). At each step, the load was applied at a constant rate of 5 N/sec, hold at the maximum stress for 30 seconds and unloaded at a constant rate of 10 N/sec.

Figure 4.1: Miniature tensile test specimen design (all dimensions are in mm)
a) Tensile test specimen design (thickness = 1 mm) b) Specimen loading and its fixture assembly.

4.1.2 Fatigue Crack Initiation (FCI) test

The tensile specimen explained in section 4.1.1 was also designed considering fatigue testing. So, the same miniature specimen design was used for the fatigue crack initiation tests. Similar to the tensile test, the specimens were ground and polished before the FCI tests. ASTM standard E466 [158] was referred for the FCI tests. Constant amplitude fatigue tests were conducted at load ratios (R) of 0.1 (maximum stress = 70 % of the UTS) and 0.4 (maximum stress = 85 % of the UTS) to ensure crack initiation at a few thousands load cycles. As miniature specimen geometry with gauge length of 3 mm was used, the deformation was measured with the displacement sensor in the tester (accuracy = $\pm 0.1\%$ of ± 25 mm). However, all the results were explained with respect to stress evaluated from accurately measured load and specimen size. The crack initiation tests were aimed to identify possible initiation sites and to understand the influence of graphite nodules and their morphology on the crack initiation mechanisms. Here, OM and SEM were used to study crack, however recent advance techniques like Digital Image Correlation (DIC) and Direct Current Potential Drop (DCPD) have also been used to accurately measure crack initiation and growth [159]. For each test, the test was first stopped for OM observation after 200,000 cycles. Both surfaces of the specimen were studied. Understanding the extent of damage in SGI microstructure, the sample was re-tested for additional load cycles of 50,000, or 30,000, or 20,000. The sample was again studied under OM to observe cracks initiation and microcracks propagation before it was reloaded. The re-testing and OM observation were repeated until sufficient cracks were initiated with clear microcrack growth. The sample was finally studied thoroughly in the SEM to have a clear view of the crack initiation sites and mechanisms.

4.1.3 Fatigue Crack Propagation (FCP) test

The miniature Compact Tension (CT) specimen, as illustrated in Figure 4.2 a) was designed considering ASTM standard E647 [160] for the FCP tests. ASTM standard E647 provides a comprehensive guideline for the FCP test. The CT specimens were metallographically polished before the test for clear visualization of cracks. Load ratios of $R = 0.1$ and 0.4 were selected, and an initial stress intensity factor range (ΔK_{start}) of $13 \text{ MPa}\sqrt{m}$ was used based on FCP test results reported in previous work [161]. Displacement is measured with the in-built displacement sensor. For the FCP studies, two intermediate crack sizes were considered; short

Figure 4.2: Miniature crack propagation test specimen design (all dimensions are in mm) a) CT specimen design (thickness = 1 mm) b) Fatigue crack propagation fixtures with front and back microscopes to measure crack length.

crack size with the crack tip still in the stable crack region was considered short intermediate crack, and the crack with the crack tip approaching the unstable region was considered long intermediate crack. During the test, the crack was observed and measured by OM placed on two faces of the specimen (Figure 4.2 b)). The lengths of cracks on both faces were measured during the test. The average crack length and the applied load were used in Eq. 4.1 to evaluate stress intensity factor (ΔK).

$$\Delta K = \frac{\Delta P}{B\sqrt{W}} \frac{(2 + \alpha)}{(1 - \alpha)^{3/2}} (0.886 + 4.64\alpha - 13.32\alpha^2 + 14.72\alpha^3 - 5.6\alpha^4) \quad (4.1)$$

where,

$\alpha = a/W$, a is the average crack length

ΔP is the applied load range

B is the thickness of the CT specimen

W is the characteristic length of the CT specimen

The calculated stress intensity factors were plotted against fatigue crack growth rate (da/dN). Secant method outlined in ASTM E647 was used to determine da/dN , which was determined by calculating slope between two adjacent data point in experimental crack length (a) vs. number of cycle (N) data. After sufficient growth of the cracks, both the specimen surfaces were studied under the SEM. Some of the specimens were tested to complete fracture to study the role

of graphite nodules at different fatigue stages along and near the crack path. The fracture surface of the failed specimens was also studied to understand the fracture pattern at different fatigue crack propagation regions.

4.1.4 Fatigue test for S-N curve

In addition to fatigue damage mechanisms tests, fatigue tests were performed on a standard half-sized fatigue specimens (Figure 4.3 a)) to obtain the S-N curve of EN-GJS-500-14. As part of an undergraduate final year project [162], the specimens were polished to an average surface roughness of around $0.6 \mu\text{m}$ before the test, which allowed to represent material fatigue strength rather than component fatigue strength. Usually, the component fatigue strength is less than material fatigue strength as the fabrication processes like machining and forging could introduce surface roughness and tensile residual stress. In this S-N test, it was attempted to capture material fatigue properties. The accuracy of the estimated S-N curve and endurance limit improve with the number of test specimens. But, due to the lack of sample material and time, a test method with minimum specimen requirement was used to obtain the S-N curve from a total of 14 test specimens. Out of different methods, JSME S 002 standard [163] simplified method to deter-

Figure 4.3: Fatigue test method for S-N curve and the specimen used a) design and dimension of standard half size fatigue test specimen (all dimensions in mm and thickness = 2 mm) b) polished specimen before the test c) JSME S002 standard procedure to obtain S-N curve using minimum test specimens (numbers represent test sequence).

mine S-N curve from a minimum number of fatigue tests was used. It involves testing of eight specimens at finite life region; two fatigue tests at four stress levels, and six test specimens for determining the endurance limit using the staircase method as illustrated in Figure 4.3 c). The fatigue life was estimated by taking the average in the finite life region, whereas for the endurance limit, staircase method was adopted. In this method, fatigue tests were conducted at different stress ranges with uniform stress step (maximum stress step of 5 % of UTS at $R = 0.1$ was used). If the specimen failed before 2 million cycles (stipulated as fatigue limit), the next specimen was tested at a lower stress amplitude; however, if the specimen survived 2 million cycles, the test was suspended, and the next specimen was tested at higher stress value (5 % higher than the current). For the statistical determination of the mean endurance limit and standard deviation, the Dixon-Mood method was used. The equations to calculate mean endurance limit and standard deviation are presented in Eq. 4.2 to Eq. 4.5, where the choice of the equation depends on the less frequent event in the test; i.e. survival or failure.

When survival is the less frequent event,

$$\mu_s = S_0 + d. \left(\frac{A_{DM}}{\sum \eta_{DM,i}} + \frac{1}{2} \right) \quad (4.2)$$

When failure is the less frequent event,

$$\mu_s = S_0 + d. \left(\frac{A_{DM}}{\sum \eta_{DM,i}} - \frac{1}{2} \right) \quad (4.3)$$

where, $A_{DM} = \sum(i)(\eta_{DM,i})$, $\eta_{DM,i}$ is the number of the less frequent event at the i^{th} maximum stress step, d is the stress interval used in the test, and S_0 is the lowest stress amplitude used.

For the standard deviation, if $\frac{B_{DM} \sum \eta_{DM,i} - A_{DM}^2}{(\sum \eta_{DM,i})^2} \geq 0.3$,

$$\sigma_s = 1.62d \left[\frac{B_{DM} \sum \eta_{DM,i} - A_{DM}^2}{(\sum \eta_{DM,i})^2} + 0.029 \right] \quad (4.4)$$

Else,

$$\sigma_s = 0.53d \quad (4.5)$$

where, $B_{DM} = \sum(i^2)(\eta_{DM,i})$

4.2 Tensile Damage Mechanisms

Tensile damage mechanisms in SGIs have been reported by many authors (cf. section 2.1.7). The understandings of tensile damage mechanisms in those studies mostly agreed on decohesion of graphite nodules by plastic deformation of the matrix and final fracture by coalescence of those graphite voids in the ferritic SGI. Recently, Di Cocco et al. [10] have shown additional internal cracking of graphite nodules in their in-situ tensile test. In high silicon Solution Strengthened Ferritic (SSF) SGIs, the properties of the ferrite matrix have been changed as compared to usual SGI grades. Most of the graphite nodules are spheroidal in SGIs; however, degenerated graphite particles also exist in the SGI microstructure. The effect of such degenerated graphite particles on tensile crack initiation and the fracture is studied in 500-14 SGI.

Table 4.1: Details of the specimens used in tensile damage mechanism studies.

Specimen	Gauge width (mm)	Thickness (mm)	Fracture stress (MPa)
TDM-1	2.96	0.98	496
TDM-2	2.94	0.82	580
TDM-3	2.89	0.81	545

Three specimens were tested, and details of the specimens along with the stress at fracture are reported in Table 4.1. The specimen TDM-1 was used as preliminary test specimen to estimate maximum force and tentative stress-strain response to plan intermediate stop tensile damage mechanism study tests. The specimen was not fine polished like actual test specimens. Specimens TDM-2 and TDM-3 were tested and stopped intermediately for crack initiation and propagation studies. The higher fracture stresses reported in TDM-2 and TDM-3 were due to three reasons; a) the specimen was fine polished minimizing surface defects, b) work hardening in stress-strain behavior with incremental loading, and c) miniature specimen has less chance of defects in it reported as size effect.

4.2.1 Crack initiation and propagation

As explained in section 4.1.1, tensile tests were stopped intermediately. Figure 4.4 illustrates the general tensile damage mechanisms in 500-14 SGI. The four points in stress-strain curves indicate the point of damage mechanism studies. Just before yield, the specimens showed slight decohesion of spheroidal graphite nodules

without matrix cracking when observed under OM, so detailed SEM studies were focused on microcracks initiation and fracture after yield point. Decohesion is clearly visible above yield point showing decohesion of most of the graphite nodules, which further grows with continuous plastic deformation. The graphite nodule voids and initiated microcracks coalesce at the final fracture of the specimen. Out of the tested specimens, SEM images of TDM-2 were used to explain the tensile damage initiation and fracture behavior. The tensile damage mechanisms were studied with respect to the graphite shape and the nature of the damage observed in the graphite nodules and the ferrite matrix.

Figure 4.4: Microstructure damage mechanisms at different parts of the tensile test.

The optical microscope observations after 400 MPa (onset of yield) started to show slight decohesion of some of the graphite nodules; however, no crack initiations were observed in the ferrite matrix. The subsequent SEM studies after yielding showed clear decohesion of most of the graphite nodules (Figure 4.5 a), Figure 4.6 a) and Figure 4.7 a)). Spheroidal graphite nodules showed decohesion from the ferrite matrix without crack initiation in the ferrite matrix (Figure 4.6 a)). Compacted graphite nodules due to its elongated graphite shape, started to

Figure 4.5: SEM images of TDM-2 specimen microstructure after different loading levels a) after 420 MPa, b) after 450 MPa and c) after fracture at 580 MPa.

Figure 4.6: SEM images of spheroidal graphite nodules damage in tensile test of TDM-2 a) after 420 MPa, b) after 450 MPa and c) after fracture at 580 MPa.

show slip bands and microcracks as shown in Figure 4.7 a). These graphite nodules showed combinations of the matrix-nodules decohesion and internal cracking of the graphite nodules. With the additional load on the specimen, the plastic deformation of the ferrite matrix caused graphite nodule voids to grow at 450 MPa (Figure 4.5 b), Figure 4.6 b) and Figure 4.7 b)). At this point, most of the graphite nodules showed decohesion from the ferrite matrix (Figure 4.5 b)); spheroidal graphite nodules mostly showed growth in the decohesion gaps (Figure 4.6 b)); and compacted and irregular graphite nodules showed microcrack initiation from the slip bands created by graphite decohesion or internal cracking of the graphite nodules (Figure 4.7 b)). The specimen was further loaded to fracture and the microstructure images of the specimen after fracture were presented in Figure 4.5 c), Figure 4.6 c) and Figure 4.7 c). The overall microstructure after fracture (Figure 4.5 c)) exhibited damage of most of the graphite nodules with slip bands and microcracks around the graphite voids in the ferrite matrix. Because of large plastic deformation of the specimen, it was difficult to identify graphite

Figure 4.7: SEM images of compacted and irregular graphite nodules damage in tensile test of TDM-2 a) after 420 MPa, b) after 450 MPa and c) after fracture at 580 MPa.

nodules and microstructure near the fracture region; however, at some distance from the fracture region, the microstructure could be studied under the SEM. At a short distance from the fracture region, most of the graphite voids grew in size with a clear sign of plastic slip bands at an angle of 45° with the loading direction (vertical). The spheroidal nodules suffered less damage as compared to degenerated graphite nodules. Some of the spheroidal nodules suffered internal cracking either circumferentially (as reported in [10]), or from the center of the nodule referred as graphite nucleation site (Figure 4.6 c)). The circumferential internal crack of spheroidal nodules is due to the mechanical properties difference within the nodules caused by different graphite growth occurring circumferentially in the solidification and cooling stage of SGI as illustrated by Di Cocco et al. [67] and Muhmond [63] in their studies. It was also reported that the graphite nucleated and grew from the melt is softer than that grew in the later solidification stage, which correlates with nodules damage at the center. Compacted, flake and

irregular graphite nodules have different growth morphology than the spheroidal nodules. Elongated graphite growth was reported along the basal plane in the hcp crystal structure of the graphite. This resulted in relatively easier fracture along the graphite mono-layer, demonstrated by internal fracture of most of the elongated graphite nodules at the fracture point of the specimen (Figure 4.5 c) and Figure 4.7 c)).

The observed damage mechanisms are in agreement with similar tensile damage characterization reported by several researchers [9, 79, 84]. The studies by Dong et al. [79, 164] in specific have performed detail investigation of tensile damage and fracture characterization in a ferritic nodular cast iron. The experimental damage mechanisms had been studied in Part I [79], and the tentative quantification of these damage mechanisms on fracture toughness had been explored in Part II [164]. The reported damage characterization states; a) no damage of the graphite nodule interface in the elastic part, b) first decohesion appeared when the microscopic yield stress was reached, c) increasing macroscopic plastic deformation induced void growth in the stress direction, d) final failure occurred by shear instabilities linking adjacent voids, and e) majority of the graphite nodules displayed the decohesion mechanism. These damage mechanisms were the very similar mechanisms illustrated in Figure 4.4. However, some other additional mechanisms observed in current studies like the internal cracking of spheroidal graphites (also reported by Di Cocco et al. [9]), and the damage of non-spheroidal graphite particles, degenerated graphite particles and shrinkage cavities were not reflected in that study. The modeling of mechanical behavior explained in that work was impressive, which was further extended to thermomechanical fatigue by Seifert and Riedel [165].

Comparison of spheroidal and compacted graphite nodules damages in Figure 4.6 and 4.7 showed slight difference in the tensile test. Compacted and irregular graphite nodules due to their higher stress concentration effect showed plastic slip bands in the ferrite matrix just after material yield point has been crossed (Figure 4.7 a)). At the same stress level, spheroidal nodules showed decohesion from the ferrite matrix, but no slip bands accumulation in the ferrite matrix were perceived. Another noticeable difference was on the graphite decohesion and cracking as explained above. However, at the fracture point, both graphite nodule types demonstrated similar mechanism of nodule voids growth and slip bands accumulation around the voids without any significant growth of the initiated microcracks.

Figure 4.8: SEM images of degenerated graphite particles damage in tensile test of TDM-2 a) after 450 MPa, b) and c) after fracture at 580 MPa.

Degenerated graphite particles as shown in Figure 4.8 were also observed on the TDM-2 specimen surface. Such degenerated graphite particles formed due to lack of sufficient nodulizer at localized regions in thick casting. The observed degenerated graphite phase appeared as dispersed graphite particles in the ferrite matrix. Even though the casting process and nodulizer content were optimized; some degenerated graphite particles were inevitable. Unlike graphite nodules, degenerated graphite particles did not show decohesion from the ferrite matrix; instead, they directly initiated microcracks in the matrix. However, it could not be understood whether the crack initiated from the graphite particle or the ferrite matrix in this experiment. Up to yield point degenerated graphite particles did not show any damage, then with continuous plastic deformation of the ferrite matrix, microcracks were observed when the applied bulk stress on the specimen

gauge section was 450 MPa. The microcracks grew over the degenerated graphite region, but did not show much growth at the fracture point as shown in Figure 4.8 b) and c). However, the crack faces opened to a larger extent at the fracture of the specimen.

Figure 4.9: SEM images of the ferrite matrix cracking in tensile test of TDM-2 a) after 420 MPa, b) after 450 MPa and c) after fracture at 580 MPa.

Another usually reported damage mechanism in SGIs was crack initiation within the ferrite matrix. Similar cracks were observed in TDM-2 specimen, one of them is illustrated in Figure 4.9 at different stress levels. Because of minimum plastic deformation of the ferrite matrix at the onset of yield, no crack in the ferrite matrix was reported. The ferrite crack was clearly visualized after the material yield point as shown in Figure 4.9 a). The crack was initiated in the middle of the ferrite matrix without any influence of the graphite distribution. Further loading the specimen to 450 MPa did not show much growth of the ferrite crack and the crack only showed slight opening of the crack faces (Figure 4.9 b)). When the same crack was observed after the fracture of the specimen, no much growth in crack length was noticed; however, the crack faces opened up to a large extent (Figure 4.9 c)). And it could be clearly seen that the crack plane was not perpendicular to the specimen surface; instead, it was at some angle. Because of the inclined crack plane, it was not possible to study what initiated the crack in the ferrite matrix. But, it was predicted that such cracks on the ferrite matrix might be due to casting defects and graphite nodules below the surface.

In a joint study with the project collaborator in Jonkoping University, a similar miniature tensile test was performed on 500-7 SGI grade. Using Digital Image Correlation (DIC), the strain concentration in the microstructure was estimated at the onset of microcracks initiation [33]. Further in-situ tests helped to estimate the overall stress range at the onset of graphite decohesion and microcrack initiation on

the stress-strain response [166]. The study showed that for 500-7 SGI with 0.2 % yield stress of 280 MPa, the bulk stress at which graphite nodules decohesion and microcrack initiation occurred was in the range of 280–330 MPa. This stress range corresponded to the kink (at yield stress region) on the stress-strain response. In this current study on 500-14 SGI, the SEM study just before yield stress (400 MPa) and just after yield stress (420 MPa) showed that most of the graphite nodules showed decohesion from the ferrite matrix in that stress range. So, it can be stated that in SGIs, graphite nodules decohesion mostly occurs at the stress range around the kink observed in the material stress-strain response. The change of slope at the kink is related to the dissipation of energy in the plastic deformation and crack initiation in the deformed matrix around graphite nodules.

4.2.2 Fracture behavior

The fracture surface of TDM–2 specimen was studied in the SEM, and images are shown in Figure 4.10. The overall fracture surface in Figure 4.10 a) indicates black graphite nodules in the highly deformed ferrite matrix. Most of the graphite nodules were observed undamaged inside graphite voids that are larger than the graphite nodules, indicating higher deformation of the ferrite matrix around the nodules. Figure 4.10 b) offers a clear view of graphite nodules inside larger graphite voids, and it could be noticed that even if the neighboring nodule is more than 10 μm apart, the ferrite matrix separating these nodules is just around 2 μm (indicated by the arrows). Another interesting feature to notice on the fracture surface was ferrite fracture regions (indicated inside ellipse). After sufficient plastic deformation, the strain hardened ferrite matrix between graphite nodules fractured, indicating rough ductile fracture surface. Such ferrite fracture regions were common in the fracture surface, and a clear image of one of them is shown in Figure 4.10 c). In the picture, the ferrite region could be characterized with micro-voids, which were commonly seen in the ferrite fracture region. From the fracture surface studies, it could be stated that matrix-nodules decohesion and graphite voids growth were the major tensile damage mechanisms in SGIs. Micro-voids were nucleated in the ferrite matrix during plastic deformation, and these micro-voids coalesced at the final fracture of the specimen to connect the graphite nodule voids.

Figure 4.10: SEM images of the fracture surface of TDM-2, a) overall fracture surface (ellipse indicating the ferrite fracture regions); b) micro-voids on the ferrite fracture region; c) graphite nodules void growth due to large plastic deformation of the ferrite matrix.

