Co-Cr-Ni-Mo wires for medical applications : processing, mechanical, microstructure and fatigue characterization

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**NANYANG
TECHNOLOGICAL
UNIVERSITY**

**Co-Cr-Ni-Mo WIRES FOR MEDICAL
APPLICATIONS: PROCESSING,
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GVK SAI SRIKANTH

SCHOOL OF MECHANICAL AND AEROSPACE

ENGINEERING

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ENGINEERING**

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

2019

Statement of Originality

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

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I have reviewed the content and presentation style of this thesis and declare it is free of plagiarism and of sufficient grammatical clarity to be examined. To the best of my knowledge, the research and writing are those of the candidate except as acknowledged in the Author Attribution Statement. I confirm that the investigations were conducted in accord with the ethics policies and integrity standards of Nanyang Technological University and that the research data are presented honestly and without prejudice.

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Authorship Attribution Statement

This thesis contains material from four papers published in the following peer-reviewed journals where I was the first and the corresponding author.

Chapter 4 is published as **GVK, S.S.**, M.J. Tan, and Z. Liu, Influence of drawing practices on the mechanical, texture and work hardening characteristics of Co-Cr-Ni-Mo wires. *Materials Science and Engineering: A*, 2018. **713**: p. 94-104. <https://doi.org/10.1016/j.msea.2017.12.063>.

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The contributions of the author and co-authors for the papers mentioned above are as follows:

I performed a detailed literature review on the material and the process and identified the potential research gaps in the processing of the material. I designed all the experiments in this study, with the support of Dr. Liu. I prepared and collected all the samples required for the mechanical, microstructure and fatigue characterization, and conducted all the mechanical testing at Heraeus Materials Singapore Pte Ltd. For the electron microscopy studies such as EBSD, FIB, and TEM, I conducted the characterization work personally using the research facilities and equipment's at Institute of Materials Research and Engineering, Singapore. I have analyzed and post-processed the data, and the data was discussed with A/Prof Tan Ming Jen and Dr. Liu for their views and opinions. I have prepared the manuscript drafts and took responsibility for being the corresponding author for all the communications with the journal editors and reviewers.

A/Prof Tan Ming Jen, reviewed all the manuscripts drafts and also the corrected versions after the reviewer's comments, and guided me in improving my writing and vocabulary skills. He has guided me during the alignment of the concept phase and provided valuable inputs on the investigation methodologies. He also provided his technical expertise and knowledge in the area of wire drawing, during our technical meeting sessions and supported me in interpreting the results and discussions correctly and drawing a proper and justified conclusion for all the experimental studies.

Dr. Liu provided the initial project direction and arranged for the necessary equipment and materials at Heraeus Singapore Pte Ltd, for all the experimental work and mechanical testing. All the manuscript drafts and the proof versions were reviewed and commented by Dr. Liu, before the final submission. The experimental data were analyzed and discussed with Dr. Liu, and his recommendations were duly implemented in all the manuscripts.

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Abstract

MP35N (35% Co-35% Ni-20% Cr-10% Mo) is a cobalt-based face-centered cubic (FCC) superalloy which is commonly used as conductor in cardiac and neurostimulation leads due to its good combination of strength, ductility, fatigue life, and corrosion resistance. Thin wires for the conductor leads are manufactured from this material using the cold drawing process which involves plastic deformation of the material and subsequent heat treatments to get the desired properties. As the process involves substantial to severe plastic deformation, it is necessary and very critical to understand the relationship between the process and the microstructure and to correlate them to mechanical and fatigue performance. The crystallographic texture is the link correlating the deformation and thermal treatments to their properties and performance.

In the present study, an attempt was made to understand the influence of the drawing practices i.e. Full Die Drawing (FDD) and Half Die Drawing (HDD) on their mechanical and electrical properties, deformation homogeneity, plastic instability, strain rate sensitivity, strain rate hardening and cyclic fatigue behavior of MP35N wires, for different cold work (CW) reductions. The difference in properties observed, are associated and compared to its microstructure, which has been characterized by Field Emission Scanning Electron Microscope (FESEM), Electron Beam Scattered Diffraction (EBSD), and Transmission Electron Microscope (TEM).

The results showed that wires drawn with FDD practice demonstrated a higher yield strength (σ_{YS}) ultimate tensile strength (σ_{UTS}) higher work hardening rate and different work hardening regimes when compared to the HDD wires for a similar amount of CW. It was observed that FDD wires had a smaller grain size, more homogenous texture and a higher number of twins and dislocations, whereas the HDD wires exhibited coarse grains, non-uniform texture and a lower amount of lattice defects. The Inhomogeneous Factor (IF) and electrical conductivity was measured on the wires, and the results concluded that the FDD drawn wires exhibited homogenous deformation, uniform microstructural and hardness gradient across the wire when compared to HDD wires. The electrical conductivity of the HDD drawn wires was higher than the FDD wires, and the level of inhomogeneity and the variation of conductivity decreased with the increase of CW.

Plastic instability (PI) in MP35N wires, was determined from the logarithmic plots of the true stress and strain curves and the strain hardening exponent (γ) and strain rate sensitivity (SRS) (m) values were computed. It was found that wires drawn with FDD practice had a lower instability until 75% CW, after which the PI increased significantly when drawn to 95% CW. The lower instability in the FDD wires at 75% CW, was attributed to the smaller grain size, higher dislocation density and lower twin spacing which contributed to higher γ and m , as characterized by TEM. Several shear bands were observed in the 95% CW FDD wires, which led to plastic strain localization and increased instability.

The influence of strain rate (SR) on the SRS, strain rate work hardening (SRWH) in MP35N wires, was investigated by subjecting the wires to uniaxial tensile tests, and by varying the SR from 10^{-6}s^{-1} to 10^{-2}s^{-1} . The experimental results illustrate that the strength, SRWH, and the m values were observed to be higher in the FDD drawn wire. The increase in strength and hardening rate of the FDD drawn wire with the rise in SR was ascribed to increased dislocation density and reduced twin thickness, and the increased SRS and ductility at low SR were attributed to the increased grain boundary (GB) activities. An abnormal SRWH was observed in the HDD drawn wire tested to an SR of 10^{-2}s^{-1} , where a Stage II hardening peak was observed at a very high strain, due to the solute segregation of the Mo atoms to the GB.

Finally, the fatigue behavior of the wires was compared, and the results demonstrated that the FDD drawn wires had a higher endurance limit until 75% CW, but there was a slight drop in the fatigue performance when the CW was increased to 95%. This was attributed to the different grain sizes, dislocation structures, fracture surfaces and crack morphologies observed between the two wires.

It can be concluded from the current study that the drawing process and its relationship to texture are highly significant and the selection of the appropriate process should be determined based on the functional requirements and application of the product.

Table of Contents

Contents	Page No.
Acknowledgements	viii
Abstract	ix
Table of Contents	xi
List of Figures	xiv
List of Tables	xx
Abbreviations & Notations	xxi
List of Publications	xvii
Chapter 1 Introduction	1
1.1. Background of Research.....	1
1.2. Research objective and scopes.....	6
1.3. Thesis Outline.....	7
Chapter 2 Literature review	9
2.1 MP35N Composition and its Phase Transformations.....	9
2.2 Strain Hardening and Strain Rate Sensitivity (SRS) in metals.....	13
2.2.1 Influence of intrinsic material properties (SFE, dislocation and twin density) on the Strain Hardening	15
2.2.2 Influence of intrinsic material properties (SFE, dislocation and twin density) on the SRS... ..	18
2.2.3 Influence of Grain Size on the Strain Hardening and the Strain rate sensitivity	23
2.3 Mechanics of Wire Drawing	31
2.3.1 Effect of drawing force on the Strain Hardening behavior of the material	33
2.3.2 Effect of drawing parameters on the texture evolution in wires	36
2.4 Fatigue and Microstructure.....	45
2.4.1 Fatigue behavior of MP35N.....	57
Chapter 3 Material and Experimental Methods	66
3.1 Material	66
3.2 Experimental Methods.....	66
3.2.1 Wire Drawing Experiments.....	66
3.2.2 Tensile Testing	68
3.2.3 Hardness Testing	69
3.2.4 Electrical Conductivity Measurements	70
3.2.5 Fatigue testing	70
3.2.6 Field Emission Scanning Electron Microscope (FESEM) and Electron Beam Scattered Diffraction (EBSD) characterization.	71
3.2.7 Focused Ion Beam (FIB) and Transmission Electron Microscopy (TEM) characterization..	72

Chapter 4 Influence of drawing practices on the mechanical, texture and work hardening characteristics of Co-Cr-Ni-Mo wires.....	73
4.1 Introduction	73
4.2 Materials and Methods	74
4.3 Results	74
4.3.1 Mechanical Results.....	74
4.3.2 Microstructure Results.....	76
4.4 Discussions	79
4.4.1 Effect of drawing practices on the Yield Strength of the material	79
4.4.2 Effect of drawing practices on the work hardening of the material	88
4.5 Summary	91
Chapter 5 Study of deformation homogeneity and electrical conductivity in Co-Cr-Ni-Mo wires drawn with different drawing practices.	92
5.1 Introduction	92
5.2 Materials and Methods	94
5.3 Results and Discussions.....	95
5.3.1 Influence of drawing practice on the hardness distribution.	95
5.3.2 Influence of drawing practice on their electrical conductivity.....	103
5.3.3 Influence of drawing practices on the Inhomogeneous Factor (IF)	106
5.4 Summary	110
Chapter 6 Plastic Instability in Co-Cr-Ni-Mo alloy wires drawn with different drawing practices.....	111
6.1 Introduction	111
6.2 Materials and Methods	113
6.3 Results	113
6.3.1 Mechanical Results.....	113
6.3.2 FIB, TEM, and EDX characterization.....	116
6.4 Discussions	119
6.4.1 Influence of drawing practices on the plastic instability of a 50% CW wire.....	119
6.4.2 Influence of drawing practices on the plastic instability of a 75% CW wire.	126
6.4.3 Influence of drawing practice on the plastic instability of a 95% drawn wire.	131
6.5 Summary	137
Chapter 7 Strain rate effects on the strain rate sensitivity and strain rate hardening in Co-Cr-Ni-Mo wires.....	139
7.1 Introduction	139
7.2 Materials and Methods	140
7.3 Results	141
7.3.1 Mechanical Results.....	141
7.3.2 Microstructure Characterization	144
7.4 Discussions	148
7.4.1 Strain rate effects on the strength, ductility, and strain hardening rate of MP35NLT wire.	148

7.4.2 Strain rate effects on the SRS of MP35NLT wire.....	158
7.5 Summary	163
Chapter 8 Fatigue behavior in Co-Cr-Ni-Mo alloy wires drawn with different drawing practices.....	165
8.1 Introduction	165
8.2 Materials and methods.....	166
8.3 Results	168
8.3.1 Microstructure and tensile results	168
8.3.2 Cyclic Performance (S-N curve) of MP35N wires	170
8.4 Discussions	172
8.4.1 Influence of drawing practice on the cyclic behavior of 50%CW wires.	172
8.4.2 Influence of drawing practice on the cyclic behavior of 75 and 95%CW wires.	179
8.4.3 Fatigue fractography and fracture surface analysis	192
8.5 Summary	206
Chapter 9 Conclusions and Recommendations for further study	208
9.1 Conclusions	208
9.2 Recommendations for Future Work	212
References	214

List of Figures

Figure 1.1: Example of a Pacemaker Implanted into the heart [4].....	2
Figure 1.2: Components of a pacing lead made from wires and coiled to a lead	4
Figure 1.3: Components of a pacing lead made from strands and coiled to a cable [3]	4
Figure 2.1: Microstructure of annealed MP35N alloy [56]	10
Figure 2.2: Phase transformation of MP with varying % Co [57].....	11
Figure 2.3: Co-Ni pseudo-binary phase diagram [59].....	11
Figure 2.4: DTA curve of MP35N showing the dissolution temp as 980°C [59].....	12
Figure 2.5: Schematic representation of the strain hardening regimes between high and low SFE materials [80]	16
Figure 2.6: Four stages of the strain hardening regimes of low SFE materials [65]	16
Figure 2.7: Comparison of the AV and the total dislocation density for aluminum alloy deformed to a similar SR.[105]	20
Figure 2.8: Influence of SRS exponent(m) with varying strain rate for different strain levels in an aluminum alloy.[106].....	20
Figure 2.9: Comparison of the AV and the SRS (m) for Ni and Ni alloys for different stress and SR [107].	21
Figure 2.10: Influence of twin spacing on the (a) SRS and (b) Activation Volume (AV) for nanotwinned Cu [110]	22
Figure 2.11: Influence of the twin density and twin spacing on the SRS of TWIP steel.[112].....	23
Figure 2.12: Influence of grain size on high SFE material on (a) Stress-Strain Curve (b) Work hardening rate [78].....	25
Figure 2.13: Influence of grain size on low SFE material on (a) Stress-Strain Curve (b) Work hardening rate [78].....	25
Figure 2.14: Variation of twinning stress in the FCC materials as a function of slip length at a constant strain imposed.[78]	27
Figure 2.15: Schematic representation of the Hall-Petch relationship for twinning and dislocation slip in coarse grain size materials.[124]	27
Figure 2.16: Deformation mechanism model in micro and nanocrystalline materials with varying SR and its contribution to strength. [137]	29
Figure 2.17: Effect of SRS on the grain size for (a) FCC-Cu[100] (b) FCC-Ni[100] (c) Various BCC metals[100] (d) Various HCP metals [145].	30
Figure 2.18: Single die drawing machine with the wire being pulled by the rotating drum and the cross-sectional area of the wire being reduced by the die placed in the die holder. [23].....	32
Figure 2.19: (a) Schematic view of the wire being pulled by the die and the representation of various forces [33]. (b) Free body diagram showing the primary forces acting on the wire drawing process [154]	32
Figure 2.20: Drawability limit curve [31]	34
Figure 2.21: Sample directions and symmetry to study the texture in wire samples. [161]	37
Figure 2.22: Tensile strength vs. Temperature for the Cu wire drawn with different die angles. [22]...	39
Figure 2.23: EBSD IPF maps for the 0.178mm MP35N wire from different suppliers, different UTS and different grades of raw material [7, 17].....	41
Figure 2.24: IPF maps comparison of the texture components of the standard and the NDR MP35N wire [162]	42
Figure 2.25: Volume fraction comparison of the texture components of the standard and the NDR MP35N wire [162].....	42
Figure 2.26: OIM maps and pole figures comparison of the 37% CW sample in (a) As drawn (b) As drawn wire subjected to 700°C for the 60s [163].....	43
Figure 2.27: OIM maps and pole figures comparison of the 60% CW sample in (a) As drawn (b) As drawn wire subjected to 700°C for the 60s [163].....	43
Figure 2.28: Area fractions of the (a) <111> and (b) <100> texture components in MP35N wire samples subjected to different CW% as a function of the aging temperature [163].....	44
Figure 2.29: OIM maps of the wire annealed at (a, c) 800C (b, d) 900C; (a, b) is in the transverse direction (c, d) is in the longitudinal direction [165].....	45
Figure 2.30: Example of a macroscopic fatigue crack and its growth from tensile to shear mode.[169]	47
Figure 2.31: Formation of extrusions/intrusions along the slip bands in materials.[168]	48

Figure 2.32: Formation of PSB and the PSB gliding through the GB with the increased stress forming intrusions and extrusions along the GB.[174]	48
Figure 2.33: Mechanisms of PSB crack formation in a Cu single crystal. [175]	49
Figure 2.34: Fatigue Crack mechanisms along the GB for Cu material (a) Micro-crack formation at the interface between the PSB and the HAGB (b) Intergranular cracking along the GB.[175].....	50
Figure 2.35: Influence of SFE and DSF on the TB cracking for Cu alloys with different SFE.[178]	51
Figure 2.36: TB cracks formation in a Cu-8%Al and Cu-16% Al.	51
Figure 2.37: Different regimes of fatigue life and the relevant factors [169]	53
Figure 2.38: S-N diagram showing the variation of the stress amplitude to the number of cycles[207].	53
Figure 2.39: Strain life-based approach for prediction of fatigue life.[207]	54
Figure 2.40: Fatigue limit of the material based on the no of cycles to failure [208]......	55
Figure 2.41: No of cycles required for crack initiation and propagation as a function of total stress amplitude [209].	55
Figure 2.42: Stages of fatigue crack propagation with varying ΔK [211].	57
Figure 2.43: Crack growth data for an MP35N material subjected to different stress ratio [212]......	58
Figure 2.44: S-N curve comparison between 35NLT and MP35N [213]	59
Figure 2.45: Inclusions in MP35N material (a)Cracked TiN (b) De-bonded TiN (c) Al_2O_3 particle [214]	60
Figure 2.46: S-N curve for the (a) As-drawn 0.101mm 35NLT wire (b) Annealed 0.101mm wire.[164, 165].....	61
Figure 2.47: Fracture zone of (a) RBBF test sample (b) TTF test sample [164].....	62
Figure 3.1: Multiple die drawing machine with the die bed and the stepped cone. (Ref: Wire drawing machine at Singapore-Heraeus facility)	67
Figure 3.2: Schematic of an FDD drawing Process	68
Figure 3.3: Schematic of an HDD drawing Process.....	68
Figure 3.4: Schematic representation of the wire cross-section with the indentation points.	69
Figure 3.5: (a) Schematic representation of the wire (shown in purple) connected between the two terminals (b) Detail view of the 4-point terminal shown in RED.	70
Figure 4.1: Comparison between the FDD and the HDD wire samples (a) σ_{YS} (Yield Strength) (b) σ_{UTS} (Ultimate Tensile Strength) (c) EL%	75
Figure 4.2: True stress-strain curve for FDD drawn wires subjected to various CW% (a) FDD (b) HDD	75
Figure 4.3: FDD vs. HDD Work Hardening curves for the wires subjected to different CW%	76
Figure 4.4: (a) EBSD band contrast image of the 0.62 mm wire (b) EBSD grain analysis distribution (histogram) of the 0.62 mm wire	77
Figure 4.5: Inverse Pole figures (IPF) orientation map of the 0.62 mm wire along the (a) X-Direction (b) Y-Direction (c) Z-Normal Direction (Wire Axis).....	77
Figure 4.6: Pole figure of the 0.62 mm wire along the three-crystallographic axis	78
Figure 4.7: MOD (Misorientation Distribution) histogram across the different grains in (a) Transverse direction (b) Longitudinal Direction.	79
Figure 4.8: FESEM -BED Images of wires drawn to 16% CW (a) FDD wire (b) HDD wire.	80
Figure 4.9: Grain size histogram of the wires drawn with 16% CW (a) FDD wire (b) HDD wire.	80
Figure 4.10: MODF histogram for the wires deformed to 16% CW (a) HDD sample (b) FDD sample.	81
Figure 4.11: Pole figure for the as drawn samples drawn to 16% CW (a) FDD wire (b) HDD wire.	82
Figure 4.12: FESEM -BSE image of the FDD drawn sample at 50% CW (a)7000X (b) 30000X.....	83
Figure 4.13: Inverse Pole figures orientation map for the HDD sample subjected to 50% CW along the (a) X-Direction (b) Y-Direction (c) Z-Normal Direction (d) Pole Figure Scattered data.....	84
Figure 4.14: 50% HDD CW sample (a) Grain size histogram (b) MODF histogram.	84
Figure 4.15: FESEM-BSE Image of the drawn sample at 75% CW (a) FDD (b)HDD.....	86
Figure 4.16: Work hardening curve comparison: FDD vs HDD for (a) 50% CW (b) 75% CW (c) 95% CW.....	89
Figure 5.1: Microstructure of the as-received wire (a) TEM BF Image (b) STEM image	94
Figure 5.2: Hardness distribution comparison of the FDD vs HDD wire samples subjected to various CW% (a) 0-50% CW Axis-1 (b) 75-95% CW-Axis-1 (c) 0-50% CW Axis-2 (d) 75-95% CW Axis-2.	95
Figure 5.3: EBSD Orientation Map of the FDD 16% CW sample (a) Surface of the wire (b) Center of the wire along the drawing direction	96
Figure 5.4: Grain Histogram of the 16% CW FDD sample (a) Surface of the wire (b) Center of the wire	97

Figure 5.5: FESEM images of the HDD 16% CW sample (a) Surface of the wire (b) Center of the wire (c) GH-Surface of the wire (d) GH-Center of the wire.	97
Figure 5.6: Orientation Distribution Function (ODF) of 16% CW wires at $\phi_2=45^\circ$ (a) FDD-Surface of the wire (b) FDD-Center of the wire (c) HDD- Surface of the wire (d) HDD-Center of the wire.....	98
Figure 5.7: FESEM Images for the 50%CW FDD sample (a) Surface of the wire (b) Center of the wire.	99
Figure 5.8: EBSD images of the 50%CW HDD sample (a) Surface of the wire (b) Center of the wire (c) GSD-Surface (d) GSD-Center (e) ODF plot-Surface (f) ODF plot-Center	101
Figure 5.9: FESEM -BSE Images of the 75% CW samples (a) FDD-Surface (b) FDD-Center (c) HDD-Surface(d) HDD-Center	102
Figure 5.10: Variation in UTS and electrical conductivity for the various CW% reduction.	105
Figure 5.11: TEM bright-field (BF) image showing the number of dislocations in the (a) FDD wire (b) HDD wire samples drawn to a similar cold work (CW) of 50%.....	105
Figure 5.12: IF comparison plots between the FDD and HDD wires drawn to different CW%.	107
Figure 5.13: Schematic drawing of the wire drawing process.	107
Figure 5.14: TEM BF Image of the 95% CW drawn wires (a) FDD (b) HDD.....	109
Figure 6.1: Engineering stress-strain curves of the wires subjected to different CW% and drawn with different drawing practice.	114
Figure 6.2: Influence of drawing practice on the (a) Calculation of γ from Ln (True Stress) vs Ln (True Strain) for a 50% CW drawn wire (b) Strain hardening exponent (γ) plotted for different CW% (c) Calculation of m from Ln(True Stress) vs. Ln (True Strain Rate) for a 50% CW drawn wire (d) Strain rate sensitivity (m) plotted for different CW%.	115
Figure 6.3: FIB sample preparation of the as-received sample (a) Pt deposit onto the bulk area (b) Omniprobe Lift out Technique (c) Final polished image of the milled lamellae.	116
Figure 6.4: TEM images of the annealed wire (a) Thick annealing twins and SAD inset of the twin (b) Thin annealing twins.....	117
Figure 6.5: EDX pattern of the as-received sample.	118
Figure 6.6: Plastic Instability comparison of the FDD and HDD drawn wires to a similar CW reduction.	118
Figure 6.7: Comparison between the FDD and HDD wires (a) Ductility (EL%) (b) Reduction in cross-sectional area (RA%).	118
Figure 6.8: TEM images of the FDD wires drawn to 50% CW reduction (a) Grain size STEM image (b) Grain Size Distribution (c) Bright Field (BF) TEM Image showing highly deformed microstructure (d) BF TEM image showing the dislocation density of the sample. (e) Darkfield (DF) image showing macro twins (f) Bright Field (BF) Image with SAD inset showing the macro twins. (g) BF image with SAD inset showing the micro twins (h) BF image with SAD inset showing the nano twins at the grain boundary.	124
Figure 6.9: TEM images of the HDD wires drawn to 50% CW reduction (a) Grain size STEM image (b) Grain Size Distribution (c) Bright Field (BF) TEM Image showing less deformed microstructure (d) BF TEM image showing the dislocation density of the sample. (e) Darkfield (DF) image (f) Bright Field (BF) Image with SAD inset showing the presence of slip bands. (g) BF image with SAD inset showing the misorientations in the grains (h) BF image with SAD inset showing misorientations at the grain boundary.	126
Figure 6.10: TEM images of the FDD wires drawn to 75% CW reduction (a) Grain size STEM image (b) Grain Size Distribution(c) Bright Field (BF) TEM Image showing highly twinned microstructure (d) BF TEM image showing the dislocation density and twinned microstructure of the sample. (e) Darkfield (DF) image showing macro twins with reduced twin spacing (f) Bright Field (BF) Image showing the closely spaced macro twins. (g) BF image with SAD inset showing the high density of micro twins (h) BF image with SAD inset showing the increased density of nano twins in the microstructure and formed inside the grains.....	128
Figure 6.11: TEM images of the HDD wires drawn to 75% CW reduction (a) Grain size STEM image (b) Grain Size Distribution (C) Bright Field (BF) TEM Image showing the increased deformed microstructure (d) BF TEM image showing the increased dislocation density of the sample. (e) Darkfield (DF) image showing the dislocation twins (f) Bright Field (BF) Image showing the increased dislocation density in the microstructure. (g) BF image with SAD inset showing the twinned microstructure with the dislocated structure (h) BF image with SAD inset showing different misorientations at the grain boundary and within the grain.	130

Figure 6.12: TEM images of the FDD wires drawn to 95% CW reduction (a) BF Image showing the shear bands formed in the microstructure with fewer twins (b) BF TEM image showing the grain size and the shear bands microstructure aligned to the drawing direction. (c) Grain Size Distribution(d) High magnification image of the shear band with SAD inset confirm the texture profile for the bands.	132
Figure 6.13: TEM DF images of the FDD wires drawn to 95% CW reduction (a) Multiple thin shear bands originated in the microstructure (b) High magnification image of the shear bands intersecting the twins and the dislocations (c) Shear bands formed are 20-30nm in thick which are relatively equal to the grain size in the sample.	133
Figure 6.14: : TEM images of the HDD wires drawn to 95% CW reduction (a) Grain size BF image (b) Bright Field (BF) TEM Image showing the microstructure with fewer twins and dislocation annihilated microstructure (c) BF TEM high magnification image showing the increased dislocation annihilation in the cell structure.	135
Figure 6.15: TEM images of the HDD wires drawn to 95% CW reduction (a) DF image showing the highly distorted structure with less twin density. (b) BF TEM image showing the dislocation penetrating the grains through its grain boundaries (GB). (c) Triple junction formed at the intersection of the three GB.	136
Figure 7.1: True Stress-True Strain curves for the wire subjected to different strain rates (a) FDD (b) HDD	141
Figure 7.2: Influence of strain rate on the strain hardening rate of (a) FDD wire (b) HDD wire.....	142
Figure 7.3: SRS (m) comparison for the FDD and HDD drawn wires subjected to various strain rates.	143
Figure 7.4: Microstructure of the annealed wire (a) IPF (Inverse Pole Figure) (b) Grain Size Distribution	144
Figure 7.5: TEM images of the annealed wire (a) Bright Field Image (b) SAD Pattern	144
Figure 7.6: Microstructure of the as drawn FDD wire (a) STEM image showing the grains (b) Grain size distribution (c) BF TEM image showing the deformed microstructure (d) BF TEM image with the SAD inset showing the twins between the grains.....	145
Figure 7.7: Microstructure of the as drawn HDD wire (a) EBSD IPF image (b) Grain Size Distribution (c) BF TEM Image (d) DF TEM Image.	146
Figure 7.8: Fracture Sample after a tensile test (a) Cutting position for the FIB (b) Magnified Image of the FIB cut sample.	147
Figure 7.9: Influence of strain rate on the (a) ductility (elongation%) (b) True fracture strain of the FDD and HDD drawn wires.....	148
Figure 7.10: TEM images of the FDD drawn wire inflicted to an SR of 10^{-6}s^{-1} (a) BF TEM Image of the deformed microstructure (b) BF TEM showing the dislocation pile up at the GB (c) TEM BF image showing the dislocation annihilation at the GB (d) BF TEM image showing the severely annihilated dislocation microstructure.	150
Figure 7.11: TEM images of the HDD drawn wire inflicted to an SR of 10^{-6}s^{-1} (a) BF TEM image of the microstructure (b) BF TEM image of the parallel deformation bands (c) High magnification image of the deformation hands (d) SAD pattern of the deformation band showing the presence of strong texture.	151
Figure 7.12: TEM images of the FDD drawn wire drawn to different strain rates (a) 10^{-5} (b) 10^{-4} (c) 10^{-3} (d) 10^{-2}s^{-1}	152
Figure 7.13: TEM images of the HDD drawn wire drawn to different strain rates (a) 10^{-5} (b) 10^{-4} (c) 10^{-3} (d) 10^{-2}s^{-1}	153
Figure 7.14: STEM image of the 10^{-2}s^{-1} of the HDD drawn wire (a) HADDF STEM image indicating the dark bands in the deformed microstructure along with the EDX points (b) HADDF STEM image of the dark area inside the slip band (c) High magnification image of the area indicated in red (d) EDX spectrum for the point 1.	157
Figure 7.15: Effect of twin thickness and twin spacing of the FDD draw wire with differing strain rate (a) 10^{-6} (b) 10^{-4} (c) 10^{-3} (d) 10^{-2}s^{-1}	159
Figure 7.16: Fracture samples of the FDD drawn wires deformed to various SR (a) 10^{-6}s^{-1} (b) 10^{-5}s^{-1} (c) 10^{-4}s^{-1} (d) 10^{-3}s^{-1} (e) 10^{-2}s^{-1}	161
Figure 7.17: Fracture samples of the HDD drawn wires deformed to various SR (a) 10^{-6}s^{-1} (b) 10^{-5}s^{-1} (c) 10^{-4}s^{-1} (d) 10^{-3}s^{-1} (e) 10^{-2}s^{-1}	162
Figure 8.1: RBFT fatigue test setup used for the current experiments.	167
Figure 8.2: Inverse pole figure maps (a) 0.141 annealed wire (b) 0.101 as-drawn HDD wire (c) 0.101 as-drawn FDD wire.	168

Figure 8.3: Tensile properties comparison between the FDD and HDD drawn wires drawn to different CW% (a) UTS (b) RA and EL% .	169
Figure 8.4: S-N curve comparison between FDD and HDD wires drawn to (a) 50% CW (b) 75%CW (c) 95%CW.	171
Figure 8.5: TEM post fatigued FDD LCF sample (a) Microstructure of the fatigued sample (b) Deformation cells formed in the microstructure (c) Nano twins observed at the area between the dislocation pileup and the GB.	175
Figure 8.6: TEM post fatigued HDD LCF sample (a) Microstructure of the fatigued sample (b) Dislocation pile-up along the slip band (c) Absence of nano twins at the interface between the dislocation-pile up and the GB.	176
Figure 8.7: TEM post fatigued FDD HCF sample (a) Microstructure of the fatigued sample (b) Deformation twins in the microstructure (c) Dislocations being obstructed at the TB (d) Dislocation pileup at the TB.	177
Figure 8.8: TEM post fatigued HDD HCF sample (a) Microstructure of the fatigued sample (b) Fewer dislocations observed in the microstructure (c) Fewer TB with no dislocation pile-up (d) Surface crack blocked at the GB.	178
Figure 8.9: TEM post fatigued FDD 75%CW LCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1350MPa (b) Magnified image of the dislocation cell structure highlighted in red box (c) Microstructure of the fatigued sample put to a stress amplitude of 850MPa (d) Magnified image of the dislocation cell structure in 8.9(c)	181
Figure 8.10: TEM post fatigued HDD 75% CW LCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1350MPa (b) Magnified image of the dislocation cell structure having cross-slip in 8.10(a) (c) Microstructure of the fatigued sample put to a stress amplitude of 850MPa (d) Dislocations crossing the TB, whereas the PSB terminating at the TB	182
Figure 8.11: TEM post fatigued FDD 75% CW HCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 685MPa (b) Higher twin density in the sample subjected to a stress amplitude of 685MPa (c) Lower twin spacing and thickness in the sample subjected to a stress amplitude of 430MPa.	184
Figure 8.12: TEM post fatigued HDD 75% CW HCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 685MPa (b) Higher twin density in the sample subjected to a stress amplitude of 685MPa (c) Lower twin spacing and thickness in the sample subjected to a stress amplitude of 430MPa.	185
Figure 8.13: TEM post fatigued FDD 95% CW LCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1550MPa (b) Shear bands observed in the fatigued microstructure (c) High magnification image of the shear bands intersecting with the TB and the dislocations piling up at the SB d) DF image of the shear bands.	187
Figure 8.14: TEM post fatigued HDD 95% CW LCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1550MPa (b) Dislocation Cell (DC) structure with a high pile-up of the dislocations at the walls of the DC (c) Nanotwinned structure and twins hindering the movement of the dislocations.	188
Figure 8.15: TEM post fatigued FDD 95% CW HCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1110MPa (b) Deformation twins observed in the fatigued microstructure (c) Tangled dislocation structure and several pile-up of dislocations at the GB d) Dislocations moving from the grain exterior to the interior.	190
Figure 8.16: TEM post fatigued HDD 95% CW HCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1110MPa (b) IDS and several pile-up of dislocations at the GB (c) Deformation twins observed in the fatigued microstructure with a reduced twin thickness d) High magnification image of the dislocation debris accumulated at the GB shown in (b).	191
Figure 8.17: Fracture surfaces of the as-drawn FDD wire subjected to a 50% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1170MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 670MPa (c) High magnification image of the striations in the LCF sample. (d) High magnification image of the HCF sample showing the striation and steps formation and the crack deflection mechanisms.	193
Figure 8.18: Fracture surfaces of the as-drawn HDD wire subjected to a 50% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1170MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 670MPa (c) High magnification image of the LCF wire exhibiting a cleavage fracture (d) High magnification image of the HCF wire showing the microcracks and the cracks propagating as a transgranular through the grains	194

Figure 8.19: Fracture surfaces of the as-drawn FDD wire subjected to a 75% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1350MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 430MPa (c) High magnification image of the LCF sample with visible striations (d) High magnification image of the HCF sample showing the formation of tongue steps. (e) High magnification image of the inset in (b) showing the shear lips formation at the circumference of the wire.	195
Figure 8.20:: Fracture surfaces of the as-drawn HDD wire subjected to a 75% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1350MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 430MPa (c) High magnification image of the LCF sample showing a visible crack extending from region 1 to region 2 (d) High magnification image of the LCF sample showing cleavage fracture mechanisms and formation of extensive microcracks in the sample . (e) High magnification image of the HCF sample exhibiting the step formed in the sample.....	196
Figure 8.21: Fracture surfaces of the as-drawn FDD wire subjected to a 95% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1550MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 1110MPa (c) High magnification image of the LCF sample exhibiting a shear mode fracture with many brittle facets (d) High magnification image of (c) exhibiting transgranular micro cracks fracture within the cracks initiating from multiple sites (e) High magnification image of the HCF sample exhibiting no striations and a micro cracked structure.	197
Figure 8.22: Fracture surfaces of the as-drawn HDD wire subjected to a 95% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1550MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 1110MPa (c) High magnification image of the LCF sample (a) near the yellow box showing the crack propagation through the GB (d) High magnification image of crack in image (c) showing fracture (e) High magnification image of the HCF sample exhibiting striations.	198
Figure 8.23: SEM analysis of the fractured outlier specimens drawn with different drawing practices (a) 50% CW HDD wire subjected to a stress amplitude of 586MPa displaying the presence of Al ₂ O ₃ inclusion at the fractured area. (b) 50% CW FDD wire (Sample 1-349,599 cycles) subjected to a stress amplitude of 721MPa, displaying the presence of MgO inclusion at the fractured area. (c) 50% CW FDD wire (Sample 5-333,849 cycles) subjected to a stress amplitude of 721MPa, shows a possibility of fallout inclusion at the fractured area. (d) 75% CW HDD wire subjected to a stress amplitude of 760MPa displayed a longitudinal split at the fractured area. (e) 75% CW FDD wire subjected to a stress amplitude of 685MPa fractured at 27M cycles due to crack originating from deep drawing lines on the wire surface. (f) 95% CW HDD wire (Sample 2-1,529,617 cycles) subjected to a stress amplitude of 1111MPa, shows a possibility of fallout inclusion at the fractured area. (g). 95% CW FDD wire (Sample 3-5,164,549 cycles) subjected to a stress amplitude of 1111MPa, shows a possibility of fallout inclusion at the fractured area.....	205

List of Tables

Table 2-1: Chemical Compositions (in Weight Percent) of MULTIPHASE Co-Ni alloys.[12]	9
Table 2-2: As drawn fiber wire texture drawn with different die angles [22].....	39
Table 2-3: Texture components and volume fraction of MP35N wires [7]	41
Table 2-4: Chemistry comparisons between the standard MP35N and 35NLT alloy [213]	59
Table 3-1: Chemical composition of the alloy as per, ASTM F 562 [217]	66
Table 7-1: EDX quantification analysis of the points (P1-P6) indicated in Figure 7.14	157
Table 8-1: Mechanical properties of the annealed wire at different diameters.	167
Table 8-2: Comparison of the grain boundary characteristics between FDD and HDD 50%CW wire	173
Table 8-3: List of outliers observed during the cyclic fatigue testing of the wires drawn with different drawing practices and CW conditions.....	203

Abbreviations & Notations

A

AD- As Drawn

AV – Activation Volume

B

BC- Band Contrast

BCC-Body Centered Cubic

BED- Back Scattered Electron Detector

BF-Bright Field

BL-Bearing Length

C

CRM-Cardiac Rhythmic Management

CTB-Coherent Twin Boundary

CW-Cold Work

D

DBS-Deep Brain Stimulation

DC- Dislocation Cell

DF-Dark Field

DSF-Difference of Schmidt Factor

DT- Deformation Twinning

E

EBSD- Electron Beam Scattered Diffraction

ECAP- Equal Channel Angular Pressing

EDX/EDS- Energy Dispersive X-ray Spectrum

EL- Elongation

F

FCC- Face Centered Cubic

FCG- Fatigue Crack Growth

FCI- Fatigue Crack Initiation

FCPR- Fatigue Crack Propagation Rate

FDD- Full Die Drawing

FEA- Finite Element Analysis

FESEM- Field Emission Scanning Electron Microscopy

FFT- Fourier Fast Transformation

FIB- Focused Ion Beam

G

GB- Grain Boundary

GBD- Grain Boundary Diffusion

GBS- Grain Boundary Shearing

GH- Grain Histogram

GS- Grain Size

GSD- Grain Size Distribution

H

HAD- Has as Drawn

HAADF- High Angle Annular Diffraction

HAGB- High Angle Grain Boundary

HCF- High Cycle Fatigue

HCP- Hexagonal Closed Pack

HDD- Half Die Drawing

HP- Hall Petch

HRFESEM- High-Resolution Field Emission Scanning Electron Microscopy

I

IDS- Incoherent Dislocation Structure

IF- Inhomogeneity Factor

IPF- Inverse Pole Figure

L

LAGB- Low Angle Grain Boundary

LCF- Low Cycle Fatigue

LT- Low Titanium

M

MOD- Mis-Orientation Distribution

MODF- Mis-Orientation Distribution Function

MP- Multi Phase

N

NC- Nano Crystalline

ND- Normal Direction

NDD-Natural Diamond Dies

NDR- Nanograin Damage Resistant

O

ODF- Orientation Distribution Function

OFHC- Oxygen Free High Conductivity

OIM- Orientation Image Maps

OPS- Oxide Polishing Suspension

P

PF- Pole Figure

PSB- Persistent Slip Band

PI- Plastic Instability

R

RA- Reduction in Area

RBBF- Rotary Beam Bend Fatigue

RBT- Rotating Beam Test

RBFT- Rotary Beam Fatigue Tester

RD- Rolling Direction

RT- Room Temperature

S

SAD- Selective Area Diffraction

SB- Slip Band

SEM- Scanning Electron Microscope

SFE- Stacking Fault Energy

SHRS- Strain Hardening Rate Sensitivity

SPD- Super Plastic Deformation

SR- Strain Rate

SRS- Strain Rate Sensitivity

STEM- Scanning Transmission Electron Microscope

T

TB- Twin Boundary

TCP- Topologically Closed Packed

TD- Tangential Direction

TEM- Transmission Electron Microscope

TTF- Tension-Tension Fatigue

TWIP- Twinning Induced Plasticity

U

UHCF- Ultra High Cycle Fatigue

UTS- Ultimate Tensile Strength

V

VF- Volume Fraction

VHCF- Very High Cycle Fatigue

W

WD- Working Distance

X

XRD- X-ray Diffraction

Y

YS- Yield Strength

List of Publications

1. **GVK, S.S.,** M.J. Tan, and Z. Liu, Influence of drawing practices on the mechanical, texture and work hardening characteristics of Co-Cr-Ni-Mo wires. *Materials Science and Engineering: A*, **2018. 713:** p. 94-10.
2. **GVK, S.S.,** M.J. Tan, and Z. Liu Study on the Deformation Homogeneity and Electrical Conductivity in Co-Cr-Ni-Mo Wires Drawn with Different Drawing Practices. *Journal of Materials Engineering and Performance*, **2019. 28(1):** p. 330-342.
3. **GVK, S.S.,** M.J. Tan, and Z. Liu, Plastic Instability in Co-Cr-Ni-Mo alloy wires drawn with different drawing practices. *Materials Science and Engineering: A*, **2019. 747:** p. 80-97.
4. **GVK, S.S.,** M.J. Tan, and Z. Liu, Analysis of strain rate sensitivity and strain rate hardening in Co-Cr-Ni-Mo wires drawn with different drawing practices. *Metals and Materials International*, **2019. p.1-16.**
5. **GVK, S.S.,** Z. Liu, and M.J Tan, Fatigue behavior in Co-Cr-Ni-Mo medical alloy wires drawn with different drawing practices. *Journal of Mechanical Behavior of Biomedical Materials*, **2019. 99:** p.134-152.
6. **GVK, S.S.,** Z. Liu, and M.J Tan, Fractography study of Co-Cr-Ni-Mo alloy fatigue wires drawn with different drawing practices. *International Journal of Fatigue*, **2020, 130.**
7. **GVK, S.S.,** M.J. Tan, and Z. Liu, Homogeneous deformation in cold drawn Co-Cr-Ni-Mo wires, *The Ninth Pacific Rim International Conference: Advanced Materials and Processing (PRICM9)* Ed. by T. Furuhashi, M. Nishida and S. Miura. The Japan Institute of Metals and Materials, 2016. p.561-566.

Chapter 1 Introduction

1.1. Background of Research

The selection, design, and processing of the materials for medical applications are very important, and the relationship between their properties and their processing must be fully understood to design a final component or product as per its intended application. Most of the wires manufactured involve cold or hot drawing process, which involves cross-sectional area reduction from bar or tubes of a larger diameter which is in the range of 30 to 20 mm to a wire of smaller diameter which is in micron-scale ($>10\text{ }\mu\text{m}$). The wires are then assembled or manufactured into many auxiliary components which are implanted into the human body, for medical applications such as cardiac rhythmic management (CRM), neurostimulation, endovascular treatments and many other. Among them, the largest market share of these components is for CRM applications which involve the manufacture of leads for pacemakers.

Pacemaker technology is one of the most significant milestones in the area of medical applications [1]. Over the past five decades, millions of patients benefited from the advantages of this masterpiece of engineering. Pacemaker therapy [1] is based on the delivery of current pulses, which causes artificial depolarization of some cardiac cells. Sudden cardiac death kills up to 350,000 people a year [2]; and it is caused by a malfunction in the heart's electrical system that can occur when the lower chambers of the heart suddenly start beating in an uncoordinated fashion, preventing the heart from pumping blood out to the lungs and body. Unless the heart is shocked back into normal rhythm, the person rarely survives.

A pacemaker is a small device which is placed in the chest or abdomen of the human body, as shown in Figure 1.1, to help correct abnormal heart rhythms [3]. This device uses electrical pulses to prompt the heart to beat at a regular rate. Pacemakers are used to treat arrhythmias, which involve problems associated with the rate or rhythm of the heartbeat. During an arrhythmia, the heart can beat too fast, too slow, or with an irregular rhythm. A heartbeat that is too fast is called tachycardia, and a heartbeat that is too slow is called bradycardia [3]. A pacemaker can relieve arrhythmia symptoms, such as fatigue and fainting, and can help a person who has abnormal heart rhythms resume a more active lifestyle.

Figure 1.1: Example of a Pacemaker Implanted into the heart [4]

As shown in Figure 1.2, leads are the critical component of the pacemakers, leads are highly reliable devices, but over time due to the repeated bending and flexing inside the human body, the devices are subjected to repeated cyclic stresses which causes them to wear through their insulation or even break. Cardiac motion or any neurostimulation requires that these leads must withstand hundreds of millions of bending cycles over their life [5]. So, designing the properties of the wire per the intended application is very important for reliable performance of the device.

The material selection plays a significant role in the design of the pacemakers. Currently, implantable leads are constructed mainly of metals, and polymers being used to provide insulations to the implanted device. The critical requirements needed for the selection of material for medical applications are listed below [6]. The materials should have the following characteristics,

1. Low resistance or high conductivity.
2. High strength and flexibility.
3. High fatigue behavior.
4. Good biocompatible properties.

5. High corrosion resistance.

The following characteristics stated above are used as guidance for the selection of the raw material during the development phase of the product, and most of the materials which do not satisfy the characteristics are outset from the material list. The potential available raw materials of designers choice and which are best suited for medical applications are listed below [3, 6]. Among these materials, MP35N and their alloys have gained significant importance in the manufacture of leads for different applications.

- Stainless steels (ISO 5832/1 and AISI 316L)
- MP35N (35Co-35Ni-20Cr-10Mo) (ASTM F362 alloy), MP35NLT (35NLT)
- MP35N/Ag clad alloys or Drawn Filled Tube Alloys (DFT)
- Titanium materials, including technically pure titanium (ISO5832/2) and titanium alloys such as TiAl6V4, TiAl5Fe2.5, and TiAl6Nb4 (ISO 5832/3);
- Niobium
- Tantalum (ASTM 560-78) or MP35N/Ta alloys.
- Platinum and its alloys. (e.g. Pt/Ir-10, Pt/Ir-20)

MP35N is cobalt-based superalloy which has been used in the medical industry for more than 30 years, and it has been widely used to draw wires to manufacture leads for medical applications such as pacemakers and neurological stimulators because of their outstanding properties as described above [7-9]. The wires are manufactured by the cold wire drawing process which involves a reduction in the cross-section of the wire or diameter, by passing the wires through dies of different diameter. This process involves increasing the strength of the material and the reduction in ductility, with the increase in the cold work (CW %) or reduction in area (RA %). The leads are manufactured by the assembly of thin diameter wires or strands to form coils or a cable of a larger diameter and then post-processed to various designs based on their applications. An example of the lead coil and cable assembled from thin wires and strands are shown in Figure 1.2 and Figure 1.3.

Figure 1.2: Components of a pacing lead made from wires and coiled to a lead

Figure 1.3: Components of a pacing lead made from strands and coiled to a cable [3]

Although MP35N is widely used in the medical industry, the relationship between the process-microstructure-property has not been well established and understood, which plays an essential role in the design of the wires for the final product applications. To date, most of the research done or experimented by many investigators on this material is on bulk samples [10-16] in tube and rod forms, where the processing techniques are significantly different when compared to the processing of thin wires. The technique used to manufacture fine wires contributes to a different microstructure and mechanical properties in the wire which needs a detail characterization. Secondly, the type of plastic deformation and work hardening imparted to the material by the wire drawing process, also depends upon the intrinsic material property such as stacking fault

energy (SFE), which determines the material response to the generation of lattice defects like dislocations and twins [17-21], to achieve the final desired properties.

In the last 30-40 years, several investigations have been performed to study the influence of drawing factors such as reduction angle [22, 23], drawing speed [24-26], and die geometry [27-29] on the mechanical properties of high SFE material wires like copper. However, limited work has been done to examine the influence of drawing parameters on the low SFE materials like MP35N in wire form.

Several researchers have reported numerous analytical theories and models to understand the mechanics [23, 30-35] involved during the wire drawing process. Based on the prior research findings, it can be concluded that the amount of plastic deformation or the total reduction significantly influences the mechanical properties [10, 23, 26, 32, 36-38], work hardening [34, 39, 40], hardness distribution [28, 29, 31, 41, 42], and also on the texture and microstructural characteristics of the wire [18, 20, 21, 43-51] .

Current techniques in the wire drawing industry involve two types of drawing practices which can be categorized as (a) Half Die Drawing (HDD) where the cross-sectional reduction per pass is between 6-10% and (b) Full Die Drawing (FDD) where the cross-sectional reduction per pass is higher than 20%. To the best of the author's knowledge, there were no prior studies carried out on MP35N or 35NLT wires, enacting a relationship between the process, properties, and performance of the wires.

So, in the current study, the influence of drawing practices (FDD vs. HDD) on the mechanical, electrical, work hardening, microstructure, and fatigue properties of MP35NLT wires are investigated. There were also no prior reports, examining the strain rate sensitivity (SRS) and plastic instability (PI) response of MP35NLT material, to its plastic deformation and strain rate (SR), which is very important from a manufacturing point of view.

1.2. Research objective and scopes

The objectives of the current research are:

1. To investigate the influence of the drawing practice (FDD vs HDD) on the mechanical properties of MP35NLT wires such as, Yield Strength (Y_s), Ultimate Tensile Strength (UTS), ductility (EL%), and to understand the work hardening capability and capacity of the material and correlate their properties and hardening response to its crystallographic texture and grain size.
2. To understand the deformation homogeneity, inhomogeneity factor (IF) and electrical conductivity of MP35NLT wires subjected to different drawing practices, by measuring the hardness distributions and electrical conductivity along the wire cross-section and correlate to their microstructure, texture and grain size distribution.
3. To evaluate the plastic instability (PI) of the MP35NLT wires drawn with different drawing practices and with different CW%, and correlate its deformation response to intrinsic properties, such as dislocation density, twin density, twin spacing, and grain size.
4. To understand influence of strain rate (SR) on the strain rate sensitivity (SRS) and strain hardening rate sensitivity (SHRS) of MP35NLT wires by subjected the wires to different strain rates in the range of 10^{-6}s^{-1} to 10^{-2}s^{-1} and interpreting the achieved mechanical results to microstructural information characterized by scanning electron microscope (SEM), electron beam scattered diffraction (EBSD) and transmission electron microscopy (TEM).
5. To investigate the influence of drawing practices, on the fatigue response and behavior of MP35NLT wires, and compare and plot the S-N curves and link their performance to the observed microstructural features such as dislocation structures, grain size, fracture surfaces and crack morphology.

1.3. Thesis Outline

Chapter 1 provides the background of pacemaker technology, its usage, and the requirements in the selection of the material for the desired application. It discusses as for why MP35N material is used mostly for pacemakers when compared to other materials, and as of how the material is being processed into thin wires, to manufacture the final product. The formability techniques of the material are discussed, and the objectives and scope of this research are defined.

Chapter 2, comprises a comprehensive literature review of the MP35N material, the inter-relation between the material properties to the SRS and SRWH, for various materials, the mechanics of the wire drawing and its relation to hardening and texture formation in FCC materials, discussing the relationship between microstructure and fatigue, and the prior research conducted on fatigue performance of MP35N wires. The key findings from the current state of research and the research gaps in the processing of MP35N material are outlined and discussed.

Chapter 3 lists the material and experimental details, test methods and conditions, characterization techniques, and the sample sizes used in the current scope of the study.

Chapter 4, details the influence of drawing practices on the mechanical, work hardening and texture in MP35N wires, and the difference in the properties observed are explained by relating the microstructure variables such as grain size, dislocation density and twins to its yield strength (σ_{YS}), ultimate tensile strength (σ_{UTS}), and strain hardening.

Chapter 5, outlines the study of the deformation inhomogeneity in MP35N wires, by comparing the hardness gradient across the wire, and calculating the inhomogeneous factor (IF), and correlating the variation to its observed microstructural differences in the wire. The electrical properties of the wire were also analyzed, and the relation between the mechanical and electrical properties are disclosed.

Chapter 6, analyses the plastic instability of MP35N wires, by calculating the SRS and the SRWH through monotonic tensile tests and interpreting the results to the observed differences in the intrinsic microstructural variables such as dislocation density, twin spacing and width, shear bands formation and their mechanisms.

Chapter 7, discusses the effects of strain rate (SR) on the SRS and SRWH in MP35N wires, which are subjected to uniaxial tensile tests by varying the SR in the range of 10^{-6}s^{-1} to 10^{-2}s^{-1} , and determining the effect of prior deformation of the material on the hardening and load-bearing resistance of the material.

Chapter 8, examines the influence of the drawing practices on the low cycle fatigue (LCF) and high cycle fatigue behavior (HCF) of MP35N wires drawn to different CW reductions, and the fatigue performance has been corresponded to its fracture surface and cracks morphology. The cyclic deformation response has been connected to the changes in the microstructure observed such as dislocation structures, twin spacings, and formation of slip and shear bands. Finally, the summary of conclusions and recommendations for future work are given in Chapter 9.

Chapter 2 Literature review

2.1 MP35N Composition and its Phase Transformations

MP stands for “MULTIPHASE” alloys and is a trademark of SPS Technologies, Inc. The alloy MP35N (35% Ni-35% Co-20% Cr-10% Mo) is a cobalt-nickel based superalloy which was first developed by Smith in 1968 [16]. Alloys in which Co, Ni, Cr, and Mo are the main components are also referred to as “Co-Ni- based superalloys.” This alloy is characterized by a remarkable combination of ultrahigh strength, high ductility, and high resistance to fracture combined with high corrosion resistance [6, 52-54]. The family of the alloys which have been developed along with MP35N as listed in Table 2-1 have been used in many other industries for their capability of high strength retention suited for high-temperature applications [12].

Table 2-1: Chemical Compositions (in Weight Percent) of MULTIPHASE Co-Ni alloys.[12]

	Co	Ni	Cr	Mo	Ti	Nb	Fe	Al	C
MP35N	35	35	20	10	0	0	0	0	<0.02
MP159	35.6	25.5	19	7	3	0.6	9	0.2	<0.02
MP210	36.3	30.9	19.4	7.3	3.8	1.2	1.04	0	<0.02

These alloys, despite their high costs, have such valuable properties that they are widely used for critical applications, such as aerospace fasteners, oil extraction and underwater drilling equipment's, and many other applications. The alloys were initially designed from the FCC (α) $\sim$ HCP (ϵ) (FCC, face-centered cubic; HCP, hexagonal close-packed) martensite phase transition that occurs on cooling pure cobalt below 420⁰C [55]. Addition of nickel stabilizes the FCC phase and depresses the martensite start temperature, M_s , so that at 35% Ni, the M_s falls below ambient temperature. The molybdenum and chromium additions have the opposite effect, as they both raise the M_s in the binary Co-Mo and Co-Cr alloys, and Mo is known to stabilize the HCP phase in the Co-Mo binary system [12, 13]. Among all the MP alloys listed in Table 2-1, MP35N alloy was found to be the best material of choice for medical applications due to its excellent biocompatibility, high corrosion resistance and non-magnetic features besides its high strength and toughness [6, 9, 52].

MP35N, when fully annealed in the range of 1000-1050⁰C for 60 minutes exhibits an FCC structure with a grain size of 30-40 μm , as shown in Figure 2.1. The physical metallurgy of the cobalt-based alloys is very interesting due to its blending with different elements having different phase structures. However, very little is being understood on its phase transformations and their contributions to the properties at various temperatures and deformations, till date for its inherent complexity in the crystallography of the alloying elements used.

Figure 2.1: Microstructure of annealed MP35N alloy [56]

As per Raghavan [57, 58] who first proposed the transformation curve as shown in Figure 2.2, there was evidence of two phases in the material, with the increasing percentage of cobalt, the transformation of FCC phase to HCP phase is seen with the increase of temperature until 327⁰C. Between 327-827⁰C, the material exists as an HCP phase and between 827-1027⁰C, it exists as a mixture of FCC+HCP phase, and above 1027⁰C, it exists as a single FCC phase. However, X-ray diffraction (XRD) and TEM studies on the deformed structure [58] at room temperature or the deformed and heat-treated alloy could not detect any HCP (ϵ) phase which contradicts with the below phase diagram.

Figure 2.2: Phase transformation of MP with varying % Co [57]

Recent work by Li, B.Q et al., [59] have also calculated a pseudo-binary MP35N phase diagram using a thermo-calc software and the Ni-database as shown in Figure 2.3.

Figure 2.3: Co-Ni pseudo-binary phase diagram [59]

Since MP35N has four alloying elements, the phase diagram was calculated as Co versus Ni while Cr and Mo were fixed at 20 wt.% and 10 wt.% respectively. The

area of the phase diagram corresponding to MP35N alloy is at 35% Ni in the diagram. The phase diagram shows that when the temperature is above 350°C, the alloy exists as a two-phase region, namely the FCC and μ phase. Below 350°C, the diagram predicts a three-phase region, i.e. FCC + μ phase + σ phase. The dissolution temperature of μ phase at 35 wt.% Ni (MP35N) is 980°C in Figure 2.4. The μ and σ phases are topologically close-packed (TCP) phases which are a source of intermetallic compounds with complex crystallographic structures. Based on phase diagram calculation, the μ and σ phases are energetically preferable to form in MP35N. TCP phases have been widely observed in alloys such as Fe-Cr-Ni-Mo-W, Ni-Cr-Mo-W-Co-Al, and other refractory metal superalloys. The μ and σ phases have been reported in Ni-based superalloys [60, 61] and the μ phase was found to precipitate at elevated temperatures and had a plate-like shape.

Figure 2.4: DTA curve of MP35N showing the dissolution temp as 980°C [59]

The phase diagram constructed in Figure 2.2 looks different from Figure 2.3, and as per the findings of the researchers [16, 57, 59, 62], they have concluded that there is a precipitation reaction occurring in the temperature range of 140°C to 480°C as indicated by the μ phase, in the matrix. However, TEM observations and Selective Area Diffraction (SAD) patterns [10-14, 58, 63, 64] on the deformed, and the heat-treated samples did not exhibit any precipitates in the matrix, and only highly dense platelet-like features are observed. So, based on the above experimental and

microstructural data analyzed, the phase transformation of the MP35N alloy is still very unclear and ambiguous, and no objective evidence of the existence of the HCP phase in the alloy is obtained.

2.2 Strain Hardening and Strain Rate Sensitivity (SRS) in metals

Strain hardening [65] is one of the most widely used techniques for providing high strength to metallic components which involves subjecting the material to severe plastic deformation. The method of plastic deformation in metals involves extensive movement and interaction of the atoms along the crystallographic planes called the slip planes. The slip plane is the plane with the highest atomic density, and the slip direction is the close-packed direction along which the slip propagates or moves. Slip happens when the shear stress on a particular slip in a particular slip direction reaches a maximum value called the critical resolved shear stress [66]. One of the chief characteristics of the plastic deformation is that the shear stress required to produce slip continuously increases with the increasing shear strain and the increase in stress required to cause slip because of the prior plastic deformation is called as strain or work hardening. At the microscale, this aspect of plastic deformation is intrinsically coupled with all other aspects of plastic deformation, such as the development of preferred lattice orientations, the formation of localized shear bands, the formation of sub-grains, and development of grain-scale residual stresses [31]. The strain hardening in a material can be determined by a simple Holloman law [67] as $\sigma = K\varepsilon^\gamma$, where σ is the strength of the material, K is the strength coefficient, and γ is the strain hardening coefficient which varies from 0.1 to 0.6 for most metals. As the γ value varies, the shape of the true stress-strain curve also varies. Materials with high strength or mechanically stronger materials have low γ and vice versa[68].

However, this is applicable only when the material is free from lattice defects, with the increase of the lattice defects in the material such as twins and dislocations, the γ value varies. There were modifications done to the power-law by Ludwik [69] and Swift [70] by adding the stress and strain constants, but the equations do not include the effect of the prior deformation, so an empirical equation was defined by Voce[71] which includes the saturation stress, and handled large strains. The strain hardening rate is defined as $\gamma = (\partial\sigma/\partial\varepsilon)\dot{\varepsilon}$, which is obtained from the slope of the stress-

strain diagram. If γ is low, the hardening rate is initially high, but it decreases with the increase of strain, however, if γ is high, the hardening rate is initially low, but increases with the increase of strain.