4.3 Fatigue Damage Mechanisms

Most of the engineering components fail by fatigue, so good understanding of fatigue damage mechanisms is required in components design against cyclic loads. Fatigue damage basically includes three stages; fatigue crack initiation, fatigue

crack propagation and rapid fracture. Out of these three stages, fatigue crack initiation and propagation are of interest in damage studies as these two stages account for the service life of the component. Damage mechanisms studies have reported the significant influence of graphite nodules (cf. section 2.1.7). However, the effect of different graphite nodule shapes in fatigue crack initiation and propagation has not been well reported. From the microstructure study, it has been identified that some of graphite nodules deviates from the spheroidal shape to form chunky, compacted and irregular graphite nodules, which is common in most of the SGIs. Considering the effect of these degenerated graphite particles in crack initiation and propagation process, SGI damage mechanisms were studied in this work.

4.3.1 Fatigue crack initiation test

Series of fatigue crack initiation tests were performed on miniature tensile samples as explained in section 4.1.2. The summary of the tested specimens is presented in Table 4.2. Total of fifteen specimens were tested, out of which six were tested

Table 4.2: Summary of the FCI specimens and tests.

Specimen	Gauge section		Load ratio R	Max. stress (MPa)	No. of cycles at failure N
	Width (mm)	Thickness (mm)			
FCI-as-cast-1	2.96	1	0.1	$0.7 \times \text{UTS}$	325,312
FCI-as-cast-2	2.99	1.02	0.1	$0.7 \times \text{UTS}$	304,464
FCI-as-cast-3*	2.96	0.87	0.1	$0.6 \times \text{UTS}$	2,000,000+
FCI-as-cast-4	2.94	0.95	0.1	$0.7 \times \text{UTS}$	474,047
FCI-as-cast-5	2.98	0.96	0.1	$0.7 \times \text{UTS}$	121,507
FCI-as-cast-6	2.96	0.98	0.1	$0.7 \times \text{UTS}$	254,308
FCI-as-cast-7*	2.95	0.97	0.1	$0.7 \times \text{UTS}$	330,000+
FCI-as-cast-8	2.96	0.98	0.1	$0.7 \times \text{UTS}$	191,964
FCI-as-cast-9*	2.98	0.98	0.1	$0.7 \times \text{UTS}$	350,000+
FCI-as-cast-10*	2.98	1.02	0.4	$0.7 \times \text{UTS}$	2,000,000+
FCI-as-cast-11*	2.93	0.98	0.4	$0.8 \times \text{UTS}$	1,000,000+
FCI-as-cast-12	2.9	0.91	0.4	$0.85 \times \text{UTS}$	335,080
FCI-as-cast-13	2.93	0.92	0.4	$0.85 \times \text{UTS}$	331,256
FCI-as-cast-14	2.93	0.91	0.4	$0.85 \times \text{UTS}$	441,767
FCI-as-cast-15*	2.94	0.88	0.4	$0.85 \times \text{UTS}$	300,000+

* Samples that were not tested to final failure, no. of cycles reported was the load cycle tested.

at a load ratio (R) of 0.4 and rest of them at 0.1. Specimens FCI-as-cast-1, 2 and 3 were preliminary tests at $R=0.1$ to estimate the appropriate loading conditions and inspection intervals for the analysis of crack initiation mechanisms. Specimens FCI-as-cast-4 to 9 were well polished before the test, and the undeformed microstructure characterized in the gauge section. Out of the tested specimens, FCI-as-cast-4, 7 and 9 were observed under OM and SEM to understand crack initiation in SGI microstructure and the role of graphite nodules. Some specimens failed earlier than 200,000, on which cracks initiation could not be studied. Fatigue cycles to failure results showed large variations, illustrating the scatter nature of fatigue life. Such scatter of fatigue life is also reported in the literature and is due to the random distribution of graphite nodules and larger defects in the specimen. Specimens FCI-as-cast-10 and 11 were intermediate specimens at a load ratio from 0.4. With the higher load ratio, decrease in the amplitude of the cyclic load caused fatigue life to increase. So, to balance for the change in load ratio, the maximum stress was increased to $0.85 \times \text{UTS}$, which resulted into crack initiation at similar load cycles as in $R = 0.1$. Specimens FCI-as-cast-12 to 15 were tested at $R = 0.4$. Two load ratios were considered in this study, so that effect of load ratio (also referred to as mean stress effect) in fatigue damage could be included in this study. Furthermore, in FCI-as-cast-15 specimen, quantitative study of graphite nodules damage was done with respect to the Roundness Shape Factor (RSF) of graphite nodules.

Fatigue crack initiation mechanisms

Fatigue cracks initiation was studied on miniature tensile specimens after different load cycles. Out of the three specimens on which cracks initiation was studied after different load cycles, mostly images of specimen FCI-as-cast-4 was used to illustrate initiation mechanisms. Based on the observed results, fatigue crack initiation mechanisms could be divided into three basic types in the ferritic SGI, as discussed below.

Firstly, cracks were initiated from elongated compacted and irregular graphite nodules. In Figure 4.11, initiation of cracks and their growth is illustrated in two of the degenerated graphite nodules after different fatigue cycles. The occurrences of these degenerated graphite nodules were found to be vulnerable from the point of crack initiation, as those graphite nodules initiated early microcracks. Cracks initiated from such graphite nodules at the extreme edge without prior debonding

Figure 4.11: Images showing fatigue crack initiation in elongated graphite nodules on specimen FCI-as-cast-4 (bold arrows indicates graphite cracks and line arrows indicates ferrite cracks) a) and b) after 200,000 cycles, c) and d) after 250,000 cycles, e) and f) 310,000 cycles, g) and h) after 390,000 cycles.

and graphite cracking as shown in Figure 4.11 a), c), e) and g). Due to high stress concentration at the end of compacted graphite nodules, a sign of microcracks initiation was observed after 200,000 load cycles. There was no sign of graphite decohesion at the point of crack initiation. With additional load cycles, the initiated microcracks started to grow slowly, and additional internal cracking of the graphite nodules become more vivid (Figure 4.11 e) and g)). On the other degenerated graphite particles shown in Figure 4.11 b), d), f) and h), damage initiated in the form of internal graphite crack (Figure 4.11 b)), that grew within the graphite particle at the beginning (Figure 4.11 d)) and ultimately initiated

cracks in the ferrite matrix (Figure 4.11 f) and h)). In other cases cracks initiated in the ferrite matrix with internal cracking of the graphite nodules. Thus, it can be stated that early cracks initiation in degenerated graphite nodules were either by internal cracking of graphite nodule followed by crack growth into the matrix, or by decohesion followed by crack initiation from the interface, or by the combination of these mechanisms. It should be noted that graphite nodules with their major axis perpendicular to the loading direction were preferred initiation sites. The initiated cracks grew perpendicular to the loading direction, and one of these initiated cracks grew as the main crack that led to the final failure of the specimen. In the case of specimen FCI-as-cast-4, the crack shown in Figure 4.11 g) further propagated as to cause final failure of the specimen.

Figure 4.12: Images showing damage of spheroidal graphite nodule under cyclic load on specimen FCI-as-cast-4 a) after 250,000 cycles b) after 310,000 cycles c) after 390,000 cycles.

Secondly, internal cracking of spheroidal graphite nodules into onion-like damage was observed. As shown in Figure 4.12, larger graphite nodules tend to internally crack into graphite core and shield. However, it should be noted that at the time of crack initiation from compacted graphite nodules (200,000 cycles), spheroidal graphite nodules did not show any effect. Signs of internal circumferential crack (Figure 4.12 a)) and decohesion started after 250,000 load cycles. The internal cracks did not show much increase in the crack after further loading (Figure 4.12 b) and c)). Not all spheroidal graphites showed such internal cracking; many were still intact in the ferrite matrix. Comparing the size of graphite nodules, it can be assumed that those larger nodules might have nucleated earlier during the solidification stage, grown from the melt and then by diffusion of carbon from austenite; causing property difference within each graphite nodule. The resulting property difference inside those graphite nodules had caused internal cracking of fully grown spheroidal graphite nodules. Decohesion of graphite-matrix interface was reported

as one of the common damage mechanisms in SGI microstructure. At the point of cracks initiation from degenerated graphite nodules, most of the spheroidal graphite nodules did not initiate cracks even though they were debonded from the ferrite matrix. So, graphite-matrix decohesion was not necessarily the influencing mechanism during early crack initiation, but it was one of the important mechanisms in crack propagation. On few occasions, cracks were also initiated from graphite-ferrite interfaces. Such crack initiation was uncommon, and mostly the spheroidal graphite nodules were intact in the ferrite matrix at the early crack initiation stage when the compacted and irregular graphite nodules already showed signs of initiation.

Figure 4.13: SEM images of fatigue crack initiation and growth from shrinkage cavity on specimen FCI-as-cast-7 a) after 200,000 cycles b) after 330,000 cycles.

Lastly, crack initiations were also observed from shrinkage porosities present in the microstructure. Casting defects like shrinkage porosity have been reported as one of the major causes of early cracks initiation in cast irons. The casting process for the investigated SGI was optimized, so shrinkage porosities of a size comparable to graphite nodules were only observed occasionally. However, in specimen FCI-as-cast-7, a shrinkage cavity of size few times that of the graphite size was observed to initiate a crack as shown in Figure 4.13 a). The initiated crack further propagated towards a near graphite nodule forming a relatively larger crack when observed after 330,000 cycles (Figure 4.13 b)). Other shrinkage porosities of a size comparable to the graphite nodules size showed crack initiation at similar fatigue cycles as for the compacted graphite nodules. Shrinkage porosities were voids in the matrix that gave rise to a stress concentration effect leading to cracks initiating earlier at the edge of the voids. So, larger sizes of such shrinkage defects will have a dominant effect on the fatigue crack initiation and the overall fatigue

life.

Figure 4.14: SEM images of fatigue crack initiation from the ferrite matrix on specimen FCI-as-cast-7 a) and b) after 330,000 cycles.

In addition to the above-mentioned damage initiation mechanisms, cracks initiation was also reported for the ferrite matrix in ferritic SGI (cf. Section 2.1.7). Similar fatigue cracks were also observed in current studies. The SEM images in Figure 4.14 shows two of such cracks initiated in the ferrite matrix after 330,000 cycles in specimen FCI-as-cast-7. However, it should be noted that such ferrite cracks were not noticed when early cracks initiation from degenerated graphite nodules and shrinkage defects were observed. So, it could be proposed that such ferrite cracks could be due to the accumulation of dislocations and slip bands in the microstructure with sufficient load cycles. Some of the possible areas where dislocations could pile-up are grain boundaries and a thin layer of the surface ferrite matrix just above submerged graphite nodules, both of which were not considered in this study. So, based on the limited surface microstructure study, it will not be possible to properly understand the underlying mechanisms. Further study with proper facilities to study the effect of submerged graphite nodules and grain boundaries in microstructural crack initiation is needed.

After the series of OM observations of the specimen FCI-as-cast-4 after different load cycles, complete gauge section of the specimen was studied in detail in the SEM. Figure 4.15 and Figure 4.16 show images and distribution of the major cracks and damage observed on face 1 and face 2 of the specimen FCI-as-cast-4. The distribution of the initiated cracks was spread all over the gauge section, and mostly those degenerated graphite particles with thier major axis oriented perpendicular to the loading direction showed initiation of microcracks. For each of the graphite nodules presented in Figure 4.15 and 4.16, RSF was also reported. Crack

Figure 4.15: SEM images of fatigue cracks initiation on face 1 of the specimen FCI-as-cast-4 after 390,000 cycles.

Figure 4.16: SEM images of fatigue cracks initiation on face 2 of the specimen FCI-as-cast-4 after 390,000 cycles.

initiation mechanisms were correlated to the RSF values of the graphite nodules, and it was observed that most of the graphite nodules with RSF values less than 0.5 were responsible for fatigue cracks initiation in the ferrite matrix. Spheroidal graphite nodules with RSF values greater than 0.8 showed decohesion from the graphite matrix but did not initiate ferrite cracks even though microcracks were observed inside degenerated graphite particles. Comparatively larger spheroidal graphite nodules with RSF values greater than 0.9 showed circumferential internal cracks due to property gradient within the graphite nodules originated from different graphite growth regions (cf. Section 2.1.3). Some shrinkage cavities of a size comparable to graphite nodule size were observed on both faces of the specimen, which initiated microcrack of a size comparable to the microcracks from the degenerated graphite nodules.

Comparison of the observed crack initiation with the tensile damage exhibited a particular difference in the matrix crack initiation and deformation. In tensile damage, matrix-nodule decohesion due to large matrix deformation was the major damage initiation mechanism with less frequent cracks before final fracture (cf. Section 4.2), whereas in fatigue, cracks were initiated at many graphite nodules and the initiation was highly dependent on the graphite shape. The initiation and growth were due to progressive damage of the matrix in fatigue. However, some similarities were also noticed in the tensile and fatigue damage. Nodule shape showed the dominant effect on both loading types as compacted and degenerated graphite particles were the one to mostly initiate an early crack in the load bearing ferrite matrix. The nature of internal damage of the graphite nodules was also similar. Cracks initiation in the ferrite matrix reported in the literature were also visible in both the loading types in current studies. Moreover, due to rapid fracture of the specimen in the tensile test, it was difficult to study propagation of cracks; whereas in fatigue test, slow progressive damage permitting to give an insight of crack initiation mechanisms in ferritic SGI.

Fracture of the specimen

After the crack initiation observations, the specimen FCI-as-cast-4 was further tested to a final failure occurring at 474,047 cycles. The crack in Figure 4.15: b) on face 1 and Figure 4.16: q) on face 2 propagated to form the main crack, which grew by connecting initiated cracks and debonded graphite nodules. Soon after the main crack started to grow, the specimen fractured and could not provide details

on propagation mechanisms. Therefore, to clearly understand crack propagation micromechanisms, separate FCP tests were conducted on miniature CT specimens. From the crack initiation study, it was difficult to predict which crack among those initiated would result into final failure, but it could be estimated that one of the cracks initiated from larger defects (casting defects or degenerated graphite nodules) would further propagate to final fracture of the specimen.

Figure 4.17: SEM images of degenerated graphite particles and initiated cracks after final failure of the specimen FCI-as-cast-4.

Figure 4.17 shows the state of compacted and irregular graphite nodules after the final failure. Internal cracks of compacted nodules resulting into microcrack in the ferrite matrix could be clearly seen in the SEM images. When the SEM images in Figure 4.17 b), c) and d) after final fracture were compared to the SEM images after 390,000 cycles in Figure 4.16 j), l) and p) respectively, large crack opening with only slight growth in crack length was detected. The internal damage of compacted graphite nodules in Figure 4.17 a) and d) clearly show multiple layers of graphite damage along their longer axis. These multiple layers of damage suggested weak bond between the monolayers in compacted graphites as the

Figure 4.18: SEM images of spheroidal graphite nodules after final failure of the specimen FCI-as-cast-4.

preferred growth direction for such graphite nodules was along the monolayer direction. Differences in the graphite growth resulted into different graphite damage mechanisms in spheroidal nodules and the SEM images of the spheroidal graphite nodules after final failure is shown in Figure 4.18. It was observed that graphite nodule decohesion was the dominant mechanism for spheroidal graphite nodules. Spheroidal graphite particles after final failure showed an increase in decohesion gap with a clear view of circumferential internal cracks (Figure 4.18 b), c) and d)). However, crack initiation for the spheroidal graphite nodule decohesion was rarely noticed. The observed microstructural damage was similar to the damage in tensile test but with the higher frequency of microcracks of slightly larger

size. Similarities of the damage after fracture suggested that after microcracks had started to grow in the ferrite matrix, the miniature specimen fractured by connecting those initiated microcracks.

Figure 4.19: SEM imaged of large degenerated graphite particles on the fracture surface of the specimen FCI-as-cast-4 a) stable region fracture surface, b) unstable region fracture surface, c) and d) degenerated graphite nodules on the fracture plane.

The fracture surface of the specimen FCI-as-cast-4 was also observed in the SEM. The fracture surface showed two distinct types of surfaces presented in Figure 4.19 a) and b). The fracture surface in Figure 4.19 a) corresponds to the stable crack propagation region in the specimen and Figure 4.19 b) corresponds to the rapid fracture region. The crack growth in the stable region was due to progressive material fatigue; however, the fatigue striations were not obvious on the fracture surface. The unstable fracture surface resembles well with the fracture surface in the tensile test, demonstrating failure due to large plastic deformation of the ferrite matrix. In few occasions, larger degenerated graphite particles were also observed on the fracture surface (Figure 4.19 c) and d)). Such graphite particles seemed to change crack plane and initiate crack branching on the fracture surface (indicated

by a arrow in Figure 4.19 d)). The larger degenerated graphite particle behaved like a defect in the ferrite matrix and could be responsible for subsurface crack initiation and propagation. So, overall, degenerated graphite particles whether present on the surface or subsurface have a significant effect on the fatigue crack initiation and propagation behavior.

Fatigue crack initiation test at $R = 0.4$

Similar to the FCI tests at $R = 0.1$, FCI tests were also performed at $R = 0.4$. The results of the tests were presented in LCF8 conference [167]. The observed results were quite similar to that for $R = 0.1$ and could not find much of the difference as the mechanism investigations were mostly focused on qualitative SEM studies. Different types of crack initiation observed are summarized in Figure 4.20 a)–i).

Figure 4.20: SEM images of the fatigue crack initiation and graphite nodules damage observed in specimen FCI-as-cast-14 after 250,000 cycles ($R = 0.4$) a)–d): crack initiation from degenerated graphites, e)–g): spheroidal graphite debonding and internal cracking, and h)–i): crack initiation in the ferrite matrix and from the shrinkage porosities.

Similar to the case for $R = 0.1$, three distinct damage mechanisms were observed from the crack initiation study. Firstly, cracks were initiated from degenerated graphite particles (compacted, irregular and flake). Cracks were initiated either by decohesion followed by the ferrite matrix cracking (Figure 4.20 a)), or by internal cracking of graphites that further propagated into the ferrite (Figure 4.20 b) and d)). Combination of these mechanisms (Figure 4.20 c)) were also found in some of the graphite nodules. Secondly, decohesion of spheroidal graphite nodules were observed (Figure 4.20 e), f) and g)), some of them showed internal cracking in the circumferential direction (Figure 4.20 f) and g)). Lastly, crack initiated from shrinkage porosities when present in the microstructure (Figure 4.20 h) and i)). Some cracks initiated within the ferrite matrix (Figure 4.20 d) and h)), this phenomenon could be influenced by submerged graphites or defects.

Quantitative study of graphite damage

Graphite nodule damage was among the major damage initiation mechanisms observed in FCI tests performed. To further study and quantify graphite damage mechanisms in SGI, the specimen FCI-as-cast-15 was further tested to 300,000 cycles. At this point, OM observation showed some sorts of damage on most of the graphite nodules, and the specimen was used for the quantitative study of the damage mechanisms. Six regions (three on each face), within the gauge section were considered. For each graphite nodule, the RSF values were evaluated as explained in an earlier chapter (cf. Section 2.1.2). The graphite nodules were studied, and damage mechanisms were classified into debonding, internal cracking and combined mechanisms for different graphite forms. Table 4.3 summarizes the percentage of damage mechanisms for different graphite forms. Graphite damage was observed to depend on RSF. For spheroidal graphites ($RSF = 0.8-1$), ferrite-nodule debonding was the most common damage mechanism, accounting for a total of 59 % of the graphite nodules studied. For compacted graphite ($RSF <$

Table 4.3: Quantitative analysis of damage mechanisms for different graphite forms in specimen FCI-as-cast-15.

Graphite Form	Roundness shape factor (RSF)	Graphite count	Damage (%)		
			Decohesion	Internal cracking	Combined damage
Spheroidal (ISO form VI)	0.81 - 1	186	58.93	11.92	29.16
Intermediate (ISO form V & IV)	0.46 - 0.8	220	41.83	22.74	35.43
Compacted (ISO form III)	0.16 - 0.45	74	17.33	36.58	46.08

0.45), combined damage with internal cracking of graphite dominated by 46 % and internal cracking dominated by 36.5 % of the total compacted graphite nodules studied. For the intermediate graphite shape, either debonding or combine damage was reported for most of the observed graphite nodules.