The other important parameter in defining the rate-dependent behavior of the metals when subjected to plastic deformation is the Strain Rate Sensitivity (SRS). It defines or measures the resistant of the material to plastic necking, and it describes how a material will behave when inflicted to different deformation strains and strain rates (SR). The SRS is also mathematically obtained from the power-law relationship of stress versus strain rate and is defined [72] by the equation $\sigma = \beta \cdot \dot{\epsilon}^m$, where m is the SRS parameter, σ is the applied stress and $\dot{\epsilon}$ is the strain rate applied for a particular strain ϵ , but the above equation does not consider the effect of temperature on the SR. So another equation was proposed by [73-75], which considers the effect of temperature on the SR, and is defined by $m = (\partial \ln \sigma / \partial \ln \dot{\epsilon})|_{\epsilon, T}$, where T is the deformation temperature. As reported by Caillard [76], the SRS parameter m can be approximated by plotting the logarithmic stress-logarithmic strain rate data, for a certain amount of strain, or from a group of stress-strain curves at different SR. The data is linearly fitted by the regression technique and the slope obtained from the fitting technique is used to determine m .

The SRS and the strain hardening depend on the intrinsic material properties such as SFE, dislocation density, twin density, twin thickness and spacing, and various grain boundary (GB) related mechanisms such as GB sliding, shearing, and migration. They also depend on the grain size of the material, applied strain, and also on the change of strain rate from quasistatic to high strain rates. It has been proposed by Kocks et al, [77], that the integral response of the material to the plastic deformation depends on many internal and external variables and is expressed by the relation as;

$$\dot{\epsilon} = f(F, T, \sigma, \rho, d, \delta, \lambda, x_1, x_2, \dots) \quad (2-1)$$

where $\dot{\epsilon}$ is the SR, F is the force applied, T is the temperature, σ is the applied stress, ρ is the dislocation density or the twin density, d is the grain size of the material, δ is the twin or the dislocation thickness, λ is the twin or the dislocation spacings, and x_1 and x_2 are other structural variables such as SFE and other GB related variables. So,

understanding the relationship between the intrinsic and external variables to the desired properties is very critical from a deformation standpoint.

2.2.1 Influence of intrinsic material properties (SFE, dislocation and twin density) on the Strain Hardening

All crystalline and polycrystalline materials have their atoms arranged in a regular or a periodic pattern along a crystallographic plane thus repeating over large atomic distances stacking the atomic planes one over another. For the FCC materials, which is the current scope of the study, the stacking sequence is ABC, ABC, ABC, i.e., the atomic alignment repeats for every third plane. When the FCC materials are subjected to plastic deformation, the periodic arrangement of the atoms are disturbed because of the atomic slip on the planes thus leading to faults or errors in the stacking sequence which are known as “Stacking Faults” [66], these faults or irregularities carry an energy known as “Stacking Fault Energy (SFE)”. The stacking fault energy is an intrinsic property of the material, and it has a strong influence on the plastic deformation of the materials, metals with low SFE strain harden more rapidly when compared to metals with high SFE due to a different substructure of dislocation tangles and cells [66].

The amount of plastic deformation in a crystal depends upon the symmetry and the potential slip systems, MP35N being an FCC alloy has a high symmetry with 12 potential slip systems. Recent studies conducted by Asgari, S, et al.,[56, 64, 65] Danaf, E.L, et al.,[78, 79] Kalidindi, S.R., et al.,[80] and Chiba, A., et al.,[81, 82] have reported that the amount of SFE plays a significant role in the strain hardening of the material, and the behavior of the low SFE material is distinctly different from the high SFE material. Figure 2.5 below shows the comparison of strain hardening curve between the high SFE and low SFE materials.

Figure 2.5: Schematic representation of the strain hardening regimes between high and low SFE materials [80]

As seen from Figure 2.5, the high SFE energy materials exhibited a two-stage hardening response, whereas the low SFE materials exhibited a four-stage hardening response. The difference in the curves can be attributed to the plastic deformation, with the high SFE materials exhibiting plastic deformation by slip alone whereas the low SFE materials deforming by both slip and twinning. Asgari, S., Danaf, E.L., Kalidindi, S.R., Doherty, R.D., et al., [65, 79] explained the 4-stage hardening regime observed in the low SFE energy materials to four different stages of twinning mechanisms as shown in Figure 2.6.

Figure 2.6: Four stages of the strain hardening regimes of low SFE materials [65]

The first regime or Stage A is called as the dynamic recovery regime [83, 84] which is similar to Stage III in Figure 2.6, where there was a decreasing rate of strain hardening with the increase of strain. In this region, the stresses are high enough so that the dislocations can take place in processes that are suppressed at low stress [65, 66, 85]. Cross slip and annihilation of dislocations of opposite sign, is believed to be the primary mechanism by which the dislocations can move and pile up at the obstacles thus leading to increased stresses, and this lasts only up to small strains. The second regime or Stage B is a region of approximately constant strain hardening, and it corresponds to the onset of deformation twinning in the material. These twins were found to be parallel to the slip markings in the grains, and so these can be reasonably described as primary twins. The newly created twin boundaries act as sites for dislocation pile-ups and storage [65].

Mahajan, S., and Chin, G., et al. [86-88] and Remy, L., et al. [89-91] analyzed the barriers to slip caused by twinning and concluded that the strain hardening rate increased by the formation of twins as twins acted as barriers to slip propagations. As discussed by Raghavan, M., et al. [57, 58, 92] and Remy, L., et al. [89-91] in their work, the formation of twins has the effect of reducing the grain size, at least for the slip systems that are non-coplanar with the active twin system. This, causes the overall strain hardening rate to stop falling and to remain almost constant in stage B. The third regime or Stage C of decreasing rate of strain hardening with the increasing of strain is attributed to the difficulty of producing deformation twins with the further increase of strain. The difficulty of producing the new twins can be due to the increase of inhomogeneous strain distribution within the grains with the increase of strain, which can lead to the misorientation within the grains thus impeding the formation of new deformation twins in the grains. As proposed by Duggan, B., et al., [93], the misorientation in the fiber textures lead to the loss of coherency between the deformation twins thus contributing to the decreasing rate of strain hardening, as the new deformation twins which are created upon further strain hardening are no longer parallel to the earlier primary twins. The last regime of the strain hardening curve or the Stage D corresponds to another constant or uniform strain-hardening regime which correlates to the formation of a significant density of secondary twins that intersect the primary twins that inhibit slip and promote high work hardening rates [65, 94-96].

Failing to observe ϵ -martensite by the TEM studies [11-13, 58, 62, 65, 78, 79, 82] conducted on MP35N bulk samples after the room temperature strain hardening led to the conclusion that the high work hardening rates and high tensile ductility observed in Co-Ni superalloys is primarily because of deformation twinning [10, 56, 64, 65, 78, 79] and not due to strain-induced martensitic transformation as it was initially assumed [12, 13, 16, 57, 58, 62]. The contribution of deformation twinning to the strain hardening is derived mainly from the fact that the twin boundaries act as sites for dislocation pile-ups and storage like grain boundaries, so their slip lengths are significantly reduced by the formation of twins leading to dislocation accumulation and storage. From the above discussions, it can be inferred that the SFE and its role in deformation in contributing to the dislocation and twin density are significant and it plays a major part in the strain hardening behavior of the material.

2.2.2 Influence of intrinsic material properties (SFE, dislocation and twin density) on the SRS

It is well known that the plastic deformation of metals under applied stress and SR, via the dislocation-based mechanisms, can be thermally activated and the relation of the shear strain rate ($\dot{\gamma}$) to the applied shear stress can be described by the well-known Arrhenius equation [97-99] as;

$$\dot{\gamma} = \dot{\gamma}_0 \exp\left(\frac{-\Delta G(\tau^*)}{k_B T}\right) \quad (2-2)$$

where $\dot{\gamma}_0$ is a pre-exponential factor which determines the number of possible activation sites, ΔG is the activation energy which is a strong function of the shear stress (τ^*), k_B is the Boltzmann's constant and T is the absolute temperature. The above equation is derived based on the assumption of deformation by a slip in single crystals, but in polycrystals where there are multiple slip planes involved, the activation energy can be split into two parts as [100];

$$\Delta G = \Delta G^* - \tau^* \cdot v^*, \quad (2-3)$$

where the first term on the right side of the equation represents the activation energy to be provided for the thermal activation alone in the absence of the applied stress and the second term represents the activation volume. The activation volume [101, 102] is defined as the rate of decrease of the activation enthalpy for the applied stress at a constant temperature. It is defined as;

$$v^* = - \left(\frac{\partial(\Delta G)}{\partial \tau^*} \right) \quad (2-4)$$

combining equation 2-2 into equation 2-4, we obtain,

$$v^* = \frac{\left[kT \ln(\ddot{\gamma}/\ddot{\gamma}_0) \right]}{\partial \tau^*} = \left(\frac{kT}{\tau} \right) \left(\frac{\partial \ln \ddot{\gamma}}{\partial \ln \tau} \right) = \frac{kT}{\tau \cdot m}. \quad (2-5)$$

Thus,

$$m = \frac{kT}{\tau \cdot v^*} \quad (2-6)$$

Rewriting equation 2-6 in terms of the uniaxial stress, the value of m can be written as

$$m = \frac{\sqrt{3}kT}{\sigma \cdot v^*}, \quad (2-7)$$

since as per von Mises theory, $\tau = \sigma/\sqrt{3}$. However, since the mechanisms derived are based on the dislocation-mediated mechanisms, based on the microscopic physical model proposed by Schoeck [103], the activation volume can be inferred as the activation distance swept by the dislocation of length l over a distance w so that the activation volume can be written as,

$$v^* = l \times b \times w \quad (2-8)$$

putting equation 2-8 into equation 2-7, we get,

$$m = \frac{\sqrt{3}kT}{\sigma \cdot l \cdot b \cdot w} \quad (2-9)$$

as per Dalla et al.,[104], the dislocation length can be approximated as,

$$l = \frac{1}{\sqrt{\rho}} \quad (2-10)$$

where ρ is the dislocation density, putting equation 2-10 into equation 2-9, we can derive the relation between m and ρ as,

$$m = \frac{\sqrt{3} \cdot kT \cdot \sqrt{\rho}}{\sigma \cdot b \cdot w} \quad (2-11)$$

From equation (2-11), we can infer that the SRS varies with the square root of the dislocation density. Based on the above constitutive equations, Yan et al.[105], has studied the influence of SR on the activation volume(AV) and total dislocation density for an aluminum alloy and noticed that the AV and dislocation density are inversely related to each other, as in Figure 2.7, which concludes that higher the AV, lower is the dislocation density and vice versa. Moreover, the dislocation density depends upon the type of deformation and the material type, with the tension tests having lower AV when compared to the compression tests.

Figure 2.7: Comparison of the AV and the total dislocation density for aluminum alloy deformed to a similar SR.[105]

Figure 2.8: Influence of SRS exponent(m) with varying strain rate for different strain levels in an aluminum alloy.[106]

Figure 2.9: Comparison of the AV and the SRS (m) for Ni and Ni alloys for different stress and SR [107].

Ma Huijuan et al.[106], studied the influence of SR on the m and the flow stress for the Al alloy subjected to different strains as in Figure 2.8 and observed that lower the SR, higher the strength and m . Li Heng et al. [107], had also done similar studies on the Ni and Ni alloys and compared the AV and the m values for different SR and noticed that lower AV contributes to higher m and vice versa. The observed differences in the AV and its variation to m can be attributed to the different dislocation mechanisms which contribute to different dislocation density and dislocation spacing and lengths in the material.

The above equations are defined for materials where dislocation movements are the primary mechanism contributing to increased strength and hardening in the material, but in most of the FCC metals, the materials also undergo deformation twinning which also plays a significant role in determining the strengthening mechanics [90, 108] and

instability of the material. Similar to the dislocation density, the twin density or the twin volume fraction, the twin spacing and the twin thickness, are the critical parameters which affect the SRS behavior of the material. It is well known that the formation of nanotwins in the grains, hinder the dislocation movement and increase the critical stress required for the plastic deformation [109]. Ye, et al. [110], analyzed the influence of the twin spacing (λ) and the orientation of the twin boundaries (TB) on a nanotwinned Cu and proposed the following relation correlating the deformation stress to the twin spacing as;

$$\sigma = \sigma_0 + \alpha M \mu b (\sqrt{\rho_0}) + \frac{k_y}{\sqrt{d}} + \frac{k_{TB}}{\sqrt{\lambda}} \quad (2-12)$$

where σ_0 is the frictional stress, M is the Taylor factor, μ is the shear modulus, b is the burgers vector, d is the grain size in the material, ρ_0 is the initial dislocation density in the material and k_y is the hardening factor from ρ_0 and d , and k_{TB} is the hardening factor from the TB. From, equation 2-12, it can be inferred that the stress or strength in the material increases with the decreased twin spacing. Combining equation 2-12 with 2-7, it can be implied that the m value varies inversely with λ .

Figure 2.10: Influence of twin spacing on the (a) SRS and (b) Activation Volume (AV) for nanotwinned Cu [110]

Figure 2.10 shows that the SRS decreases with the increase of twin spacing, whereas the AV increases. As per Liang et al.[111], the twin spacing depends upon the twin volume fraction and the twin thickness, and it is defined as;

$$\lambda = 2\delta \frac{1-F}{F}, \quad (2-13)$$

where δ is the twin thickness, and F is the volume fraction of the twins. Champion [112] in his work investigated the effect of m on the twin density and twin spacing on TWIP steel and concluded that higher twin density and lower twin spacing contributes to high m as shown in Figure 2.11.

Figure 2.11: Influence of the twin density and twin spacing on the SRS of TWIP steel.[112]

From the above discussions, it can be clearly understood that the development of twins during the plastic deformation strongly influences the SRS behavior of the material, by improving the deformation homogeneity and thus delaying the plastic instability.

2.2.3 Influence of Grain Size on the Strain Hardening and the Strain rate sensitivity

Polycrystalline metals and alloys with high strength and ductility have been the preferred choice for designers and researchers in all industries such as aerospace, automobile, structural and of late in the medical industry. It is being acknowledged for several decades that the strategy to improve the strength in the material, is by refining the grain

size to ultra-fine (100-500nm) or nanometer range (<100nm). The technical route used for producing the ultra-fine and nanograin materials involves subjecting the material to extensive plastic deformation processes such as cold rolling, wire drawing, forging, equal-channel angular pressing (ECAP) or superplastic deformation (SPD) techniques. There were also other techniques such as ball milling, electrodeposition, surface attrition treatments, cryogenic rolling, and dynamic plastic deformation at cryogenic temperatures to refine the grain size [113]. So, the processes and methods used to govern the plastic deformation and improve the properties in the material, has raised considerable interest among the researchers worldwide.

The classical relationship between the grain size and strength was proposed by Hall [114] and Petch [115] and is defined as;

$$\sigma_Y = \sigma_0 + k_{H-P} \times d^{-1/2} \quad (2-14)$$

where;

σ_Y = yield strength of the material, σ_0 = lattice friction of the material, d = grain size of the material, and k_{H-P} =Hall Petch slope. Based on equation 2-14, it can be predicted that the strength in the material varies inversely with the grain size. Some mechanisms such as dislocation pile-up against the GB [116], GB delegating as both dislocation sources for the creation of new dislocations and as sinks for the storage of dislocations [117], and the enhanced interactions between the GB and the dislocations, contribute to the increased plastic deformation and improved formability. This affects the strain hardening, and strain rate sensitivity of the material and the mechanisms controlling the two factors needs to be fully understood.

As per Romhanji, E. et al., Lahaie, D, et al., Song, S and Gray, G., [118-120], increasing the grain size also results in lower twin nucleation sites for the material. To confirm and study the influence and effect of grain size of low SFE materials on the strain hardening rate, El-Danaf, E, et al.,[78] conducted tests on various low SFE materials like 70/30 brass, MP35N, 316 stainless steel, with various grain sizes ranging from 9 μ m to 250 μ m and compared against the high SFE Oxygen Free High Conductivity Copper (OFHC). Figure 2.12, and Figure 2.13 below shows their comparison.

Figure 2.12: Influence of grain size on high SFE material on (a) Stress-Strain Curve (b) Work hardening rate [78]

Figure 2.13: Influence of grain size on low SFE material on (a) Stress-Strain Curve (b) Work hardening rate [78]

As seen from Figure 2.12, there was a minimal effect of the grain size on the stress-strain curve and the strain hardening curve for the high SFE copper material, but, for low SFE 70/10 brass in Figure 2.13(a), it can be seen that the grain size has a significant effect on the stress-strain curve as well on the strain hardening curve.

Increasing the grain size has resulted in a significant decrease of strain hardening in Stage A, and influenced the strain hardening plots at Stage B. For the 250 μm grain-sized 70/30 brass, the strain hardening increased with the strain in Stage B, while for the medium grain sized brass of 30 μm , the strain hardening rate remained constant. For the small grain size brass (9 μm) the strain hardening rate was found to decrease with strain or stress continuously, and the Stage B and Stage C can hardly be differentiated from each other. Optical investigations conducted by El-Danaf, E, et al., [78] for the low and high SFE samples revealed that the deformation markings produced by the twin clusters are more extensive in the large grain sized brass when compared to small grain-sized brass. From the above results, it has been concluded that more deformation twins are produced in the large grain sized materials when compared to the small-sized grain materials for a fixed amount of strain.

The trend of increased strain hardening rates with smaller grain sizes in the material is consistent with the established theories of strain hardening by Thompson, A.W, et al. [20, 121-123] where he explained based on the influence of plastic strain on slip lengths. The work reported that higher amount of slip length decrease with strain or reduced slip lengths are observed in the small grain-sized materials when compared to the medium and the high grain sized materials. As the length scale of twinning [78] is governed by the average slip length or the length of the homogenous slip, this effect in-turn affects the overall twinning stress and the hardening behavior of the material. The variation of twinning stress in the material as a function of the slip length and the grain size for various high and low SFE materials is shown in Figure 2.14.

In Figure 2.14, the actual slip lengths decreased with the increase in grain size and the materials which exhibit twinning are low SFE materials of various grain sizes with the twinning stress decreasing with the reduction of the grain size. Experimental results discussed by Zhu, Y, et al., [124] on comparing the grain size effect on twinning also indicated that the critical stress for twinning also shows a Hall-Petch type behavior with the following relation;

$$\sigma_T = \sigma_0 + k_T \times d^{-1/2} \quad (2-15)$$

where;

σ_T = Critical stress for twinning, and k_T =Hall Petch slope for the twinning.

It was concluded that for coarse or large grain sized materials with $k_T > k_S$, so with the decreasing grain size in the material, the critical stress required for twinning increases faster than dislocation slip, leading to difficulty in twin formations than dislocation slip. Schematic representation of the Hall-Petch relationship for twinning and dislocation slip for coarse-grain sized materials as defined by Zhu, Y, et al.[124] is shown in Figure 2.15.

Figure 2.14: Variation of twinning stress in the FCC materials as a function of slip length at a constant strain imposed.[78]

Figure 2.15: Schematic representation of the Hall-Petch relationship for twinning and dislocation slip in coarse grain size materials.[124]

Based on the above discussions it can be stated that the grain size plays a dominant role in the determining the strain hardening ability of the material and is determined by the competing regimes between the dislocation creation and annihilation, with the increase of the plastic deformation. It is also determined by the ability of the material to form twins, which help in further refining the grain size of the material and increasing the propensity for the hindrance of dislocation motion during deformation, by acting as an obstacle to the mean free path of the mobile dislocations and forming a dynamic Hall-Petch effect [125].

The materials subjected to an extensive plastic deformation follow a general rule that the strength of the material increases with the decrease of ductility [126-128]. This is because as the material is deformed to higher strains, the grain refinement process happens, which reduces the strain hardening capacity in the material and contributes to an early onset of necking, and a reduced plastic strain. This phenomenon is termed as “Plastic Instability” (PI) [129-131] in the material and the parameter to govern or control the PI in the material is the SRS (m) [132, 133]. However, with the development of new technologies and promising future for metals in various applications, there is a great need in the development of the materials, which can fulfill the requirements of strength and ductility. Wang and Ma [134, 135], have discussed in their work several strategies to improve the strength and ductility in the materials simultaneously by providing a bimodal grain distribution, or by deforming the materials at high strain rates and low temperatures, increasing the amount of nano twins on the grains, or by increasing the SRS of the material by offering more resistance to necking. It has been shown [136, 137] that different regions of deformation behavior evolve with different deformation mechanisms, by varying the SR which contributes to different strength at different regimes as shown in Figure 2.16.

Figure 2.16: Deformation mechanism model in micro and nanocrystalline materials with varying SR and its contribution to strength. [137]

It has been proposed by Humphrey, R.T, et al. [137] that in Region I, which corresponds to the deformation at low SR between 10^{-5} to 10^{-3}s^{-1} , which is considered region above the creep or the quasistatic regime. The strengthening in the material is attributed to the solution chemistry or the alloy content, which increases the internal friction strength of the material with the increase of the alloy content. In the Region II, which is defined by the SR between 10^{-3}s^{-1} to 10^3s^{-1} , the structural state of the material, the dislocation-based mechanisms and their mutual interactions contribute to the strengthening of the material as shown in Figure 2.16 for both micro and nanocrystalline materials. It has also been shown experimentally [138-140], that the SRS exponent (m) to be higher in the nanocrystalline materials when compared to the microcrystalline materials. Region III, which is defined by the SR of $>10^3\text{s}^{-1}$ is called the dynamic test regime, where the applied SR is rapid, the strengthening is attributed to the increase in the effective mass due to phonon drag. A phonon drag is described on an atomic scale, as the change in the electron mobility, due to the distortion or polarizing of the lattice by the fast-moving electrons.

It was also shown by several researchers that the SRS (m), not only depends on the grain size distribution in the material but also on the lattice structure. It was reported that the SRS of the FCC [100, 141-144] and HCP [145-147] materials, increased with

the decrease of grain size whereas for the BCC [148-150] materials the SRS had an inverse effect as shown in Figure 2.17.

Figure 2.17: Effect of SRS on the grain size for (a) FCC-Cu[100] (b) FCC-Ni[100] (c) Various BCC metals[100] (d) Various HCP metals [145].

The above discussions show that the effect of the strain rate and the grain size have coalesced together with the crystal lattice. The reason for the divergence of m with different materials was credited to the different deformation behavior of the material. In FCC metals, with the increase of the plastic deformation the enhancement of dislocation activities such as dislocation nucleation and dislocation pinning at the GB [101, 143], with no dislocation interactions at the grain interior [151], bestowed to an enhanced m

with the decrease in grain size. In contrast, with the BCC metals, the diminished m with the decrease of the grain size was associated to the low mobility of the screw dislocations [152], due to the increased Peierls-Nabarro stress [153], which opposes the motion of the screws and increases the spread width of the double kinks and thus reducing the overall kink nucleation.

2.3 Mechanics of Wire Drawing

Wire drawing is classified under the category of metal forming operations which involves pulling the metal through the die or a converging channel to reduce the cross-sectional area and to increase the length with the volume of the material being constant [23]. The pulling force exerted on the material is through a tensile force applied to the exit of the die. Most of the plastic flow is caused by the compression force, which arises from the reaction of the metal with the die. In most cases, the cross-section is circular, although non-circular cross-sections may be drawn by drawing. Compared to other metal forming operations such as rolling and extrusion, wire drawing offers much better dimensional control, lower capital equipment cost, and the capability to manufacture parts of small cross-sections, with continuous processing [23]. Drawing is usually conducted at the room temperature which is otherwise called as the “Cold Drawing” process but with materials with limited ductility or brittle materials “Hot Drawing” process is employed where the workpiece or the dies are heated before the reduction.

The cross-sectional reduction in the area can be made with a single pass which is called as a single die drawing process or the reduction in area can be obtained in a sequence of passes which is called as multi-die drawing process. The drawing die is the critical component of the wire drawing process as the final dimensions, surface finish, and the strength of the material are determined by its die geometry and their tolerances. Lubricants are used to reduce the coefficient of friction between the workpiece and the die, thus minimizing the frictional force and the die wear.

A schematic diagram of the single die drawing process is shown in Figure 2.18, the rotating drum is pulling the wire, and the wire is reduced in cross-section by the die placed in the die holder, and a soapbox is used as a lubricant during the drawing

process. The free-body diagram of the various forces acting on the material during the drawing operation is shown in Figure 2.19.

Figure 2.18: Single die drawing machine with the wire being pulled by the rotating drum and the cross-sectional area of the wire being reduced by the die placed in the die holder. [23]

Figure 2.19: (a) Schematic view of the wire being pulled by the die and the representation of various forces [33]. (b) Free body diagram showing the primary forces acting on the wire drawing process [154]

In Figure 2.19(a), the pulling force P is exerted by the rotating drum which is also called as the drawing force, and because of the friction between the die and the workpiece, there is a frictional force acting between the workpiece and the die as shown in Figure 2.19 (b). Due to the combined effect of the pulling force (F) and the die force (N), exerted on the wire, causes the wire to extend and reduce in cross-sectional area, while passing through the die, as schematically illustrated in Figure 2.19 (a). The various

variables involved during the wire drawing process and as seen in Figure 2.19 are defined below.

d_o = Initial wire diameter

d_1 = Final wire diameter

P = Drawing Force

τ_1 = Shear stress is acting between the workpiece and the die, which in turn is dependent on the frictional stress.

α = Die semi-angle or the reduction angle

h = Mean thickness or the average diameter of the workpiece.

L = Contact length of the workpiece with the die.

As the wire is pulled through the stationary die the initial diameter of the wire as stated by d_o is reduced to the final dimension of the die or the final wire diameter as stated as d_1 . This cross-sectional reduction happens in the converging channel of the die or otherwise known as the reduction zone as defined by the die reduction angle α , to obtain the final dimensions of the workpiece as shown in Figure 2.19 (a). Based on the diameter of the final drawn wire, the wire drawing process can be classified as big wire drawing (7.0 mm-1.0 mm), intermediate wire drawing (0.8 mm-0.1 mm), and fine wire drawing (0.05 mm-0.0030 mm)[25]. Depending on wire diameter drawn, different machines are used, single die drawing machines or otherwise called as “Bull Block,” are used for drawing big wires, and for drawing intermediate and fine wires, multiple-die drawing machines are used.

2.3.1 Effect of drawing force on the Strain Hardening behavior of the material.

Cold wire drawing [23, 27, 30, 155] is a metal forming process that imparts severe plastic deformation to the metal by progressive strain hardening. Numerous equations have been proposed to predict drawing force, and many of these calculations involve the use of empirical constants and lengthy calculations [30]. Often there is a simpler equation to estimate the force that is needed. However, it should be noted that equations based only on homogeneous deformation [23] should not be used alone, as frictional work and redundant deformation [27, 30, 31, 33, 35, 40, 156, 157], have a significant effect and will seriously underestimate drawing force if not included [40].

The drawing force involving the redundant work ϕ and the frictional effect μ is calculated using the equation;

$$F = \sigma_a A_f \left(1 + \frac{\mu}{\tan \alpha} \right) \phi \ln \left(\frac{A_0}{A_f} \right) \quad (2-16)$$

Where σ_a = Average flow stress or the yield stress in the wire.

A_0 = Initial cross-section at the entrance of the die

A_f = Final cross-section at the exit of the die

μ =Coefficient of friction

α =Die semi-angle or the reduction angle.

Deformation by wire drawing process involves stretching the material through the die and involves significant strain hardening, as per Holloman's law ($\sigma = K\epsilon^n$), it is known that the materials cannot be stretched uniformly to strains much larger than the strain hardening exponent (n) [31]. Figure 2.20 shows the plot of the flow curve of the material subjected to a steady-state wire drawing process with various conditions.

Figure 2.20: Drawability limit curve [31]

As seen from the Figure 2.20, for the ideal process without friction and redundant work the drawing stress (σ_{xa} or σ_d) acting on the material is determined by the curve highlighted in red, but in the actual case the curve in green shows the actual hardening

of the metal. The stress-strain curve includes a term η which is called the efficiency of the deformation process or deformation efficiency. Backofen defined it [31] as;

$$\eta = U_P / U_T \quad (2-17)$$

where U_P = Useful work required for wire drawing

U_T = Total work required for wire drawing

On being deformed through the die, the material is strain hardened. For an extremely strain hardened material, the formation of a neck is quickly followed by a fracture. Therefore the drawing limit as shown in Figure 2.20, is defined as the point where the drawing stress reaches the flow stress in the material (σ_ϵ), i.e., $\sigma_d = \sigma_\epsilon$. This is attributed to the wire stretching outside of the die leading to the necking in the material which contributes to the drawn length breaking off [23, 30, 31]. As the material hardens during the drawing process and follows a power hardening relationship, the drawing stress is then determined by [31];

$$\sigma_d = \frac{K \epsilon^{n+1}}{\eta(n+1)} = \frac{\sigma_\epsilon \epsilon}{\eta(n+1)} \quad (2-18)$$

where σ_ϵ = flow stress in the material after strain hardened.

σ_d = drawing stress applied to the material.

n = strain hardening exponent of the material.

η = deformation efficiency of the material as defined in Equation (2.17).

ϵ = strain or deformation applied to the material

Substituting the criterion for the maximum allowable single pass deformation, the drawability as defined by Backofen [31] is;

$$\epsilon_{\max} = \eta(n+1) \quad (2-19)$$

since $\epsilon = \ln(A_0/A_f)$ and $r = 1 - (A_f/A_0)$, the maximum reduction or the drawability based on the strain hardening can be determined as;

$$r_{\max} = 1 - e^{-\eta(n+1)} \quad (2-20)$$

For the wires subjected to multiple pass drawing, as the n value decreases towards zero, the allowable reduction decreases with the increase of deformation, as the wire passes through a series of dies. Then the maximum drawing strain is determined by;

$$\frac{\varepsilon_d}{\varepsilon_{\max}} = \frac{\eta(n+1)}{n} \quad (2-21)$$

Based on the concepts discussed by Backofen [31] it can be clearly seen that the drawing method and its parameters such as the Δ (deformation factor), α (reduction angle) ε (effective strain rate), total area reduction, and reduction per pass, have a significant impact on the strain hardening behavior of the material. It may influence the flow instability in the material and contribute to different properties and texture in the material.