4.3.2 Fatigue crack propagation test

Fatigue crack propagation tests were conducted on miniature CT specimens to study crack propagation mechanisms and Fatigue Crack Growth Rate (FCGR) in EN-GJS-500-14 SGI. Specimens and test conditions used in the experiments are summarized in Table 4.4. Series of FCP tests were conducted on CT specimens fabricated from as-cast and thermally cycled 500-14 SGI. At each material condition, tests were conducted to investigate short crack propagation, long crack propagation and FCGR. For as-cast material, ten specimens from FCP-as-cast-1 to FCP-as-cast-10 were used, out of which five specimens were tested at load ratio $R = 0.4$, another three specimens were tested at $R = 0.1$ and K -decreasing tests were conducted in an attempt to estimate fatigue crack growth threshold (K_{th}) for the investigated ferritic SGI. Specimens from thermal cycled SGI blocks after 16 and 45 cycles were used to study the effect of the thermal cycling process on the fatigue crack growth rate and its mechanism. The study of microstructure on thermal cycled SGI showed signs of matrix-graphite decohesion (cf. Section 3.3.1). This change in interface state and other unforeseen material changes might influence crack growth rate in SGI microstructure. The FCGR results were compared to the as-cast SGI crack growth rate. In addition to the separately performed FCGR tests, the specimens used for crack propagation mechanism studies (e.g. FCP-as-cast-2, FCR-as-cast-6) were further loaded to collect additional data point for the FCGR plot. FCP test results were studied into three sections and are explained in the paragraphs below.

Stable crack propagation mechanisms

Fatigue crack propagation mechanisms are illustrated in Figures 4.21 to 4.25. Spheroidal graphite nodules being the most common graphite form, matrix-graphite decohesion was the most frequent crack growth mechanism observed in the investigated ferritic SGI. The fatigue crack propagated by partial decohesion of the spheroidal graphite nodules and connecting those graphite nodules along the crack

Table 4.4: Summary of FCP specimens and tests.

Specimen	Thickness $B(\text{mm})$	Load ratio (R)	ΔK_{Start} ($\text{MPa}\sqrt{m}$)	Cycles N	Crack length $a(\text{mm})$	Comments
FCP-as-cast-1	0.99	0.4	13	67357	-	Preliminary test
FCP-as-cast-2	0.99	0.4	13	41000	0.53	Short crack study
FCP-as-cast-3	0.93	0.4	13	60000	1.65	Long crack study
FCP-as-cast-4	0.98	0.4	13	42974	-	FCGR test
FCP-as-cast-5	0.98	0.4	13	57000	1.05	Long crack study
FCP-as-cast-6	0.92	0.1	13	61000	0.75	Short crack study
FCP-as-cast-7	0.97	0.1	13	97050	1.67	Long crack study
FCP-as-cast-8	0.97	0.1	13	110528	-	FCGR
FCP-as-cast-9	0.99	0.4	13	155000	1.76	K-decreasing test
FCP-as-cast-10	0.97	0.4	12	470000	1.91	K-decreasing test
FCP-TC16-1	0.92	0.4	13	40000	0.76	Short crack study
FCP-TC16-2	1.03	0.4	13	51000	1.84	Long crack study
FCP-TC16-3	0.98	0.1	13	114500	2.46	FCGR test
FCP-TC16-4	0.97	0.4	13	64576	-	FCGR test
FCP-TC45-1	0.95	0.4	13	52000	0.83	Crack study
FCP-TC45-2	0.97	0.1	13	98386	-	FCGR test
FCP-TC45-3	0.96	0.4	13	41000	1.54	Long crack and FCGR test

path. Figures 4.21 a) and b) show the example of crack propagation by partial decohesion of the spheroidal graphite nodules at the load ratios of 0.4 and 0.1 respectively. It was observed that such partial decohesion of the spheroidal graphite nodules while a crack approaching them provided crack tip blunting effect to the crack. Because of the crack tip blunting effect, the stress at the crack tip drastically reduced, stopping further propagation of the crack for a short interval. In some occasions, when multiple cracks were initiated at the CT sample notch, some of them were completely arrested at the spheroidal graphite nodules, suggesting graphite nodules as possible crack arrester in SGI microstructure. The partial decohesion mechanism of the crack propagation leaves embedded graphite nodules on one crack surface and empty graphite voids on the other crack surface (Figure 4.21 a)). Not only the graphite nodules along the crack showed decohesion, but also those graphite nodules near the crack path showed decohesion from the ferrite matrix. Such nodule decohesion was due to the strong crack tip stress field allowing the weak matrix-nodule interface to debond.

Although most of the graphite nodules were of spheroidal shape, some nodules

Figure 4.21: Fatigue crack propagation by partial decohesion of the spheroidal graphite nodules a) $R = 0.4$ (FCP-as-cast-5) b) $R = 0.1$ (FCP-as-cast-8).

Figure 4.22: Fatigue crack propagation through degenerated graphite particles a) $R = 0.4$ (FCP-as-cast-5) b) $R = 0.1$ (FCP-as-cast-8).

degenerated to form compacted and irregular nodules in the investigated SGI material. Compacted and irregular graphite particles present in the microstructure behaved differently than spheroidal graphite nodules. As shown in Figure 4.22 a) and b), compacted graphite particles present in the crack path were damaged, and the crack passed through narrow sections of the compacted graphite. Due to the irregular and elongated shapes of non-nodular graphite particles, they could not provide effective crack tip blunting effect as spheroidal graphite nodules, resulting in the crack passing through the graphite particles. Compacted graphite particles were reported to have irregular surface allowing stronger interface bond than spheroidal graphite nodules, which might be one of the reasons for internal fracture of compacted and irregular graphite particles. Additionally, when the major axis of such graphite nodules was perpendicular to the crack growth direction (Figure 4.22 b)), the crack would have to change its direction by a larger angle in

order to decohere from the matrix. So, the crack propagated by damage of the graphite particles instead of matrix-graphite decohesion. Such damage mechanism will result into fractured graphite particles in both crack surfaces.

Figure 4.23: Fatigue crack branching observed around graphite nodules a) $R = 0.4$ (FCP-as-cast-2) b) $R = 0.1$ (FCP-as-cast-8).

Cracks branching were observed around graphite nodules. SEM images in Figure 4.23 a) and b) demonstrate crack branching mechanism when multiple graphite nodules were present near the crack. Crack branching could occur either from the main crack or by initiation of secondary crack in front of the crack tip area. When multiple graphite nodules were present in front of the crack tip, the crack would propagate to one of the nearest graphite. Instead of the crack propagating further, a kink crack could appear on the main crack and grow toward another graphite nodule to form a crack branch. Secondary cracks might form in the microstructure prior to stable crack propagation or in front of the crack tip due to higher stress. Such secondary cracks growth towards the main crack could result into a crack branch as well. However, it was difficult to understand which crack branching mechanism dominated in the high silicon SFF SGI grade.

To explain overall crack growth mechanisms in CT specimen, Figures 4.24 and 4.25 presents SEM images of both surfaces of specimens FCP-as-cast-6 (short crack) and FCP-as-cast-7 (long crack) were presented. In the stable propagation region, crack growth was highly influenced by the microstructure features in front of the crack tip. The crack mostly propagated by connecting graphite nodules, resulting in zig-zag crack path (Figure 4.24 A) and B)). The crack tip stress field and the plastic zone size in this region was smaller, so only a few graphite nodules near the crack tip were influenced by the crack tip stress intensification. The above-mentioned crack propagation mechanisms; crack propagation by par-

Figure 4.24: SEM images of short fatigue crack growth in specimen FCP-as-cast-6 ($R = 0.1$, $\Delta K_{Start} = 13 \text{ MPa}\sqrt{m}$, $a = 0.75 \text{ mm}$, $\Delta K = 27 \text{ MPa}\sqrt{m}$).

tial decohesion of spheroidal graphite nodules (Figure 4.24 b), (e) and (f)), crack passing through degenerated graphite nodules (Figure 4.24 c)), and crack branching around graphite nodules (Figure 4.24 d) and (e)) could be observed along the stable crack propagation. Different from this, in some occasions the crack passed through the matrix between two nearby graphite nodules without connecting them to the main crack. Such crack propagation was believed to be influenced by the submerged graphite nodules. As the crack size increases, the crack growth is

Figure 4.25: SEM images of long fatigue crack growth in specimen FCP-as-cast-7 ($R = 0.1$, $\Delta K_{Start} = 13 \text{ MPa}\sqrt{m}$, $a = 1.67 \text{ mm}$, $\Delta K = 35 \text{ MPa}\sqrt{m}$).

mainly influenced by the loading direction, and the crack propagated perpendicular to the loading direction as shown in Figure 4.25. In microstructure level, the crack propagation is still influenced by the graphite nodules. Some crack branches showed larger opening towards the main crack (Figure 4.25 b)). In Figure 4.25 c),

the crack passing through two graphite nodules was captured. SEM observation of the crack tip revealed the presence of submerged graphite nodule just below the crack face, suggesting crack propagation is also influenced by the invisible submerged graphite nodules. The stress field and the plastic zone around the crack tip grows with increasing crack size. When the crack tip approaches the unstable region, the larger crack tip stress region could be witnessed by larger numbers of graphite decohesion and multiple microcracks initiation in front of the crack tip as shown in Figure 4.25 f). In this region, the crack propagation was by coalescence of nucleated voids and secondary cracks, resulting in rapid fracture of the remaining section.

Fracture study of the specimen

The completely fractured specimens were studied to understand the role of graphite nodules in different regions of fatigue failure. Figure 4.26 presents a complete SEM image of the fracture specimen FCP-as-cast-1 along with the images of graphite nodules in and near the crack path. Two distinct crack regions, stable crack propagation region and unstable crack propagation region, were observed. The stable crack path was characterized by a steady crack surface as compared to the unstable crack propagation region characterized by a more random crack path. Figure 4.26 a) shows partial decohesion of graphite nodule at the crack initiation site. The presence of a graphite nodule at the crack initiation site suggests that graphite nodules when present at the stress concentration area could assist in crack initiation. The compacted graphite nodules when present in the crack path were damaged by fracture, retaining half of the fractured graphite on the fractured specimen (Figure 4.26 b) and d)). Secondary cracks and crack branching explained in section 4.3.2 is evident in Figure 4.26 c) as well. Figure 4.26 e)–h) shows graphite particles at and around the crack path in the unstable region. Sign of graphite nodule decohesion with graphite void growth due to large plastic deformation was clearly seen. These voids could coalesce by fracture of the ferrite matrix to form a microcrack. In the beginning, the graphite nodules acted like defects that can assist during crack initiation. Then in the stable crack propagation region, they led to a crack tip blunting effect by partial decohesion that stopped crack growth for a while. And finally in the unstable region, they acted like a void in the matrix that gradually grew in size with continuous plastic deformation of the ferrite matrix.

Figure 4.26: SEM studies of graphite particles along the crack path of completely fractured FCP-as-cast-1 ($R = 0.1$, $\Delta K_{Start} = 13 \text{ MPa}\sqrt{m}$).

Figure 4.27: Fracture surface and magnified SEM images of fracture surface of FCP-as-cast-8 ($R = 0.1$, $\Delta K_{start} = 13 \text{ MPa}\sqrt{m}$).

Figure 4.27 shows the fracture surface of the whole CT specimen with an enlarged view of fracture surfaces in different zones. The fracture surface was characterized by graphite nodules and empty graphite voids. Fracture surface in the stable region exposed partially disintegrated graphite nodules with a narrow decohesion gap (Figure 4.27 A)-a) and b)). The size of the empty graphite pockets was similar to the graphite nodule size, which suggested slow decohesion of graphite nodules with less plastic deformation of the surrounding ferrite matrix. Although the crack propagation was stable, fatigue striations commonly reported for fatigue crack growth was not clearly visualized on the ferrite matrix. The ferrite matrix in the fracture surface was characterized by a combination of progressive damage region (random striation like lines) and smooth fracture region. Some secondary cracks were also detected in the ferrite matrix between graphite nodules (Figure 4.27 a)), indicating internal crack branching and the complex crack growth in SGI. The decohesion gap gradually increased at the transition region, where the fracture surface was partly similar to the stable region and partly similar to the unstable region (Figure 4.27 B-c) and d)). Fracture surface at this region showed some progressive damage regions and slight growth of the graphite voids showing clear decohesion of most of the graphite nodules. The fracture surface in the unstable region was characterized by the complete disintegration of graphite nodules due to large void growth (Figure 4.27 C)-e) and f)). The size of the graphite pockets was larger than the graphite nodule size illustrating large plastic flow of the ferrite matrix. Due to higher plastic deformation, reduction of thickness and elongation of the specimen could be clearly seen in the unstable region. The unstable fracture surfaces were similar to the tensile fracture surface reported in tensile fracture surface (cf. Section 4.2.2).

Fatigue crack growth rate

The Fatigue Crack Growth Rate (FCGR) test provides useful information to estimate crack growth rate at different Stress Intensity Factor (SIF) values which is a function of applied load, crack size and specimen geometry. FCGR tests were conducted at two load ratios of $R = 0.4$ and $R = 0.1$, and results are presented in Figure 4.28. With the starting SIF of $13\text{MPa}\sqrt{m}$, fatigue pre-crack length of around 0.3 mm was made before collecting FCGR data. The fatigue crack growth data were collected at the interval of 5000 cycles at the beginning and reduced to 2000 cycles, 1000 cycles and 500 cycles as the crack advances to a larger size. The FCGR gradually increased with increasing SIF at constant force test. Comparing

Figure 4.28: Fatigue crack growth rate for EN-GJS-500-14 with Paris' law fit.

the test results at different load ratios ($R = 0.4$ and 0.1), higher FCGR is observed at $R = 0.4$ which is obvious due to higher mean tensile load at higher load ratio. It is commonly reported in many FCGR tests that FCGR curves shift upper left (indicating higher FCGR) with increasing load ratio, also termed as mean stress effect in fatigue analysis. It is a common practice to use Fatigue Crack Growth (FCG) model to fit experimental FCGR results. Here Paris' law fit valid for stage II (stable region) is used. Such FCG model helps to understand data trend and also helps in easy comparison of different test results. Paris' fit equations along with the respective R-square values are presented in Figure 4.28. R-square values of 0.92 and 0.72 for FCGR data at $R = 0.1$ and 0.4 respectively showed fairly good fit to the experimental data. The FCGR were plotted for SIF values of 14–24 MPa√m. It was difficult to continue FCGR tests at higher SIF due to the miniature specimen design, leading to rapid unstable fracture. So, it was not possible to estimate critical SIF from the FCGR tests. The FCGR data and the FCG model like Paris' fit equation could be used to define the crack evolution criteria in a fatigue crack growth simulation. One such implementation is the use of Paris' law to define damage evolution in low cycle fatigue analysis using direct cyclic analysis in ABAQUS/Standard.

4.3.3 Fracture toughness

In a system with cracks, the stress field at the crack tip is correlated to the applied force and crack size by the stress intensity factor (K). The crack will start to propagate when the SIF value corresponding to the load and crack is larger than threshold SIF (K_{th}). The crack propagation will be stable until the SIF value reaches a critical value of SIF (K_{Ic}). This critical value of SIF at which the crack will grow rapidly to final fracture is known as fracture toughness. Fracture toughness is a material property which can be tested by different methods stated in ASTM standard E1820 [168]. One widely used method is to use a CT specimen that is fatigue pre-cracked to estimate mode I plane strain fracture toughness designated as K_{Ic} . The evaluated fracture toughness could guide on determining the stable crack propagation region and is also an important material property in fracture simulation.

In this work, the miniature CT specimen design used for the FCP tests was used to evaluate mode I fracture toughness of as-cast EN-GJS-500-14 SGI. Although the standard method stated in ASTM E1820 was referred, the test could not be conducted completely within the standard due to the miniature specimen design. A total of five specimens were tested, out of which the first specimen was tested without prior pre-crack and failed at the loading pin hole. For the remaining four specimens, fatigue pre-crack was performed at $R = 0.4$ and $\Delta K_{Start} = 13 \text{ MPa}\sqrt{m}$. One of the specimens failed during pre-cracking, and the remaining three specimens showed pre-cracks of length 2.9 mm, 3.5 mm and 4.2 mm. These specimens were tested in force controlled mode, whereby continuously monitoring of the force and the load point displacement was done. The maximum force attained before rapid fracture of the CT specimen was used in Eq. 4.1 to evaluate the corresponding fracture toughness. The average fracture toughness from the tests was $42.37 \text{ MPa}\sqrt{m}$ with the standard deviation of $4.35 \text{ MPa}\sqrt{m}$. The evaluated average fracture toughness value correlated well with the fracture toughness value of $45 \text{ MPa}\sqrt{m}$ obtained in similar fracture toughness test conducted by project collaborator at Jonkoping University.

4.3.4 Stress-life (S-N) curve for EN-GJS-500-14

The stress-based fatigue life estimation approach is the earliest technique used for fatigue life prediction, but still the frequently used one as it is relatively easy

Figure 4.29: Stress-life (S-N) curve obtained from 14 fatigue tests according to JSME S 002 standard.

and requires only a few parameters. In this approach, fatigue life (N cycles) is related to the applied stress (mostly stress amplitude, but for tension-tension fatigue maximum stress) as;

$$S_{max} = aN^b \quad (4.6)$$

where, a is the fatigue strength coefficient and b is the fatigue strength exponent.

Fourteen specimens were tested according to JSME S 002 standard to obtain a complete S-N curve for high silicon EN-GJS-500-14 SGI. Eight specimens were tested at four finite life stress ranges and six specimens tested to estimate endurance limit as explained in section 4.1.4. All the fatigue tests were performed at a load ratio of 0.1 and maximum stress at steps of 5 % of UTS, the highest being 90 % of UTS and the lowest being 75 % of UTS for the finite range. The overall S-N curve obtained is shown in Figure 4.29. For the finite life part of the curve, a straight line fit was determined for the average life at each stress level. The S-N fit equation is also presented in the plot. Comparison of the fit line and individual test data points showed some scattering even at the same stress range which is commonly reported in this type of fatigue test. This S-N curve serves as a basic tool to estimate fatigue life at a given applied stress; however, to acquire high ac-

curacy S-N fit, a larger number of fatigue test data is desirable. For the endurance limit, the staircase method was adopted, and out of six tested specimens, three failed and three survived. Using lowest stress value (S_o) = 65 % UTS, stress step (d) = 5 % of UTS and selecting survival as less frequent $A_{DM} = 1$, $\sum \eta_{DM,i} = 3$ and $B_{DM} = 1$ in Eqs. 4.2 and 4.5, the average endurance limit was evaluated to be 353.44 MPa with a standard deviation of 13.54 MPa. The estimated endurance limit is also plotted in the S-N curve. Based on the obtained values, it suggests that at the load ratio of 0.1, if the applied maximum stress is kept below 354 MPa, the component will have an infinite life (higher than 2 million cycles in this case). In this way, a complete S-N curve is plotted with a minimum number of specimen tests. The S-N curve provides quick and easy method to estimate life of the component for a given load. Stress-life approaches are most useful at High Cycle Fatigue (HCF), where the applied stresses are elastic, and no plastic strain occurs anywhere other than at the tips of the fatigue crack. However, at Low Cycle Fatigue (LCF), scatter of fatigue data makes this approach increasingly less reliable and inaccurate.

4.3.5 Effect of thermal cycling on FCGR

The details of thermal cycling and its effect on the SGI material and microstructure have been discussed in the previous chapter (cf. Section 3.3). The initial thermal cycling showed a change in the thermal expansion behavior and a sign of matrix-nodule decohesion was also noticed after 45 thermal cycles. However, no significant changes in the phase material properties were observed in the nanoindentation study. The observed change in the matrix-nodule interface, thermal expansion and other unforeseen changes could have an effect on the crack propagation behavior and growth resistance. So, FCP tests were also performed on the CT specimens machined from the thermal cycled SGI blocks. Study of the fatigue crack propagation did not show significant difference as the crack growth by decohesion of spheroidal graphite nodules with crack branching similar to that reported for the as-cast material in the previous section (cf. Section 4.3.2). The crack propagation results were mainly focused on the qualitative study of the OM and SEM images, making it difficult to compare as-cast material and thermal cycled material. Thus, FCGR tests were performed on the thermal cycled material to quantify the crack growth resistance and compare it to the FCGR of as-cast material.

Figure 4.30: Comparison of fatigue crack growth rates of thermal cycled SGI with as cast material ($R=0.1$).