2.3.2 Effect of drawing parameters on the texture evolution in wires

Most of the materials are crystalline in the solid-state, and a majority of them are polycrystalline. In a polycrystalline material, each grain is an individual crystal whose orientation differs from that of its neighbors. Therefore, a poly-crystal can be considered as an aggregate of many individual single crystals, which may be differently oriented [158]. In materials science, the word texture [158-160] refers to the orientational aspects of the grains or crystals in a polycrystalline material. In scientific literature, texture describes the preferred orientation of grains. Since many of the physical, mechanical, and chemical properties of single crystals vary with the crystallographic direction or plane, a textured polycrystal, which contains a combination of such single crystals, will naturally have an important bearing on the material properties [158].

Wire drawing involves the plastic deformation of individual metal grains; each grain can be considered as a crystal wherein deformation involves slip or shearing on close-packed planes in close-packed directions [21]. With wire drawing the shear strains applied on the material because of the applied deformation or due to the frictional conditions developed at the die-material interface, play an essential role in the metal flow and on the development of a crystallographic texture gradient across the wire [50].

Wire drawing is considered to be a uniaxial deformation process undergoing asymmetric deformation [50, 158], where the deformation processes are assumed to be applied along one direction and axis, and the analysis of microstructure along the particular direction can depict the crystallographic texture satisfactorily.

As discussed by Rajan et al. [19], to study the texture in the wire, because of its axisymmetric property the wire can be assumed to be unrolled from a cylindrical section to a flat section as shown in Figure 2.21 by cutting the wire along a section of a plane in the axial direction.

Figure 2.21: Sample directions and symmetry to study the texture in wire samples. [161]

As per [19], to define the directions of the plane of the specimen with the crystallographic directions. The axial direction (x) of the wire is aligned to the rolling direction (RD) or the drawing direction axis, the transverse direction of the wire (y) to the tangential direction (TD) of the specimen, and the radial direction (z) of the sheet is associated to the normal direction of the specimen (ND). Texture components are represented by the following notation as $(hkl)[uvw]$ [19], where (hkl) represents the crystallographic planes parallel to the given sample directions.

Most of the prior research on the texture measurements involving wire drawing as the deformation process was performed using copper (Cu) as a base material, which is an FCC material of high Stacking Fault Energy. It has four (111) slip planes and three $\langle 110 \rangle$ slip directions, so the total number of active slip systems in the material is twelve. The slip always happens on the most densely packed plane in the structure and the slip direction being the closely packed direction of the crystal [66]. As discussed by Wright [21, 23] during the Cu wire drawing process, the crystal or the grains are elongated which leads to the slip in the crystal planes and thus permitting the crystal to rotate so

that the angle between the slip direction and the direction of elongation decreases. Since there are several slip directions in operation as the deformation proceeds the crystal or the grain seeks an orientation relative to the wire axis that minimizes the angles between the slip directions and the wire axis.

For, the Cu wire drawing it was reported by Wright [21] that the angle between the $\langle 110 \rangle$ direction and the wire axis is minimized at 33° when the crystal aligns a $\langle 111 \rangle$ direction with the wire axis. Given this tendency, each grain in the wire aligns a $\langle 111 \rangle$ direction with the wire axis as plastic deformation proceeds, and because of the initial orientation of the grain differing from the required $\langle 111 \rangle$ direction, the wire needs to be subjected to a substantial amount of cold work to align all the grains $\langle 111 \rangle$ axes to the wire axis. This preferred grain orientation along the $\langle 111 \rangle$ is called the fiber texture, which is the natural texture found in heavily drawn Cu wires [19, 21]. But not all grains are rotated along the $\langle 111 \rangle$ direction; there are grains which are rotated to align the $\langle 100 \rangle$ direction with the wire axis, leaving the slip directions at a 45° to the wire axis. This was determined as the twinning mechanism contributing to a double fiber texture of $\langle 111 \rangle$ and $\langle 100 \rangle$. Wright [21] also concluded that the wire manufactured with low Δ (plastic deformation zone) values of ≤ 1 produced a homogeneous deformation and uniform texture across the wire cross-section when compared to wires manufactured with higher Δ values of ≥ 2 . The wires deformed with higher Δ contributed to a non-homogeneous deformation leading to a non-uniform redundant work near the outer portions of the wire, which slows down the texture development in the outer portions of the wire cross-section.

Kraft, F., et al. [22] has studied the influence of die angle on the texture and annealing response of the Cu wire and concluded that the die angle (α) plays a vital role in the development of the (111) fiber texture. Wires drawn with low die angle developed a higher volume fraction of (111) and a low amount of random texture whereas the wires drawn with high die angle developed a significant amount of random texture. Table 2-2, shows the texture components of the as drawn wire drawn with different die angles.

Table 2-2: As drawn fiber wire texture drawn with different die angles [22]

Die Angle (°)	$I_{\max}$ (111)	$I_{\max}$ (200)	Volume Fraction		
			(111)	(200)	random
8°	24.4	8.0	0.41	0.19	0.39
16°	19.1	6.0	0.34	0.22	0.44
24°	5.9	2.2	0.20	0.15	0.65

It can be observed that the wire drawn with 8° (low) die angle had the maximum intensity ($I_{\max}$) or volume fraction of (111) texture component, and it required higher annealing temperatures to recrystallize the material whereas the material which is drawn with high die angle of 24° required lower temperature for the recrystallization process. The results were consistent with the findings from Wright [21, 23] who observed a similar effect. Figure 2.22 shows the response of the as drawn wire drawn with different die angles to the recrystallization temperature. Deformation textures bias the nucleation and growth of recrystallized grains so that the respective annealing texture developed is strongly dependent on the previous cold-worked or the deformed structure. Wright has stated that the wire drawn with 99% CW which had a significant amount of <111> fiber texture generally changes to a <100> fiber texture upon annealing, however if subjected to a lower cold work, it leads to the formation of a double texture component involving <111> and <100> may recrystallize to form a <100> and <112> double annealed texture.

Figure 2.22: Tensile strength vs. Temperature for the Cu wire drawn with different die angles. [22]

Kraft, F. et al., [22] has also compared the effect of drawing speed on the formation of the texture component in the Cu wire, keeping the Δ value constant, and he noticed that the wires drawn with high drawing speeds exhibited a different texture component comprising of $\langle 111 \rangle$, $\langle 100 \rangle$ and $\langle 112 \rangle$ whereas the wire drawn with low speeds exhibited only $\langle 111 \rangle$ and $\langle 100 \rangle$ components. Based on the above discussions it can be inferred that in the case of the wire with a strong fiber texture, the properties along the wire axis approach the properties along the crystal orientation associated with the fiber texture orientation.

MP35N, unlike Cu being a material with low SFE [11, 46, 78, 79] has a different work hardening or deformation characteristics when compared to high SFE material Cu. Limited research has been conducted on the MP35N fine wires, unlike Cu, where a relationship between the drawing parameters, to their properties and crystallographic texture have been well defined. Bernard Q .Li., et al. [7, 17] have conducted an EBSD study on MP35N as-drawn (AD) wire of 0.178mm processed by different suppliers, but the prior cold work and the thermal and processing details of the wire are not disclosed. As shown in Figure 2.23, the EBSD IPF maps of Sample 1 and Sample 2, manufactured to the same UTS of 2100 MPa, but from different wire suppliers, looked significantly different. Sample 1 had a higher volume fraction of (111) and a lower fraction of (113), whereas sample 2 had a lower fraction of (111) and a higher fraction of (113) as shown in Table 2-3. Sample 3 and sample 4 were obtained from the same supplier manufactured with a similar wire drawing process, but with different grades of MP35N, sample 3 being MP35NLT (Low Titanium) and sample 4 being MP35N. He concluded that there is a significant texture difference observed between the wires from the different supplier and with different grades of the material as shown in Figure 2.23. This was attributed to the different requirements of strength needed for the material which might have led to the different processing conditions at the wire manufacturer leading to a different texture, but the major texture components in all the wires are identified to be $\langle 111 \rangle$, $\langle 100 \rangle$ and $\langle 113 \rangle$.

Figure 2.23: EBSD IPF maps for the 0.178mm MP35N wire from different suppliers, different UTS and different grades of raw material [7, 17]

Table 2-3: Texture components and volume fraction of MP35N wires [7]

Sample No	$\langle 111 \rangle$	$\langle 001 \rangle$	$\langle 101 \rangle$	$\langle 113 \rangle$	Total
1	0.589	0.149	0.005	0.239	0.982
2	0.473	0.108	0.025	0.358	0.964
3	0.66	0.199	0.009	0.119	0.987
4	0.655	0.145	0.013	0.167	0.98

Bernard Q .Li., et al. [162] has also analyzed the texture of an MP35N/Ag wire of 0.101mm diameter which has been manufactured using a special proprietary process for a unique application and compared with the standard process wire shown in Figure 2.24. Figure 2.25 shows the volume fraction comparisons of the wire manufactured with different processes. It can be noticed that the standard wire exhibited a higher volume fraction of $\langle 111 \rangle$ whereas the NDR (Nanograin Damage Resistant) process wire displayed $\langle 100 \rangle$ as the major component. This confirms that the type of the manufacturing process and deformation imparted to the material has a strong influence on the development of texture which interrelates to their properties.

Figure 2.24: IPF maps comparison of the texture components of the standard and the NDR MP35N wire [162]

Figure 2.25: Volume fraction comparison of the texture components of the standard and the NDR MP35N wire [162]

Sorensen, D., et al. [163] has also conducted EBSD studies on 0.178 mm wires subjected to prior cold work (CW) of 37% and 60%. Orientation Image Maps (OIM) and the corresponding pole figures are analyzed to study the preferred crystallographic orientations and to measure the grain sizes in the samples. The samples were also subjected to aging treatment to study the influence of heat treatments on the texture components.

Figure 2.26: OIM maps and pole figures comparison of the 37% CW sample in (a) As drawn (b) As drawn wire subjected to 700°C for the 60s [163]

Figure 2.27: OIM maps and pole figures comparison of the 60% CW sample in (a) As drawn (b) As drawn wire subjected to 700°C for the 60s [163]

From Figure 2.26 and Figure 2.27, it can be inferred that the samples with 60% CW had higher [100] oriented grains when compared to the samples with the 37% CW samples, which had higher volume fraction of [111] and lesser fraction of [100], this is quite the opposite to the trend observed in the high SFE Cu materials [20-23, 26, 49].

Figure 2.28: Area fractions of the (a) $\langle 111 \rangle$ and (b) $\langle 100 \rangle$ texture components in MP35N wire samples subjected to different CW% as a function of the aging temperature [163]

Sorensen, D., et al.[163] has also concluded that the area fraction components of the $\langle 111 \rangle$ and the $\langle 100 \rangle$ did not change with the temperature until 900°C . However, there was a sharp drop in the area fraction of the $\langle 111 \rangle$ for the 60% CW sample and a marginal drop in $\langle 100 \rangle$ fraction after 900°C as shown in Figure 2.27 and Figure 2.28. The observed trend has been attributed to the onset of recrystallization for the MP35N samples where a change in texture is observed, with the higher CW% wire recrystallizing faster than the low CW wire.

Prasad, M., et al. [164, 165] has conducted the annealing tests on a 0.102 mm diameter MP35N wires subjected to a temperature of 800°C and 900°C for a duration of 60 minutes as shown in Figure 2.29, and concluded that a change of microstructure from the as drawn condition was observed only after 900°C . For both annealing conditions, the crystallographic orientation in the longitudinal direction was almost random whereas, in the transverse direction along the cross-section of the wire, there was a strong presence of $\langle 111 \rangle$ fiber texture along with a significant fraction of the $\langle 100 \rangle$ component. The texture in the transverse direction after annealing has not significantly changed from the texture in the as-drawn condition although recrystallization and grain growth has been observed.

Figure 2.29: OIM maps of the wire annealed at (a, c) 800°C (b, d) 900°C; (a, b) is in the transverse direction (c, d) is in the longitudinal direction [165]

Based on the above discussions, it can be inferred that the texture development during the wire drawing process is highly anisotropic and strongly dependent on the process parameters, and the material. High SFE materials such as Cu developed a different texture and responded differently to deformation and heat treatments when compared to low SFE materials like MP35N. The texture development in the wire strongly depends on the deformation imparted to the wire during the drawing process which depends on the process parameters like the die angle (α), Δ (the plastic deformation zone), total reduction, strain rate, drawing speed and importantly the prior condition of the material.

2.4 Fatigue and Microstructure

The word fatigue emanated from the Latin dictionary “fatigare” which means getting tired, although this expression has been used to describe the human behavior, it has become a broadly accepted synonym in the engineering and the material industry to describe the failure of the machines and materials when subjected to cyclic loads. Fatigue failures are generally classified as failure under dynamic loading, and the mode of failure can be astute with no possible sign of gesture or warning. As per ASTM

standard E1828 [166], the fatigue has been defined as the process of progressive localized permanent structural change occurring in a material subjected to conditions that produce fluctuating stresses and strains at some point or points and that may culminate in cracks or complete fracture after a sufficient number of fluctuations. The study and characterization of fatigue failures are generally considered multidisciplinary as it involves scientists and engineering from various disciplines. Although the initial studies on fatigue date back to as old as the 18th century [167], over the past 150 years this field of research has gained significant importance, and significant advances have been made in the area of fatigue over the past centuries.

Initially, fatigue failure was considered to be a macroscopic phenomenon [168] with many failures or cracks, thought to be originating from the surface, when subjected to different loading conditions such as tension, compression, torsion, bending. So, the factors which affect the surface conditions of the specimen during its processing such as surface roughness, residual stress, the strength of the material, and other extrinsic factors such as oxide formation and corrosion of the metal surface during its working condition are given utmost importance. The fatigue cracks nucleate at the surface region with the interaction of the surface asperities and grow in the plane of maximum tensile stress. An example of a macroscopic fatigue crack and its propagation is shown in Figure 2.30, where macroscopic shear lips are formed on the fractured surface, the formation of shear lips is considered to be a surface phenomenon as the crack growth is not constrained in the thickness direction [169]. However, it was later realized [170, 171] that the fatigue properties of the material are also microstructure dependent and the cyclic hardening or softening observed in the material upon application of the applied plastic strain is dependent on the intrinsic properties of the materials such as dislocation density, grain size, and SFE of the material.

Figure 2.30: Example of a macroscopic fatigue crack and its growth from tensile to shear mode.[169]

Materials, when subjected to cyclic plastic strain, generate energy during its deformation which stores inside the material, and leads to a drastic change in the microstructure, by altering the defect mechanics and mechanisms. With the cyclic hardened metals, the increased plastic strain leads to an extended dislocation density in the structure. It is acknowledged that in metals subjected to plastic deformation, the dislocations in the material move along the crystallographic planes along a particular direction by a mechanism known as slip. With the increase of the applied strain, the mobility of the dislocation decreases and the dislocations concentrate along the slip planes, forming slip bands in the microstructure. The deformation in the material also changes from reversible slip to irreversible slip [172, 173] with the stress and strain localization along the slip bands, leading to the formation of extrusion/intrusion pairs as shown in Figure 2.31. The dislocations glide through the slip bands and get trapped in the slip bands, which result in the formation of Persistent Slip Bands (PSB) as shown in Figure 2.32. This extrusion/intrusion pairs act as stress risers in the materials, increasing the local stress concentration and contributing to the formation of micro and macro cracks along the slip bands as shown in Figure 2.33.

Figure 2.31: Formation of extrusions/intrusions along the slip bands in materials.[168]

Figure 2.32: Formation of PSB and the PSB gliding through the GB with the increased stress forming intrusions and extrusions along the GB.[174]

Figure 2.33: Mechanisms of PSB crack formation in a Cu single crystal. [175]

But not all cracks develop at the slip bands, and there are many other sources and discontinuities in the material such as voids, inclusions, grain boundaries, twin boundaries and secondary precipitates which all can affect the crack formation in the material. Zhang et al. [176, 177], reported that GB also act as the preferential sites for fatigue crack formation and the fatigue crack behavior of the Low Angle Grain Boundary (LAGB) is different when compared to High Angle Grain Boundary (HAGB). The LAGB allowed the dislocations and PSB to pass through the grain boundary whereas at HAGB; the dislocations were blocked, contributing to an enhanced dislocation pile up and stress concentration at the interface between the GB and the PSB driving the micro-cracks formation as seen in Figure 2.34(a). With further increase of the cyclic deformation the fatigue crack propagation transits to intergranular cracking along the GB and the cleavage planes as shown in Figure 2.34(b).

(a)

Figure 2.34: Fatigue Crack mechanisms along the GB for Cu material (a) Micro-crack formation at the interface between the PSB and the HAGB (b) Intergranular cracking along the GB.[175]

The other factor which was found to have a dominant effect on the crack propagation in the material is the SFE. As discussed in Section 2.2, it is explained that SFE plays a vital role in the evolution of twins in the material. The decrease of SFE in the material contributed to an increased twinning in the material, and the Twin Boundary (TB) enacted the same role as the GB in blocking the dislocation mobility in the materials. Zhang [178], studied the influence of SFE and Difference of Schmidt Factor (DSF) on the TB crack behavior of various Cu alloys and concluded that decrease of SFE or increased DSF contributed to more TB cracking in the material as shown in Figure 2.35. This was attributed to the increased dislocation pile up at the TB, and the change of slip mechanism from wavy mode to a planar mode which reduced the cross slip in the material, thus contributing to an increased micro-crack formation along the TB as shown in Figure 2.36. It was also concluded that increased DSF or the crystallographic orientation had a severe effect on the TB cracking, due to the increased blockage of dislocations, because of the difference in relationships of the slip systems to the TB.

Figure 2.35: Influence of SFE and DSF on the TB cracking for Cu alloys with different SFE.[178]

Figure 2.36: TB cracks formation in a Cu-8%Al and Cu-16% Al.

The critical microstructure factor which was found to have a significant influence on the fatigue mechanisms, namely the crack initiation and crack propagation is the grain size of the material. It has been proven that finer grain size improves the fatigue crack initiation (FCI) [179, 180] in the material and the coarser grain size enhances the fatigue crack growth (FCG) [181] resistance of the material. The influence of grain size on the cyclic deformation in metals has been explained concerning the strain compatibility, because of the grain boundary effects [182, 183]. There were major disagreements reported regarding the influence of the grain size to the cyclic behavior in different metals, with some investigations reporting that the reduction of the grain size decreased the cyclic saturation stress [184-186] while others reported the opposite effect [187-189]. It was also mentioned that the effect of grain size was more significant at low strain amplitude than the high strain amplitude [190], this was attributed to the slip behavior of the material where multiple slip systems operate at the low strain amplitude. As the strain amplitude increases, the need for operation of multiple slip systems is needed to maintain the overall strain compatibility which causes a difference in the saturation stress. It was described that at high strain amplitudes the slip length approaches the dislocation cell size with the grain size having little effect on the cell diameter whereas at low strain amplitudes the slip length is a function of the grain size [191]. There were also numerous models proposed by several researchers [192-206] discussing the effect of microstructural parameters on the FCI and FCG, to reduce the time for the material development and develop the desired microstructure for the intended application. The developed models helped to visualize the inter-relationship between the crack mechanisms and microstructural parameters and improve the fatigue life of the material.

From the above discussions, it can be inferred that the influence of the grain size on the fatigue life is both material and deformation dependent, and it can vary from material to material. So a generalized effect on the fatigue response, cannot be concluded based on the grain size alone especially in the nanocrystalline regime, where the influence of other microstructural features seems to be interfacing with the grain size effect.

The fatigue failure in materials or the fatigue life can be categorized into the following stages as shown in Figure 2.37, where a cyclic plastic deformation in the

material, introduces irreversible slip in the material, which leads to a crack initiation of microscopic crack, which upon nucleation and coalescence grows into a macroscopic crack, before final failure or fracture.

Figure 2.37: Different regimes of fatigue life and the relevant factors [169]

The fatigue life or the endurance limit in the material is determined either by stress-based or strain-based approached approach. The stress-based approach or otherwise known as Wohler or S-N diagram is used to quantify the fatigue data by plotting the applied stress (S) to the number of cycles to failure (N). The S-N relationship in the material is determined by subjecting the material to cyclic stress at a specified value of, stress ratio (R), stress amplitude (A), or applied normal stress (σ_a). A typical S-N curve is shown in Figure 2.38. This approach has been used in the fatigue analysis of the specimen, under the low-stress amplitudes or in the elastic range of deformation, where the number of cycles to failure is higher.

Figure 2.38: S-N diagram showing the variation of the stress amplitude to the number of cycles[207].

Due to the limitations in accurately predicting the fatigue life under high-stress amplitudes, a strain-based approach has been developed by Coffin and Manson, where the total plastic strain amplitude applied to the specimen is resolved into both elastic and plastic components as shown in Figure 2.39.

Figure 2.39: Strain life-based approach for prediction of fatigue life.[207]

Based on the number of cycles to failure, the material can withstand to; the fatigue limit can be categorized into three regions, as shown in Figure 2.40. If the no of cycles to failure is less than $<10^4$ cycles, the region is called Low Cycle Fatigue (LCF) region where the applied stress or strain amplitudes are high, the region between $10^4 < N < 10^7$, cycles to failure is termed High Cycle Fatigue (HCF), where the applied stress and strain are low. For the $10^7 < N < 10^9$, it is called as Very High Cycle Fatigue (VHCF) region where the applied stress and strain amplitudes are very low, and for $N > 10^9$, it is known as Ultra High Cycle Fatigue (UHCF) or gigacycle region. For most of the materials, the fatigue tests are performed between the conventional limits of the LCF and HCF, due to time and cost constraints.

Figure 2.40: Fatigue limit of the material based on the no of cycles to failure [208].

Figure 2.41: No of cycles required for crack initiation and propagation as a function of total stress amplitude [209].

Figure 2.41 shows the various stages of the crack growth for a required number of cycles, as a function of the stress amplitude. It can be inferred, that in the LCF region, the failure of the specimen is dominated by the crack growth since initiation occurs early in the process and the propagation is highly plastic, however in the HCF region, the crack initiation is followed by a crack propagation period before the specimen fails.

Campbell [210], proposed that once a crack is initiated in the material, the stress field ahead of the crack can be quantified and characterized by the stress intensity (K). The fatigue crack propagation in the materials can be divided into three regions as observed in Figure 2.42. In region I, the ΔK_{th} stress is lower; there is no crack propagation observed in this region, and the cracks behave as non-propagating cracks. As the crack advances, the stress intensity at the crack tip increases and the crack growth increases until it approaches region II. In region I the crack growth is sensitive to microstructure, applied stress, and environment. This region is dominated by inhomogeneous crack growth along the crack front and may produce faceted fracture surface features, although such details depend upon the microstructure, environment, and load ratio (R) and is marked by the appearance of facets on the fatigue specimens. Once the crack reaches region II, the crack propagation rate increases linearly with the increase of ΔK_{th} stress, and follows a power-law regime defined by the Paris Law shown in Figure 2.42. In this region, the microstructure and the other variables have limited effect on the crack propagation, and they are very much dependent on ΔK , crack length, and the load ratio. Region II is characterized by the appearance of striations on the specimen surfaces. This is followed by region III, where the crack growth accelerates with the increase of ΔK and contributes to a fracture or failure of the material. The fracture appearance in this region can be in the form of extensive striations or dimples in the case of specimen failed by the ductile overload or by static mode of fractures (cleavage and intergranular fracture).

From the above discussions, it can be noted that the determination of endurance or fatigue limit of the material is very important to understand the load-bearing capacity of the material and also the response of the microstructural parameters to the applied load.

Figure 2.42: Stages of fatigue crack propagation with varying ΔK [211].

2.4.1 Fatigue behavior of MP35N

As discussed in Section 2.1 before, the primary reason for the selection of the MP35N material for the medical applications is its high fatigue strength when compared to other metals, due to its high fracture toughness [11]. Figure 2.43 shows the fatigue crack growth of the MP35N material subjected to different stress ratios, as can be seen, increase of stress ratio does shift the curve slightly to the right, thus contributing to increased crack growth in the material, but the variance observed for the two different stress ratios did not change significantly. The final ΔK at catastrophic fracture approached and exceeded $100 \text{ MPa m}^{1/2}$ which is an evidence of high fracture toughness in the material.

Figure 2.43: Crack growth data for an MP35N material subjected to different stress ratio [212].

The ASTM 562 (MP35N) alloy system was initially designed as a high fatigue resistant fastener alloy for aerospace applications, and it could not meet the necessary standards and performance requirements for the medical industry due to the presence of higher inclusions in the metal. Due to the increased need for micro cleanliness in the material, a new variant of F562 material known as 35NLT has been designed to cater for the needs of the medical industry with a low inclusion content and sizes. The optimization and processing of the 35NLT alloys are discussed in [213], where it was corroborated that the principal failure mechanism for the low fatigue behavior of the MP35N material is the TiN cubical particles which are not eliminated by the typical thermo-mechanical treatments. So, the melt techniques have been improved to remove down the size of the TiN particles and reduce the overall inclusion distribution in the material. As can be seen from Table 2-4, the Ti content in the material has been reduced from 1.0% to 0.01%. The overall percentage of other metallic and non-metallic inclusions have been also reduced.

Table 2-4: Chemistry comparisons between the standard MP35N and 35NLT alloy [213]

Element	ASTM F 562		35N LT Alloy
	Min.	Max.	
Carbon	-	0.025	0.006
Manganese	-	0.15	0.01
Silicon	-	0.15	0.01
Phosphorus	-	0.015	0.003
Sulfur	-	0.010	0.0006
Chromium	19.0	21.0	20.06
Nickel	33.0	37.0	36.75
Molybdenum	9.0	10.5	10.32
Iron	-	1.0	0.12
Titanium	-	1.0	0.01
Boron	-	0.015	0.008
Cobalt	Bal.	Bal.	32.56

Figure 2.44: S-N curve comparison between 35NLT and MP35N [213]

The S-N plot between the two alloy systems is shown in Figure 2.44, there was no significant difference observed in the high-stress amplitude region, but there was a shift in the S-N curve, with the material able to withstand to higher number of cycles to failure at lower stress amplitudes. This was accredited to the reduced inclusions in the 35NLT alloy when compared to MP35N as shown in Figure 2.45. From the point of inception of 35NLT alloy system for the medical device industry, there was a significant

shift in the consumption of this material, with most of the designers and the material scientists preferring the 35NLT alloy system over the MP35N system.

Figure 2.45: Inclusions in MP35N material (a)Cracked TiN (b) De-bonded TiN (c) Al₂O₃ particle [214]

Prasad et al. [164, 165], has studied the cyclic deformation response of the 0.101mm 35NLT wire as drawn and annealed wire, using a rotary beam bend fatigue (RBBF) and tension-tension fatigue (TTF) tests and concluded that the endurance limit for the material was $< 500\text{MPa}$ for a fatigue life of 10^7 cycles. They also noticed that the sensitivity of the test method was more predominant in the LCF region when compared to the HCF region.

Figure 2.46: S-N curve for the (a) As-drawn 0.101mm 35NLT wire (b) Annealed 0.101mm wire.[164, 165]

The fatigue life of the as-drawn wire deformed with RBBF test method was higher when compared to the TTF test method as shown in Figure 2.46, it can be also observed that the fatigue life of the as drawn wire was higher in the HCF region when compared to the annealed wire, and the fatigue life of the annealed wire was higher in the LCF region when compared to the as drawn wire, this was ascribed to the different microstructure in the material processed to different conditions which led to different crack initiation mechanisms. It was also observed that there was a considerable difference in the fracture morphology of the material tested with different tested methods, the RBBF tests samples exhibited two fracture zones, namely the fatigue fracture zone and the fast fracture zone, whereas the TTF test samples exhibited an additional ductile fracture zone on top of the two fracture zones observed in the RBBF test samples as shown in Figure 2.47.

Figure 2.47: Fracture zone of (a) RBBF test sample (b) TTF test sample [164]

Based on the observed findings as per discussions in the sections above, the following points can be summarized;

- Plastic deformation applied to the material depends on several intrinsic properties of the material such as SFE (Stacking Fault Energy), dislocation density, grain size, and type of deformation (compressive or tensile) applied, which influences the strain hardening or the work hardening characteristics of the material, that contribute to variation in the mechanical and fatigue properties of the material.
- High SFE FCC materials like Cu work-harden distinctly when compared to the low SFE FCC materials like MP35N and α -Brass, this is because of the difference in the mechanisms of plastic deformation like slip and twinning which contribute to different movements or displacements in the atomic planes of the crystal leading to different work-hardening behavior.
- SRS is an essential factor in regulating the rate-dependent behavior of the materials and is controlled by their grain size, applied strain rate on the material, and deformation temperature. It also depends on microstructural parameters such as dislocation density and the length of the dislocation segment, which determine the activation volume and the activation energy of the material. Higher

dislocation density reduces the activation volume of the material, therefore, increasing the SRS in the material. The SRS determines the propensity of the material to the onset of necking or the instability in the material because of strain localization. The SRS is a rate-controlled mechanism which alters between different materials, owing to the disparity in plastic deformation kinetics, which is driven by the slip and dislocation interactions in the material.

- Cold wire drawing is a metal forming process which applies a mixed-mode of deformation on the material, with tensile forces being applied along the wire axis due to the pulling force applied when stretched through the die and compressive forces applied due to the interaction between the die and the workpiece. This process is best suited for cross-sectional area reduction of the specimens to a smaller dimension under a micron-scale when compared to the other metal forming operations like forging and rolling which are used for the bulk sample production.
- The amount of deformation imparted to the material by the wire drawing process depends on the drawing parameters like the Δ (deformation zone factor), α (die angle), $\dot{\epsilon}$ (effective strain rate), cold work applied, and the extent of reduction per pass (r). These parameters affect the properties of the material such as flow stress, strain hardening capacity and deformation of the material. It was emphasized that drawing with a lower Δ contributes to a more uniform work hardening with less redundant work, whereas higher Δ increases the amount of redundant work, which leads to non-homogenous strain hardening throughout the wire cross-section. However, the parameter depends upon the material drawn and its response to the applied deformation.
- The drawing parameters such as die angle (α), Δ , and the drawing speed contributed substantially to the disparity in the mechanical and texture properties of the high SFE Cu wires. Wire drawing with a low Δ and low α contributed to a significantly higher amount of (111), and less random structure, therefore, requiring the need for a higher recrystallization temperature, when compared to the wire drawn with high Δ and high α .