Figure 4.30 presents the FCGR plot for as-cast, 16 thermal cycled (TC-16) and 45 thermal cycled (TC-45) material at $R = 0.1$. The individual data points overlap each other, and it was difficult to compare the results. So, Paris' fits were also plotted in the figure to show the average trend of experimental data. R-square values higher than 0.8 for all the cases indicate fairly good fit. Comparing the Paris' fit lines for as-cast and thermal cycled cases, a slight decrease in crack growth rate could be noticed for thermal cycled samples at lower SIF value. At higher SIF, the FCGR curve is almost same indicating no change in the crack growth rate in the material. The decrease in the crack growth rate at lower SIF is more for TC-45 sample than for TC-16 sample. Such reduced crack growth rate could be mainly due to two reasons. Firstly, the increasing microcracks in the thermal cycled samples and their coalescence ahead of the crack tip could have reduced the crack growth rate. And secondly, the ferrite grain refinement by diffusion of residual carbides into graphite nodules during the thermal cycling process could have improved the crack growth behavior. Similar FCGR result comparison at $R = 0.4$ did not follow the same change in the crack growth rate as at $R = 0.1$; instead, the Paris' fits were almost same for all the cases. So, the reduction in crack growth rate could not be well verified. However, from all the

crack propagation study and FCGR comparison, it could be concluded that the initial thermal cycling process did not influence crack propagation mechanisms and did not reduce the material resistance to crack growth, even if some changes in the thermal expansion behavior and microstructure were observed.

4.4 Damage Criteria

In the previous section, failure mechanisms in SGI microstructure have been thoroughly investigated. The observed damage mechanisms could work as a basis to define mechanisms based damage criteria, which is an essential part of the material definition in any kind of failure simulation. In any failure predictions and simulations, it is crucial to define the damage process by considering experimentally observed damage mechanisms to account for the undergoing mechanisms wherever possible. Failure mechanism definition usually consists of three parts; a) definition of the effective (undamaged) material response, b) damage initiation criterion, and c) damage evolution criterion. In this work, damage criteria for the investigated SGI material are defined based on the previously explained experimental observations. Mechanisms based tensile and fatigue damage criteria for the high silicon SSF SGI are discussed in following sections.

4.4.1 Tensile damage criteria

In tensile damage studies, plastic deformation of the ferrite matrix and inhomogeneous stress due to graphite nodules played an important role in damage initiation and fracture. The shape of graphite nodules was decisive on the nature of their role in the damage initiation and growth process. The matrix-nodules interface was a common damage initiation point in the SGI microstructure. So, any attempt to model failure in SGI microstructure should include graphite nodules and their interface with the ferrite matrix. The ferrite matrix properties should be able to represent the elastic-plastic behavior as plastic deformation of the matrix is the driving mechanism to the final fracture. Thus, in FE simulation of tensile damage in SGI material, three damage processes should be defined; a) the matrix-nodules interface decohesion criteria, b) ferrite crack initiation criteria, and c) crack growth criteria.

From the experimental damage studies, the matrix-nodules decohesion was ob-

served to initiate slightly before the yield stress, and it was the main mechanism at the bulk stress range of around 20 MPa higher than the yield stress [166]. But, the stress and strain around graphite nodules were higher than the bulk stress and strain due to the stress concentration around graphite nodules. It was difficult to exactly measure the matrix-nodules interface properties, including decohesion initiation and growth, in the current experiment. From the in-situ tensile test with DIC by the project collaborator, strain around graphite nodules was estimated when the interface decohesion and microcracks initiation were noticed. However, the ferrite-graphite interface properties could not be estimate. So, the interface properties were estimated by FE simulation (which will be discussed in next chapter). It was suggested that some iteration on the interface properties and damage parameters should be performed to match the interface damage with the experimentally observed damage. So, the interface properties with decohesion initiation stress range around 410 MPa (yield stress) would be adequate to define the interface decohesion.

It was observed from the DIC system that the local strain around graphite nodules at the point of crack initiation was approximately 2 % with an overall strain of 0.21 % on the microstructure [33]. From the microstructure observation, the early crack initiations were mostly at the maximum stress region. So, a simple Maximum Principal Strain (MAXPE) could be used to define crack initiation in the ferrite matrix as below.

$$f = \frac{\langle \varepsilon_{max} \rangle}{\varepsilon_{max}^0} \geq 1 \quad (4.7)$$

Here, f is the damage parameter, ε_{max}^0 represents the maximum allowable principal strain (equal to 2 %), and the Macaulay brackets ($\langle . \rangle$) signify that a purely compressive strain does not initiate damage. Damage is assumed to initiate when the ratio reaches a value of one.

Once the crack has been initiated in the model, growth of the crack could be defined based on the energy that is dissipated as a result of the damage process in an area near the crack tip. The maximum amount of dissipation energy at the onset of crack growth is known as fracture energy (G_c). Fracture energy is a material property and could be experimentally evaluated using fracture and impact test. In the previous section 4.3.3, the fracture toughness (K_{Ic}) value has been evaluated from fracture toughness test. The fracture energy per unit area

could be evaluated from fracture toughness using Eq. 4.8 below.

$$G_c = \frac{K_{Ic}^2}{E'} \quad (4.8)$$

where, $E' = E$ for plane stress and $E' = E/(1 - \nu^2)$ for plane strain.

However, it should be noted that the fracture toughness evaluated is for the bulk material, and in the investigated SSF SGI, the fracture process is mostly related to the ferrite matrix fracture. So, it could be assumed that the fracture toughness is valid for the ferrite crack growth. Using the fracture toughness of $42.37 \text{ MPa}\sqrt{m}$ and plane stress condition ($E' = E = 170 \text{ GPa}$ and $\nu = 0.3$ for EN-GJS-500-14), the fracture energy is evaluated to be 10560 J/m^2 . Thus, it could be defined in the simulation model that the crack will start to grow when the strain energy dissipation at the crack tip exceed 10560 J/m^2 .

4.4.2 Fatigue damage criteria

In fatigue damage, fatigue crack initiation and propagation were due to progressive damage in the repeated cyclic loading. So, the fatigue damage criteria should concisely state fatigue crack initiation and propagation laws. The plastic deformation of the ferrite matrix was not large as compared to tensile damage; however, at the crack tip a small plastic zone is developed, and the final fracture is also assisted by large plastic deformation. So, the ferrite material properties must define the elastic-plastic behavior. Graphite nodules shape and the matrix-nodules interface play an important role in fatigue crack initiation and propagation. Graphite nodules shape showed a dominant role in the crack initiation stage, and the interface decohesion provided crucial crack tip blunting effect during crack propagation. So, it is desirable to have accurate shape representation of the graphite nodules in the microstructure and bonded matrix-nodules interface at the beginning of the fatigue load simulation.

ABAQUS provides an analysis method to simulate low cycle fatigue. Here, the crack initiation and propagation criteria were evaluated so that they could be directly implemented in the software. In ABAQUS, the number of fatigue load cycles to initiated damage is defined by Eq. 4.9.

$$N_0 = c_1 \Delta w^{c_2} \quad (4.9)$$

where, c_1, c_2 are material constants, Δw is the inelastic hysteresis energy per cycle.

The materials constants in Eq. 4.9 could be estimated from the crack initiation test results. Figure 4.31 shows the plot of hysteresis curves for fatigue crack initiation at the load ratios of 0.1 and 0.4. From the crack initiation test, the crack initiation lives at $R = 0.1$ and 0.4 were reported around 200,000 cycles and 250,000 cycles respectively. For each load ratio, five hysteresis curves were plotted in Figure 4.31, which were used to estimate the average inelastic hysteresis energy per cycle. The area between loading and unloading curves of the load cycle was evaluated using the trapezoidal rule in *Matlab*. The average estimated inelastic energies per cycle were 0.876 mJ and 0.248 mJ respectively at $R = 0.1$ and 0.4. Using these two sets of inelastic energy and initiation life in Eq. 4.9, the material parameters c_1 and c_2 were estimated to be 1.95×10^5 and -0.1768 respectively.

Figure 4.31: Cyclic force vs. deformation curve in fatigue crack initiation test.

For the fatigue crack growth, ABAQUS uses Paris' law in the form of relative fracture energy release rate (ΔG) as in Eq. 4.10.

$$\frac{da}{dN} = c_3 \Delta G^{c_4} \quad (4.10)$$

where, c_3 and c_4 are material constants.

Using Eq. 4.8, the FCGR plot in Figure 4.28 could be converted in terms of the relative fracture energy release rate and by fitting the data; the materials constants

c_3, c_4 were evaluated to be 1.46×10^{-17} and 2.9108 respectively at the load ratio of 0.1.

4.5 Summary

Tensile and fatigue damage mechanisms were studied on miniature tensile and CT specimens respectively. Damage mechanisms were studied based on OM and SEM inspection of the specimen at different intervals during the loading and fracture surface examination. In the previous chapter, it was determined that the SGI microstructure could be characterized by graphite nodules and the matrix structure with their phase properties, so the crack initiation and propagation were firmly investigated with respect to graphite nodules and ferrite matrix damage. Overall, the presence of graphite nodules in the ferrite matrix, causing inhomogeneous stress and strain distribution in the microstructure, played a major role in the crack initiation and propagation in both tensile and fatigue loading. Fatigue loading in particular illustrated the role of graphite nodules shape in the crack initiation and propagation mechanisms.

In the tensile test, the matrix-nodules interface decohesion and plastic deformation of the ferrite matrix were the dominant mechanisms. Less influenced by nodule shape, graphite nodules showed decohesion from the ferrite matrix at the overall stress equivalent to the yield stress. The matrix voids due to graphite decohesion started to grow with plastic deformation of the ferrite matrix, and the elongated graphite nodules due to higher stress concentration showed microcrack initiation more frequently. The degenerated graphite particles in the microstructure showed crack initiation in the ferrite matrix without much of the interface decohesion. Some cracks were also observed in the ferrite matrix as the matrix started to deform plastically. At the point of fracture, one of the larger cracks grew rapidly by coalescence of the graphite voids and initiated microcracks to fracture the specimen. All the graphite nodules showed large decohesion gap with many slip bands around the enlarged graphite voids. The initiated microcrack from the degenerated graphite particles and in the ferrite matrix did not show much growth in the crack size; instead, they showed large crack opening. Study of the fracture surface showed undamaged graphite nodules with the ferrite forming graphite voids around the graphite particles in the ferrite fracture surface. The graphite voids were much larger than graphite nodules size. The ferrite fracture region was

characterized by ductile fracture with the presence of micro-voids.

Fatigue damage was investigated by separately performed fatigue crack initiation and propagation tests. FCI tests exhibited the substantial influence of graphite nodules shape on the early crack initiation as most of the fatigue microcracks were initiated from the compacted and irregular graphite nodules; either by internal graphite cracking or by decohesion or by a combination of decohesion and internal cracking. Spheroidal graphite nodules on the other hand did not initiate microcracks at the time of crack initiation from degenerated graphite particles, but they showed matrix-nodules interface decohesion and some of the larger spheroidal nodules showed circumferential internal cracks. A quantitative study of graphite nodules damage exhibited internal cracking and combine damage of graphite nodules with RSF less than 0.5, whereas the spheroidal nodules with RSF higher than 0.9 showed matrix-nodules interface decohesion. The fracture surface study of the FCI specimen revealed the presence of large degenerated graphite nodules, signifying the influence of such degenerated graphite nodule as a defect on the ferrite matrix. FCP tests were performed on miniature CT specimens to investigate stable fatigue crack propagation. Spheroidal graphite-matrix decohesion inducing a crack tip blunting effect was the most frequent damage mechanism during the stable crack propagation in the SGI microstructure. The compacted and irregular graphite nodules were mostly fractured when present in front of the crack tip. Crack branching was observed either by the main crack kinking towards nearby graphite nodules or by secondary cracks growth toward the main crack.

Based on the experimentally observed damage mechanisms, tensile and fatigue damage criteria were established. The evaluated damage parameters could be useful in FE fracture simulation of SGI microstructure. It was highlighted that in addition to crack initiation and propagation criteria; the interface decohesion should be modeled in SGI microstructure. For the tensile damage, initiation was defined as MAXPE with a maximum allowable strain of 2 % in the ferrite, and fracture energy based evolution of the crack when the strain energy release rate reaches the material fracture energy of 10560 J/m². From the experimental observation, it was recommended to use yield stress as the matrix-nodule decohesion initiation criteria, but for the interface specific properties, it was suggested to perform some iterations as these properties were not able to be estimated from the experimental test. For the fatigue damage, crack initiation and propagation laws were established that could be directly implemented in the commercial software ABAQUS. The initiation criteria were expressed in terms of inelastic hysteresis

energy per cycle as in Eq. 4.9, and the crack growth was expressed by Paris' law in terms of relative fracture energy release rate as in Eq. 4.10.

Separately performed tensile tests, fatigue crack initiation tests and fatigue crack propagation tests contributed insight on the roles played by graphite nodules in tensile loading and at a different stage of fatigue loading. Microstructure damage micromechanism studies provided clear perception of tensile and fatigue damage mechanisms in SGI. The observed damage mechanisms from graphite particles showed dependency on the graphite growth morphology, illustrating the critical role of the compacted and degenerated graphite particles on material damage.

Chapter 5

SGI Microstructure Modeling and X-FEM Crack Simulation

In Chapter 3, the microstructure of Spheroidal Graphite Iron (SGI) was characterized, and the effect of thermal and mechanical process on the microstructure was studied. Microstructure study at different conditions showed that graphite morphology has a significant influence on the mechanical properties of SGI, and it was concluded that the overall SGI microstructure could be characterized by graphite nodules size, shape and distribution in the matrix structure with proper phase properties. In Chapter 4, microstructure damage micromechanisms were investigated in detail for tensile and fatigue loads, which demonstrated the important role of graphite nodules on the damage mechanisms. The matrix was illustrated as major load carrying part in SGI, which fractured by a ductile failure in tensile and by progressive damage and fracture in fatigue loading. Although graphite nodules did not show load sharing tendency, the presence of graphite nodules in a spheroidal form reduced early crack initiation and also assisted in better crack growth resistance by providing the crack tip blunting effect. At the end of Chapter 4, damage criteria were also established based on the observed experimental damage mechanisms. These experimental studies of microstructure and damage mechanisms establish an essential basis to micromechanisms modeling. With the development of advance computational method and technology, Finite Element (FE) modeling of micromechanisms in material microstructure will create an efficient method to analyze fracture problems and predict component failure.

In literature, many approaches had been studied to model SGI micromechanisms. Cell model with Gurson void nucleation and growth is one of the commonly

used methods, where a Representative Volume Element (RVE) was formulated by modeling graphite particles as primary voids, or rigid spheres that were bonded and debonded, or elastic spheres that were bonded or debonded by different researchers. Another approach is to develop FE model from the real microstructure image that could exactly represent surface microstructure in the RVE model. In such FE models, ductile fracture based damage models were used in tensile load, and cumulative fatigue crack growth models were used to predict fatigue failure. Usually, in conventional FEM approximation, fracture and discontinuities modeling requires rigorous re-meshing and result projecting, which is computationally very expensive. With the development of advanced methods like Galerkin method and eXtended Finite Element Method (X-FEM), fracture and discontinuities modeling is significantly easier and accurate. In X-FEM, additional enrichment functions are defined to represent discontinuities and track the crack tip without the requirement of recursive re-meshing (cf. Section 2.4). Methods like X-FEM is getting popular in many commercial FEM software package, and ABAQUS is one of the commonly used FEM software for all purpose fracture modeling, which was also used in this work.

In this chapter, SGI microstructure modeling approach was developed starting from the homogenized RVE model to multiscale material model. After briefly stating the X-FEM implementation in ABAQUS, a three-graphite representative volume element is studied to establish experimental damage mechanisms based SGI microstructure modeling strategy. The stress-strain evolution, graphite-nodules interface properties and X-FEM crack initiation and propagation were thoroughly analyzed in the three graphite RVE model. The implemented modeling approach was further implemented to develop multiscale model, where the microstructure model was developed by image based FEM method: OOF2 to represent real SGI micrograph. The multiscale model was also analyzed for the stress-strain evolution which was validated by Digital Image Correlation (DIC) maps. The validate multiscale SGI material microstructure of the CT specimen was used to simulate X-FEM crack initiation and propagation. Finally, based on the X-FEM crack growth simulation studied in this work, the capabilities and limitations of the current formulation in ABAQUS were discussed.

5.1 Background: ABAQUS X-FEM

The basic theory of X-FEM and its implementation in ABAQUS has been reviewed in section 2.4. In ABAQUS, the X-FEM enrichment is implemented in two approaches using X-FEM based cohesive behavior approach, and X-FEM based LEFM approach (VCCT technique). Later approach being more suitable for modeling brittle fracture like composite delamination problems, X-FEM based cohesive approach was used to simulate crack initiation and propagation in SGI microstructure. The ABAQUS analysis user's guide (section 10.7.1) provides details of the X-FEM approaches to model discontinuities [17]. Matthew in his webpage [169], provided tutorials to basic X-FEM analysis for crack initiation and propagation modeling in ABAQUS. The XFEM-based cohesive approach is very similar to the cohesive element approach with traction-separation constitutive behavior. In this cohesive traction-separation model, it initially assumes linear elastic behavior, which is followed by crack initiation and evolution. The cohesive approach consists of a damage initiation criterion and a damage evolution law. A crack initiation occurs when the stress or strain reaches specified maximum value, which can be specified by one of the available Maximum Principal Stress (MAXPS) or Maximum Principal Strain (MAXPE), or Maximum Nominal Stress (MAXS) or Maximum Nominal Strain (MAXE), or Quadratic Traction-interaction (QUADS) or Quadratic Separation-interaction (QUADE) criteria. The damage evolution is based on the fracture energy, which is the area under the traction-separation law, and can be defined with linear or exponential evolution. In this modeling work, the damage parameters identified in chapter 4 from the experimental damage studies were used to define MAXPE initiation and fracture energy based evolution.

5.2 Homogenized RVE Approach

Ductile iron microstructure contains randomly distributed graphite particles of different sizes and shapes. However, many studies have been carried out in SGI micromechanisms modeling, assuming the homogeneous distribution of graphite nodules. Usually, the experimentally determined average graphite parameters are used to represent SGI microstructure. The major advantage of using uniform graphite distribution is the simplicity of the RVE model, in which detail micromechanisms can be studied. Use of single cell as the RVE benefits simplicity and efficiency; but, compromises its versatility of modeling randomness present

in the microstructure. In this work, the aim of RVE model study is mainly to study the graphite particle decohesion using surface based cohesive interface, and attempt XFEM crack initiation and propagation. The RVE model is to be loaded in uniaxial tension to study strain concentration around graphite particles and interface decohesion. The interface specific parameters for the surface based cohesive approach could not be measured directly from experiments, so it is targeted to acquire those parameters by RVE model study. After the graphite/ferrite interface is well represented, the RVE model is further used in an attempt to study XFEM capabilities in commercial ABAQUS code for crack initiation and propagation in tensile load. In current RVE model, three graphite nodules size were considered; one of average size, other two plus and minus maximum standard deviation in the average size to account for the overall range of graphite nodules observed. Modeling multiple graphite nodules also considers the interaction between the surrounding graphite particles. However, other factors like nodules shape were not incorporated in the current RVE model. Formulation of three graphite RVE model is explained systematically in the following section. Beginning from the RVE model geometry, material properties, constitutive equations used and boundary conditions are detailed. With the model implemented the RVE model was verified for the stress-strain evolution, interface properties, and finally X-FEM crack initiation and propagation was simulated.

5.2.1 Model geometry and boundary conditions

The two-dimensional plane stress RVE model and dimensions are illustrated in Figure 5.1. In the RVE model, the sizes of the graphite circles were based on the microstructural characterization result in chapter 3. With the average graphite particles diameter of $27 \pm 8 \mu\text{m}$, three graphite particles of size $20 \mu\text{m}$, $27 \mu\text{m}$ and $35 \mu\text{m}$ were represented in the RVE. The overall size of the RVE model (square) was then evaluated considering total area of three graphite particles equal to 10 % of the model size, which was identified to be a square plate of $136 \mu\text{m}$ with graphite particles as showed in Figure 5.1. This model size estimation was established from the microstructure characterization result for EN-GJS-500-14 demonstrating 9–10 % of graphite area in the 2D micrograph. Another important information to finalize RVE model was the distance between the graphite particle centers and their orientation termed as Nearest Neighbor Distance (NND). NND was roughly estimated to be around $70 \mu\text{m}$, so the graphite circles were retained $70 \mu\text{m}$ apart

from each other. The orientation of the graphite network was placed in the center of the RVE model to help minimize the effect of the edge boundary conditions. So, the final design of the RVE model consists of a three graphite networks forming an equilateral triangle of 70 μm side.