- Higher cold work (CW) and higher recrystallization temperatures developed a higher volume of (111) along the wire axis for the MP35N material whereas, the Cu material generated a higher volume of (100) material upon annealing, and the recrystallization temperatures decreased with the increase of CW.
- Fatigue is an essential and critical criterion for medical devices, and a thorough understanding of the material variability during its service should be performed before its application. The fracture of the specimen ought to happen if the material loses its capacity to withstand or sustain the applied load, and it depends on many macroscopic and microscopic parameters. The macroscopic variables such as surface finish and roughness affect the variation and prediction of the crack initiation mechanisms, whereas the microstructural parameters such as grain size, grain boundaries, slip bands, twin bands affect both the crack initiation and growth.
- The MP35NLT wires demonstrated a higher fatigue life when compared to MP35N, due to the reduced TiN inclusion in MP35NLT material, which improved the endurance limit of the material.

From the above findings, the following research gaps were identified which will be investigated during this study;

- Most of the preceding studies on MP35N alloy, involved analyzing the effect of plastic deformation on the intrinsic material properties of the material such as SFE, grain size, on bulk samples involving processes like cold forging and rolling process, where the mode of deformation process is compression, and the sample dimensions were in the range of 6mm to 20mm. So, the current study focusses on examining the influence of the cold drawing process on the deformation aspects of samples in micron-scale (618 μ m-101 μ m) which are drawn using a multi-die drawing machine, which employs a different drawing design and technique when compared to the single die drawing machine.

- All the previous studies conducted involved investigating the effect of drawing parameters like die angle (α), overall reduction, drawing speed, Δ , and die design on the mechanical, texture and recrystallization characteristics of high SFE materials such as copper, but no studies were performed examining the effects of the drawing practices on the deformation attributes in the material. So, the current work investigates the influence of drawing practices namely, FDD and HDD on MP35NLT alloy which is a material of low SFE and to understand their impact on the mechanical, microstructural and the work hardening characteristics of the material upon deformation with various amount of cold work (CW) or total reduction.
- The amount of strain applied in the wire drawing process is defined by the relation; $\varepsilon = 2 \ln \frac{d_i}{d_f}$; where d_i is the initial diameter, and d_f is the final diameter, and the SR is defined as the variation of strain over time. In the actual drawing process, the SR applied to the material is differed by changing the drawing speed, which changes the applied strain over time. There were no prior studies reported examining the influence of SR on the SRS and the SRWH on MP35N wires, so in the current study the impact of the SR on the strength, ductility and hardening capacity of the material will be analyzed by varying the SR from 10^{-6}s^{-1} to 10^{-2}s^{-1} in both the FDD and HDD drawn wires subjected to uniaxial tensile tests and correlate the observed mechanical aspects to their microstructure.
- Finally, as discussed in Chapter 1, since MP35N is the most preferred material for medical applications because of its high strength and excellent fatigue resistance, an investigation would be conducted to determine the influence of the drawing practice on the cyclic deformation characteristics of the alloy. The test would be conducted on the fine wires using a Rotating Beam Fatigue Test (RBFT) technique as per ASTM standard [215], and a relationship between mechanical, cyclic and microstructure would be established by comparing the fatigue performance of the wires in the LCF and HCF regions, to their fracture surfaces and morphology.

Chapter 3 Material and Experimental Methods

This chapter deals with the materials used in this research and a brief description of the different characterization techniques employed in this work.

3.1 Material

MP35N® (a trademark of SPS Technologies Inc., PA) is a multi-component alloy which was developed by Smith [216]. The chemical composition of the alloy as per ASTM 562-13 [217], is shown in Table 3-1. Since the alloy has a low titanium content, it is known as 35NLT. In the current study, the material was fully recrystallized before the wire drawing operations, and the raw material diameter was 0.62 mm. The wire has been annealed in a temperature range of 1000° C to 1100°C, which is usually the recrystallization temperature range for the material. The wire had a yield strength (YS) of 870MPa, UTS of 1400MPa and a ductility (EL%) of 42% after annealing.

Table 3-1: Chemical composition of the alloy as per, ASTM F 562 [217]

Element	Composition, Wt. %	
	Minimum	Maximum
Titanium (Ti)	...	0.010
Chromium (Cr)	19.0	21.0
Nickel (Ni)	33.0	37.00
Molybdenum (Mo)	9.0	10.5
Cobalt (Co)	Balance	Balance

3.2 Experimental Methods

3.2.1 Wire Drawing Experiments

Experiments on wire drawing were done at Heraeus Materials Singapore Pte Ltd under two different conditions using a multistep drawing machine shown in Figure 3.1.

Figure 3.1: Multiple die drawing machine with the die bed and the stepped cone. (Ref: Wire drawing machine at Singapore-Heraeus facility)

The first approach involves attenuation of the wire cross-section at a reduction per pass ratio of 6-10% and at a die semi-angle of (α) $6-8^\circ$ which is considered being an HDD drawing practice. Moreover, the second technique involves receding the cross-sectional area at a reduction per pass ratio of 20-30%, with the die semi-angle being the same to be known as FDD drawing practice. For an illustration purpose, to complete a total area reduction of 50% from 0.8mm to 0.565mm, the FDD drawing practice as shown in Figure 3.2 requires only two to three dies to complete the process with a reduction of 20-30% per pass whereas the HDD drawing technique shown in Figure 3.3 requires at least five to ten dies to draw the wire with a reduction of 6-12% per pass. The wires were drawn at a speed of 30m/min to 100m/min depending upon the diameter of the wire drawn, i.e., slower speeds for drawing big diameter wires and faster speeds for drawing smaller diameters. Natural Diamond Dies (NDD) were used for the drawing operations, and the wires are drawn using an oil-based lubricant. Both the condition wires had been drawn from an initial diameter of 0.62mm to a final diameter of 0.141mm, with a total reduction ratio of 95%. The FDD method was completed in a shorter time with the total number of dies used is less than 10, while the HDD technique took 32 dies to complete the run. Wire samples from different cold work reduction (CW%) were collected after drawing.

Full Die Drawing Practice

0.618mm

Figure 3.2: Schematic of an FDD drawing Process

Half Die Drawing Practice

Figure 3.3: Schematic of an HDD drawing Process

3.2.2 Tensile Testing

The as-drawn wires were deformed in uniaxial tension as per ASTM E8, using an Instron 6400 test frame, with a load cell of 500 N and by pneumatic yarn grips. The cross-head speed was set at 12.7 mm/min, and the gauge length was maintained at 254 mm for all the samples tested. The strains were calculated by the cross-head movement of the tester,

and the data is recorded for every 0.005 intervals per second. The samples were subjected to a strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$. The testing was performed at room temperature, and the samples were tested until fracture. True stress-strain curves were calculated from the raw data of the tensile curve, and the necking portion was excluded from the measurements.

3.2.3 Hardness Testing

The wire samples were mounted in bakelite, and they were grounded and polished with a $9\mu\text{m}$ grit paper until $1\mu\text{m}$, using a Struers grinding and polishing machine. An Oxide Polishing Suspension (OPS) polishing was performed as the final processing step with colloidal silica as the reagent. The polished samples were mounted on an HM-210 Mitutoyo Hardness tester, and a diamond indenter was used for indentation. Accurate positioning of the indentation was performed with a digital micrometer attached to the X-Y stage of the machine. The hardness test was performed as per ASTM standard E384-17 [218], and the load was differed from 100 to 150 g based on the diameter of the indented wire. The indentation was performed on the cross-sections along both the axis of the wire, as shown in Figure 3.4, and the indentation time was around 15-20 seconds for each sample. Five points are intended along both the axis which is equidistant from each other and evenly dispersed along their length. Hardness values were recorded in Vickers Hardness Scale (H_V).

Figure 3.4: Schematic representation of the wire cross-section with the indentation points.

3.2.4 Electrical Conductivity Measurements

The electrical resistivity of the wires was measured using Burster 2316 resistomat. The device accords to the proven 4-wire sensing method or by 4-point probe method which is an electrical impedance measuring technique that uses separate pairs of current-carrying and voltage-sensing electrodes to make more accurate measurements than traditional two-terminal sensing. For each CW% condition, wire samples of 1m long were cut and clamped between the two connecting ends of the clamping device, as shown in Figure 3.5(a). The wires have been tested at room temperature with the temperature compensation set to 20⁰C and the resistivity data recorded in units of ohm/m. The obtained values are then converted into the conductivity measurements and plotted in SI derived units of Siemens/meter.

Figure 3.5: (a) Schematic representation of the wire (shown in purple) connected between the two terminals (b) Detail view of the 4-point terminal shown in RED.

3.2.5 Fatigue testing

The wire specimens drawn with different drawing practices are subjected to cyclic fatigue tests on a rotary beam fatigue tester (RBFT) as per ASTM standard E2948-16a [82]. The tests were conducted at Heraeus Materials Singapore Pte Ltd, on a custom-built fatigue test machine with a design similar to positool valley instrument fatigue tester, but the former machine occupying a lesser space when compared to the latter. Before the fatigue loading, the drawn wires were straightened by a roller straightening machine, to remove the residual curvature known as “cast,” in the wire which is a geometrical attribute inherited from the cold wire drawing process. This step needs to

be done to minimize the variation in the test data, and it was observed during the fatigue set-up that the wires without straightening would spin out of the chuck and fracture at the chucks leading to erroneous results. The straightened wires were cut to the desired length based on the input variables of the chuck distance, wire diameter, young's modulus (E), applied stress and the length of the wire was determined from the respective calculations stated in the standard E2948-16a [82]. One end of the wire is clenched to a hollow bush, and the other end of the wire is clamped to a rotating chuck which rotates at a speed of 3000RPM and operates at a frequency of 50Hz. The vibration supports are mounted along the curvature of the wire, and the break detection probes are placed along the wire. A cyclic counter fixed to the machine records the number of cycles to failure, and for each revolution of the chuck, the wire specimens are subjected to an alternate compression and tension cycles during its course of cyclic loading. The tests were performed at room temperature using air as a medium.

3.2.6 Field Emission Scanning Electron Microscope (FESEM) and Electron Beam Scattered Diffraction (EBSD) characterization.

For the microstructural characterization, the wire samples are mounted in G2 epoxy and hardener mixed in the proportion of 10:1 and cured for 20 to 30 min. The samples are then polished using an IB-19500 CP Ion miller [219-221] with a voltage of 5-10 kV and at a current of 70-110 μ A for 2-3 hours using Ar as the ionizing gas medium.

The ion-milled samples were examined in a High-Resolution Field Emission Scanning Electron Microscope (HR-FESEM) – JSM-7800FLV Prime in a Back Scattered Detector mode (BED). For the crystallographic texture study, Electron Beam Scattered Diffraction (EBSD) analysis was performed on the specimens using a NordlysF detector connected to the FESEM. The specimens were tilted to a 70° angle, and the machine was operated at an accelerating voltage of 15 kV to 20 kV. The beam size was varied between 6nm to 10nm depending upon the specimen size, and the step size was altered from 0.01 μ m to 0.4 μ m. The working distance was maintained between 12-13mm and the aperture size around 60 μ m. The indexing and the recommended pre-processing steps like the tilt correction, background noise correction, and the raw data collection were done using the Aztec software. Data analysis like grain size histograms, grain boundary distributions and texture information like the pole figures, Inverse pole figures (IPF) across the respective sample and the crystal directions are plotted, from the

raw data using the HKL fast acquisition software. For the determination of grain size statistics, the critical misorientation or the tolerance angle was kept to 10° , and the grain filter minimum area was kept to 10 pixels. For plotting the misorientation profile, a point on the orientation map is identified, and a straight intercept line is drawn across the map in an either horizontal or vertical direction to find the misorientation across the grains. For the calculation of the pole figures, the half-width is set to 10° and the cluster size to 5° .

For the SEM analysis, to observe the fracture zones of the tensile test and fatigue samples, the wires were immersed in Iso Propyl Alcohol beaker for 1-2 minutes and air-dried for few minutes. The dried samples were mounted vertically onto the copper stub, using double-sided conductive tape. The samples were then loaded into the JOEL JSM-6010LV SEM chamber and were examined at a working distance (WD) of 10-15mm and a spot size of 60nm. For the fractography examination of the fatigue samples, the wires were probed initially at lower magnification between 200 to 1000X, to detect the crack initiation point or the fractured area, and for identifying the possible mechanisms of fracture. An Energy Dispersive X-ray Spectrum (EDX) would be performed to ascertain for any inclusions observed at the fracture point.

3.2.7 Focused Ion Beam (FIB) and Transmission Electron Microscopy (TEM) characterization.

Due to the inherent difficulties in the sample preparation of thin wires by the conventional methods such as broad ion beam milling, electro-polishing, the wire samples were prepared by Focused Ion Beam (FIB) milling technique [222-225]. A DA300 dual-beam FIB, equipped with an in situ Omniprobe micro-manipulator was used for the lifting out the specimen onto the Cu grid [226, 227]. Ga ion source with a specimen cutting voltage of 30keV was used for the FIB milling. A particular area along the wire drawing direction was selected, and the samples were transferred to the TEM half grid using the micro-manipulator [228, 229]. A protective layer of Pt was deposited on the extracted lamella, and the sample was further thinned down to 70-80nm for better transparency under TEM. An ion beam current of 150pA was used for the serial cuts, and a current of 9pA was used for the thin cuts. Microstructural characterization was performed using Tecnai X Twin Transmission Electron Microscope, operating at a voltage of 200keV and equipped with an EDAX X-ray detector.

Chapter 4 Influence of drawing practices on the mechanical, texture and work hardening characteristics of Co-Cr-Ni-Mo wires.

4.1 Introduction

This research investigates the influence of the cold drawing practices on the mechanical, micro-texture, and work hardening behavior in Co-Cr-Ni-Mo wires. Cold wire drawing [23, 27, 30, 155] is a metal forming process that imparts severe plastic deformation to the metal by progressive strain hardening. The mechanics of wire drawing [35] involve a reduction in the cross-section of the material sequentially by passing it through a series of conical dies of different diameters. Deformation-induced by wire drawing depends on several factors like the material drawn, reduction ratio, die geometry, drawing speed and friction between the wire and the die interface [23, 24, 26, 32, 33, 40].

In the last 30-40 years [23, 30-33, 35, 155], much research had been done to study the influence of drawing factors and their combinations on the mechanics of plastic deformation by wire drawing. It was established that the total reduction ratio significantly influences the mechanical, strain hardening, and deformation mechanisms of the wire [23, 30, 34, 35, 156]. Most of the research to date have focused on the effect of total reduction, die angle, drawing speed, and their influences on the mechanical and texture characteristics of the wire using copper, steel and aluminum as raw materials [14, 22-29, 32, 34, 35, 38, 40, 42, 49, 79, 156, 157]. However, less research has been done on Co-Cr-Ni-Mo (MP35N) alloy, which is an FCC material of low SFE (Stacking Fault Energy), unlike copper and aluminum which are FCC materials of high SFE.

MP35N (a trademark of SPS Technologies Inc., PA) a multi-component alloy developed by Smith [16] has a composition of 35% Co, 35% Ni, 20% Cr and 10% Mo. It is known to exhibit high fracture strength, toughness, and excellent corrosion resistance [1, 3, 52, 54, 230, 231]. Because of its excellent fracture resistance and corrosion properties, it finds its place as the best material of choice for many designers in the medical industry for many implantable devices like cardiac pacemakers, defibrillators, and neurostimulators.

Prior studies [10, 11, 13, 14, 57, 58, 64, 65, 78-80] and research conducted on the material, concludes that the material derives its high strength upon plastic deformation while maintaining a relatively high amount of ductility. The literature [12, 56, 64, 78, 82] reported to-date has focused on the material properties in the bar, and rod forms but not in the wire forms as the thermo-

mechanical processing of the wires are different when compared to rods. Of late, there were a few studies [17, 59, 162-165, 232, 233] performed on MP35N wires in as-drawn and annealed conditions, but details on the drawing methods and their relationship to properties are not divulged.

Current industrial drawing practices involve two types of drawing which can be categorized as (a) Half Die Drawing (HDD) where the cross-sectional reduction per pass is between 6-10% and (b) Full Die Drawing (FDD) where the cross-sectional reduction per pass is greater than 20%. The objectives of the current research are to understand the deformation behavior of MP35NLT alloy subjected to different drawing practices and study their influences on the mechanical and microstructural characteristics of the wire.

4.2 Materials and Methods

MP35NLT wire with an initial diameter of 0.62mm and in annealed condition was drawn to different cold work (CW) reductions using FDD and HDD drawing practices, and the samples at the respective CW reductions of 16%, 50%, 75%, and 95% were collected and characterized for their mechanical and microstructural behavior. The reason for the selection of 16% CW as one of the deformation conditions was to analyze if there is any contribution of the different drawing practice on its microstructure even at such a low CW reduction. Six batches of wire were manufactured to ensure the repeatability of the runs (three batches from FDD and three batches from HDD). There were no wire breaks or any abnormalities encountered during the wire drawing operation, and both the operations were completed. The tensile tests were performed at room temperature, and the samples were tested until fracture. Three samples from each batch and nine samples for each drawing condition were tested. The average and standard deviation of the obtained results are illustrated in their respective plots.

4.3 Results

4.3.1 Mechanical Results

Mechanical properties of the wires such as Yield Strength σ_{YS} , Ultimate Tensile Strength σ_{UTS} , Elongation EL% have been tested. Figure 4.1 below shows the comparison of the mechanical properties between the FDD and HDD wires subjected to different CW%.

As seen from Figure 4.1 the σ_{YS} and σ_{UTS} , of the FDD drawn wires were higher than the HDD wires until 75% CW; however after 75%, the σ_{YS} and σ_{UTS} , of the FDD wires dropped unto 80% CW, after which there was a gain in σ_{UTS} and σ_{YS} until 95%, reaching values similar to HDD

wires. True stress-strain curves were calculated from the raw tensile data and plotted in Figure 4.2. The curves indicated that the stress increased monotonically with strain as the amount of CW% increased.

Figure 4.1: Comparison between the FDD and the HDD wire samples (a) σ_{YS} (Yield Strength) (b) σ_{UTS} (Ultimate Tensile Strength) (c) EL%

Figure 4.2: True stress-strain curve for FDD drawn wires subjected to various CW% (a) FDD (b) HDD

However, the relative increase of stress for an applied amount of strain was higher for the FDD wires when compared to the HDD wires. The work hardening rates ($\delta\sigma/\delta\epsilon$) for the drawn wires are calculated from the true stress, and strain data and the work hardening curves are plotted in Figure 4.3. The work hardening rate ($\delta\sigma/\delta\epsilon$) for the FDD wires were relatively higher than the HDD wires, drawn to the same amount of CW%. An HDD wire subjected to 50% CW had a ($\delta\sigma/\delta\epsilon$) rate of 88000MPa, whereas the FDD wire had a ($\delta\sigma/\delta\epsilon$) of 180000MPa. The FDD wires exhibited different hardening regimes when compared to the HDD wires. The curves plotted for the FDD wires were in close resemblance to the low SFE material hardening curves as discussed in prior work [56, 65, 78-80, 234], whereas the HDD curves had a mixture of both the high and low SFE materials hardening curves for different CW%.

Figure 4.3: FDD vs. HDD Work Hardening curves for the wires subjected to different CW%

4.3.2 Microstructure Results

EBSD analysis was performed on the received wire at 0.62mm, on a sample area of $300 \times 500 \mu\text{m}^2$ with a step size of $0.2 \mu\text{m}$. Figure 4.4(a) shows the EBSD band contrast image taken at 3000 times magnification, a mixture of coarse and fine grain sizes was seen, with a manifest presence of annealing twins inside the grains. Grain size histogram plotted from the mapped area is displayed in Figure 4.4(b). As determined from the histogram, the grain size was between $4\text{-}12 \mu\text{m}$, and the highest frequency was observed between $5\text{-}6 \mu\text{m}$.

(a)

(b)

Figure 4.4: (a) EBSD band contrast image of the 0.62 mm wire (b) EBSD grain analysis distribution (histogram) of the 0.62 mm wire

(a)

(b)

(c)

Figure 4.5: Inverse Pole figures (IPF) orientation map of the 0.62 mm wire along the (a) X-Direction (b) Y-Direction (c) Z-Normal Direction (Wire Axis)

Figure 4.6: Pole figure of the 0.62 mm wire along the three-crystallographic axis

Inverse Pole Figure (IPF) orientation map shown in Figure 4.5(c) along the normal direction exhibited a double fiber texture with a strong presence of (111) fiber texture component and a weak component of (100) and (110). The other two directions along the transverse and the rolling direction exhibited a strong presence of (110) and (111) fiber component, and a weak texture of (100) component. Overall, the (100) component in the texture after annealing was weak unlike the standard FCC metals like Cu, where a strong presence of (100) and a weak presence of (111) components are observed after annealing [21, 50]. The pole figures in Figure 4.6 indicates that the material is textured with a high density of (111) poles lying clustered at distinct locations of the pole figures along the $\langle 100 \rangle$ and $\langle 111 \rangle$ directions. Band contrast image in Figure 4.4(a) indicated that there were several twins embedded inside the grains. A Mis-Orientation Distribution (MOD) histogram profile, between the grains was plotted. The histograms plotted in Figure 4.7, exhibited a strong presence of High Angle Grain Boundary (HAGB), where the misorientation between the neighboring grains was observed to be higher than 15° , along with the misorientation peaks of less than 15° which are Low Angle Grain Boundary (LAGB). This confirms that the parallel bands enclosed within the grains to be annealing twins due to the presence of LAGB noticed in the histogram.

Figure 4.7: MOD (Misorientation Distribution) histogram across the different grains in (a) Transverse direction (b) Longitudinal Direction.

4.4 Discussions

4.4.1 Effect of drawing practices on the Yield Strength of the material

The average flow stress σ_{YS} of the material [33, 66] determines the deformation behavior or the amount of stress the material can withstand during drawing. The increase in σ_{YS} of the material to the applied plastic deformation is governed by the Hall Petch (HP) equation [31, 66], which in turn is defined by the grain size and the number of twins generated in the microstructure. As per HP relationship, the smaller the grain size, the higher should be the σ_{YS} , so the samples deformed to various CW% were analyzed for their grain size through FESEM and EBSD analysis.

4.4.1.1 Influence of drawing practice on σ_{YS} for 16% CW

The 16% CW FDD wires as shown in Figure 4.1(a), had a higher σ_{YS} of 218MPa when compared to the HDD wires; and to understand the reason for the variation in the strength, the samples were subjected to microstructural analysis.

Figure 4.8: FESEM -BED Images of wires drawn to 16% CW (a) FDD wire (b) HDD wire.

As observed from Figure 4.8, the FDD wire microstructure is more deformed when compared to the HDD wire. EBSD analysis was performed on the deformed samples, and the GSD (Grain Size Distribution) histogram is plotted in Figure 4.9.

Figure 4.9: Grain size histogram of the wires drawn with 16% CW (a) FDD wire (b) HDD wire.

Figure 4.9(a) indicates that in FDD wires, there was a higher distribution of grains between 0.1-0.6μm when compared to the HDD drawn wire shown in Figure 4.9(b). The frequency or distribution of grains with a grain size of greater than 1.0μm was noticed to be higher in the HDD drawn wire. As seen from Figure 4.8(a), there were deformation markings observed in the microstructure of the FDD 16% CW sample, when compared to the HDD sample in Figure 4.8(b). To confirm their characteristic, the MODF was performed on both the samples, as shown in Figure

4.10, and a comparison has been made on the amount of HAGB and LAGB. As seen in Figure 4.10(b), sample deformed with FDD drawing practice exhibited a higher fraction of HAGB ($>15^\circ$), when compared to HDD sample in Figure 4.10(a) where a higher fraction LAGB ($<15^\circ$), were observed. This implies that the planar defects inherent in FCC materials such as twinning or dislocations which contributes to increased misorientation between the grains have commenced in the FDD drawn samples at such a low CW, whereas the HDD drawn samples drawn to the same CW, does not exhibit such defects in the microstructure.

Figure 4.10: MODF histogram for the wires deformed to 16% CW (a) HDD sample (b) FDD sample.

The pole figures have been compared between the two wires to determine the variation in texture. As seen from Figure 4.11, there was a noticeable difference in the texture; the FDD drawn wire had a higher fraction of (111) and a lower fraction of (100), whereas the HDD wire had a higher fraction of (100) and lower fraction of (111), when observed along the deformation axis. The texture results corresponded well with the microstructure displayed in Figure 4.8, which affirm that significant deformation has begun along the $\langle 111 \rangle$ direction for the FDD wires, which is the slip plane for the FCC metals. The noted results illustrate that the difference in grain size between the two samples together with the variation in the microstructure and texture contributed to a higher σ_{YS} in the FDD wire when compared to HDD wire. To further understand the significance of the rapid increase in σ_{YS} with strain or CW%, the samples deformed to 50, 75 and 95% CW were characterized.

Figure 4.11: Pole figure for the as drawn samples drawn to 16% CW (a) FDD wire (b) HDD wire.

4.4.1.2 Influence of drawing practice on σ_{YS} for 50% CW

The FDD wire deformed to a 50% CW was unable to be indexed by EBSD, whereas the HDD wire was indexed, so a FESEM- Back Scattered Electron Detector (BED) images were collected for the FDD sample, as shown in Figure 4.12. The FDD wire microstructure was highly distorted, with the grains being severely deformed, and misoriented along the drawing axis. Using the image analysis software, the grain sizes were measured, which were in the range of 100-200nm. Several parallel striped deformation markings as illustrated by red arrows in the microstructure were observed throughout the wire sample. The microstructure of the FDD wire, correlated well with the results of earlier investigations [10, 12, 64, 65, 78, 80, 163, 164] on MP35N rods and other low SFE alloys, where the parallel markings noticed in the microstructure were confirmed to be deformation twins.

(a)

(b)

Figure 4.12: FESEM -BSE image of the FDD drawn sample at 50% CW (a)7000X (b) 30000X

The observed results justify as for why EBSD could not index the FDD wire, as described by many researchers [159-161, 235, 236] on the limitations of the spatial and angular resolution of the EBSD equipment, in working with deformed samples of nano or sub-micron scale. However, the HDD drawn wires subjected to a similar CW reduction of 50% could be indexed by the EBSD. As noticed from IPF images of the HDD drawn wire shown in Figure 4.13, the microstructure was less deformed, with the grains and the grain boundaries visible in the microstructure, unlike the FDD microstructure in Figure 4.12, where the microstructure was highly distorted and deformed.

Figure 4.13: Inverse Pole figures orientation map for the HDD sample subjected to 50% CW along the (a) X-Direction (b) Y-Direction (c) Z-Normal Direction (d) Pole Figure Scattered data

Figure 4.14: 50% HDD CW sample (a) Grain size histogram (b) MODF histogram.

Pole figure scattered data shown in Figure 4.13(d) exhibited a strong presence of (110) and (111) planes and a weak presence of (100) plane, this indicates that the grains have started rotating towards the (110) direction from the initial (111) direction shown in Figure 4.11(b). Grain size histogram observed in Figure 4.14(a), indicated that the grains were between 0.1 to 1.6 μm , with a mixture of coarse and fine grains, unlike the FDD wire microstructure in Figure 4.12(b) where the grains were mostly fine in the nanometer range. MOD profile in Figure 4.14 (b), shows an increased fraction of HAGB when compared to 16% HDD CW sample; however, a considerable amount of LAGB could still be observed. The observed findings of increased distribution of fine grains and a higher number of deformed twins in the microstructure, explain as for why an FDD drawn wire had a higher σ_{YS} when compared to HDD drawn wire.

4.4.1.3 Influence of drawing practice on σ_{YS} for 75% and 95% CW

For a wire drawn to 75%, CW, both the wires could not be indexed by EBSD. Therefore, samples were evaluated by FESEM, and high magnification BED images were collected as shown in Figure 4.15. Both the microstructures looked distorted with the severity being higher for the FDD sample. The FDD wire had grains between 50-100nm, whereas the HDD wire exhibited grains higher than 100nm. Deformation markings as indicated by red arrows in Figure 4.15(b) seem to originate across the grain boundaries for the HDD drawn wire, whereas the deformation markings seem to intersect and annihilate each other in the FDD drawn sample, with the FDD microstructure having a significantly higher number of twins than the HDD wire. The deformation markings and the grain size of the HDD sample correlated well with the FDD 50% CW BED image in Figure 4.12, where the measured grain size was in the range 100-200nm, while the HDD wire drawn to 75% CW had a grain size in the range of 100-300nm. The σ_{YS} of the HDD drawn wire at 75% CW, was around 1760MPa which was similar to the σ_{YS} of the 50% CW FDD wire, at 1840MPa. This affirms the conjecture that smaller grain sizes and higher number of twins are formed in the FDD drawn wire at a lower CW%.

After 75% CW, as shown in Figure 4.1, the σ_{YS} of the FDD drawn wire decreased from 2280MPa to 1945MPa with a simultaneous increase in EL% from 4.27% to 4.98% up to 87% CW, after which the σ_{YS} increased again. This phenomenon can be explained as follows; the reduction in the grain size to a certain critical level contributed to a reduction in the interfacial energy at the grain boundaries (GB), which reduced the hindrance of dislocation motion at the GB. This also reduced the formation of new twins and with the application of further stress, the twins, and dislocations present at the GB, changed their mobility from intergranular to trans-granular leading

to a reduction in the strength of the material with a simultaneous increase in the ductility. This phenomenon can be defined as the “Inverse Hall Petch effect” [237-240], which can be ascribed to the grain softening and local stress localization in the material [241], because of the inadequacy of the material to generate new dislocations.

(a)

(b)

Figure 4.15: FESEM-BSE Image of the drawn sample at 75% CW (a) FDD (b)HDD.

The results correlated well with the theory proposed by Zhu, Y, et al. [124] and Chiba, A., et al. [81, 82], on the impact of plastic deformation to the slip mechanisms in the material leading to a reduced twinning and dislocations in the material, because of the reduction in grain size. This regime of recovery existed only for a certain amount of critical strain, and as the strain exceeded the critical twinning stress, the misorientation and twinning phenomenon emerged again as viewed with the increase of σ_{YS} and the decrease of EL% in Figure 4.1. This softening effect was not observed in the HDD samples and the increase of σ_{YS} was linear with strain. The σ_{YS} of HDD wire after 95% CW was slightly higher than the σ_{YS} of the FDD drawn wire. For the 95% CW FDD and HDD samples, the FESEM imaging was not possible because of the high plastic deformation which led to a severely distorted microstructure.

Hence, the material drawn with the FDD contributes to a higher σ_{YS} , when compared to the HDD process. The reason behind this can be attributed to the difference in the amount of plastic deformation imparted to the wires by the different drawing practices. As defined by Panteghini [40] the amount of plastic strain that can be applied to the material during its plastic deformation is given by the following equation;

$$p = \ln\left(\frac{1}{1-r_A}\right) \quad (4-1)$$

$$\text{where } r_A = 1 - \left(\frac{R_0}{R_f}\right)^2 \quad (4-2)$$

p = average plastic strain

R_0 = Radius of the wire at the entrance of the die;

R_f = Radius of the wire at the exit of the die.

From Equation (4-1), a higher reduction per pass as such in FDD drawing practice, the amount of plastic strain imparted to the wires is higher when compared to the HDD drawing process, which contributes to a higher plastic deformation on the wires. With increasing CW%, the deformation becomes cumulative due to an increased area of reduction, and the amount of applied strain increases. The high plastic deformation applied in FDD drawn wires contributed to smaller grain sizes and a higher amount of lattice defects like twins and dislocations in the microstructure, therefore contributing to higher strength in the material.

4.4.2 Effect of drawing practices on the work hardening of the material

In the current work, an attempt was made to study the work hardening behavior of the MP35NLT alloy wires, drawn with FDD and HDD drawing practice. The work hardening rate ($d\sigma/d\epsilon$) has been calculated from the plots of true stress and strain data and plotted against $\dot{\epsilon}$ (True Strain rate). The data from the necking portion has been truncated as this could mislead data.

Figure 4.16 shows the work hardening curves of the FDD and HDD drawn wire subjected to various CW%, it can be observed that the work hardening rate for the FDD wires was three times higher than the HDD wires, and the hardening curves appeared different from each other for a similar CW reduction. There was a three-stage hardening regime observed for the 50% CW FDD and 75% HDD wires, whereas the 95% FDD and HDD wires exhibited only a two-stage hardening curve. For the 50% HDD wire, there was no hardening regime observed, and the hardening rate decreased with an increase in strain, as shown in Figure 4.16(a). However, for the 50% FDD wire, a decrease in the curve's slope or hardening rate until a strain of 0.0025, was observed. This was indicated as Stage A in the curve, followed by an increase in slope between a strain of 0.0025 to 0.0075, which is shown as Stage B. With further increase of strain, the hardening curve decreased continuously, specified as Stage C in Figure 4.16(a).

The hardening curve for the 50% HDD wire looked similar to the curves of high SFE FCC materials like copper and 90/10 brass, where there were no 4-stage hardening regimes observed in the material, and the work hardening rate decreased with the increase of strain [65, 78, 79]. This can be attributed to the crystallographic slip in the material along the atomic planes where there is a decrease of slip length with the increase of strain. However, for the 50%, FDD wire samples, there was a three-stage hardening regime noticed in Figure 4.16(a). Stage A corresponds to the crystallographic slip in the material which is the most common mechanism for the materials undergoing plastic deformation until a strain of 0.0025, this was followed by an onset of Stage B, where the work hardening rate increased with strain until a strain of 0.0075, where the twins originate in the material, and finally Stage C corresponds to twin intersections, leading to increased twin misorientation and dislocation density in the sample, thus inhibiting the production of new twins and also contributing to the reduced grain size in the material as shown in Figure 4.12.

(a)

(b)

(c)

Figure 4.16: Work hardening curve comparison: FDD vs HDD for (a) 50% CW (b) 75% CW (c) 95% CW.

This difference in the hardening behavior perceived could also be attributed to the variation in microstructures observed between the two wires. As seen in Figure 4.12, the FESEM image of the 50% FDD CW sample displayed a highly deformed and distorted structure with severe deformation markings intersecting the grain boundaries, whereas 50% HDD CW sample seen in Figure 4.13 exhibits a less deformed and homogenous structure, with less or no deformation markings observed in the microstructure. The difference in the number of deformation markings observed between the two-wire samples coincides to the amount of twinning and dislocation density in the material which contributed to the difference in the work hardening behavior.

Figure 4.16(b) shows the work hardening comparison between the FDD and HDD samples subjected to 75% CW reduction. The HDD samples exhibited a three-stage hardening regime like 50% CW FDD wire in Figure 4.16(a). This infers that twin initiation or deformation twinning in the HDD drawn wire can be observed only after 75% CW. This phenomenon is confirmed by

Figure 4.15(b), where there was clear evidence of slip and deformation markings observed in the microstructure of the HDD wire. Hence, to induce twinning in the HDD drawn wires, the samples need to be deformed to a higher CW of 75%, whereas the origination of twinning can be noticed in FDD samples at a low CW%.

Only two stages of work hardening were observed in the 75% CW FDD drawn samples, as shown in Figure 4.16(b). Stage A corresponds to the dynamic recovery in the material where there is an extensive cross slip between the existing twins, leading to twin annihilation and rearrangement along the deformed planes. This contributed to a decreased strength and reduced work hardening in the material. Stage A is followed by Stage C where the decreasing grain size in the matrix completely inhibits the formation of new twins and dislocations, thus contributing to a decreased work hardening rate. At 95% CW, as shown in Figure 4.16(c), the FDD and HDD work-hardening curves are comparable to each other, with evidence of Stage C for the HDD sample and presence of Stage A and Stage C for the FDD sample. This can be attributed to inhibition of twin formations in the material and the observed results correlated with the theory proposed by Mahajan, S., and Chin, G., [86-88] on deformation twinning and its contribution to work hardening.

Based on the above results, it can be summarized that the work hardening rate in the FDD wires is relatively higher when compared to the HDD wires. The initiation or on-site of twin formation is observed at a lower strain in FDD samples whereas, in HDD wires, the twins were noticed only at higher strains. So, it can be concluded that the FDD drawing process had a stronger effect in reducing the grain size and assisting to a higher deformation twinning in the material, thus contributing to higher work hardening rates and increased strength in the wire.

4.5 Summary

From this work, the following conclusions can be made.

- (1) Higher σ_{YS} was obtained from the wires drawn with FDD practice when compared to the wires drawn with HDD practice. This was attributed to the enhanced grain refinement in the material, and to the difference in the number of twins and dislocation density generated in the microstructure by the drawing practice, with the FDD practice generating a higher frequency of fine grains and twins when compared to HDD wires for a similar amount of CW%.
- (2) A softening effect in the material was observed in the wires drawn with FDD practice between 75-90% CW, which led to a reduction of material strength and increased ductility. This phenomenon was attributed to the “Inverse Hall Petch Effect.
- (3) Higher work hardening rates was noticed in the FDD drawn wires (three times higher) when compared to HDD wires; this was attributed to the higher deformation induced by the FDD process which leads to an early onset of twinning at a very low strains (16-50% CW) when compared to HDD process where the twinning started only after 75% CW.

Chapter 5 Study of deformation homogeneity and electrical conductivity in Co-Cr-Ni-Mo wires drawn with different drawing practices.

5.1 Introduction

Wire drawing depends on several parameters like (a) type of material drawn, (b) reduction ratio (c) die characteristics like the total reduction angle (2α) and the bearing length (BL) (d) drawing speed (e) and friction between the wire and the die interface [24, 26, 242, 243]. The interaction and control of parameters during the drawing process determine the deformation homogeneity in the wires, which is very critical for the understanding of stress and strain distributions across the wire. The amount of inhomogeneity incurred in the wire determines the final product characteristics and has a direct impact on the product quality and also on the subsequent post-processing operations.

Prior studies by Luis, et al. [244] compared the influence of die angle, die length and total area reduction on the deformation homogeneity on aluminum alloy by both the analytical and FEA methods and concluded that die angle plays a significant role in the contribution to inhomogeneous deformation and internal distortion in the wire. Vega, et al. [26] studied the influence of the process parameters on the copper wire drawing and noticed that an increase in effective strain makes the deformation more inhomogeneous over the cross-section, especially near the die wire interface, thus leading to severe wire damage. Coser, et al. [245] investigated the influence of the residual stress distribution in steel rods by changing the bearing geometry and length through numerical analysis and concluded that differing the bearing length (BL) in the ratio of $0.4D$ to $0.6D$ (where D is the diameter of the wire drawn) produced less residual stress on the wire surface. The research was analogous to the work of Overstam [246] who altered between two bearing geometry namely circular and tapered and concluded that tapered profile bearing has a better effect in reducing the inhomogeneous deformation in the wire. Lin, et al. [156] has explained the analysis of inhomogeneous deformation by the modification of the Backofen hardness inhomogeneity factor to an effective strain factor and concluded that drawing with a smaller die angle in the range of 2° to 4° , the deformation was homogeneous and uniform because of the reduction of the effective strain and the inhomogeneity increased with the increase of die angle. Hassan & Hashim [247] studied the influence of die angle, bearing length and drawing velocity on the homogeneity in deformation using DEFORM-3D on AL-1100 material, and concluded that the drawing force increased with the increase of die angle and BL, and decreased with the increase of drawing

velocity, with higher drawing force contributing to severe in-homogenous deformation. Luksza, et al. [248] investigated the influence of the single die and multi-die drawing process on the non-uniformity of strain and mechanical properties and concluded that significant differences exist in the effective strain distributions for the wire drawn with a single and multi-die drawing processes and they need to be evaluated independently using different approaches.

It is well-known that in the design of wires for medical applications such as cardiac pacing and defibrillation leads, on top of its mechanical strength, the electrical properties of the wire such as conductivity and resistivity are very critical owing to the transferring of pacing pulses, electric shocks and energy from the device to the required site or tissue [249]. Electrical conductivity is defined as the ability of the material to transfer an electric current or charge in the material through an area and is determined by the volume of the free valence electrons and their movement in the material. Several investigations [250-253] have been performed to study the influence of the cold plastic deformation techniques such as upsetting, cold forging, rolling on the electrical conductivity in materials such as aluminum, copper, brass, and their alloys and the reviews concluded that electrical conductivity is sensitive to the deformation ratio and differs from a pure metal to an alloy. It is further established that increasing the strength of the wire by the cold deformation leads to a drop in the electrical conductivity and vice versa [254, 255].

Most of the research accomplished to date using numerical and analytical approaches on the wire drawing process have focused on the total reduction and their effects, adopting different combinations of drawing speed, friction, and die design on bulk rods and tubes using a single die drawing process. Limited work has been conducted to examine the impact of drawing practices and their effects on the deformation homogeneity in thin wires drawn with a multiple-step drawing process. Prior investigations have also focused on high Stacking Fault Energy (SFE) materials like copper, aluminum, and steel but not on low SFE materials like MP35N, which exhibit different work-hardening behavior. No compelling evidence was provided to illustrate for the variation in the deformation homogeneity in the wire when drawn with different drawing parameters, and the conclusions to date have been established either with analytical or FEA methods.

The objective of the current study is to understand the deformation homogeneity and electrical conductivity of MP35NLT wires subjected to different drawing practices namely Full Die Drawing (FDD) and Half Die Drawing (HDD), by measuring the hardness distributions and electrical conductivity along the wire cross-section and correlate their relationship to their microstructure, texture and grain size distribution.

5.2 Materials and Methods

MP35NLT wire with an initial diameter of 0.62mm and in annealed condition was used as a starting material for the experiments. The TEM (Transmission Electron Microscope) and STEM (Scanning Transmission Electron Microscope) bright-field (BF) images of the as-received wire are shown in Figure 5.1. The grain size of the annealed wire was in the range 2500 to 6000 nm, and there were several annealing twins observed in the microstructure which were dispersed between the grains. The annealed wire was drawn to different cold work (CW) reductions using FDD and HDD drawing practices, and the samples at the various CW reductions of 16%, 50%, 75%, and 95% were collected and characterized for the hardness and EBSD measurements. The materials and the experimental methods, for wire drawing, hardness testing, electrical conductivity measurements and microstructural methods (EBSD, TEM, and FESEM) were discussed in Chapter 3.

Figure 5.1: Microstructure of the as-received wire (a) TEM BF Image (b) STEM image

For the hardness measurements, three readings from each batch of wire and eighteen readings from the six batches (three batches from FDD and three batches from HDD) of wire, are averaged and plotted for each measurement point. For the electrical resistance measurements, three samples of one meter each from each CW% condition was tested, and the data was averaged. The resistivity values were measured in (ohm/m) and transformed to Siemens per meter (S/m) using the standard relation of $\rho = \frac{L}{(A \times G)}$ where ρ is the resistivity of the material and G is the conductance of the material, L is the length of the specimen, and A is the cross-sectional area.

5.3 Results and Discussions

5.3.1 Influence of drawing practice on the hardness distribution.

For the inherent difficulties encountered in measuring the plastic strain on the fine wires using the conventional measurement techniques, microhardness testing was used to compare the effective strain on the wires quantitatively. Hardness measurements have been used as a useful tool by many researchers [256-259] to study the influence of drawing parameters on the inhomogeneity of strain and redundant deformation.

Figure 5.2: Hardness distribution comparison of the FDD vs HDD wire samples subjected to various CW% (a) 0-50% CW Axis-1 (b) 75-95% CW-Axis-1 (c) 0-50% CW Axis-2 (d) 75-95% CW Axis-2.

Figure 5.2 shows the hardness results (H_v) of the FDD and HDD drawn wires subjected to different CW reductions along Axis-1 and Axis-2. As seen from Figure 5.2, the hardness value at

the surface, was higher for the FDD wires when compared to the HDD wire until 50% CW, and the gradient in hardness ΔH (Hardness at the surface-Hardness at the center) was higher for the HDD drawn wires until 75% CW, after which it reduced at 95% CW. For the FDD samples, the hardness gradient for the 50% and 75% CW samples were comparatively lower than the HDD drawn wires. To understand the reason behind the disparity in the ΔH gradient between the FDD and HDD samples, EBSD and FESEM analysis was performed on the samples deformed to different CW reductions.

Figure 5.3: EBSD Orientation Map of the FDD 16% CW sample (a) Surface of the wire (b) Center of the wire along the drawing direction

Figure 5.3 shows the comparison of the orientation maps between the surface and center, for an FDD sample deformed to a 16% CW. As examined in Figure 5.3, the IPF (Inverse Pole Figure) map at the surface of the wire had a mixture of coarse and fine grains whereas the IPF map at the center had a higher frequency of coarse grains. For a quantitative size comparison, a grain histogram (GH) plot has been obtained from the IPF map and plotted in Figure 5.4. The frequency of fine grains in the range of 0.1 to 0.5 μm was higher, and the distribution of coarse grains between 0.7 to 1.0 μm was lower at the surface of the wire whereas the center of the wire exhibited an inverse effect with a higher frequency of coarse grains and a lower frequency of fine grains. Figure 5.5 shows the band contrast (BC) images, and the grain size comparison for the 16% CW HDD

sample, between the surface and the center of the wire. As seen in Figure 5.5(a) the microstructure at the surface of the wire resembled to be severely deformed with a higher amount of fine grains, whereas the center of the wire, as seen in Figure 5.5(b), appeared homogenous and un-deformed with a higher amount of coarse grains.

Figure 5.4: Grain Histogram of the 16% CW FDD sample (a) Surface of the wire (b) Center of the wire

Figure 5.5: FESEM images of the HDD 16% CW sample (a) Surface of the wire (b) Center of the wire (c) GH-Surface of the wire (d) GH-Center of the wire.

The GH obtained for the HDD wire, is shown in Figure 5.5(c) and (d) and the histogram distributions well correlated with the BC images, where a higher frequency of fine grains in the range of 0.1 to 0.4 μm was noticed at the surface of the wire, however, at the center, the distribution of grains was between 0.5 to 2.0 μm .

Figure 5.6: Orientation Distribution Function (ODF) of 16% CW wires at $\varphi_2=45^\circ$ (a) FDD-Surface of the wire (b) FDD-Center of the wire (c) HDD- Surface of the wire (d) HDD-Center of the wire

Figure 5.6 shows the ODF contour comparisons for the 16% CW FDD and HDD wires, the ODF plots have been used to understand the difference in texture along the respective crystallographic orientations defined by the rotation of their three Euler angles. For the FCC materials subjected to plastic deformation, as the relative orientations appear along the $\varphi_2=45^\circ$ axis [158, 160], the ODF contour plots were compared at $\varphi_2=45^\circ$, $\varphi_1=180^\circ$ and $\phi=90^\circ$. The ODF contours examined, appeared to be significantly different between the FDD and HDD wires, for the FDD wires, the ODF maximum perceived at the surface as well as on the center of the wire as noticed in Figure 5.6(a) & (b). However, for the HDD wires, the ODF maxima were seen only at the wire surface as displayed in Figure 5.6(c) and the center of the wire had zero maxima as seen in Figure 5.6(d), which indicates that there is less deformation at the center for the HDD wire.

This describes as for why a higher ΔH gradient was observed for the 16% HDD sample when compared to FDD wire. The significant difference in the grain size distribution along with the microstructural gradient observed between the surface and the center of the wire would have contributed to the difference in wire's strength, with fine grains contributing to higher strength and hardness, and coarser grains contributing to a lower strength. The marked trend corresponds well with the Hall- Petch relationship, on the enhanced strength in the material with a reduction in grain size, since the average of the surface and center grains dominate in a tensile test and the surface grains alone in a bending test [260, 261]. A smaller microstructural gradient was noticed for the FDD wires between the center and the surface of the wire, thus contributing to a smaller ΔH gradient. The ODF contour plots also display that there exists a texture difference between the surface and center of the wire, with the difference being more significant for the HDD wire.

The ΔH gradient decreased with the increase of CW for both the FDD and HDD wires, with the gradient being lower for the FDD wires and higher for the HDD wires. Microstructure and grain size analysis were repeated on the deformed samples to understand for the drop in ΔH gradient. The 50% CW HDD wire was able to be indexed by EBSD; however, the 75% CW HDD wire was unable to be indexed by EBSD. For the FDD wires, both the 50% and 75% CW wires were unable to be indexed, by EBSD, so FESEM with back-scattered electron detector (BED) mode was used to characterize the grain size and the microstructure of the wires.

Figure 5.7: FESEM Images for the 50%CW FDD sample (a) Surface of the wire (b) Center of the wire.

Figure 5.7 shows the microstructure of the 50% CW FDD wire imaged by FESEM, and Figure 5.8 displays the EBSD image of the HDD wire. The microstructure of the FDD wire looks severely distorted and deformed with a lot of parallel deformation markings across the grains, whereas in HDD wire, less deformed structure and no parallel bands were observed intersecting the grains and grain boundaries. The grains measured in the FDD wire were between 100-200 nm, with the distribution of grains being uniform across the wire surface and the center. However, with the HDD wires as seen in Figure 5.8(c) and (d) there was a greater dispersal of fine grains between 0.1 to 0.45 μm at the surface of the wire, and a larger volume of coarser grains in the range of 0.6 to 1.2 μm at the center of the wire.

The ODF plots shown in Figure 5.8(e) & (f) demonstrated that there was a change of texture orientation with the increase of CW or strain. The ODF plot in Figure 5.8(f) coincided with the ODF plot for the FDD wire in Figure 5.6(b), but the ODF maxima was higher for the 50% CW HDD wire than the 16% FDD wire, which can be due to the increased strain applied by the higher CW reduction. The highly distorted microstructure of the FDD wire demonstrates as why the samples were unable to be indexed by EBSD, as described by researchers in their investigations [159, 160] on the limitations of the spatial and angular resolution of the EBSD equipment in working with samples deformed to a high plastic strain whose grains are in the nano or sub-micron scale. Deformation markings observed in the images of the FDD wires corresponded well with the studies of Kalidindi, Asgari et al. [65, 78, 80, 262] on the deformation of MP35N rods, where they have determined that the deformation markings observed in the microstructure to be the deformation twins and dislocations by TEM.

Figure 5.8: EBSD images of the 50%CW HDD sample (a) Surface of the wire (b) Center of the wire (c) GSD-Surface (d) GSD-Center (e) ODF plot-Surface (f) ODF plot-Center

Figure 5.9 shows the FESEM image comparison for the 75% CW FDD and HDD wires, both the wire microstructures appeared highly deformed, but the severity of deformation was higher for the FDD wire. Deformation markings were observed in the HDD wires at 75% CW both at the surface and center of the wire, with the intensity being higher at the surface as shown in Figure 5.9(c) and (d).

Figure 5.9: FESEM -BSE Images of the 75% CW samples (a) FDD-Surface (b) FDD-Center (c) HDD-Surface(d) HDD-Center

However, for the FDD wires shown in Figure 5.9(a), the deformation markings on the surface of the wire seem to annihilate against the grain boundaries and form a closed-loop structure, when compared to the center. The FDD wires also exhibited a uniform GSD between the surface and the center, with the grain size between 50-150nm, however, for the HDD wires, the grains at the surface of the wire were relatively finer when compared to the center. The following pattern was observed for all the HDD wires, where the grain sizes at the wire surface of the wire were smaller when compared to the center of the wire.

With further increase of plastic deformation to 95%, both the wires could not be characterized even by HRFSEM, but based on the hardness plots it can be identified that the increase of ΔH gradient for the 95% FDD wire was marginally higher than the 75% CW wire but lower than the 50% FDD wire, while the ΔH gradient decreased in the HDD wire. This can be due to the homogeneity in the grain size attained between the surface and the center of the wire with an enhanced plastic strain, which contributed to a reduced gradient in the 95% HDD drawn wire. However, the slight increase of ΔH gradient, observed in FDD drawn wires could be rationalized to the proposition explained in chapter 4, to the “Inverse Hall Petch Effect” which led to decreased strength in the material with the increased plastic strain because of the dynamic recovery of the material. This phenomenon was noticed only in FDD wires and not in HDD wires. With the increased deformation to 95% CW, the FDD wire lost its penchant for dislocation generation, pointing to the suppression of dislocation pile-ups and elevated dislocation sliding at the grain boundaries. This contributed to an increased grain softening [263, 264] in the wire and led to a lower hardness at the center of the FDD wire.

Based on the above experimental findings, it can be concluded that the wires drawn with FDD drawing practice have a homogeneous deformation throughout the wire cross-section as noticed by the uniform hardness and microstructural gradient observed between the center and the surface of the wire. The FDD drawn wires also generated higher strength and hardness when compared to the HDD processed wires.

5.3.2 Influence of drawing practice on their electrical conductivity

Electrical conductivity is an essential property in the design of leads, and it is desirable for the wires built into leads to have a high electrical conductance or low electrical resistance in the order of 5-50 Ω [249, 265]. The reciprocal of the electrical conductivity (G) is termed as the electrical resistivity (ρ), and it measures the degree through which the conductor opposes the flow of current per unit length. As per Matthiessen’s rule [266], the increase in lattice defects is directly proportioned to the increased total electrical resistance or decreased electrical conductivity, and is expressed by the following equation as; $\rho = \rho_{GB} + \rho_d + \rho_v$, where ρ_{GB} , ρ_d , ρ_v are the factors contributing to an increase in total resistivity in the wire due to grain boundaries (low and high angle), dislocations and vacancy-type defects originated in the material upon deforming the material. It was suggested by Hong, et al. [267] that contribution of electron scattering to the vacancy type of defects is minimum during the cold drawing process, so this indicates that the grain boundary scattering and the dislocation scattering are the major sources contributing to an

increased resistivity in materials. For the polycrystalline materials, Mayadas and Brown [268, 269] have proposed that the electron scattering at the Grain Boundaries (GB) and dislocations depends on the electron mean free path (ϕ) and its reflection coefficient (r) which is correlated to its grain size (d) by the following equation;

$$\frac{\rho}{\rho_0} = 1 + \frac{3}{2} \left(\frac{r}{1-r} \right) \frac{\phi}{d} \quad (5-1)$$

Equation (5-1) shows that the grain size and its role in contributing to the GB and the dislocation density is substantial in effecting the scattering of electrons and thus the electrical resistivity because of the variation of ϕ with d . Hence, it is very critical in achieving a balance between the mechanical and the electrical properties which depend on the type and extent of cold work or the plastic deformation the material is put forth. Figure 5.10 presents the electrical conductivity comparison between the FDD and HDD wires drawn to different CW%. It can be observed that the electrical conductivity of the wire reduced with the increase of CW% and the conductivity of HDD drawn wires are higher than the FDD drawn wires until 70% CW, after which the gap between them diminished. This can be attributed to the variation in the deformation mechanics of the wire drawn with different drawing practices, which lead to different strengthening and different microstructures in the wire.

As explained in chapter 4, FDD wire reduced to a 50% CW had higher strength than the HDD wire; however, the electrical conductivity of the wire is lower than the HDD. To understand the reason behind the variation in the electrical and mechanical properties detected in Figure 5.10, between the FDD and HDD samples, the 50% CW samples have been characterized by TEM as shown in Figure 5.11.

The HDD wires had a lower volume of lattice defects (dislocations and twins) in the microstructure when compared to the FDD drawn wires as seen in Figure 5.11(b), and as observed in the FESEM images of the FDD and HDD wires shown in Figure 5.5, Figure 5.8 and Figure 5.9. From Equation (5-1), it can be inferred that lower dislocation density (ρ), and larger grain size (d) contributed to less scattering of electrons and thus an increased ϕ and r in the HDD wires. The results correlated with the observations of [270] who perceived that increased strength in the material by the generation of defects in the wire leads to a loss of electrical conductivity in the wire. This is because of the increased scattering of the conductive electrons, which results in a decreased electron mean free path and increased reflective coefficient, as noticed in the FDD wire.

Figure 5.10: Variation in UTS and electrical conductivity for the various CW% reduction.

Figure 5.11: TEM bright-field (BF) image showing the number of dislocations in the (a) FDD wire (b) HDD wire samples drawn to a similar cold work (CW) of 50%.

With the increase in CW to 75%, the conductivity plots of the FDD and HDD wires merged and the conductivity being reduced. This could be ascribed to the reduced diameter and the grain size in the wire with the increase of the CW%. The electron mean free path [271] in the wires is lowered with the length scale approaching the grain size of the material and thus contributing to a

lowered conductivity due to reduced electron scattering, irrespective of the difference in the amount of dislocation density and lattice defects between the two wires drawn.

5.3.3 Influence of drawing practices on the Inhomogeneous Factor (IF)

In metal forming processes, such as wire drawing, because of its high plastic deformation, there is a severe contribution of redundant work to the stress flow in the workpiece which makes the distribution of stress and strain to be non-uniform. This leads to a condition known as “Inhomogeneous Deformation,” which brings about a heterogeneity in the texture and microstructure of the wires, thus affecting the mechanical and physical properties of the wire. In the current work, the influence of the drawing practices on the inhomogeneous deformation was investigated by calculating the Inhomogeneous Factor (IF). For the experimental approach, as defined by Backofen [31], the hardness values measured across the wire cross-section were used to determine the inhomogeneity in the wire, and the equation for calculating the inhomogeneity is defined by;

$$IF = \left(\frac{H_S - H_C}{H_C} \right) \quad (5-2)$$

where H_S is the hardness at the surface of the wire and H_C is the hardness at the center.

Figure 5.12 shows the comparison of the IF value between the FDD and HDD wires. The ordinate represents the calculated IF values, and the abscissa represents the normalized distance of the wire position, with (0) being the wire center and (1.0) being the surface. The IF value for the FDD drawn wire was lower than the HDD drawn wire, until 50% CW, after which the variation minimized. The IF values increased from the center of the wire to the surface, but the relative increase was higher in the HDD wires when compared to the FDD drawn wires. At 75% CW the curves were very similar to each other, and the gradient between the surface and the center for both the drawn wires was minimal with the HDD drawn wire having a slightly higher IF value at the surface. However, at 95% CW the IF value and the hardness gradient of the HDD drawn wires increased again when compared to FDD drawn wires.

Figure 5.12: IF comparison plots between the FDD and HDD wires drawn to different CW%.

The deformation homogeneity in the wire is described by the internal and mean stress distributions across the wire which depends on the diameter reduction and the die parameters, as shown in Figure 5.13. Nakagiri [272] studied the influence of the die parameters on the chevron crack formation and stress distribution in the wire and concluded that the die to wire contact length or the contact ratio is critical in the design of the drawing process. The contact ratio is a dimensionless parameter as defined by the equation (5-3), and alters with the die angle (α) and the contact length (L) between the wire and the die as shown in Figure 5.13.

Figure 5.13: Schematic drawing of the wire drawing process.

$$\frac{L}{D_0} = \frac{D_0 - D_1}{2D_0 \sin \alpha} \quad (5-3)$$

where L= contact length between the die and the wire

α = die semi-angle

D0=Diameter at the entrance of the die

D1=Diameter at the exit of the die.

In the current work, with α being constant, the contact length is determined by the diameter reduction with FDD drawn wire having higher contact length ratio when compared to the HDD wire due to a higher reduction in FDD drawing practice. Wright and Avitzur [30, 273], have defined the correlation between the contact length ratio and the deformation factor (Δ) as,

$$\Delta = \frac{D_0}{L} \quad (5-4)$$

where Δ is the deformation zone geometry factor, and L is the contact length as defined in equation (5-3).

The Δ parameter defined above is very crucial in determining the level of homogeneity transmitted to the wire by the drawing process. It can be perceived that in HDD wires due to a lower reduction per pass ($D_0 - D_1$), and with a constant α , the contact length (L) between the die and the wire decreases, thus leading to a lower L/D_0 ratio, and thus a higher Δ . The higher Δ leads to an increased amount of the redundant work in the wire, where the deformation is localized only to the surface of the wire thus dominating the inception of tensile stress or the increase of internal stresses in the wire, contributing to the high plastic strain on the wire surface. This causes high deformation at the wire surface and less deformation to the center; this explains why the IF values were identified to be higher in the HDD wires. However, for the FDD drawn wire samples with a higher contact length ratio, due to a higher reduction per pass ($D_0 - D_1$), the deformation penetrates through the thickness of the workpiece, unlike the HDD drawing practice where the deformation is localized to the surface. This contributes to a lower redundant deformation factor and increased compressive stresses on the wire surface, leading to lower Δ values and a homogenous deformation in the wire.