Figure 5.1: Three graphite RVE model and the model dimensions (all dimensions are in μm).

The boundary conditions prescribed in the RVE model are illustrated in Figure 5.1. Periodic Boundary Conditions (PBC) was used on the left and right vertical edges to eliminate the existence of free surface. PBC are a set of boundary conditions that forms an infinite model by simply repeating the RVE cell throughout the space. The periodic boundary conditions were defined using ***Equations** constraints in ABAQUS. The bottom edge of the RVE model was constrained to move in y-direction and translation boundary condition was defined along x-direction. Then, the top edge of the model was prescribed with a uniform displacement boundary along y-direction to apply load on the RVE model. Even if the RVE model and the applied boundary conditions represented the microstructure, the RVE model was very small compared to the real test specimen. So, relative displacement was applied on the top edge by constraining the bottom edge and the

results discussed and compared based on the nominal strain over the RVE model.

The RVE model was meshed with a course mesh of $2\text{ }\mu\text{m}$ at the four edges and a finer mesh of $0.5\text{ }\mu\text{m}$ at the interface. A 4-node bilinear plane stress quadrilateral elements with reduced integration (CPS4R) were used to discretize the model. Total of 21,006 elements on the ferrite square and 9,128 elements on three graphite circles were present on the RVE model. For the ferrite-graphite nodule interface, different states were modeled. Starting off the RVE model stress-strain evolution without graphite nodules, the RVE model was analyzed with purely elastic graphite not bonded to the graphite and the interface was modeled with surface based cohesive behavior to simulate interface decohesion. The RVE model with graphite nodules modeled at different state was compared and the stress evolution evaluated. For the crack initiation and propagation simulation, only the ferrite matrix was defined as enriched X-FEM region with damage criteria. Although graphite particles damage were usually reported in damage mechanisms studies and observed to be weaker than ferrite, the load carrying capacity of the SGI matrix was mostly associated with the ferrite matrix damage. The response of the graphite particles was mostly elastic and did not show plastic deformation. So, the graphite nodules were modeled as purely elastic behavior without defining damage criteria.

5.2.2 Material properties and constitutive equations

The three major constituents in presented RVE were the ferrite matrix, graphite particles and the ferrite-graphite contact interface. Some assumptions were made while defining the material response of these constituents. The list below states the assumptions made.

- The ferrite matrix was modeled as a main load sharing phase, which was defined as elastic-plastic power law hardening material.
- Graphite particles were represented as purely elastic phase without damage criteria. So, the internal damage of graphite particles was not considered in this simulation.
- The ferrite-graphite interface was modeled as surface-based cohesive behavior, which retains connectivity of the ferrite and graphite nodules until maximum allowable stress is reached. After complete dissociation of the interface,

the interface behaved like general contact, which shared load during compression only as in decohesed graphite particles.

- Only the ferrite phase was enriched as X-FEM crack region. So, the crack can only initiate and propagate in the ferrite phase. The enriched elements were modeled with cohesive constitutive behavior available in ABAQUS.

The material properties and constitutive framework for each phase and interface are explained below.

Graphite phase

The elastic material property was completely defined by stating Young's modulus (E) equal to 18 GPa from nanoindentation test and Poisson ratio (ν) equal to 0.2. The elastic constitutive behavior of graphite was represented by simple Hooke's law in Eq. 5.1.

$$\sigma = E_G \varepsilon \quad (5.1)$$

where , E_G is the Young's modulus of graphite particles.

Ferrite phase

In the investigated high silicon EN-GJS-500-14 SGI, the matrix was completely ferritic, which was the only phase undergoing plastic deformation and sharing the applied load in the microstructure. The elastic-plastic phase properties estimation has been discussed in section 3.1.4. In those studies it was revealed that the properties measured in nanoindentation test were local properties within a grain without consideration to grain boundaries and other defects, and the properties estimated from tensile test optimization were bulk phase properties considering all the defects. So, it was decided that the bulk phase properties estimated from tensile curve optimization were adequate to represent bulk phase in microstructure. In the approach using tensile test to optimize phase properties, Ramberg-Osgood relation was used to represent ferrite and pearlite phase, and the graphite particles were considered purely elastic. All the parameters in Ramberg-Osgood relation including Poisson's ratio were estimated for both the matrix phase in the detail study formulated as an inverse analysis problem. In that published work [21], the estimated Young's modulus of each phase were in good agreement with the Young's

modulus from the nanoindentation test, and the hardening behavior of the matrix composition well estimated the bulk hardening behavior in the tensile test result. Based on the similar framework, the elastic-plastic phase properties for the ferrite matrix in EN-GJS-500-14 (ferritic SGI) were approximated. However, power law hardening behavior (Eq. 5.2) was used for the ferrite matrix as the *Deformation Plasticity* using Ramberg-Osgood relation in ABAQUS modeled material as a non-linear elastic behavior instead of true plastic behavior. The problem was simpler for the ferritic SGI as it only has one matrix phase. Nanoindentation test was used to evaluate the Young's modulus of the ferrite matrix. For the elastic-plastic hardening behavior, it was considered that the bulk hardening behavior in tensile test was completely due to the ferrite phase as it's the only matrix phase. The graphite particles behaved elastically with lower strength as compared to the matrix phase, and also most of the graphite particles were already debonded from the matrix as the bulk material yield point approached in the tensile test. So, the hardening part above yield in tensile test was fitted into power law relation to estimate Holloman strength coefficient (K) and Holloman strain hardening exponent (n) for the ferrite matrix. For the Poisson's ratio, the value optimized for the ferrite phase in EN-GJS-500-7 SGI was used. It was considered that the Poisson's ratio will much influence the material behavior as soon as it is close and representative of the phase.

$$\sigma = \begin{cases} E_F \varepsilon, & \sigma \leq \sigma_Y \\ K \varepsilon^n, & \sigma > \sigma_Y \end{cases} \quad (5.2)$$

where, E_F is the Young's modules of the ferrite phase = 227 GPa, $\nu = 0.327$, K is the Holloman strength coefficient = 787 MPa, and n is the Hollomon strain hardening exponent = 0.148

For the ferrite matrix, cohesive behavior based X-FEM enrichment was defined, which was based on linear elastic traction-separation behavior. The available traction-separation model in ABAQUS v6.14 assumes linear elastic behavior followed by the damage initiation and evolution. As the damage initiates, the enriched elements start to show increase in cohesive damage variable following one of the evolution laws illustrated in Figure 5.2. The elastic behavior was expressed in terms of normal and shear stresses in relation to normal and shear separations

Figure 5.2: X-FEM based cohesive traction-separation behavior in ABAQUS a) linear and b) nonlinear [17].

of a cracked element. The elastic behavior was expressed by Eq 5.3 below.

$$\mathbf{t} = \begin{Bmatrix} t_n \\ t_s \end{Bmatrix} = \begin{bmatrix} K_{nn} & 0 \\ 0 & K_{ss} \end{bmatrix} \begin{Bmatrix} \delta_n \\ \delta_s \end{Bmatrix} = \mathbf{K} \boldsymbol{\delta} \quad (5.3)$$

where, $\mathbf{t}$ is the nominal traction stress vector with normal and shear stress components t_n and t_s respectively, $\mathbf{K}$ is the penalty stiffness matrix with normal and shear components K_{nn} and K_{ss} respectively, $\boldsymbol{\delta}$ is the nominal separation vector with normal and shear separation δ_n and δ_s respectively. The terms K_{nn} and K_{ss} are calculated based on the elastic properties for an enriched element.

For the damage initiation in the ferrite matrix, a Maximum Principal Strain (MAXPE) criterion was used with the experimentally determined allowable maximum principal strain of 2 % at the onset of crack initiation (cf. Section 4.4.1). The initiation criterion was expressed by the Eq. 4.6. The damage initiation and evolution criteria were defined as material properties in X-FEM based cohesive approach.

For the damage evolution, energy based linear damage evolution law was used. The damage evolution law defines the material stiffness degradation rate after initiation criterion has been reached. The damage evolution rate was determined in such a way that the area under the traction-separation curve was equal to the fracture energy specified for the ferrite material. The experimentally determined critical fracture energy was 10560 J/m². With the initiation criterion reached, a scalar damage variable (D) monotonically evolves from 0 to 1 leading to fracture of the

enriched element. Evolution of the damage variable affects the stress components following Eq. 5.4 below.

$$t_n = \begin{cases} (1 - D)\bar{t}_n, & \bar{t}_n \geq 0 \\ \bar{t}_n, & \text{otherwise (no damage to compressive stiffness)} \end{cases} \quad (5.4a)$$

$$t_s = (1 - D)\bar{t}_n \quad (5.4b)$$

where, $\bar{t}_n$ and $\bar{t}_s$ are the stress components predicted by the elastic traction-separation behavior for the current strains without damage.

Ferrite-graphite interface

The ferrite-graphite interface decohesion is one of the mechanisms to initiate crack in the ferrite matrix. It also plays a crucial role of crack tip blunting during crack propagation. Thus, the interface state changes from bonded to debonded state during crack initiation and growth. But, the interface was modeled as either debonded or tie from the initial state. In this work, a different approach was used to model the interface as surface-based cohesive behavior, which enabled to specify contact properties. The surface-based cohesive interface behavior allowed modeling of the intact interface at the beginning, which could debond to permit free movement of graphite and ferrite surfaces. The microstructural stress and strain evolution with the cohesive interface were compared with the unbounded interface model to realize the improvement in the RVE model. The constitutive framework of the surface based cohesive behavior was also similar to that of cohesive element approach explained in above section, and illustrated by similar constitutive Eq. 5.3 and 5.4. However, separation components were reinterpreted as contact separations, which were the relative displacement between the corresponding slave and master nodes along the contact normal and shear directions. Stresses were redefined as forces acting along the contact normal and shear directions divided by the current contact area. The penalty stiffness can be defined manually, or default contact enforcement method based on the elastic properties of slave and master elements could be selected. It was very difficult to evaluate the interface specific properties, so default contact enforcement was used at the beginning which was further updated by iterative simulation at varying contact properties. With the redefined stress, the damage initiation criterion was redefined based on nominal

stress as in Eq. 5.5.

$$\max \left\{ \frac{\langle t_n \rangle}{t_n^0}, \frac{t_s}{t_s^0} \right\} = 1 \quad (5.5)$$

where, t_n^0 and t_s^0 are the allowable maximum nominal stresses along the normal and shear directions. Macaulay brackets ($\langle . \rangle$) signify that a purely compressive strain does not initiate damage.

The interface decohesion starts when the criterion in Eq. 5.5 was reached. Assuming isotropic properties, equal allowable nominal stresses in normal and shear directions were used. It was very challenging to experimentally measure interface specific stresses, so rough estimation of the $t_n^0 = t_s^0$ was obtained from the experimental damage study. Decohesion of graphite particles were reported to occur at the stress range near the yield stress of the material. Thus, the yield stress value itself was implemented as the allowable maximum nominal contact stresses. It was believed that use of the surface-based cohesive behavior with approximate damage parameters predicted from damage studies would provide better approximation of the interface modeling than modeling either unbound or completely tied interface.

Unlike cohesive behavior, the surface-based cohesive behavior was defined as interaction property. However, the damage evolution criterion was defined by the similar framework as explained in the previous section and represented by Eq. 5.4. The critical fracture energy required to completely define linear damage evolution was initially guessed to 3000 J/m², which was much smaller than the ferrite fracture energy and larger than reported fracture energy for carbon in graphite phase. Further, the iterative study was performed in an attempt to understand the effect of the interface fracture energy.

5.2.3 Stress and strain evolution

The RVE models with different state of graphite particles and interface were applied with uniform displacement field on the top edge. The evolution of the stress and strain in the RVE model at increasing displacement field for different RVE configurations were presented in Figures 5.3 and 5.4 respectively. In all the simulation results, all the model parameters other than stated were kept constant for all cases. Same mesh seeding, material properties and loading were used in all cases for the basis of identical comparison.

Figure 5.3: Stress evolution in the RVE model at different displacement load (top edge) a) graphite particles modeled as void, b) elastic graphite particle unbound to the ferrite matrix, c) elastic graphite particles bounded to by surface-based cohesive behavior.

Figure 5.3 shows the evolution of stress in the three graphite RVE models with graphite particles modeled as voids, unbound elastic particles and surface-based cohesive behavior bound elastic particles. Legends for all the cases were made uniform, so that the stress maps could be compared. Uniform displacements (d) of $0.125\ \mu\text{m}$, $0.25\ \mu\text{m}$, $0.375\ \mu\text{m}$ and $0.5\ \mu\text{m}$ were applied, which corresponds to ε_{yy} equal to 0.092% , 0.184% , 0.276% and 0.368% respectively. At $d = 0.125$ (equivalent to $\varepsilon_{yy} = 0.092\%$), the stresses were in elastic range, which was almost similar for all the RVE models irrespective of graphite particles and their interface state. It reflected that in the elastic range, the SGI microstructure could be modeled as the matrix structure with graphite voids as well. The elastic stress was mostly distributed within the ferrite matrix. However, noticeable change in the evolution of stress with plastic deformation of the matrix depending on the presence of graphite particles and their interface state could be seen. In the RVE model with graphite voids, higher stress concentration was observed around larger graphite voids as the material was loaded to the plastic range and a higher stress band could be noticed connecting larger graphite voids. Periodic boundary implementation enabled influence of graphite particles at the left and right sides, forming a zig-zag high stress band connecting larger graphite particles similar to that observed in experiment. Comparing the stress distribution in RVEs with graphite voids and unbound elastic graphite particles, similar stress distributions were observed around all the graphite particles (Figure 5.3 a) and b)). Normal hard contact and friction with a coefficient of 0.1 were defined between the ferrite and graphite particles. The maximum stress band connecting larger graphite particles were observed similar to the graphite void RVE model. So, no noticeable change in the RVE model behavior was observed with unbound graphite particles, illustrating no improvement in representation of graphite nodules in the model. Further, to represent the interface, surface-based cohesive behavior was formulated with the ferrite surface as master and graphite particles as slave surfaces. With the cohesive behavior defined for the interface, no much difference in overall stress distribution was observed. slightly higher stress concentrations were observed around larger graphite particles as evident in Figure 5.3 c). With the effective cohesive surface contact, graphite particles shared some contact stresses slightly reducing the ferrite stress concentration around the graphite particles. With the surface based cohesive interface, the graphite nodules exhibited partial decohesion. All the RVE models with different graphite states represented the stress concentration around graphite nodules well and form continuous link of high stress band connecting larger graphite particles. Unbound graphite nodules

did not show any improvement; however, cohesive interface helped to improve the RVE model to capture graphite decohesion mechanism and partial load shearing with the graphite nodules.

Figure 5.4: Strain evolution in the RVE model a) graphite particles modeled as void, b) elastic graphite particle unbound to the ferrite matrix, c) elastic graphite particles bonded to by surface-based cohesive behavior.

The strain evolution at displacement (d) equal to 0.25 μm ($\varepsilon_{yy} = 0.184\%$) and 0.5 μm ($\varepsilon_{yy} = 0.368\%$) in the RVE models were illustrated in Figure 5.4. In the graphite void model, similar to the stress distribution, larger strain concentration

Figure 5.5: Strain concentration at the edge of the decohesed interface after degradation of the cohesive behavior ($\varepsilon_{yy} = 0.368$ %) (deformation scale = 1).

was observed around larger graphite particles and a clear higher strain band connecting larger graphite particles could be noticed. This strain band is most probable crack initiation and fracture region. For the RVE model with unbound elastic graphite particles also similar strain distribution was evident, justifying similarity in modeling graphite particles as voids or unbound elastic particles. However, the presence of graphite particles and normal contact behavior showed partial separation of the graphite particles. In the RVE model with the cohesive interface, the graphite particles were slightly strained at the region of the compressive, which showed similarity to the experimental observation. The strain distribution in the ferrite matrix were similar, only difference was that the bound interface slightly reduced the strain concentration around smaller graphite nodules, so the high strain band connecting larger graphite nodules became only preferable fracture region. Figure 5.5 presents zoomed view of the the strain concentration around the larger graphite nodules. For the larger graphite particle, the maximum strain point corresponds to the point of ferrite compression and its direction affected by the larger graphite nodules around. The matrix between two larger graphite nodules seemed to be highly strained due to thinner matrix in-between. Such high strain concentrations were observed on either side of the graphite nodules, forming continuous link of high strain band connecting larger graphite particles, which is

typical for most of the cast irons. The maximum separation was noticed at the top region of the graphite particle with the approximation separation gap of $0.35\ \mu\text{m}$. This partial separation of the graphite particles and the strain concentration at the region of compression was one of the main damage initiation mechanisms in tensile tests (cf. Section 4.2) and lead to crack initiation at the maximum strain point. Although the use of cohesive interface behavior is well justified, the effect of interface properties need to be studied, and suitable properties must be identified from simulation trials to better represent SGI microstructure.

5.2.4 Interface properties and decohesion

Surface based cohesive interface definition requires interface stiffness, damage initiation and evolution criteria. Damage initiation was defined as nominal stress equivalent to the yield stress of the material based on the experimental study at the point of graphite particles decohesion. Contact stiffness and evolution energy were studied here to investigate their effects on stress, strain and interface decohesion in the RVE model. For the contact stiffness, tentative estimation was referred to the stiffness used in delamination problem using cohesive zone model. Turon et al. [170] have summarized the stiffness used in many works reporting it in the range of 1×10^5 to $1 \times 10^8\ \text{N/mm}^3$. Similar stiffness range of 1×10^5 to $1 \times 10^{10}\ \text{N/mm}^3$ was used, and the resulting stress and strain evolution were studied. Figure 5.6 illustrates the stress and strain distribution in the RVE model with different contact stiffness. It can be clearly noticed that at lower stiffness (Figure 5.6 b)); there was almost no load transferred to the graphite particles resulting into stress-free graphite particles. Whereas at higher stiffness (Figure 5.6 c) and d)), there was partial load sharing to the graphite particles indicated by relatively lower stress value in the graphite particle than the ferrite matrix. Such behavior was due to the fact that lower stiffness corresponds to lower load transfer from the interface at the same interface separation before its complete decohesion. The stress and strain distribution at interface stiffness of 1×10^5 and 1×10^6 were similar to the Figure 5.6 b), which slightly changes at the stiffness above 1×10^7 demonstrating dominant higher stress and strain bands connecting larger graphite particles as shown in Figure 5.6 c) and d). It can be expected that in SGI microstructure, the graphite particles will be able to share some fraction of the load until it is decohesed from the matrix. Especially, graphite particles have good load bearing capacity in compression, which is well represented in the

Figure 5.6: Stress and strain evolution at different cohesive interface stiffness a) default contact enforcement, b) stiffness = 1×10^5 , c) stiffness = 1×10^8 , d) stiffness = 1×10^{10} .

RVE model at interface stiffness of 1×10^8 N/mm³ and higher. Also, it can be seen that at the region of larger separation, graphite particles showed no stress sharing and localized stress concentration at the region between tension and compression (referred to as shear band). So, it can be pointed out that the higher end of the cohesive interface stiffness recommended by Turon et al. for cohesive zone model works fairly well to define graphite/matrix interface using surface based cohesive approach. The stress and strain distribution at default contact stiffness (Figure 5.6 a)) matches closely with the stiffness value of 1×10^8 N/mm³ (Figure 5.6 c)). Although the stress and strain distribution in the RVE did not change much at different contact stiffness, a significant effect was observed on the interface state and damage criteria.

Figure 5.7: Overall stress-strain plot for the RVE model from FE simulation at different cohesive contact stiffness.