The perceived differences in the IF results observed in Figure 5.12 harmonized well, to the microstructural gradient and to the differences in the plastic strain observed between the FDD and HDD wires, with the gradient being reduced with the increase of CW%, thus conferring to a lower IF. The rationale behind high IF value for the 16% and 50% CW HDD wire can be ascribed to the difference in plastic strain disseminated on the wire by the different drawing practices, with HDD drawn wire inflicting higher strain on the surface of the wire when compared to the center thus contributing to a small grain structure at the surface and coarse grain structure at the center as shown in Figure 5.8, thus transcending to higher IF values. However, at 75% CW a similar microstructure observed between the wires drawn as shown in Figure 5.9 might have contributed to the flat IF peaks in Figure 5.12.

At 95% CW the IF value for the HDD drawn wire increased again and was higher than the FDD drawn wire. This can be attributed to the inability of the material drawn with the FDD process, to absorb or store the deformation energy in the material after a particular plastic strain leading to plastic strain localization [274], with the material ceasing to deform with the increased plastic strain and thus transiting to failure [275] with further deformation.

Figure 5.14: TEM BF Image of the 95% CW drawn wires (a) FDD (b) HDD

However, with the HDD drawn wire, due to the smaller reduction per pass, the materials ability to withstand further plastic strain was higher as observed from the TEM images in Figure 5.14, where the FDD microstructure appeared to be severely deformed with a lot of dislocations and twins in the microstructure whereas, in HDD drawn wire, the number of twins and dislocations are comparatively lower. The higher IF value implies existence of higher redundant work in the

workpiece with an increased level of non-homogenous deformation. However, for the FDD wires, the deformation and stress distribution between the surface and the center of the wire surface was uniform which contributed to uniform microstructure, lower hardness gradient and smaller IF values, therefore, inducing a homogenous deformation in the wire.

5.4 Summary

The effect of drawing practices on the level of deformation inhomogeneity and electrical conductivity in MP35NLT wires was experimentally studied. Based on the above findings, the following conclusions can be outlined:

- Full Die Drawing (FDD) wires had a lower hardness gradient (ΔH) gradient across the wire cross-section when compared to Half Die Drawing (HDD) wires for a similar cold work reduction. The ΔH gradient decreased with the increase of CW% up to 75% CW, after that increasing marginally for the FDD wire and decreasing for the HDD wire.
- FDD drawn wires had a uniform microstructure and grain size distribution between the center and the surface of the wire, whereas the HDD drawn wires had a non-uniform microstructure with fine grains at the wire surface and coarser grain at the center. The difference in the microstructure gradient reduced with the increase of CW%. The FDD wires had a higher frequency of fine grains and twins in the microstructure, which contributed to higher hardness and strength in the wires.
- The Orientation Distribution Function plots (ODF) for the FDD and HDD wires varied considerably between the wire surface and the center, with the maxima observed at the wire surface, but the difference of texture orientations was higher in the HDD drawn wire when compared to FDD wire.
- The electrical conductivity of the HDD wires was higher than the FDD drawn wires, but the relative increase of strength to the loss of electrical performance was minimal, which makes the FDD process best suited for drawing Co-Cr-Ni-Mo wires.
- The difference in the electrical conductivity between the two-drawing process was seen only until 50% CW, after which the two curves merged, irrespective of the higher amount of dislocations and lattice defects observed in the FDD microstructure.
- The Inhomogeneous Factor (IF), which determines the level of inhomogeneity in the wire was lower for the FDD wires and the value decreased with the increase of CW%.

Chapter 6 Plastic Instability in Co-Cr-Ni-Mo alloy wires drawn with different drawing practices.

6.1 Introduction

Metals when plastically deformed by traditional forming methods such as rolling, forging, extrusion, wire drawing, significantly improved the strength of the material, however, the exceptional increase in strength has been compensated by the loss of ductility in many materials [276, 277]. This has been attributed to the flow localization in the material, which contributed to an expedited necking in the material during its tensile deformation in monotonic test conditions. This localized phenomenon of strain localization [278] causes the deformation inhomogeneous driving to a phenomenon called plastic instability (PI) [279-281]. On the onset of the plastic instability, the material loses its load-bearing capacity, and the stress continues to decrease with the increase of strain, leading to a catastrophic fracture [282].

The first exemplary standard for measuring the plastic instability was adduced by Considere [283] who proposed, necking onset as the point where the strain hardening coefficient (γ) drops below the value of the flow stress (σ) for a given plastic strain rate ($\dot{\epsilon}$). It is defined as;

$$\gamma = \left(\frac{\partial \sigma}{\partial \epsilon} \right)_{\dot{\epsilon}} \quad (6-1)$$

The above equation is applicable only to strain rate insensitive materials, and the relation was defined entirely based on the true stress and true strain data. Hart [74] later extended the criterion, to include the strain rate sensitivity (m) of the flow stress and defined plastic instability as:

$$\gamma + m \leq 1 \quad (6-2)$$

where m is defined as;

$$m = \frac{\partial(\ln \sigma)}{\partial(\ln \dot{\epsilon})} \bigg|_{\epsilon, T} \quad (6-3)$$

Equation (6-2) illustrates that the plastic stability in the materials is defined based on the strain hardening rate and the strain rate sensitivity of the material. Several authors [129, 284-290]

have revisited the Hart and the Considere criteria and proposed many models to include the intrinsic defects generated during the plastic deformation, and derived many constitutive equations for the various testing conditions of constant load, stress, strain rate and reduction in area. However, all the governing principles still conclude the instability dependence on m and γ , as defined by Hart, but with different state variables. Most of the instability criterion propounded is based on the assumption that the plastic deformation is on a macroscopic scale and homogeneous [83, 291, 292]. However, in the actual case, the strain is inhomogeneous and needs to be analyzed on the microscopic level to include the effects of intrinsic state variables such as dislocations, shear bands, slip bands, twins, and their contributions to the plastic strain localization.

With the plastic instability defined by the strain hardening exponent (γ) and strain rate sensitivity (m), the factors governing the γ and m , such as material type, grain size, texture, orientation of the crystal lattice, dislocation density, strain rate, temperature, processing method, and prior plastic deformation, play an essential role in controlling the plastic instability. E. Ma [133] studied the instability in nanocrystalline and ultrafine-grained Cu and concluded that plastic instability increases with the reduction of the grain size, and attributed this effect to the inability of the material to store the dislocations, thus losing its strain hardening capacity. However, she proposed several strategies [134] to reduce the plastic instability of the materials with a low γ , by increasing the strain rate sensitivity (m), through the optimization of the grain size distribution and the grain boundary sliding mechanisms.

Most of the prior research conducted on the PI of metals, involved deformation processes such rolling, forging, equal channel angular pressing (ECAP) and superplastic deformation (SPD) techniques, where the method of deformations is entirely different when compared to cold wire drawing. Earlier work on the PI involved deformation on bulk samples in the diameter range of 50-10mm, where the deformation and the heat treatment methods are undoubtedly different when compared to fine wires as in the current scenario. To the best of the author's knowledge, there were no prior plastic instability studies reported on Co-35Ni-20Cr-10Mo LT (MP35NLT) alloy, which is considered being a superalloy with a low stacking fault energy (SFE).

So, in the present work, investigation of plastic instability of MP35NLT was performed. The material is cold drawn to fine round wires, with different drawing practices namely the Full Die Drawing (FDD) and Half Die Drawing (HDD) and the necking instability is determined by varying the prior deformation rate or the cold work (CW%) on the samples. The deformed samples

were characterized using a Transmission Electron Microscope (TEM) to understand the relationship between the deformation behavior and plastic instability.

6.2 Materials and Methods

The materials and the experimental methods, for wire drawing, tensile testing, and TEM characterization are discussed in Chapter 3. For the tensile tests, the gauge length was maintained at 254 mm, and the samples were subjected to a strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$. The testing was performed at room temperature, and the samples were tested until fracture. Six batches of wire are manufactured (three batches with FDD practice and three from HDD practice), and the wires of the required length were collected at the particular operation stage before proceeding with the drawing operation. Engineering stress-strain and true stress-strain curves were calculated from the raw data of the tensile curve, and the strain hardening rate and the strain rate sensitivity values were determined from the double logarithmic plots of the stress vs. strain and stress vs. strain rate. Three samples from each batch are tested, and data from a total of nine samples were analyzed for each condition of 50, 75, and 95% CW. The mean and the standard deviation (error plots) of the obtained and calculated results are illustrated in the figures below.

6.3 Results

6.3.1 Mechanical Results

Engineering stress-strain curves of the wires subjected to different cold work reductions and drawn with different drawing practices are shown in Figure 6.1. The annealed wire had a higher ductility (EL%) and lower strength, as shown in Table 3-2, when compared to the deformed or drawn wires, with the ductility decreasing with the increase of deformation. The FDD wires had higher strength and lower ductility when compared to the HDD wires, except at 50% CW where higher strength and higher ductility was detected in the FDD drawn wire. At 95% CW, the HDD wire exhibited higher strength and ductility than the FDD drawn wire. The reason for the abnormality in the stress-strain behavior with the variation in the CW reduction will be explained through microstructural characterization in the sections below.

Figure 6.1: Engineering stress-strain curves of the wires subjected to different CW% and drawn with different drawing practice.

True stress and true strain data were calculated from the engineering stress-strain curve data, and the strain hardening coefficient (γ) and the strain rate sensitivity (m) values were determined from the double logarithmic plots of stress versus strain as shown in Figure 6.2(a), and stress versus strain rate as shown in Figure 6.2(c) for the resolution of γ and m . The computed values of γ and m for the wires drawn with different drawing practices and to various CW reductions are plotted in Figure 6.2(b) and Figure 6.2(d).

(a)

(b)

(c)

(d)

Figure 6.2: Influence of drawing practice on the (a) Calculation of γ from Ln (True Stress) vs Ln (True Strain) for a 50% CW drawn wire (b) Strain hardening exponent (γ) plotted for different CW% (c) Calculation of m from Ln(True Stress) vs. Ln (True Strain Rate) for a 50% CW drawn wire (d) Strain rate sensitivity (m) plotted for different CW%.

As seen in Figure 6.2, the γ and m values increased with the increase of cold work reduction until 75% reduction, after which a drop in the coefficients was noticed until 95% CW. The relative increase in the coefficients until 75% CW and the reduction in the coefficient's after 75% CW were observed to be higher in the FDD drawn wire than the HDD wire. Figure 6.6 and Figure 6.7 compares the PI and the EL and the RA% between the FDD and HDD drawn wires for different CW reductions.

6.3.2 FIB, TEM, and EDX characterization.

Due to the necessity for thin electron transparent samples for TEM characterization [293], the wire samples have been pre-thinned to a size of 100-300nm lamellae using a FIB milling technique.

(a)

(a)

(c)

Figure 6.3: FIB sample preparation of the as-received sample (a) Pt deposit onto the bulk area (b) Omniprobe Lift out Technique (c) Final polished image of the milled lamellae.

The as-received annealed wire was deposited with a Pt layer, as shown in Figure 6.3(a) and is milled in three steps. The first step involves milling the pre-selected area with a probe current of 20nA; then the beam current has been reduced to 3000pA to mill the lamellae to 1.5μm thick. The thinned sample was lifted out of from the bulk area using the Omniprobe lift-out technique shown in Figure 6.3(b) and attached to the TEM copper grid. A 30kV ion beam was

used for all the milling operations, and the final cleaning operation was done at 5kV and 100pA. The final thinned section of the lamellae is shown in Figure 6.3(c).

The annealed wire received at 0.62mm had a completely recrystallized structure with the grain sizes between 2600-6000nm as shown in Figure 5.1, and the microstructure had a lot of annealing twins (thick and thin) embedded within the grains as shown in Figure 6.4(a) and Figure 6.4(b), with the twins in the range of 30-180nm.

Figure 6.4: TEM images of the annealed wire (a) Thick annealing twins and SAD inset of the twin (b) Thin annealing twins

The Selective Area Diffraction (SAD) inset shown in Figure 6.4(a) confirms the indexed area to be a twin, as observed by a misoriented diffraction pattern. Energy Dispersive X-ray analysis (EDX) on the received sample was performed with the EDAX detector attached to the TEM machine, and the spectrum is shown in Figure 6.5. The four principal elemental compositions of the alloy used in the current study matched with the ASTM F562 standard composition shown in Table 3-1, with the cobalt (Co) having the highest weight percentage in the alloy followed by nickel (Ni) and chromium (Cr) and molybdenum (Mo) having the lowest weight percentage .

Figure 6.5: EDX pattern of the as-received sample.

Figure 6.6: Plastic Instability comparison of the FDD and HDD drawn wires to a similar CW reduction.

Figure 6.7: Comparison between the FDD and HDD wires (a) Ductility (EL%) (b) Reduction in cross-sectional area (RA%).

6.4 Discussions

6.4.1 Influence of drawing practices on the plastic instability of a 50% CW wire.

Plastic instability is an important factor in understanding the material behavior and its response to the plastic deformation which it is subjected to, in defining its appropriateness to the desired engineering applications. It is considered being a trade-off between the strain hardening and the strain rate sensitivity of the material which resists or delays the necking in the material when subjected to a uniaxial tensile deformation [139, 294]. So, the control of plastic instability [132] in the material is essential from an engineering standpoint of view, especially when the grain size is reduced to nanoscale for the desired strength improvements. In the current work, the plastic instability of the Co-Cr-Ni-Mo thin wires is studied, by subjecting the material to FDD and HDD drawing techniques and analyzing their response on the instability for a similar cold work reduction. The plastic instability has been studied from a microscopic viewpoint by testing the factors contributing to the instability such as grain size, dislocation density, shear band formation, twin density.

Figure 6.6 shows the plastic instability comparison of the FDD and HDD wires drawn to a similar CW reduction. As defined in equation (6-2); and as per Harts criterion, the material is considered being stable if $\gamma+m>1$. In the current study, it can be noted that the wires subjected to FDD and HDD drawing practice are relatively unstable, but the plastic instability (PI) decreases with the increase of CW until 75% CW and then increases again. The high instability can be attributed to the low surface-area-to-volume-ratio of the thin wires when compared to the bulk samples. It can be observed that the PI of the FDD wire is lower when compared to the HDD wire at 50% CW reduction, with the $(\gamma+m)$ value being 0.462 for FDD wire and 0.314 for HDD drawn wire. The higher $(\gamma+m)$ in FDD wire can be ascribed to the higher strain hardening coefficient noticed in the FDD drawn wire when compared to the HDD wire as shown in Figure 6.2(a), where a γ value of 0.435 was obtained for the FDD drawn wire, whereas the HDD wire had a smaller γ value of 0.303. The results corresponded well to the higher strain rate sensitivity values (m) observed in the FDD drawn wire when compared to the HDD drawn wire ($m=0.027$ for FDD and $m=0.011$ for HDD). The ductility (EL%) and the cross-sectional reductional (RA%) in the wire are compared between the FDD and HDD wires and plotted in Figure 6.7. It can be seen that the FDD drawn wire for a 50% CW reduction has a ductility of 4.45% and an RA of 55% whereas the HDD wire has a ductility of 4.11% and an RA of 51%. The following observation corresponded

well with the results of Wang et.al.[295] who concluded that increased m delays the onset of the necking in the material, which leads to an improved ductility as noticed in the FDD drawn wire. The deformed wire samples were characterized by TEM and compared as shown in Figure 6.8 and Figure 6.9

The higher γ observed in 50% CW FDD wire than the HDD drawn wire is explained based on the following observations: The FDD wire had a smaller grain size than the HDD wire for a similar CW reduction, as seen in Figure 6.8(a) and

(e)

(f)

(g)

(h)

Figure 6.9(a). The grain size histogram shown in Figure 6.8(b) and Figure 6.9(b) has been plotted by measuring the grain size across many TEM images (about 400-500 grains for each sample). It can be seen from the histogram plots that the FDD wire had a higher volume fraction (VF) of grains between 50-100nm when compared to the HDD wire which had a higher VF of grains in the range of 160-200 nm. Danaf et al.[78] in his study, described that the smaller the grain size of the material, the higher is the work hardening rate for the material. Also, the dislocation density of the FDD drawn wire was relatively higher than the HDD drawn wire, as noticed in Figure 6.8(d) and Figure 6.9(d). The results support well the observations of [111, 296, 297], on the FCC metals, in which increasing the plastic strain or applying higher strain on the materials contributed to higher work hardening rate (γ) because of the increase in the dislocation density and suppression of dynamic recovery.

The higher m seen in the FDD drawn wire can be explained based on the equation proposed by Conrad [126] whereby the relationship between the dislocation density, activation volume and m was shown to be as :

$$m = \frac{\sqrt{3}kT}{\sigma \cdot v} \quad (6-4)$$

where v is the activation volume of the material, which can be defined as the number of atoms involved in the thermal activation event of the moving dislocations, to overcome the localized obstacles along the slip planes. As v scales inversely with the dislocation density (ρ) [141, 298], it

can be described that the higher dislocation density detected in the FDD drawn wires, contributes to a lower activation volume in the material and thus inducing a higher m as per equation (6-4).

The increased γ and m of the FDD drawn wires can also be explained based on the observations of twins in Figure 6.8 and Figure 6.9. There were several macro, micro twins noticed in the microstructure of the FDD drawn wire as shown in Figure 6.8(e) and (f), and there were several nano twins embedded within the grains and between the grain boundaries as seen in Figure 6.8(g). The SAD (Selective Area Diffraction) and FFT (Fast Fourier Transformation) insets performed on the specific region of interest highlighted in the red box, confirm the diffraction spot to be a twinned region. However, for the HDD drawn wire examined in Figure 6.9, there were fewer amount of twins observed in the Dark Field (DF) image of the specimen as shown in Figure 6.9(e), and profuse slip bands were noticed in the Bright Field (BF) image of the HDD drawn wire as shown in Figure 6.9(f). There was an increase in the number of misorientations observed between the grains and their boundaries with the grain boundaries besieged by dislocations as shown in Figure 6.9(g) and (h).

The SAD and the FFT insets examined from the selected area, exhibit the presence of a texture rather than a misoriented twin profile as in FDD samples. The higher twin density observed in the FDD wires compared to the HDD drawn wires might have led to increased resistance of the movement of the mobile dislocations, thus acting as a barrier to the free dislocation motion, and leading to a kinematic hardening effect known as dynamic Hall Petch effect [66, 299, 300] constituted by the deformation twinning. The following observations demonstrate that the amount of plastic deformation or strain put forth on the Co-Cr-Ni-Mo wire drawn with the FDD drawing practice was higher than the HDD drawing process, which led to decreased grain size, higher dislocation density, higher twin density and an overall higher γ and m [301]. The significant plastic work retained in the FDD drawn wire, due to lower plastic instability contributed to higher strength and higher ductility in the wire.

(a)

(b)

(c)

(d)

(e)

(f)

(g)

(h)

Figure 6.8: TEM images of the FDD wires drawn to 50% CW reduction (a) Grain size STEM image (b) Grain Size Distribution (c) Bright Field (BF) TEM Image showing highly deformed microstructure (d) BF TEM image showing the dislocation density of the sample. (e) Darkfield (DF) image showing macro twins (f) Bright Field (BF) Image with SAD inset showing the macro twins. (g) BF image with SAD inset showing the micro twins (h) BF image with SAD inset showing the nano twins at the grain boundary.

(a)

(b)

(c)

(d)

(e)

(f)

Figure 6.9: TEM images of the HDD wires drawn to 50% CW reduction (a) Grain size STEM image (b) Grain Size Distribution (c) Bright Field (BF) TEM Image showing less deformed microstructure (d) BF TEM image showing the dislocation density of the sample. (e) Darkfield (DF) image (f) Bright Field (BF) Image with SAD inset showing the presence of slip bands. (g) BF image with SAD inset showing the misorientations in the grains (h) BF image with SAD inset showing misorientations at the grain boundary.

6.4.2 Influence of drawing practices on the plastic instability of a 75% CW wire.

As the amount of plastic deformation on the material increases, the grain size decreases and the work hardening capacity (γ) of the material decreases due to the inability of the material to create new dislocations, or due to the accumulation and rearrangement of the dislocations in the microstructure leading to dislocation annihilation. This leads to a lower γ and m , as explained in prior studies performed by several researchers [110, 302-308]. To corroborate this effect, the wires have been subjected to higher plastic deformation, by increasing the CW reduction to 75%. Figure 6.6, shows that the $(\gamma+m)$ value for both the FDD and HDD drawn wires surged with the increase of deformation to 75% CW, with the gain being higher for the HDD wire (60%) than the FDD wire (11%) when compared to the results at 50% CW. The FDD drawn wire had a γ of 0.473 and m of 0.044, which led to $(\gamma+m)$ value of 0.517, unlike the HDD drawn wire which had a $(\gamma+m)$ value of 0.501, with γ and m values being 0.462 and 0.039 respectively. It is noted that that the increase of γ and m value for the 75% FDD wire shown in Figure 6.2 was lower than the HDD

drawn wire when compared to 50% CW. The ductility of the FDD wire increased by 4%, and HDD wire ductility increased by 13% when compared to that of 50% CW. This explains as to why the relative increase of PI of the HDD was higher, due to the higher work hardening rate observed in the 75% CW wire. It can be observed from Figure 6.10 (a) and (b), that the grain size of the FDD drawn wire decreased with the increase of plastic deformation and a higher volume fraction of the grains were found in the range of 40-60nm. There was also a grain size reduction in the HDD drawn wire, as observed in Figure 6.11(a) and (b), where a higher volume fraction of grains in the range of 80-90nm was noticed.

(a)

(b)

(c)

(d)

(e)

(f)

(g)

(h)

Figure 6.10: TEM images of the FDD wires drawn to 75% CW reduction (a) Grain size STEM image (b) Grain Size Distribution (c) Bright Field (BF) TEM Image showing highly twinned microstructure (d) BF TEM image showing the dislocation density and twinned microstructure of the sample. (e) Darkfield (DF) image showing macro twins with reduced twin spacing (f) Bright Field (BF) Image showing the closely spaced macro twins. (g) BF image with SAD inset showing the high density of micro twins (h) BF image with SAD inset showing the increased density of nano twins in the microstructure and formed inside the grains.

(a)

(b)

(c)

(d)

(e)

(f)

Figure 6.11: TEM images of the HDD wires drawn to 75% CW reduction (a) Grain size STEM image (b) Grain Size Distribution (c) Bright Field (BF) TEM Image showing the increased deformed microstructure (d) BF TEM image showing the increased dislocation density of the sample. (e) Darkfield (DF) image showing the dislocation twins (f) Bright Field (BF) Image showing the increased dislocation density in the microstructure. (g) BF image with SAD inset showing the twinned microstructure with the dislocated structure (h) BF image with SAD inset showing different misorientations at the grain boundary and within the grain.

The average grain size of the FDD drawn wire reduced from 120nm to 47nm, as shown in Figure 6.10(b) when the CW reduction has been increased from 50 to 75%; this contributed to increased strength in the wire as shown in Figure 6.1, according to the Hall Petch effect. However, it also led to a slightly increased γ , which was an unlikely phenomenon with FCC metals. The increase of work hardening, even with a reduced grain size in the FDD drawn wire can be attributed to the increased density of twins in the microstructure as seen in Figure 6.10(e) to (h). Deformation twinning is known to play an essential role in the increased hardening of the low SFE materials [309, 310] such as the Co-Cr-Ni-Mo (MP35NLT) alloy used in the current study. The twins originated in the microstructure operated as a twin boundary, creating obstacles to the local mobile dislocation's movement, thus increasing the local stresses at the dislocation pile-ups and the grain boundaries. This leads to an increased misorientation and reorientation of the grains, which may engender to subdivide grains thus causing an increased work hardening in the material. The increase of m with the increased strain can be explained by the model proposed by Champion [112], who proposed an equation correlating the relation between m to the twin spacing (δ) as;

$$m = B(1 - \frac{\sigma_0}{\sigma}); \quad (6-5)$$

$$\text{and } \frac{1}{B} = 2 \left[1 + \frac{\delta}{bd^2v_e} \times \frac{\partial v_{gb}}{\partial \rho} \right];$$

where σ_0 =critical stress required to initiate a twin, σ = applied stress, ρ =dislocation density

δ =twin spacing, d =grain size, v_e , and v_{gb} are the frequency of dislocation emission and absorption.

As per Equation (6-5), the lower the twin spacing, the higher would be m , leading to an increased SRS and higher resistance to necking when compared to a less twinned structure. Figure 6.10(f) clearly shows that the twin spacing and the width of the twins in the 75% CW FDD sample are lower when compared to the 50% CW FDD sample in Figure 6.8. This demonstrates that the smaller twin spacing observed in the 75% CW FDD wire, would have led to lowered grain boundary properties, such as dislocation interactions, due to their hindrance to the dislocation motion and also providing higher resistance to grain boundary sliding. The increase of the twin boundaries would have also led to the redistribution of stresses in the material at the grain boundaries, thus accommodating for a higher strain in the grains, thus preventing the strain localization in the grains. The boundaries surrounding the twinned zones can act as potential dislocation sources by increasing the dislocation density and thus an increased m .

However, for the HDD drawn wire, there was a reduction in the average grain size from 170nm to 75nm, and also the dislocation density increased with the increase of CW from 50 to 75% CW as seen in Figure 6.11. So, the increased γ , m and ductility for the 75% CW HDD drawn wire can be explained on a similar note as in 50% CW FDD wire, where a smaller grain size and a higher dislocation density contributed to a higher work hardening and a higher SRS or a lower PI in the wire due to higher resistance to necking.

6.4.3 Influence of drawing practice on the plastic instability of a 95% drawn wire.

To further understand the influence of increased deformation on plastic instability (PI), the wires were subjected to a 95% CW reduction, and their influence on the drawing practice is characterized. Figure 6.6 shows that the $(\gamma+m)$ value of the FDD drawn wire decreased significantly from a value of 0.517 at 75% CW to 0.295, with the increase of CW to 95%. However, for the HDD drawn wire, the $(\gamma+m)$ value was lowered from a value of 0.501 to 0.393. The higher drop of the PI of the FDD drawn wire corresponds well with the reduced (γ) and (m) shown in Figure 6.2. Observation of the deformed microstructures under TEM revealed some interesting

findings. The FDD drawn wire had several parallel thick and thin bands distributed through the microstructure as seen in Figure 6.12(a) and (b). From Figure 6.12(c) it can be observed that the grain size reduced to a range of 20-40nm, with the average grain size being around 35nm.

Figure 6.12: TEM images of the FDD wires drawn to 95% CW reduction (a) BF Image showing the shear bands formed in the microstructure with fewer twins (b) BF TEM image showing the grain size and the shear bands microstructure aligned to the drawing direction. (c) Grain Size Distribution (d) High magnification image of the shear band with SAD inset confirm the texture profile for the bands.

(a)

(b)

(c)

Figure 6.13: TEM DF images of the FDD wires drawn to 95% CW reduction (a) Multiple thin shear bands originated in the microstructure (b) High magnification image of the shear bands intersecting the twins and the dislocations (c) Shear bands formed are 20-30nm in thick which are relatively equal to the grain size in the sample.

The following observations coincided with the investigations of other researchers [302, 311-313], who confirmed the thick parallel bands, to be shear bands. The following phenomenon can explain the formation of shear bands: with the enhanced grain refinement in the FDD wire with increasing deformation, the material loses its inability to store and create dislocations, thus reducing the strain hardening ability of the material. The reduced hardening deprives its effectiveness to resist the increased plastic flow, thus causing localization of strain in the material

due to the increased shear stress after the onset of the plastic deformation. The shear stress localization [314, 315] generates shear bands in the material which are aligned at an angle of 20-45° to the loading direction. Based on the concentration and distribution of strain, the shear bands exhibit different thickness, which can be equal to the grain size of the material, as shown in Figure 6.13(c).

The SAD pattern inset of the shear band shown in Figure 6.12(b), shows sharp maxima of the {111} reflection indicating texturing. The low values of γ and m observed in the FDD drawn wire can be due to the debilitation of the material for dislocation storage and increased plastic strain localization due to shear banding. This could also explain the drastic drop in the ductility and the RA% observed for the FDD wire shown in Figure 6.7, where the EL% lowers down to 3.96%, from 4.64% and the RA decreased to 43%. It shows that with the increase in plastic strain localization, the material loses its ability for a uniform, homogenous deformation, thus leading to the onset of early necking and high plastic instability.

There was a reduction in the γ and m values for the HDD drawn wire also, but the drop was relatively insignificant when compared to the FDD drawn wire. There were no shear bands observed in the HDD drawn wire TEM images shown in Figure 6.14 and Figure 6.15, and this confirms that there is no plastic strain localization. The average grain size of the sample decreased from 75nm, as seen in a 75% CW sample, to 60nm as shown in Figure 6.14(b), which was comparatively higher than the FDD average grain size of 35nm. However, the microstructure of the HDD sample, looked severely annihilated, with many dislocation intersections, as shown in Figure 6.14(c) and (d). The dislocations also appear to pass through the grain and form triple junctions at the grain boundaries as shown in Figure 6.15(b) and (c), and there were also a significantly lower amount of twins observed in the microstructure as seen in Figure 6.15(a).

(a)

(b)

(c)

(d)

Figure 6.14: : TEM images of the HDD wires drawn to 95% CW reduction (a) Grain size BF image (b) Bright Field (BF) TEM Image showing the microstructure with fewer twins and dislocation annihilated microstructure (c) BF TEM high magnification image showing the increased dislocation annihilation in the cell structure.

Figure 6.15: TEM images of the HDD wires drawn to 95% CW reduction (a) DF image showing the highly distorted structure with less twin density. (b) BF TEM image showing the dislocation penetrating the grains through its grain boundaries (GB). (c) Triple junction formed at the intersection of the three GB.