In Figure 5.7, average stress-strain in the y-direction is plotted for the ferrite matrix in the RVE model along with the maximum damage criteria at the ferrite/graphite interface. The stress and strain values were an average of σ_{yy} and ε_{yy} for all the ferrite elements in the RVE model. It can be noticed that the overall stress-strain values were lower than the bulk stress-strain curve in the tensile test, which was due to the fact that the σ_{yy} plot in the RVE model includes some regions in compression and inhomogeneous stress-strain. The maximum value of

the damage criteria tracks the interface decohesion initiation. The interface starts to decohere as the damage criteria (f) reaches one. The overall stress-strain was not influenced by the stiffness. However, the damage criteria were significantly influenced by the stiffness. It is because of the fact that the interface stiffness connects interface stress and separation as expressed in Eq. 5.3. At lower stiffness ($< 10^7$ N/mm³) the interface damage criteria did not reach unity, indicating intact interface in spite of contact separation due to lower contact stiffness. At the stiffness of 1×10^7 and higher, the interface damage criteria showed decohesion of the interface. From the experimental tensile damage mechanism studies (cf. section 4.2), the graphite-matrix decohesion started at the notch near yield point in the overall stress-strain curve. From the overall stress-strain and damage criteria plot in Figure 5.7, it is observed that at the interface stiffness of 1×10^8 N/mm³, the interface decohesion started at the desired point in the overall stress-strain. At the stiffness higher than 1×10^8 N/mm³, less change was noticed in the decohesion initiation. So, the interface stiffness higher than 1×10^8 N/mm³ appeared to satisfy the interface decohesion at the region near yield point in the overall stress-strain curve. However, higher interface stiffness controls interface separation to reach decohesion criteria and also contributes to higher load sharing with the graphite particles. Considering these effects, it was thought that the interface stiffness of 1×10^8 N/mm³ was suitable to model graphite-ferrite interface in SGI microstructure and was used in all the subsequent simulations.

A similar study of stress-strain evolution and effect on interface damage criteria was carried out for the interface damage evolution in terms of fracture energy. In the model, no crack initiation and evolution criteria were defined in ferrite and graphite phase to focus on the interfacial crack. Along the interface, surfaces of each element on ferrite phase and graphite were associated with damage variables that start to increase from 0 to 1 as the damage initiation criteria were achieved. Damage variables could range from 0 to 1 for the elements depending on the interface stress, so to compare the overall behavior the range of damage variables in terms of maximum value damage variables have attended was used. It was not possible to obtain any estimate of the interface fracture toughness and also no such information could be found in the literature. So, a range of fracture energy lower than that of ferrite (1000–10,000 J/m²) was used in RVE model. Results showed no influence of interface fracture energy on stress-strain and overall interface damage criteria. Although the fracture energy was changed from 1000 to 10,000 J/m², the location of complete evolution of first damage variable did not change. This

observation indicated that the fracture energy did not play a significant role in load sharing between the ferrite and graphite particles, and also did not show much influence on decohesion initiation point in the overall stress-strain plot. However, the interface fracture energy could have some influence on how damage variable for each element increases; by intuition, the evolution of damage variable will be slower for higher fracture energy and faster for lower fracture energy. Based on the experimental damage mechanism studies, it is desirable to have interface fracture energy much lower than ferrite fracture energy. So, fracture energy of 3000 J/m^2 comparable to the fracture energy for bulk graphite was used as the interface property.

5.2.5 Crack initiation and propagation

After studying stress and strain concentration around the graphite particles, and estimation of the cohesive interface properties, the ferrite region was defined as X-FEM enrichment region. Refer to section 2.4 for basic X-FEM theory, enrichment functions, discontinuity representation, numerical integration and available X-FEM formulation in ABAQUS. In this model, only crack initiation and propagation in the ferrite phase was considered as most of the load was distributed in the ferrite phase. The ferrite matrix was defined as power law hardening elastic-plastic material, and graphite particles were defined as purely elastic material. As compared to the displacement load applied in stress and strain simulations in the previous section, higher displacements up to $1.5 \mu\text{m}$ were applied to initiate crack with longer crack propagation. This crack simulation was aimed to understand X-FEM crack simulation in ABAQUS and also to examine additional adjustments required to simulate crack initiation and growth in SGI microstructure model. As compared to the stress-strain simulation, the crack simulation using X-FEM required more time with smaller time steps for convergence. X-FEM crack simulation considering purely elastic material behavior was solved with some adjustment. However, X-FEM crack simulation with elastic-plastic material behavior suffered severe convergence problem due to additional material nonlinearity. So, some changes in solver settings were required to completely converge the nonlinear simulation. Following are the list of adjustments made in an attempt to completely converge X-FEM crack simulation in ABAQUS.

- Control increment sizes were adjusted to prevent ABAQUS from approaching sudden stiffness changes. The initial, minimum and maximum increment

size was reduced. Initial = 0.001, minimum = 1×10^{-20} , max = 0.01 and maximum number of increments = 100,000.

- Modify general solution controls to allow discontinuous analysis and significant cutbacks before ABAQUS stops the analysis. $I_0 = 8$, $I_R = 10$ and $I_A = 50$.
- For local instability, use automatic stabilization of the step and monitor the damping energy (damping factor = 0.0002). Toggle on adaptive stabilization.
- Increase damage initiation tolerance to allow faster solution convergence but with slightly less accuracy of the result. Damage initiation tolerance allows to initiate damage when the calculated stress or strain was larger than the defined criterion, instead of further step cutback. (Default = 0.05) Maximum used value 0.1.
- Crack propagation process requires progressive damage that includes softening and loss of stiffness. Use viscous regularization with damage evolution to ensure that the tangent stiffness matrix remains positive.
- For contact and material related issues, use `*PRINT, CONTACT=YES` and `*PRINT PLASTICITY=YES` to get detail information on contact and material integration point with convergence problem in the message file (`*.msg`).
- Change the solution control parameters to loose the convergence criteria. However, this should be avoided if possible and should be used with cautions. Convergence criterion for the ratio of the largest residual to the average force, R_n^α (default = 0.005). Convergence criterion for the ratio of the largest solution correction to the largest incremental solution value, C_n^α (default = 0.01).

Figure 5.8 presents the crack initiation and its propagation at different overall strains. The initiation of cracks was shown along with the logarithmic strain (LE) plot. Even if the overall strain was kept less than 1.5 %, very high strain concentrations were observed around graphite particles, and the crack also initiated at one of these higher strain concentration regions. Strain concentrations were observed at the region of the ferrite matrix compression at the interface, which was very much similar to the strain distribution results explained in the previous section. The direction of strain concentrations were influenced by surrounding graphite nodules. Usually in metals $\pm 45^\circ$ shear bands are observed, but due to the presence of the graphite stress concentrations the initiation sites were not exactly at $\pm 45^\circ$. The

Figure 5.8: X-FEM crack initiation and propagation simulation in the RVE model implemented in ABAQUS a) $\varepsilon_{yy} = 0.37 \%$, b) $\varepsilon_{yy} = 0.74 \%$ and c) $\varepsilon_{yy} = 1.11 \%$.

location of the crack initiation was of interest. As prescribed by the higher strain and stress band connecting larger graphite particles, the single crack initiated at the graphite/ferrite interface and propagated in the matrix between tow larger graphite nodules. The exact crack initiation point is dictated by the strain concentration, and in the RVE model maximum strain concentration occurred at the smaller graphite nodule among the two larger graphite nodules forming high strain band. Thus, initiating crack that propagated towards the largest graphite nodule in the model (Figure 5.8 a)). It was observed that even the overall applied strain was only 0.37% , the local strain at the strain concentration was higher than 4% . In Figure 5.8 a), there were multiple regions of strain concentration on the either

sides of the larger graphite particles. However, only single crack initiated. With the increase of applied load, the initiated crack propagated and also the strain concentration on the other graphite particles increased and reached much higher than the defined crack initiation criterion, but still there was no sign of another crack initiation. This was one of the limitations of existing X-FEM in ABAQUS as it only allowed initiation of multiple cracks, if more than one elements reached initiation criterion at the same time step. In another event, once the crack was initiated in an enrichment region, another crack can only initiate after the initiated crack passed through the whole crack domain. Although multiple cracks were initiated at multiple graphite particles in the experimental crack initiation tests, current X-FEM in ABAQUS did not support multiple cracks initiation. However, possible crack initiation sites could be suggested based on the strain distribution result. The initiated single crack starts to grow with higher displacement load applied. As illustrated in Figure 5.8 b) and c), the crack growth direction deviated towards the larger graphite particle and followed the higher strain band between the two larger graphite nodules. However, the crack path did not follow the center of the strain band and did not connect to the next graphite nodule due to convergence difficulty at higher load.

In the current X-FEM formulation in ABAQUS, the crack growth direction was based on Maximum Tangential Stress (MTS) criterion. However, the crack cannot change its direction more than 90° in a single element. The clear increase in the interface gap could be noticed in the larger graphite particle initiating crack and the larger graphite particle near the crack (Figure 5.8 b) and c), however, the increased interface gaps was mostly due to the effect of graphite particle itself rather than the crack tip field. Another limiting fact in X-FEM ABAQUS was that the crack tip enrichment function was not included for the growing crack in Eq. 2.7, only the Heaviside enrichment function was used. So, the crack tip stress and strain were absent in all the plots in Figure 5.8 in spite of growing crack. It could be suggested that modeling of the crack tip field would shift the higher strain band to connect the crack tip and surrounding graphite particle, which would significantly influence the crack growth direction. The crack initiation and propagation observed in the RVE model did not have a direct comparison to the experimental result. However, the interface decohesion, crack initiation location around the larger graphite particle and propagation match fairly well with the experimental observation. And from this study, it was noticed that additional considerations to allow multiple crack initiation and growth, and crack

tip stress and strain field could improve accuracy in microstructure dependent crack initiation and propagation modeling using X-FEM ABAQUS.

In current X-FEM formulation in ABAQUS, only one crack could initiate and grow if multiple cracks initiation criteria were reached at different time. In other to allow multiple cracks initiation, the RVE model was defined into three enrichment regions (one for each graphite particles) as illustrated in Figure 5.9 a). Uniform displacement field of $1.5 \mu\text{m}$ was applied on the top surface as in the case of Figure 5.8 d), and the corresponding simulation result showed initiation of two cracks from the larger graphite particles. The first crack initiated from the larger graphite particle, similar to the crack initiated in the single enrichment problem. Just after the first crack initiation, another crack initiated in the R3 enrichment region at the interface. Both the initiated cracks were in the region between two larger graphite nodules in the model growing towards each other. These crack grew until it reaches the boundary of the XFEM enrichment region and faced convergence difficulty. The third graphite (smallest) did not show any crack initiation as the strain concentration was considerably less. The definition of one enrichment region for individual graphite particles helped to initiated one crack per graphite particles; however, there exists additional strain localization region on the other side of graphite particles where the local strain was much higher than the defined crack initiation strain.

The RVE mode was further divided into six enrichment regions (two per individual graphite particle) to account for strain concentrations on the either sides of the graphite particles. The configuration of the enrichment region and the corresponding result is shown in Figure 5.9 b). With the additional enrichment regions, multiple cracks were initiated from the strain concentrations around the larger graphite particles. In this case also, the first crack to initiate was same as that for the single enrichment cases. Additional cracks started to initiated soon after the first crack initiated, so it could be said that these cracks initiated at a short time interval in the simulation. Although multiple cracks were initiated at the enrichment regions, no clear interaction between the cracks was noticed. These cracks observed in the previous cases grew in very similar way. The crack from the largest graphite propagated in a similar way irrespective of another crack growing towards it. In the experimental crack initiation study, the growth of initiated crack was highly influenced by the graphite particles and cracks initiated from the surrounding graphite particles, which was not well represented in the current RVE simulation. But, other aspects of crack initiation on either side of the strain

a) Three X-FEM enrichment regions (R1, R2 and R3) and the corresponding crack initiation and propagation at $d = 1.5 \mu\text{m}$ ($\varepsilon_{yy} = 1.11 \%$).

b) Six X-FEM enrichment regions (R1, R2, R3, R4, R5 and R6) and the corresponding crack initiation and propagation at $d = 1 \mu\text{m}$ ($\varepsilon_{yy} = 0.74 \%$).

Figure 5.9: Definition of multiple X-FEM enrichment regions in the RVE model
a) three enrichment regions and b) six enrichment regions.

concentrations around the larger graphite particles were comparable to the experimental observations. Thus, it could be insisted that multiple enrichment regions (two per graphite particle based on the strain distribution) facilitated to model multiple cracks initiation and propagation in the SGI microstructure. However, the propagation of the initiated cracks were less influence by the cracks initiated on the other enrichment region around the surrounding graphite particles.

5.3 Microstructure Submodeling of SGI Material

In the previous section, the RVE model was studied to understand stress and strain concentration around graphite particles in SGI microstructure, and also the graphite-ferrite interface property was estimated. The RVE model study worked as a preliminary effort in an attempt for submodeling of SGI specimen. The actual components are much larger size as compared to microstructure features, so microstructure submodeling is a convenient method for modeling local microstructure in bulk component. Such approach is even more applicable to components showing a small region of stress concentration where crack usually initiated and grew. For example, in automobile engine crankshaft, stress is higher at the crankpin fillet where fatigue crack usually initiated. Such region can be developed as microstructure submodel to investigate the role of microstructure in crack initiation and its growth. Another advantage of submodeling is that the load and boundary condition of the microstructure submodel can be directly translated from component boundary conditions, which provided a valid basis to compare simulation result with experimental results. For the crack initiation and growth simulation, it usually required very fine mesh for an accurate and converging solution, which could drastically increase computation cost if such fine mesh is implemented in the whole model geometry. In that prospect, submodeling allows to locally refine the mesh at the region of crack growth. In this current work, it is attempted to use X-FEM formulation to simulate crack growth in microstructure model requiring an additional calculation for the extra degree of freedoms of the enriched nodes. Submodeling approach also helps to reduce computation time by allowing to define the submodel only as the enriched X-FEM region.

In SGI material, the crack initiation and propagation is highly depended on the microstructure of the component at the higher stress and strain region. So, any

Figure 5.10: Illustration of microstructure submodel in CT specimen.

form of failure and life prediction should consider the microstructural features and defects in the material. This explained an admirable reason why submodeling could be appropriate modeling approach to predict microstructure dependent failure in SGI components. To demonstrate microstructure dependent crack growth simulation using submodeling approach, a miniature CT specimen used for FCP test in section 4.3.2 was modeled with microstructure submodel at the notch region as illustrated in Figure 5.10. Both the global CT model and microstructure submodel were modeled with plane stress elements. For the global model, course mesh was used and modeled as bulk SGI material. For the microstructure submodel, the real undeformed micrograph at the notch of the CT specimen was used to generate FE representation mesh using image based FEM method OOF2, which is explained in detail in next section. As only submodel was defined as X-FEM enrichment region, cracks can only initiate and grow within the microstructure submodel. The model was first checked for efficient and smooth stress evolution from the global to microstructure model. The resulting strain evolution in the microstructure model was compared with the DIC result to validate the modeling approach. The model was then used to simulate crack initiation at the notch and further growth of the initiated crack was attempted using inbuilt ABAQUS capabilities.

5.3.1 SGI microstructure model using OOF2

In addition to homogeneous cell model, some researchers have used microstructure model based on real micrograph in various simulation, which were briefly reviewed in section 2.5. The reported works used image based finite element analysis method OOF2 developed by Langer et al. Similar approach was implemented using OOF2 to represent the real SGI microstructure into FE mesh.

Figure 5.11 illustrates the major steps in generation of FE representation of SGI microstructure. Although 2D-simulations of SGI microstructure do not generally capture completely the material response in the third direction, 2D-plane stress assumption was used due to the lack of 3-dimensional microstructure characterization, and also to avoid overly demanding computations. Such real micrograph based microstructure model allowed to capture the randomness of the microstructure to some extent that was hard to capture in offer used cell models. The method started with the undeformed SEM images of the CT sample at the notch region as shown in Figure 5.11 a). Even if graphite particles and the ferrite matrix were clearly distinguishable, they have similar color tone. The OOF2 software works on the principal of color code and different phases were distinguished based on the reference pixel color selected. So, it is highly desirable to use input image with very contrasting colors to different phased present in the microstructure. The SEM micrograph in Figure 5.11 a) was enhanced with contrast and brightness multiple times to obtain clear black and white microstructure image. In the black and white image, slight Gaussian blur was used to smoothen the interface without losing phase details to obtain black and white micrograph as shown in Figure 5.11 b). It should be noted that in the black and white micrograph, the graphite particles at the boundary were removed as they caused difficulty in global to submodel assembly. The graphite morphologies were well retained in the microstructure. The notch was also indicated to avoid mesh at that region. The processed image was used to generate FE discretization for each phase in the microstructure. In current ferritic SGI, two mesh files were generated for the ferrite and graphite particles. At the beginning, pixel groups were defined for each phase. These pixel groups were used to form a mesh skeleton for the whole model. From the available element library, bilinear quadratic elements were used as they were supported in existing X-FEM formulation in ABAQUS. The mesh skeleton was further refined at the graphite-ferrite interface in order to increase the accuracy of calculation near the interface. Figure 5.11 c) shows the refined mesh at the interface. The

a) SEM image of SGI microstructure at the CT notch

b) Image processed SGI micrograph

c) FE representation of SGI microstructure using OOF2

Figure 5.11: Microstructure submodel from the real SGI microstructure using OOF2 a) SGI micrograph, b) image processed micrograph, c) FE mesh generated from OOF2.

skeleton was used to generate mesh for each phase, which was combined together in ABAQUS/CAE to obtain complete microstructure model as shown in Figure 5.11 c). The cohesive interface properties estimated from the experimental studies and RVE model were implemented to define the interface. This FE representation of SGI micrograph was connected to the bulk CT sample model to establish submodel.

5.3.2 Material properties, constitutive equations and boundary conditions

In this modeling approach, there were two regions that required different bulk and phase material properties definition. For the microstructure submodel generated from the OOF2 software, ferrite and graphite properties explained in section 5.2.2 for the RVE model were used. There microstructure submodel was implemented similar to the RVE model with similar constitutive framework and material properties including the interface properties. Refer to section 5.2.2 and 5.2.4 for details of the material properties and constitutive equations. The CT sample was modeled as a bulk SGI material. The elastic-plastic material data obtained from the tensile test was used to define elastic-plastic material response of the CT model. Plastic behavior was defined as tabulated values from yield stress to ultimate tensile stress with the corresponding plastic strain and a linear hardening part with slightly positive slope to model further deformation. The modification beyond UTS was made to avoid softening part of the tensile curve in the simulation. As the main aim in the simulation was to study microstructure dependent crack initiation and propagation in SGI material, only the ferrite region in the microstructure model was defined as X-FEM enrichment region. The CT sample was not defined with any damage criterion, which significantly reduced computation time. The main are of interest was the microstructure model. The CT sample model was designed with exact dimension to the CT sample used in the experiment, So that the results could be directly compared.

The boundary conditions for the CT sample were formulated similar to the experimental constrains as illustrated in Figure 5.10. Both the top and bottom pin holes in the CT were defined as a kinematic coupling with the hole center as the reference control point. The top hole reference point was constrained in x and y direction and allowed rotation DOF about the z-axis (UR3). The load was applied to the bottom hole in the negative y-direction. The out of plane specimen thick-

ness was used from the actual specimen thickness, and the corresponding force was applied in the simulation. The global CT model and microstructure submodel was linked together by a tie constraint to deform the whole model as one part. And it was essential to verify uniform stress evolution between global and submodel, which was explained in the next section.

5.3.3 Stress and strain evolution

Before the model can be used for X-FEM crack simulation, it was accessed to ensure that the combined model was properly setup. With the boundary conditions and constraints prescribed in above section, it was necessary to verify gradual evolution of stress at the CT notch and its smooth transition from global to microstructure submodel. The concentrated force was applied to the model, and the results are presented in Figure 5.12 a) to d). The figures show stress evolution in combined model and close look at the microstructure submodel. The results were plotted for a different force that corresponded to the SIF range of 7.5 to 30 $\text{MPa}\sqrt{m}$. for the whole range of the applied load, symmetric stress profile were observed in the CT model. The stress profile at the notch matched well with the plane stress crack tip stress field, which dictated validation of the overall stress evolution. Although the overall stress was expected to be homogeneously distributed, inhomogeneous microstructure stress was expected and observed in the model. The stress inhomogeneity could be clearly evident at the lower force (Figure 5.12 a) and b)) illustrating higher stress band connecting graphite particles. Up to the load of 250 N ($K = 15 \text{ MPa}\sqrt{m}$), graphite particles showed less stress. Further increase of the applied load caused large stress concentration at the notch that reached above 500 MPa. In Figure 5.12 d), it is noticed that most part of the microstructure has uniform stress and higher stress on the graphite particles. It was due to the fact that the whole microstructure region already reached beyond the yield stress with much higher stress at the notch, but without damage criterion. One major contributing factor for higher graphite stress was the pixelated graphite-ferrite interface (see Figure 5.11 d)), the caused sharp interface. These pixelated interfaces were due to the fact that the OOF2 software used boundary between two pixel groups as the interface. The pixelated interface cannot be completely eliminated in the image based FEM analysis as the basic ingredient of all the images are the square pixels. However, increasing the number of pixels in the image and use of Gaussian blur filter helped to some extend to reduce but did not

a) Stress distribution on the standard CT at Force = 125 N ($K = 7.5 \text{ MPa}\sqrt{m}$) b) Stress distribution on the standard CT at Force = 250 N ($K = 15 \text{ MPa}\sqrt{m}$) c) Stress distribution on the standard CT at Force = 375 N ($K = 22.5 \text{ MPa}\sqrt{m}$) d) Stress distribution on the standard CT at Force = 500 N ($K = 30 \text{ MPa}\sqrt{m}$)

Figure 5.12: Stress evolution on the CT sample model and the microstructure submodel at the notch a) $K = 7.5 \text{ MPa}\sqrt{m}$, b) $K = 15 \text{ MPa}\sqrt{m}$, c) $K = 22.5 \text{ MPa}\sqrt{m}$ and d) $K = 30 \text{ MPa}\sqrt{m}$.

completely smoothen the interface. The graphite particles in the microstructure model showed clear decohesion as the ferrite matrix started to deform plastically.

From the stress evolution, it was noticed that in all the range of the applied load, almost all part of the CT sample was in the elastic region, only the microstructure model and area around showed larger stress concentration. So, the definition of X-FEM crack region within the microstructure model was justified by this observation. The stress evolution in the CT sample and the microstructure submodel also dictated that the current model setup, constraints and boundary conditions matched the expectation, and the result from the simulation could be directly compared to the experimental results to validate the simulation results.

5.3.4 DIC validation of strain distribution in the microstructure model

The strain distribution in the microstructure model was validated by comparing the simulation strain with the strain measured by Digital Image Correlation (DIC). Thanks to the project collaborator from Jonkoping University, Sweden for developing DIC system with in-situ testing capabilities and for providing the DIC strain maps. Refer to the published work [171] reporting details of the DIC technique developed and used by the project collaborator. The CT specimen was slightly changed to make the specimen compatible with the in-situ testing facility while keeping the notch length (a) and the characteristic length (W) same. The undeformed micrograph was recorded before the test, and the model dimensions

Figure 5.13: Validation of strain evolution on the microstructure submodel for CT sample 1 a) DIC strain map and b) FEA strain distribution.

were also adjusted to match the CT specimen used for the DIC measurement. Figure 5.13 and Figure 5.14 show comparison of the maximum principal strain maps obtained from the simulation and measured from the DIC for tow CT specimens. DIC strain maps for specimen 1 (Figure 5.13 a)) showed multiple regions (indicated by red arrows), where strain could not be plotted. Similar strain map for specimen 2 (Figure 5.14 a)) was more uniform. Such discontinuities in DIC maps were either due to lack of sufficient pits to track movement in the microstructure or due to larger displacement of the pits. These pits were generated intentionally by pit etching process to aid DIC mapping of the deformed images. For both specimens, the simulation over predicted strain concentration as compared to DIC strain maps. Over prediction of the strain map was more significant in specimen 1 than in specimen 2. For both specimens, the maximum values of strains were much higher for simulation. Such high strains were due to the sharp pixelated interface and occurred at the tip of the pixelated interface. So, the overall strain maps were still comparable at the same strain range. Although the simulation overestimated the strain concentration around the graphite particles, the location of the strain concentration and the strain band form between the graphite particles were comparable for the DIC map and simulation. One important factor contributing to the discrepancy of the result was the fact that DIC maps considered all the surface and subsurface effects, whereas FEM strain distribution was completely based on the the effect of surface graphite particles. Subsurface graphite particles close to the surface could cause larger deformation of the speckle pattern resulting into unmaped pixels in the DIC results as observed for the specimen 1. For specimen 2, the result was more comparable, and the locations of strain concentrations were

Figure 5.14: Validation of strain evolution on the microstructure submodel for CT sample 2 a) DIC strain map and b) FEA strain distribution.

comparable for most of graphite nodules with some differences in the magnitude of the strain value.

The discrepancies in DIC strain maps and simulation strain could be attributed to many reasons. First reason for the discrepancies could be the inefficiency of DIC to measure large strains in the matrix and at the regions near the interface, where maximum strains were expected. Such large strains were effectively distributed in the simulation. Second important reason could be due to the huge difference in the spatial resolution of DIC and FE simulation, which resulted in the higher smoothening in the DIC maps. The spatial resolution for DIC was $69\text{ }\mu\text{m}$ and for FEA were $0.55\text{ }\mu\text{m}$ near the interface and $2\text{ }\mu\text{m}$ on the remaining microstructure model. So, the spatial resolution for DIC was at least 34 times larger than for FEA strain plot. Third reason was because of the fact that the simulation estimated 2D strain field without consideration of subsurface microstructure. Unlike FES, DIC measured 2D strain field considering the effect of subsurface microstructural features. In DIC maps in Figure 5.13 a) and Figure 5.14 a), red circles indicated the higher strain regions that were mostly due to the effect of subsurface features. These high strains in the ferrite matrix were not captured in FEA. Finally, the very high strains at the interface were due to the sharp pixelated interface.

5.3.5 X-FEM crack growth simulation

The developed multiscale SGI material microstructure modeling approach verified by DIC could find many applications like microstructural inhomogeneous stress-strain analysis, analysis of different processes on the SGI microstructure, and microstructure dependent crack growth simulation. In this work, the multiscale SGI model was used in an attempt to simulate microstructure dependent crack growth within the inbuilt X-FEM formulation in ABAQUS. X-FEM technique provides specific advantages of mesh independent crack analysis, easy implementation and accurate solution as compared to conventional FEM crack analysis. Refer to section 2.4 for detail description of X-FEM technique, and refer to section 5.2.5 for the earlier implementation of X-FEM technique to model crack initiation and propagation in the RVE model. The ABAQUS X-FEM demonstrated its capabilities to simulate crack initiation and propagation in simple RVE model with some limitations. The ABAQUS X-FEM capability was further evaluated to access its applicability to model crack initiation and growth in complex microstructure model. So, the results were intended to demonstrate capabilities and limitations

of the formulation. Similar modeling approach, material properties, damage criteria and model adjustments were formulated in the multiscale SGI model. In the beginning, it was attempted to simulate crack initiation and its growth in a single step. But, the initiated crack did not show much longer propagation due to convergence problem. So, further crack propagation was attempted with a predefined crack at the notch. Series of simulations were performed on microstructure model at both faces of the CT specimen. The overall results were explained below with illustration for one representative sample face microstructure model.

Crack initiation

Out of two damage modeling approach available in X-FEM ABAQUS, traction-separation law based cohesive approach was used to simulate crack initiation and propagation in the multiscale microstructure model illustrated in Figure 5.11 and Figure 5.12. Maximum principal strain based crack initiation criterion (MAXPE) was formulated for the ferrite matrix, the critical maximum principal strain being evaluated from the experimental test. So, the crack was expected to initiate from the strain concentration regions. As displacement formulation was recommended to help converge the simulation, displacement load was applied, and the corresponding force was evaluated from a reaction force recorded at the reference point

Figure 5.15: Crack initiation result in the multiscale model with single enrichment X-FEM region (displacement applied = 0.1 mm, time step completed = 0.163).

of the fix pin of the CT model. The initiation of a crack in the microstructure model is shown in Figure 5.15 along with logarithmic strain plot. It is clearly noticed that the crack initiation (indicated by the red arrow) was at one of the strain concentration and was close to the graphite particles near the notch. The graphite particles along the higher strain band showed decohesion from the ferrite matrix; however, due to the sharp pixelated interface, the graphite particles showed larger strain sharing than on the RVE model. Although there were multiple higher strain concentration regions observed, only one crack initiated as it was identified that the current X-FEM formulation in ABAQUS does not allow multiple cracks in single enrichment. The initiated crack propagation was less influenced by the higher strain band as also observed for the RVE model. The propagation of the initiated crack was a real challenge as the rate of convergence was very slow, and it was very hard to completely converge the simulation due to the complex nature of the model.

Figure 5.16: Illustration of multiple X-FEM enrichment regions definition in the microstructure model.

In order to facilitate multiple crack initiation in the model, the ferrite matrix was divided into multiple enrichment regions. Initially, the model was divided into multiple enrichment regions as indicated by two horizontal lines in Figure 5.16. It helped to initiate additional crack at the notch region. In the series of simulations performed, one major limitation observed in X-FEM ABAQUS was when the initiated crack reaches nearby a graphite particle. The crack after reaching the graphite particle only showed large decohesion and crack opening, but no crack was propagated from the other side of the graphite particle even the strain value was much higher than the critical initiation strain. The problem was due to inability to initiate another crack on the other side of the graphite, which was not solved by the

three horizontal enrichment regions. So, the middle enrichment region (main crack domain) was further subdivided into multiple enrichment regions to divide larger graphite particles into two enrichment regions as illustrated in Figure 5.16 (vertical yellow lines representing sub division of the enrichment region). The corresponding crack initiation result is shown in Figure 5.17. With multiple enrichment regions, the strain concentration regions and the initiation of cracks at the notch did not change; however, additional cracks (indicated by red arrows) were observed from the graphite particles at the higher strain band. The initiation of cracks on either side of graphite particles and crack between two nearby graphite particles, seemed realistic as such cracks initiation were usually observed in the experimental study of the tensile damage mechanisms. From this observation, it could be justified that definition of two enrichment regions dividing it by a line parallel to the loading direction could facilitate crack initiation on either side of graphite particles. In the current model, the multiple enrichments were defined manually, which was troublesome to define for each graphite particles in the model. So, any attempt to automate the multiple enrichment definition could lead to effective X-FEM model for multiple cracks simulation in the microstructure model. Although cracks were initiated in the model they did not showed significant growth. Convergence of the X-FEM simulation was the major problem interrupting crack growth. To check for the compatibility of the crack initiation and evolution criteria used, other available damage criteria within cohesive approach were also tried in the similar simulation. The maximum principal stress (MAXPS) criterion with the maximum allowable principal estimated as UTS of the material also showed crack initiation at the same location with similar stress and strain concentration bands. No noticeable difference was observed on the result. The simulation result was compared with the experimental fatigue crack growth result in the sample, which showed difference in the crack initiation point. The crack instead initiated from the strain concentration at the middle of the CT notch. It should be noted here that the amount of load applied in the simulation and the FCP test were not the same, and the experimental test result was used just to get rough idea on the microstructure influence in the crack initiation and propagation. In the CT notch, it is most likely to initiate crack at the same location irrespective of magnitude of load, due to larger stress concentration at the middle of the notch. But in the current simulation, the microstructure stress concentration was also considered, so the comparison experimental and simulation results showed discrepancy. The influence of the subsurface graphite particles in the experimental result and crack initiation only considering surface graphite particles was one of the major reasons

for the discrepancy. It was guessed that the initiation of the crack in current simulation may have hindered initiation of a crack that would grow further to form main crack. So, it was believed that definition of pre-crack at the actual crack initiation site may help to study propagation of the crack in the model and it may help in further convergence of the simulation.

Figure 5.17: Crack initiation result for multiple enrichment X-FEM model (red arrows indicating cracks initiated) (displacement applied = 0.1 mm, time step completed = 0.163).

Crack Propagation

For the detail study of crack propagation capabilities of X-FEM in ABAQUS, pre-crack of 10 μm length was defined at the middle strain concentration in the CT notch, which also corresponds to the crack initiation in the experimental test. Initially, all the modeling approach, parameters and properties were kept similar to the X-FEM formulation used for the RVE model (cf. section 5.2.5). As stated in the crack initiation simulation, both the single enrichment and multiple enrichment models were simulated, so that the results could be compared. In the

Figure 5.18: Single enrichment X-FEM crack propagation in SGI microstructure model.

Figure 5.19: Multiple enrichments XFEM crack propagation in SGI microstructure model.

single enrichment model, the predefined crack growth was observed without any other cracks, however in the multiple enrichment regions model, the predefined crack growth was followed by initiation of additional cracks. But, in both the cases, the crack main crack faced convergence issue before it could reach near the graphite. Series of simulations were performed in an attempt to further propagate the crack, most of them showed growth of the pre-defined crack but hardly reached

Table 5.1: Summary of the effect of model, material and control parameters on the X-FEM crack growth and simulation convergence.

Parameters	Effect on the X-FEM crack growth result and convergence
Graphite particles modeling (void, bound, cohesive interface)	Crack growth direction highly influenced by the graphite and the interface state; graphite as void showed a strong influence on the crack growth direction, bound combine model mostly showed straight crack and graphite with cohesive interface showed influence based on the interface properties.
Enrichment regions	Single enrichment showed only one crack to initiate and propagate; multiple enrichments definition helped to initiate multiple cracks.
Load/displacement formulation	No significant influence on convergence was observed. In some case, displacement formulation seemed to advance few more steps.
Element type (CPS4, CPE4, CPS4R, CPE4R)	Both plane stress and plane strain element showed similar result. Plane element with reduced integration performed better as they resolved some distorted elements issues.
Mesh size	Fine mesh desirable for X-FEM crack simulation. Very fine mesh size ($< 2 \mu\text{m}$) was used, so no influence even some change in mesh.
Ferrite material properties (elastic, elastic-plastic)	Elastic material for ferrite showed faster convergence, but with excessively higher stress. Any material definition with plastic behavior suffered convergence issue.
X-FEM crack initiation criteria (MAXPE, MAXPS)	Both MAXPE and MAXPS showed effective crack initiation when the critical strain or stress value is achieved for the first time in an enrichment region.
Cohesive interface properties	Stiffness played a major role to dictate the crack growth direction; lower values ($< 1 \times 10^5$) showing similarity with graphite void model and higher values ($> 1 \times 10^8$) showing straight crack. No influence of interface damage parameters on crack path and convergence.
Initial crack size (10 μm , 15 μm , 30 μm)	In dependent of the initial crack size, all the cracks grew until it reaches a graphite nodule.
Solution control parameters, damage stabilization	Did not influence the crack path when controlled within the allowable range. Improved rate of convergence till the crack reaches a graphite nodule.

the graphite particle. After the convergence issue was not solved by changing the increment parameters, viscous damping, damage stabilization regularization, damage tolerance and other adjustments listed in section 5.2.5, the solution control parameters were also adjusted to slightly loosen the convergence criteria and the

crack size was also increased. These adjustments helped to further converge the simulation and the crack managed to reach the graphite particle. Figure 5.18 shows the propagation of the predefined crack to the near graphite in single enrichment model. The crack growth showed influence of the surrounding graphite particles as illustrated by the slight deviation in the crack path near graphite particles. The excessively high strain accumulation around the graphite particle at the crack tip showed inability to initiate another crack around the graphite nodule. Similar result for the multiple enrichment model is shown in Figure 5.19. Here, as the predefined crack start to grow, other cracks initiated at the higher strain concentrations around the graphite and they also start to grow along with the initial crack. With the initiation of additional cracks, the strain localization was much less as compared to the single enrichment region. Before the predefined crack reach the graphite nodules the cracks initiated from the graphite nodule approached next graphite particles and the simulation again suffered another convergence problem, most probably due to similar problem as explain before. Based on the series of simulations performed, it was understood that the major problem in X-FEM crack simulation of complex SGI microstructure in ABAQUS was its capability to associate only one crack with an enrichment region. Some improvement was observed by defining multiple enrichment regions and adjusting other model and simulation parameters. But, complete convergence of the simulations was not achieved despite of several attempts by changing series of tolerances, control parameters, element types available, damage criteria, material definition and interface definition. Table 5.1 summarizes model, material and control parameters changed in the microstructure model to study their influence on X-FEM crack and the model convergence. From the series of simulations, it was understood that the current X-FEM formulation in ABAQUS can accurately simulate growth of single dominant crack in elastic material with some adjustment of stabilization and solution control parameters. However, in complex problem with multiple nonlinear behavior, and multiple cracks and defects interacting with each other, the inbuilt X-FEM formulation suffered major convergence issue.

5.4 2D Approximation of 3D Microstructure: Possible Misinterpretation

In this work, 2D plane stress approximation of 3D SGI microstructure was used to study inhomogeneous stress, strain and damage behavior. The 2D plane stress state assumes non-zero stresses in the 2D plane and zeroes perpendicular stress components. Use of simplified 2D model definitely helps to ease modeling approach and significantly reduce computation time. However, the 2D model could not reflect all the mechanisms in the real 3D microstructure. It is possible that the results could be misleading in comparison to real mechanisms, so it is essential to study the possible misinterpretation of the real mechanisms based on the 2D plane model analysis. It is recommended to consider following points for avoiding possible misinterpretation in current deformation and damage study on SGI microstructure using FEM simulation.

- The 2D model well-represented stress and strain concentrations caused by the presence of surface graphite particles in the ferrite matrix. Clearly, the 2D model assumes circular (kind of cylindrical) graphite particles which in actual is spheroidal and whatever exists on the surface is the same throughout the plane stress thickness. In that sense, it does not account for the effects of subsurface graphite particles. In actual microstructure, the subsurface graphite particles also play a significant role in inhomogeneous strain distribution, and it may even initiate microcracks and assist their propagation (that appeared as ferrite matrix crack in the experimental damage mechanism study). So, based on the 2D model analysis, it cannot be ignored that additional strain inhomogeneities caused by subsurface graphite particles could result into additional damage mechanisms like matrix microcracks and crack branching in the SGI microstructure.
- The surface-based cohesive behavior helped to simulate graphite particles decohesion in the FE models. In actual microstructure, graphite decohesion occurs simultaneously at the opposite ends of the graphite nodules forming elliptical graphite void, whereas in the RVE model, interface separation initiated mostly on the top side and comparatively larger separation gap observed with further loading. So, some influence of loading boundary could not be ignored in the RVE model. However, such effect is reduced in the real micrograph based model as the model is similar to real CT specimen tested

and symmetric load applied on both ends.

- In the Finite element models, the graphite particles were modeled as purely elastic phase and no damage conditions were prescribed to them. But, in real microstructure damage, many of the irregular graphite particles and some spheroidal graphite nodules exhibited internal cracking of the graphite particles, and the prior case even leading into microcrack growth into the ferrite matrix. Even if such graphite damage were not modeled in the FE analysis, it did not much influence the model behavior as the interface decohesion results in similar stress concentration effect.
- In XFEM crack initiation and propagation simulation, cracks initiation points were predicted considerably well allowing mesh independent crack propagation. Definition of multiple enrichment regions facilitated multiple cracks initiation and propagation. These cracks showed did not show interaction with each other due to lack of crack tip stress field for growing crack in XFEM ABAQUS. But, in real microstructure there exist complex three-dimensional crack tip stress field interacting with nearby graphite nodules and initiated microcracks. Considerations should be made to include crack tip stress for growing cracks in the future study of XFEM crack studies.

5.5 Summary

A three graphite RVE model was formulated based on the average microstructure characterization result of EN-GJS-500-14 SGI. Graphite particles were assumed perfectly spherical and different sizes within the standard deviation were used. 2D plane stress problem was formulated with reduced integration elements to simplify the model. The material properties implemented in the model assumed purely elastic graphite and elastic-plastic ferrite, which well represented the SGI microstructure as the ferrite phase undergoes large plastic deformation and carries major part of the applied load. Periodic boundary conditions were implemented in the RVE model to eliminate the existence of free surface and simulate as an infinite model. The RVE model was applied relative displacement load to study the role of graphite particles modeled as void in the ferrite matrix, as unbound elastic particles and as surface based cohesive interface bound elastic particles. The RVE model with surface based cohesive interface showed better representation of the SGI microstructure. It enabled graphite particles to be modeled as

bound particles until critical stress was reached, which was later modeled as partially decohesed graphite particles. Although the cohesive interface improved the modeling approximations, the model behavior was also influenced by the interface properties. It was not possible to directly measure the interface properties for the experimental test. So, iterative simulations were performed in the RVE model to study sensitivity of the interface properties and estimate tentative value that match with the experimental observations. The plot of interface damage initiation variable on the overall stress-strain plot showed that the interface stiffness of $1 \times 10^8 \text{ N/mm}^3$ initiated decohesion at the notch region of the overall stress-strain curve. The complete RVE model was then used to study X-FEM crack initiation and propagation behavior. Only the ferrite region was defined as X-FEM enrichment region. As expected, the crack initiated at the strain concentration near the larger graphite particle. In the course of the propagation of the initiated crack, many strain concentration regions with much higher strains were observed, but no other cracks were initiated. It was identified that the existing X-FEM formulation in ABAQUS allowed only one crack to initiate in an enrichment region unless multiple initiation criteria were achieved at the very same time. So, the RVE model was further sub-divided into multiple enrichment regions to study capabilities of X-FEM ABAQUS to simulate multiple cracks. With the multiple enrichment regions, multiple cracks were initiated on either side of the larger graphite particles. The strain concentration caused by the graphite particles gave good prediction of the crack initiation sites, however the propagation of the crack did not follow the higher strain band between the graphite particles and showed less influence by the cracks initiated on the other enrichment region around the surrounding graphite particles.

After implementation of the RVE model, a multiscale SGI material microstructure model was developed. A plane stress CT specimen model was executed with microstructure submodel at the CT notch and bulk SGI material on the remaining part. The microstructure submodel was generated using image based FEM method OOF2, which represented real SGI microstructure at the CT notch into FE discretization. The material properties, interface properties and constitutive used in the RVE model were implemented in the microstructure model. The multiscale CT model was first checked for the stress and strain evolution in the whole model and microstructure submodel. The result showed smooth transition of global and submodel with efficient model interface. The stress distribution showed uniform stress evolution around the global CT model, whereas inhomogeneous stress was

observed in the microstructure submodel due to the influence of graphite particles. Further, the model was validated by comparing with DIC strain map, which showed slightly less strain magnitude with similarity in the location of the higher strain bands. It was pointed out that the developed multiscale SGI material microstructure modeling approach could be used for microstructure inhomogeneous stress-strain analysis, analysis of different processes on the SGI microstructure and microstructure dependent crack growth simulation. In this study, the multiscale model was used in an attempt to simulate crack growth in the complex microstructure model implementing X-FEM enrichments regions. Both single enrichment and multiple enrichment of the ferrite matrix were attempted. Multiple enrichments helped to initiate multiple cracks, but the initiated cracks did not show large growth due to major convergence problem. So, it was tried to simulate crack growth with predefined crack at the notch. In series of simulations ran by changing multiple model and control parameters, no complete convergence of the X-FEM crack growth was achieved. It was noticed that the major convergence problem was due to inability to initiate another crack from other side of graphite particles after a crack reached the graphite. Definition of multiple enrichment regions helped to initiate multiple cracks, however, the interaction of multiple crack and the cracks from graphite reaching another graphite caused convergence difficulty. Therefore, it was concluded that the current X-FEM formulation in ABAQUS can accurately simulate growth of single dominant crack in elastic material with some solution control adjustments. However, in complex problem with multiple nonlinear behavior, and multiple cracks and defects interacting with each other, the inbuilt X-FEM formulation suffered major convergence issue. So, it is suggested to use ABAQUS user subroutines to enable multiple crack initiations and model crack tip stress field.

Real SGI microstructure alike FE model developed by combining experimental damage mechanism criteria and representative phase properties contributed a new approach to multiscale modeling of SGI material. The surface based cohesive interface parameters estimated from the RVE study resulted in better representation of the SGI microstructure, which enabled modeling graphite particles as bonded elastic inclusions to a certain load and as debonded elastic solids after decohesion criteria has been reached. Attempt to implementation X-FEM framework on the developed microstructure model facilitated microstructure dependent damage modeling and accessed the X-FEM capabilities in ABAQUS to model such complex crack, highlighting the challenges and modification to solve some of them.

Chapter 6

Conclusions and Future Works

In this chapter, the research works done is summarized with major conclusions, contributions and highlights of possible future directions to further improve and complete this work.

6.1 Conclusions

In this work, Solution Strengthened Ferritic (SSF) Spheroidal Graphite Iron (SGI) material and microstructure have been studied, and damage mechanisms on the SGI microstructure have been comprehended. In SFF SGI, higher silicon content helped to form solid solution strengthened ferritic matrix in SGIs. The microstructures of SFF SGIs were more uniform with graphite particles in the complete ferritic matrix. Among the studied SGIs, EN-GSJ-500-14 with 3.71 % silicon showed a better balance of strength and ductility as compared to other similar grades SGIs with different silicon contents. As expected, the ferrite matrix hardness was observed to increase with higher silicon content. The microstructure phase specific properties were of interest, for which two methods were investigated; phase properties optimization from tensile test and nanoindentation P-h curve optimization for phase properties. It was noticed that the two methods captured different aspects of the material microstructure and estimated different elastic-plastic ferrite properties. The tensile test optimization method captured properties of the bulk phase including effects of all microstructure defects like porosities, shrinkage, grain boundaries, etc. Whereas the properties from nanoindentation test captured very local material response, mostly within single grain without consideration to other

microstructure defects. It was pointed out that the bulk phase properties obtained from the tensile test was more suitable to represent phase properties as it simplifies implementation of the phase properties without requiring to represent individual microstructure defects.

The EN-GJS-500-14 SGI was further Deep Cold Rolled (DCR) and thermally cycled to approximate the effects of mechanical and thermal processes on the SGI microstructure. DCR process being mechanical process caused high distortion of the surface material. The distortion induced severe plastic hardening of the ferrite matrix, which was exhibited in the nanoindentation hardness measurement. The plastic deformation in the DCR process also influenced surface microstructure, and it was remarked that graphite characterization on the deep cold rolled surface might lead to error as many spheroidal graphite nodules appeared irregular and elongated due to partial surface exposure. So, it is recommended that the deep cold rolled surface must be ground and polished before graphite nodule characterization. However, the effect of such sample preparation on residual stress must be considered. The major challenge observed for DCR process on SGI material was due to the presence of subsurface graphite particles, which caused the ferrite matrix cracking above such graphite particles. In the case of thermal cycling, although initial thermal cycling showed a decrease in thermal expansion behavior, no significant change in material microstructure and phase properties was noticed. However, the major microstructure change was decohesion of the graphite-ferrite interface due to dissimilar coefficients of thermal expansion. The microstructure should be characterized by graphite morphology, matrix structure and its properties, but additional parameter illustrating the graphite-ferrite interface state should be included in the microstructure characterization.

Understanding the crucial role of the microstructure and graphite morphology in mechanical and fracture behavior of SGI materials, microstructure dependent damage mechanisms were investigated in EN-GJS-500-14. Tensile and fatigue damage mechanisms were studied on the miniature tensile and compact tension (CT) specimens. Fatigue damage mechanisms were investigated by separately performed Fatigue Crack Initiation (FCI) and Fatigue Crack Propagation (FCP) tests. The role of spheroidal graphite nodules, degenerated graphites and shrinkage cavities in damage initiation and growth were studied explicitly for tensile and fatigue load. In the tensile test, the matrix-nodule interface decohesion and plastic deformation of the ferrite matrix were the dominant mechanisms. Less influenced by nodule shape, graphite particles showed decohesion from the ferrite matrix at

6.1. CONCLUSIONS

the overall stress of 400 MPa to 420 MPa, which is close to the yield stress of the material. The decohesed graphite voids showed significant growth with plastic deformation of the ferrite matrix, and those elongated graphite nodules showed microcrack initiation more frequently due to higher strain concentration. Degenerated graphite particles including those elongated graphite nodules dominated cracks initiation into the ferrite matrix. The initiated microcracks from the degenerated graphite particles and in the ferrite matrix did not show much growth in the crack size; instead, they showed large crack opening. In fatigue tests, graphite nodule shape played a decisive role in the crack initiation and propagation behavior. In the crack initiation region, compacted and irregular graphite nodules dominated cracks initiation, and in the crack propagation region, the spheroidal graphite-matrix decohesion inducing a crack tip blunting effect was the most frequent damage mechanism in the microstructure. FCI tests exhibited that cracks initiation were either by internal graphite cracking or by decohesion or by a combination of decohesion and internal cracking. Spheroidal graphite nodules on the other hand did not initiate microcracks at the time of crack initiation from degenerated graphite particles, but they showed matrix-nodules interface decohesion and some of the larger spheroidal nodules showed circumferential internal crack. The extent of plastic deformation of the ferrite matrix, and the growth of graphite voids were much less in fatigue load due to progressive nature of the damage. Further, quantitative study of graphite nodules damage revealed internal cracking and combine damage of most of the graphite nodules with Roundness Shape Factor (RSF) less than 0.5, whereas the spheroidal nodules with RSF higher than 0.9 in specific showed the matrix-nodules interface decohesion. The nature of graphite particle damage was observed to depend on the graphite growth morphology in the solidification process. In FCP tests, the compacted and irregular graphite nodules were mostly fractured when present in front of the crack tip. In the case of fatigue crack growth, crack branching was often observed either by main crack kinking towards nearby graphite nodules or by secondary cracks growth toward the main crack. The roles of graphite nodules were predominant on the stable crack propagation region. Other than graphite nodules, shrinkage cavities of a size comparable to the size of graphite particles were also observed, which behaved similar to the compacted graphite particles, initiating microcrack. The dominance of such shrinkage cavities is directly dependable of their size in the microstructure. At the point of the fracture; in tensile tests, one of the larger cracks grew by coalescence of the graphite voids and initiated microcracks to fracture the specimen; in fatigue tests, final fracture occurred by the rapid growth of

the fatigue crack by coalescence of graphite particles. Fracture surface study of the specimens showed similarity in the final fracture in fatigue failure and tensile fracture. The fracture surface exposed undamaged graphite particles in graphite voids whose size were much larger than the graphite size, and the ferrite fracture regions between graphite particles were characterized by ductile fracture with the presence of micro-voids initiation and growth. In addition to damage mechanisms studies, fatigue crack growth rate, fracture toughness and fatigue life of the EN-GJS-500-14 material were also characterized. Based on the experimental damage mechanisms and characterizations, tensile and fatigue damage criteria were established for the investigated SGI, which is useful in any attempt to define damage criteria of the EN-GJS-500-14 SGI.

The average graphite characterization result for EN-GJS-500-14 was used to develop a three graphite RVE model of SGI microstructure. The RVE was modeled with graphite particles as voids, unbound elastic particles and surface based cohesive interface bound elastic particles. Periodic boundary conditions were implemented in the RVE model to eliminate the existence of free surface and simulate as an infinite model. In the elastic load region, all the modeling approximation showed similar stress and strain inhomogeneity. As the ferrite matrix deform plastically, the stress and strain distribution pattern showed the particular difference on the amount and location of the strain concentration. The RVE model with surface based cohesive interface showed a better representation of the SGI microstructure. It enabled graphite particles to be modeled as bound particles until critical stress was reached, which was later modeled as partially debonded graphite particles. Further, the RVE model was simulated with different interface parameters to approximate these parameters to match experimental graphite decohesion behavior. The interface damage initiation variable was plotted on the overall stress-strain curve for the RVE model, which showed that the interface stiffness of $1 \times 10^8 \text{ N/mm}^3$ initiated decohesion at the notch region of the overall stress-strain curve (at the beginning of yield). The RVE model with surface based cohesive interface was then used to simulate X-FEM crack initiation and propagation. Only one crack initiated at the higher strain concentration region around the larger graphite particle. Even if multiple higher strain concentration regions were observed, no other cracks initiated in the RVE model. It was noticed that in X-FEM ABAQUS, it allows only one crack to initiate and grow in single enrichment region. So, the RVE model was further sub-divided into multiple enrichment regions, which allowed multiple cracks to initiate in the RVE model. With the

definition of multiple enrichment regions, the X-FEM crack initiation and growth simulation result showed similarity with the experimental results. The material properties, interface properties and constitutive were used to develop a multiscale SGI material microstructure model. The CT sample model with microstructure submodel at the notch was developed to demonstrate and validate the multiscale modeling approach. Image based FEM method OOF2 was used to generate FE representation of the SGI micrograph. The stress and strain evolution results showed a smooth transition of global and submodel with uniform stress evolution in the global CT model and inhomogeneous stress and strain in the microstructure submodel. The inhomogeneous strain in the microstructure model was validated by DIC result, which showed slightly higher strain magnitude at the similar higher strain locations. The developed multiscale SGI material microstructure modeling approach could be used for microstructure inhomogeneous stress-strain analysis, analysis of different processes on the SGI microstructure and microstructure dependent crack growth simulation. It was attempted to use X-FEM formulation in ABAQUS to simulate crack initiation and propagation in such complex model. Defining multiple enrichment regions helped to initiate multiple cracks and its growth towards nearby graphite. However, series of simulations showed that the model suffered major convergence issue due to only one crack initiation and growth in an enrichment region and due to the interaction of the initiated cracks. In the existing X-FEM formulation in ABAQUS, the convergence problem could not be solved completely by changing model, stabilization, damping and solution control parameters. So, it is suggested to use ABAQUS user subroutines to model X-FEM crack growth in complex models.

6.2 Major Contributions

The contributions of this research work are listed below:

- Microstructure damage micromechanism studies provided clear perception of tensile and fatigue damage mechanisms in SGI. Separately performed tensile tests, fatigue crack initiation tests and fatigue crack propagation tests provided insight on the roles played by graphite nodules in tensile loading and at a different stage of fatigue loading. The observed damage mechanisms from graphite particles showed dependency on the graphite growth morphology, illustrating the critical role of the compacted and degenerated graphite

particles on material damage. This finding draws attention on the importance of controlling casting defects (shrinkage cavities, porosities, etc.) and degenerated graphite particles in as-cast SGIs to improve its fatigue properties.

- Microstructure model developed by combining experimental damage mechanism criteria and representative phase properties in the FE representation of real SGI microstructure provides a new approach to perform microstructure dependent analysis. The surface based cohesive interface parameters estimated from the Representative Volume Element (RVE) study resulted in better representation of the SGI microstructure, which enabled modeling graphite particles as bonded elastic inclusions to a certain load and as debonded elastic solids after decohesion criteria has been reached. Attempt to implementation X-FEM framework on the developed microstructure model facilitated microstructure dependent damage modeling and accessed the X-FEM capabilities in ABAQUS to model such complex crack, highlighting the challenges and modification to solve some of them. Successful implementation of X-FEM in microstructure model will perceive new direction to damage modeling of engineering materials.
- Microstructure investigation of SGI after thermal and mechanical processes evaluated microstructure changes in such processes. It was pointed out that the mechanical surface processes like DCR could severely deform surface microstructure requiring additional precaution in graphite morphology characterization. For the thermal cycling, it was identified that the graphite-matrix interface state should be included in microstructure characterization. It was stated that with the additional characterization of interface state for thermal processes and residual stress for mechanical processes, the SGI microstructure could be completely characterized by graphite morphology, matrix composition and phase properties. The microstructure parameters identified in this work well represented SGI microstructure and similar framework could be used for other types of cast irons with graphite.
- Material, microstructure and damage characterization of high Si grade EN-GJS-500-14 will be a valuable addition of material data to the scientific community.

6.3 Future Works

The following future work recommendations are made to extend current research work as well as the implementation of presented works on the microstructure, damage mechanisms and modeling of spheroidal cast irons in general.

- The multiscale material modeling with microstructure submodel demonstrated in this work allows to study microstructure behavior in a specific region of the component. One of the implications of this model is in microstructure dependent damage modeling attempted in this work using in-built X-FEM capabilities in ABAQUS. Although X-FEM approach is attractive (accurate result with computational benefit) and easy (mesh independent method without the requirement of re-meshing or mesh conforming to crack face) technique, current formulation on ABAQUS has certain limitations that posed difficulty in modeling crack initiation and propagation of complex problem. It would be interesting to overcome the limitations of X-FEM formulation in ABAQUS by using user subroutines to model crack initiation and growth in the multiscale model. Further with efficient X-FEM crack growth modeling, it can be extended to simulate microstructure dependent fatigue crack propagation. The fatigue damage criteria derived from the experimental tests and damage mechanisms studies at the end of Chapter 4 in this thesis could be used to define crack initiation and propagation criteria.
- Compressive residual stress is one of the major effects of DCR process, which improve fatigue life of the rolled component by making fatigue crack initiation and propagation difficult. Earlier in this thesis, it has been discussed that the major challenge of DCR process on SGI material and microstructure is the initiation of microcracks around graphite particles. However, the initiated cracks may or may not grow due to induced compressive residual stress. Therefore, the influence of induced compressive residual stress on the crack growth rate and the ultimate effect of DCR process on fatigue life of the SGI components need to be investigated for proper understanding of the benefits of the DCR process on SGI components.
- In SGI material, crack initiation and propagation behavior is dominated by graphite particles as discussed earlier. However, the grain boundaries will also influence initiation of crack and its growth. So, Electron Backscatter

Diffraction (EBSD) studies on the crack initiation and propagation tested samples will help to understand the role of grain boundaries in cast irons with major graphite defects. Also, the crack propagation is influenced by the subsurface graphite distribution. So, with the advances of three-dimensional material characterization techniques, it will be interesting to study 3D crack profile for detail understanding of surface and subsurface crack growth and their interaction.

- Degenerated graphite particles (elongated, irregular, chunky, exploded, etc.) have shown significant influence on the fatigue crack initiation and propagation behavior. The amount of such degenerate graphite particles will have a direct influence on the fatigue life of the SGI component. Thus, quantification of average degenerated graphite particles fraction on different cast SGI, and its corresponding fatigue life is necessary. Such quantification of fatigue life will suggest the severity of degenerated graphite particles.
- Comprehensive damage mechanisms were studied based on room temperature test, however the the material behaves different at high temperature. It would be good continuation of this work to study damage mechanisms at high temperature test. At high temperature, the difference in thermal expansion behavior between the ferrite matrix and graphite particles could play significant role in the damage mechanisms. Also, the effect of heat treatment at elevated temperature on the recovery of deformed microstructure in mechanical surface treatment processes like deep cold rolling could be interesting.
- Graphite particles and the ferrite matrix in SGI microstructure exhibit a large difference in their thermal expansion behavior, which will exert higher thermal stresses at the interface. So, for the application of SGI materials in components under thermomechanical loading, it is suggested to investigate fatigue crack initiation and propagation under combined thermomechanical load.

Author's Publications

Journal Papers

1. S. Sujakhu, S. Castagne, M. Sakaguchi, K.A. Kasvayee, E. Ghassemali, A.E.W. Jarfors, and W. Wang. On the fatigue damage micromechanisms in si-solution-strengthened spheroidal graphite cast iron. *Fatigue & Fracture of Engineering Materials & Structures*, 41(3):625–641, 2018.
2. K. A. Kasvayee, E. Ghassemali, K. Salomonsson, S. Sujakhu, S. Castagne, and A. E. W. Jarfors. Strain localization and crack formation effects on stress-strain response of ductile iron. *Materials Science & Engineering: A*, 702:265–271, 2017.
3. K. A. Kasvayee, E. Ghassemali, K. Salomonsson, S. Sujakhu, S. Castagne, and A. E. W. Jarfors. Microstructural strain mapping during in-situ cyclic testing of ductile iron. *Materials Characterization*, 2018.

Conference Papers

1. S. Sujakhu and S Castagne. Microstructure and damage mechanisms in thermomechanically treated si-solution-strengthened spheroidal graphite cast iron. In *International Conference on PROCESSING & MANUFACTURING OF ADVANCED MATERIALS Processing, Fabrication, Properties, Applications (THERMEC'2018)*, Paris, France, July 2018 [Accepted].
2. S. Sujakhu, S. Castagne, M. Sakaguchi, K. A. Kasvayee, E. Ghassemali, A. E. W. Jarfors, and W. Wang. Micromechanisms investigation on fatigue crack initiation and propagation in high silicon spheroidal graphite cast iron.

In *8th Low Cycle Fatigue Conference (LCF8)*, pages 276–281, Dresden, Germany, June 2017.

3. S. Sujakhu, S. Castagne, and M. Taureza. Microstructure characterization of ductile cast iron and its phase properties detection. In *International Conference on Micromanufacturing (4M/ICOMM)*, pages 526–529, Milan, Italy, March 2015.

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