It is known that increased dislocation annihilation in the material contributes to a lowered dislocation density and reduced work hardening due to increased grain boundary shearing and sliding mechanisms [316-318]. The reduction in m for the HDD drawn wire can be illustrated by the equation proposed by Yasnikov et al. [319] which describes the relationship between m , k_2 , and ρ as:

$$\frac{\gamma}{\sigma} + \frac{1}{m} \left(k_2 M - \frac{1}{2} \frac{d \ln \rho}{d \varepsilon} \right) \leq 1 \quad (6-6)$$

where k_2 is the dynamic recovery coefficient leading to a loss of dislocation density due to dislocation annihilation,

M = Taylor factor

ρ = dislocation density and ε is the plastic shear strain rate.

As per Equation (6-6), it can be noted that the reduction of ρ due to an increased dislocation annihilation, leads to a higher value of k_2 and therefore a reduced m as noticed in Figure 6.2(b). The lowered m also decreases the resistant to necking and thus increasing the PI of the material. Based on the above results, it can be inferred that the amount of plastic deformation or strain imparted to the wire by the FDD drawing technique is higher than the HDD drawing practice. For the FDD drawn wire, the microstructural facets such as grain size refinement, dislocation induction, and the twinning mechanisms are observed to occur at a lower CW% reduction when compared to an HDD drawn wire. It was also noticed that the critical resolved stress required for the deformation twinning to initiate a slip system change and activation of multiple slip systems in the material is higher for the FDD drawn wire.

6.5 Summary

From the above results and discussions, the following conclusions can be made comparing the influence of the drawing practices, i.e., FDD vs. HDD on the plastic instability of the Co-Cr-Ni-Mo wires drawn to different CW reductions.

- (1) Wires drawn with different drawing practices contributed to various microstructural heterogeneities, such as grain size, dislocation density, twin density, shear bands for a similar CW reduction; this lead to a significant difference in the plastic instability (PI). The FDD drawn wires had a lower PI until 75% CW, which increased significantly with the increase of CW reduction to 95%. However, with the HDD drawn wire, the PI was higher at 50% CW, after which the instability margin reduced at 75% CW, and then subsequently increased back again at 95% CW, with the increase being lower than that in the FDD drawn wire.
- (2) Higher strain hardening exponent (γ) and strain rate sensitivity (m) were obtained from the wires drawn with FDD practice when compared to the wire drawn with HDD practice. For a 50% CW reduction, this was attributed to the difference in the dislocation density and

activation volume between the wires, with the FDD wires having a lower activation volume or higher dislocation density when compared to HDD wires.

- (3) There was an increase of γ and m values in the FDD drawn wire even with a reduction of the grain size to less than 50 nm; this unusual phenomenon can be ascribed to the increased density of the twins and the reduction of twin spacing, which led to an increased m and delayed the onset of necking, leading to lower PI.
- (4) Extensive shear bands were observed in the FDD wire drawn to a 95% CW reduction; this led to plastic strain localization due to the exhaustion of the dislocation density with the reduced grain size; this significantly reduced the γ and m values. This phenomenon was not seen in the HDD drawn wire, and severe dislocation annihilation was noticed in the HDD drawn wire, drawn to a 95% CW reduction.

Chapter 7 Strain rate effects on the strain rate sensitivity and strain rate hardening in Co-Cr-Ni-Mo wires.

7.1 Introduction

The deformation resistance or the fracture mechanisms in the material subjected to plastic deformation are determined by its grain size, the applied strain rate, and its processing temperature [100]. As the strain rate determines the loading sensitivity of the material over time, the understanding of the plastic behavior of the metal when subjected to different strain rates is of great importance for ensuring the dependability and endurance of the material, during its service. The influence of the strain rate on the plastic deformation of the metals is determined by a parameter called Strain Rate Sensitivity (SRS) which is defined by $m = \frac{d \ln \sigma}{d \ln \dot{\epsilon}} |_{\epsilon, T}$, where σ is the applied stress and $\dot{\epsilon}$ is the applied strain rate for a given strain and temperature [320]. Over the last few decades there, were many constitutive relations developed interrelating the influence of strain rate, strain, and temperature to the SRS [101, 105, 321], and its effect on the deformation mechanisms in the material. Many prior studies, were also performed to examine the influence of the grain size on the SRS of metals [107, 126, 322], and it was concluded that the nanocrystalline materials have a profound effect on the strain rate when compared to the conventional grain size. It was also demonstrated that the lattice structure also influences the SRS, with the bcc metals having an increased SRS and the fcc and hcp metals having a decreased SRS with the increase of grain size [141].

The other critical parameter which is found to be sensitive to the applied strain rate and the flow stress in materials is the strain hardening rate sensitivity (SHRS) of the material. It depends on, similar processes and structural variables as defined for SRS, but it also depends upon other intrinsic variables such as stacking fault energy (SFE) and the sensitivity of the material to deformation twinning. It is defined by the relation $\theta = \frac{d\sigma}{d\epsilon}$. Several researchers have proposed that in fcc metals, the strain hardening rate increases with the increase of strain rate [134, 296, 297]. However, the results published on Twin Induced Plasticity (TWIP) steel which is also an fcc material, contradicted the above statement, where the strain hardening rate was observed to decrease with the increase of strain rate [111, 300, 323, 324]. This concludes that the hardening rate in the material is highly dependent on the structural changes developed in the material enroot its plastic deformation, such as the density of mobile free dislocations, the amount of twinning in

the material and their hindrance to the dislocation motion. The active involvement of grain boundaries (GB) and its mechanisms such GB sliding and shearing also play an essential role in the hardening of the material.

Prior research performed on Co-35Ni-20Cr-10Mo (MP35N) alloy, encompassed mostly on its strain hardening behavior and its response to variables such as grain size [78, 262], deformation path [65, 79], and temperature [64, 82], in bulk tube and rods which are processed by operations such single die drawing, rolling, forging and hot forming. The processing steps involved in the manufacture of bulk tubes and rods are entirely different from that of fine wires, where multiple drawing steps, faster drawing speeds, and shorter heat treatment cycles are used for producing the thin wires [273]. As discussed in Chapter 4, 5 and 6, the influence of drawing practices on the mechanical, electrical, microstructure and the deformation homogeneity in MP35N wires, subjected to different cold work (CW) reductions, were evaluated and the results conclude that the drawing practice had a significant impact on the deformation mechanisms and mechanical behavior of the material. There were no prior studies performed comparing the effect of strain rate on the SRS and SHRS in MP35N fine wires.

So, the current study aims to understand the influence of strain rate on the SRS and SHRS of MP35N wires subjected to different drawing practices namely Full Die Drawing (FDD) and Half Die Drawing (HDD). The wires were subjected to a deformation strains of 0.64 (50% CW), and the samples collected for the particular strain are subjected to uniaxial tensile tests at room temperature, by varying the strain rate from 10^{-6} to 10^{-2} . The mechanical results were correlated to microstructural data characterized by Scanning Electron Microscope (SEM), Electron Beam Scattered Diffraction (EBSD) and Transmission Electron Microscopy (TEM) to understand the deformation mechanisms in MP35N fine wires.

7.2 Materials and Methods

The materials and the experimental methods, for wire drawing, tensile testing, and TEM, EBSD and SEM characterization are discussed in Chapter 3. The wires had been drawn from an initial diameter of 0.62 mm to a final diameter of 0.45 mm with the FDD and HDD drawing technique, for a total cold work reduction of 50%. For the tensile tests, the gauge length was maintained at 254 mm, and the samples were subjected to varying strain rates of 8.3×10^{-6} , 8.3×10^{-5} , 8.3×10^{-4} , 1.6×10^{-3} , and $3.3 \times 10^{-2} \text{ s}^{-1}$. The reason behind the selection of the strain rates in the quasi-static range was to keep the deformation history and the temperature rise in the samples to be a minimum,

so as to reduce the complexity of multiple variables and deformation mechanics interactions which occur simultaneously in high strain rate tests [325], thus convoluting the strain effects on the deformation mechanisms. The testing was performed at room temperature, and the samples were tested until fracture. True stress-strain curves were calculated from the raw data of the tensile curve and, the strain hardening rate and the strain rate sensitivity values are determined.

7.3 Results

7.3.1 Mechanical Results

Engineering stress-strain data for the 50% CW wires drawn with different drawing practices and with varying strain rates were obtained from the stress-strain plots and converted to true stress-strain data by the standard calculations defined by Dieter G.E. et al. [260]. Figure 7.1 shows the true stress and true strain curves for the FDD and HDD drawn wire. The stress of the wire increased with the increase of strain rate from 10^{-6}s^{-1} to 10^{-2}s^{-1} in both the FDD and HDD drawn wires, but the relative increase in stress for a given strain is higher in the FDD wire when compared to the HDD wire. The FDD wire had a strength of 2015 MPa, when subjected to a strain rate of $8.3 \times 10^{-6}\text{s}^{-1}$ and increased to 2100 MPa when the strain rate was incremented to $3.3 \times 10^{-2}\text{s}^{-1}$, however in the HDD wire, the strength of the wire was 1870 MPa, at low strain rate and increased to 2020 MPa with the elevation in strain rate.

Figure 7.1: True Stress-True Strain curves for the wire subjected to different strain rates (a) FDD (b) HDD

(a)

(b)

Figure 7.2: Influence of strain rate on the strain hardening rate of (a) FDD wire (b) HDD wire.

Figure 7.2 shows the strain hardening rate curves for the FDD and HDD wires, inflicted to different strain rates. The strain hardening rates were normalized by the shear modulus and plotted against the strain. There was a significant difference observed in the hardening behavior of the FDD and HDD drawn wire, for the applied strain rate. The FDD drawn wire exhibited a single-stage hardening regime, at a lower strain rate of 8.3×10^{-6} and displayed a three-stage hardening

regime between the strain rates of $8.3 \times 10^{-5} \text{ s}^{-1}$ to $3.3 \times 10^{-2} \text{ s}^{-1}$, with the stage II peak increasing with the increase of strain rate. However, in the HDD drawn wire the material exhibited a single-stage hardening curve up to a strain rate of $1.6 \times 10^{-3} \text{ s}^{-1}$ and displayed an abnormal three-stage hardening curve at a strain rate of $3.3 \times 10^{-2} \text{ s}^{-1}$. It was also noticed that the normalized hardening values for the FDD drawn wire were comparatively higher than the HDD drawn wire for a similar strain rate applied.

Figure 7.3: SRS (m) comparison for the FDD and HDD drawn wires subjected to various strain rates.

Figure 7.3, shows the SRS (m) comparison of the FDD and HDD drawn wires subjected to different strain rates, the values of m were obtained from the slope of the double logarithmic plots of true stress and true strain rate data and are plotted against the strain rate. It can be seen that the value of m decreased linearly with the increase of the strain rate for the FDD wires, however for the HDD wire, the m value increased up to a strain rate of 10^{-5} s^{-1} and then decreased with the increase of strain rate as in FDD samples. It could be seen that the FDD drawn wire had a higher m value when compared to the HDD drawn wire.

7.3.2 Microstructure Characterization

EBSD analysis was performed on the annealed wire received at 0.62mm, with a step size of 0.1 μ m. Figure 7.4(a) shows the Inverse Pole Figure (IPF) map of the wire along the wire axis direction and Figure 7.4(b) shows the grain size distribution (GSD) of the wire. It can be observed that the wire had a mixture of coarse and fine grains and the grains were in the range of 4 to 11 μ m with the highest distribution of grains in the range of 4.5 to 6.5 μ m.

(a)

(b)

Figure 7.4: Microstructure of the annealed wire (a) IPF (Inverse Pole Figure) (b) Grain Size Distribution

(a)

(b)

Figure 7.5: TEM images of the annealed wire (a) Bright Field Image (b) SAD Pattern

Figure 7.5(a) shows the Bright Field (BF) TEM image of the annealed wire, the microstructure of the wire consisted of a mixture of coarse and fine grains with several twins embedded inside the grains. The Selective Area Diffraction (SAD) image shown in Figure 7.5(b), exhibits a strong texture with a misorientated profile, which confirms the presence of twins in the microstructure. The annealed wire was drawn to a 50% CW reduction, with the FDD and HDD drawing process and the microstructure of the samples before the tensile tests are shown in Figure 7.6 and Figure 7.7. The FDD drawn wires were unable to be indexed by the EBSD, and TEM analysis was performed, however, the microstructure of the HDD drawn wire was characterized by both EBSD and TEM.

Figure 7.6: Microstructure of the as drawn FDD wire (a) STEM image showing the grains (b) Grain size distribution (c) BF TEM image showing the deformed microstructure (d) BF TEM image with the SAD inset showing the twins between the grains.

(a)

(b)

(c)

(d)

Figure 7.7: Microstructure of the as drawn HDD wire (a) EBSD IPF image (b) Grain Size Distribution (c) BF TEM Image (d) DF TEM Image.

The FDD microstructure displayed in Figure 7.6(c) looks highly deformed when compared to the HDD microstructure in Figure 7.7(c), this explains why the FDD sample was could not be indexed by the EBSD, because of its drawbacks in characterization of the highly strained samples for the limitations in its spatial and angular resolutions of the equipment [159, 160]. The FDD wire exhibited higher volume fraction of grains in the size of 60-100nm as seen in Figure 7.6(b),

whereas the HDD drawn wire had a higher volume of grains in the range of 200-350nm as in Figure 7.7(b). There were also few twins seen in the microstructure of the HDD drawn wire as shown in Figure 7.7(a) and Figure 7.7(d), while the FDD drawn wire had several twins rooted between the grain boundaries as shown in Figure 7.6(d), the Fourier Fast Transformation (FFT) inset obtained from the highlighted rectangular area confirms the existence of twins in the microstructure. For the FDD and HDD wires imposed to different strain rates, the fracture samples were collected after the tensile test, and the samples were cut near the fracture point as shown in Figure 7.8(a) using Focused Ion Beam (FIB) and separated from the cut surface for TEM characterization.

Figure 7.8: Fracture Sample after a tensile test (a) Cutting position for the FIB (b) Magnified Image of the FIB cut sample.

7.4 Discussions

7.4.1 Strain rate effects on the strength, ductility, and strain hardening rate of MP35NLT wire.

The MP35N wires drawn with the FDD and HDD drawing techniques, for a CW reduction of 50% are subjected to room temperature (RT) monotonic uniaxial tensile tests with varying strain rates from 10^{-6}s^{-1} to 10^{-2}s^{-1} . Figure 7.1(a) and (b) shows the stress-strain curves for the FDD and HDD drawn wire. It can be seen that the strength of the wire increased with the increase of SR for both the conditions, with the FDD drawn wire enduring a higher amount of strength when compared to the HDD drawn wire for a similar amount of strain. It is also noticed that the difference of strength between the low SR and high SR wire was only 80MPa in the FDD drawn wire, whereas, for the HDD drawn wire, the difference was around 153MPa. Figure 7.9(a) and (b) shows the ductility and true fracture strain (ϵ_f) comparison between the FDD and HDD wires tested to different strain rates. The true fracture strain was computed from the area reduction (RA%) of the samples, using the relation $\epsilon_f = \ln(100/(100-\text{RA}\%))$. It can be perceived that the elongation of the FDD wire reduced with the increase of strain rate. However, with the HDD drawn wire, the ductility in the wire increased between SR of 10^{-6} to 10^{-5}s^{-1} , then decreased with the further increase of SR to 10^{-4}s^{-1} and stabilized between SR of 10^{-4} to 10^{-2}s^{-1} and a similar trend was noticed with the ϵ_f .

Figure 7.9: Influence of strain rate on the (a) ductility (elongation%) (b) True fracture strain of the FDD and HDD drawn wires.

As shown in Figure 7.2(a) and Figure 7.2(b), the strain hardening curves observed were remarkably different, between the FDD and HDD wires, with the hardening rate increasing with strain rate. The stage II hardening peak of the FDD wire increased, as the strain rate increased from

10^{-5}s^{-1} to 10^{-3}s^{-1} and displayed a three-stage hardening regime, but the hardening curve at a strain rate of 10^{-6}s^{-1} exhibited a single-stage hardening curve. A similar single stage hardening response was observed in the HDD drawn wire inflicted up to a strain rate of 10^{-3}s^{-1} , but an abnormal hardening three-stage hardening curve was found in the sample exposed to a strain rate of 10^{-2}s^{-1} . TEM investigations were performed on the deformed samples to understand the variation in the mechanical response observed between the two drawn wires. Figure 7.10(a) and Figure 7.11(a) shows the comparison of the microstructure of the FDD and HDD wires; it can be noticed that the microstructure of the FDD drawn wire looks highly deformed than the HDD wire, with a higher dislocation density noticed in the microstructure of the FDD wire. It was also observed from Figure 7.10(b) and (c) that there was a severe pile-up of dislocations at the GB for the FDD wire.

However, with the HDD drawn wire, the absence of prior dislocations in the microstructure of the as drawn wire, as shown in Figure 7.7(c), the dislocation conciliated plastic deformation activities were absent in the material, which made the deformation intergranular [326, 327] because of the enhanced GB activities such as GB sliding. This limited the ability of the material to resist the large strains promoting an extremely localized deformation [132, 133], thus resulting in the formation of shear bands in the microstructure as in Figure 7.11(b) and (c), which made the deformation inhomogeneous and localized, thus contributing to severe plastic instability in the material. This resulted in reduced strength and a diminished work hardening capacity in the material, followed by low ductility as seen in Figure 7.2(b) and Figure 7.9. Diffraction patterns observed in Figure 7.11(d) show strong texturing which implies geometric softening [328, 329] as the dominant mechanism happening in the HDD wires, to accommodate the increased resolved shear stress for the slip activities. This led to an increased stress localization in the microstructure, driving to the formation of shear bands.

Figure 7.10: TEM images of the FDD drawn wire inflicted to an SR of 10^{-6}s^{-1} (a) BF TEM Image of the deformed microstructure (b) BF TEM showing the dislocation pile up at the GB (c) TEM BF image showing the dislocation annihilation at the GB (d) BF TEM image showing the severely annihilated dislocation microstructure.

The reason for two different deformation mechanisms and microstructures observed for a wire subjected to the same strain rate of 10^{-6}s^{-1} can be explained by the Orowan equation [330], which is defined as $\dot{\epsilon} = \rho b v$, where $\dot{\epsilon}$ is the strain rate, ρ is the density of the mobile dislocations, and v is the dislocation velocity. It can be inferred from the above equation that with the increase of the strain rate, the mobile dislocation density and the velocity increases and vice versa. In the current scenario, with a lower strain rate, the velocity of the mobile dislocation density is

decreased, which increases the interaction time for GB diffusion and dislocation based activities such as grain boundary shearing and sliding, thus accounting for an increased pile-up of the dislocations at the GB, as shown in Figure 7.10(b) for the FDD wire. The increased GB mechanisms at a lower SR reduces the hardening capacity of the material due to the incidence of dynamic recovery in the material as detected by a single-stage hardening curve in Figure 7.2(a) for an SR of 10^{-6}s^{-1} in the FDD wire.

Figure 7.11: TEM images of the HDD drawn wire inflicted to an SR of 10^{-6}s^{-1} (a) BF TEM image of the microstructure (b) BF TEM image of the parallel deformation bands (c) High magnification image of the deformation hands (d) SAD pattern of the deformation band showing the presence of strong texture.

On the further increase of the SR, it can be observed that the number of dislocations pile-ups at the GB and the dislocation cell size for the FDD drawn wire decreased with the increase of SR from 10^{-5} to 10^{-2}s^{-1} as shown in Figure 7.12. The wires drawn with low strain rate had a lower

dislocation density in the cell interiors, and it increased with the increase of SR, with the formation of many loose dislocation tangles in the cell structure.

Figure 7.12: TEM images of the FDD drawn wire drawn to different strain rates (a) 10^{-5} (b) 10^{-4} (c) 10^{-3} (d) 10^{-2} s^{-1}

Figure 7.13: TEM images of the HDD drawn wire drawn to different strain rates (a) 10^{-5} (b) 10^{-4} (c) 10^{-3} (d) 10^{-2}s^{-1}

The cell size of the samples also decreased with the increase of SR for the FDD wire, with the lower SR deformed samples having a clear and precise cell structure as shown in Figure 7.12(a) and the 10^{-2}s^{-1} distorted sample having no cell structure as seen in Figure 7.12(d). It was also observed that the dislocation density increased with the increase of SR for the FDD drawn wires. The observed microstructural variation can be explained as follows, with the rise in SR, the velocity of the mobile dislocations increased, and the activation energy and the driving force between the mobile dislocations decreased, which reduced the interaction time between the dislocations. This led to high misorientation between the grains, which created a barrier to the

dislocation movement and increasing the overall dislocation density. As explained by Holt, D.L, et al. and Huang, F, et al. [331, 332], the relation between the dislocation cell size and the total dislocation density can be defined as $d_{cs} = K\rho^{-1/2}$, where d_{cs} is the dislocation cell size, K is a constant, and ρ is the overall dislocation density. From the equation, it can be inferred that with the increase of the dislocation density, the dislocation cell formation reduces as perceived in the current scenario for the FDD drawn wire, with the rise in SR.

The influence of the SR on the deformation microstructures can also be explained by [333], the Zener-Hollomon parameter (Z)= $\dot{\epsilon} \exp(Q/RT)$, where $\dot{\epsilon}$ is the SR, Q is the activation energy, and T is the temperature. Based on the above equation, it can be inferred that with the increase of the SR, the activation enthalpy decreased in the metal, due to the reduction of the free path of the mobile dislocations. Hence dislocation annihilation was reduced, thereby, lowering the dynamic recovery and contributing to increased stress in the material, which is beneficial for the generation of increased storage of new dislocations thus enhancing the overall dislocation density. This increased the work hardening and strength of the material with the increased strain rate as observed in the FDD drawn wire.

However, with the HDD drawn wires, the microstructure of the wires, looked different from the FDD drawn wires, until an SR of 10^{-4} s^{-1} , there were no or very few dislocations observed in the microstructure as shown in Figure 7.13(a) Neither dislocation cells nor dislocation pile-ups are found at the GB of the HDD drawn wire, and the grains looked columnar. As explained by Lu, L et al. and Chen, X et al. [334, 335], the work hardening rate for the Cu with columnar grains was found to be lower than with equiaxed grains; this phenomenon was attributed to the different dislocation types and behaviors developed in the material during its deformation thus contributing to the different dislocation-slip mechanisms. So, in the current situation, for the HDD drawn wire, with the absence of any visible dislocation cells, and the grains observed to have a columnar structure, explains as for why such a low hardening rate was observed in the HDD wire when compared to the FDD drawn wire at a strain rate of 10^{-5} s^{-1} . The number of shear bands in the microstructure also reduced when compared to the wire at an SR of 10^{-6} s^{-1} . This might be linked to the increased strain hardening rate of the material as shown in Figure 7.2(b), which redistributed the stresses in the material, and reducing the stress localization. The increased SR also changes the misorientation between the grains, which reduces the heterogeneity in the material and contributing to a higher strength and strain hardening.

With the increase of strain rate from 10^{-5}s^{-1} to 10^{-2}s^{-1} , it can be noticed that the work hardening and strength of the material increased for the HDD wire. Until an SR of 10^{-3}s^{-1} , there was a stage I hardening curve observed, and the strain hardening rate for the 10^{-3}s^{-1} SR was comparatively higher than the other two strain rates. The higher hardening rate observed for the 10^{-3}s^{-1} is because of the increased dislocation density, as seen in Figure 7.13(d), where the dislocations seem to pile up along the slip bands. With the increase of SR to 10^{-2}s^{-1} , there was a two-stage hardening curve regime observed as shown in Figure 7.2(b). The hardening rate decreased from 0.0322 to 0.012, with the increase of strain to 0.0136, and then increased suddenly, to 0.0204 for a strain of 0.019. The appearance of the Stage II hardening curve at such a high strain was abnormal as most of the Stage II hardening peaks at a lower strain as seen in Figure 7.2(a). There was an increased dislocation density along the slip bands with an increase of SR, as seen in Figure 7.13(d), but this does not explain and justifies for the abnormal effect observed.

High Angle Annular Dark Field (HAADF), STEM images were captured in the area near the fracture point. As seen in Figure 7.14(a), they were thin dark bands observed in the HDD microstructure of the deformed sample and Figure 7.14(b) indicates the dark circular contrast embedded in the slip bands. Energy Dispersive X-ray Spectroscopy (EDX/EDS), measurements were performed on the dark lines and the surrounding area near the dark band and the circular portion indicated by P1-P6, with P1 being the point on the dark band, P3 is the point adjacent to the band, P4 is the point farthest from the band, and P2 is on the region away from the fracture area. P5 and P6 indicate the EDX points of the area highlighted in Figure 7.14(b). Figure 7.14(d) shows the representative image of the EDX spectra obtained from the machine on the dark band at point P1. Table 7-1 shows the quantitative elemental composition data of the points P1-P6.

From the compositional data in the Table 7-1 points P1, P3 and P5, had a high Mo content of 21.68%, 19.75% and 20.66% by weight, whereas the points P2 and P6 had a Mo content of 10.37% and 9.39% respectively and the point P4 exhibited a lower Mo content of 5.29%. As per ASTM-F562-13 standard for MP35N LT material [57], the standard composition for the Mo element in the alloy is around 9.0-10.5% by weight. It is well-known that HAADF-STEM technique [336], reproduces the images based on the intensity of the contrast, which is dependent on the atomic number (Z) of the material, which implies that darker the contrast, higher is the Z. In the current scenario, for the MP35N alloy, the element with the highest atomic number is Mo (Z=42), followed by Ni (Z=28), Co (Z=27) and Cr (Z=24). This confirms that the dark bands and

the circular areas observed in the deformed microstructure to be Mo as ascertained by the EDX spectrum.

(a)

(b)

(c)

(d)

Figure 7.14: STEM image of the 10^{-2}s^{-1} of the HDD drawn wire (a) HADDF STEM image indicating the dark bands in the deformed microstructure along with the EDX points (b) HADDF STEM image of the dark area inside the slip band (c) High magnification image of the area indicated in red (d) EDX spectrum for the point 1.

Table 7-1: EDX quantification analysis of the points (P1-P6) indicated in Figure 7.14

Element	Weight%	Atomic%	Weight%	Atomic%	Weight%	Atomic%	Weight%	Atomic%	Weight%	Atomic%	Weight%	Atomic%
	P1		P2		P3		P4		P5		P6	
Cr(K)	19.77	24.36	19.82	22.75	40.21	46.57	19.67	21.96	23.79	27.33	17.73	20.32
Co(K)	36.17	38.76	35.18	35.62	16.58	16.95	36.14	36.61	29.35	31.55	35.97	36.38
Ni(K)	22.36	23.14	34.6	35.17	23.45	24.07	38.8	38.23	26.18	29.02	36.89	37.45
Mo(K)	21.68	13.72	10.37	6.45	19.75	12.40	5.29	3.19	20.66	12.08	9.39	5.83

The observed phenomenon of increased Mo content, with the increase of SR, can be attributed to the different deformation and dislocation mechanisms noticed in the HDD wire when

compared to the FDD drawn wire. It can be seen from Figure 7.13(d), that the orientation and the movement of the dislocations were different in the HDD wire, with the dislocations favoring to move on parallel planes, lying next to each other and having a similar orientation. It has been stated by Messerschmidt, U [337], that dislocations moving on parallel slip planes have a higher propensity to annihilate with each other when compared to the dislocations moving on non-parallel slip planes, which obstruct the dislocation motion. Due to the increased interaction of the dislocation activities, and a reduced dislocation density it leads to the creation of the vacancies in the atomic structure, thus contributing to different energy states in the structure. This reduces the activation energy of the system and as defined by Fick's first law [338], the diffusivity in the material increases, leading to increased solute segregation to the interfaces such as GB during the deformation process. This leads to a long-range order interaction between the dislocations and the solute atoms, leading to non-equilibrium segregation [339], which results in the solute atoms pairing with the created vacancies and moving to the GB upon application of the stress. The segregation of solute atoms to the GB hinders the movement of the dislocations at the GB thus contributing to increased strength and hardening in the material as observed in the HDD drawn wire. This phenomenon has been observed in Mg-Gd alloys [340, 341] and Mg-Zn-Y alloys [342] where the Gd atoms segregate to the GB after cold rolling and extrusion, and also in FCC metals such as Al alloys [105, 343, 344], steels [345, 346] and some Co-Ni alloys [82]. So, the abnormal hardening and the higher strength observed in the HDD deformed to an $SR 10^{-2} s^{-1}$ can be associated with GB solute segregation which impelled due to the minimization of its free energy.

7.4.2 Strain rate effects on the SRS of MP35NLT wire.

Figure 7.3, shows the comparison of the SRS (m) between the FDD and HDD wires; it can be noticed that the SRS linearly decreased with the increase of SR for the FDD drawn wire. At an SR of $10^{-6} s^{-1}$, the value of m obtained was 0.0415 and with the increase of SR to $10^{-2} s^{-1}$, the value of m reduced to 0.0085. The high m at a lower SR can be explained by the increased GB mechanisms as shown in Figure 7.10 and its associated activities such as Grain Boundary Diffusion (GBD) and Grain Boundary Shearing (GBS) which are known to increase m [347-349]. The increased m noticed at low SR can be attributed to the different deformation mechanisms, where the increased time for the GB activities and a reduced movement of the mobile dislocations, would make the dislocation-based mechanisms unfavorable and thus encouraging more GB activities. The results obtained in the present study correlated well with the results of prior studies [107, 350, 351] where a similar effect has been observed.

Another dominant feature for the increase of m with the lower SR can be explained based on the twin spacing (λ) and twin thickness (δ). It is well known [110, 352] that for materials that undergo deformation twinning, decreasing the twin spacing, and increasing the twin thickness contributes to a higher m and vice versa. The decreased twin thickness with the increase of strain contributed to an improved strain hardening, and increased strength in the material [334], in the FDD drawn wire as shown in Figure 7.15.

Figure 7.15: Effect of twin thickness and twin spacing of the FDD draw wire with differing strain rate (a) 10^{-6} (b) 10^{-4} (c) 10^{-3} (d) 10^{-2} s^{-1}

This justifies the reason behind the decreased SRS with the increased SR for the wire drawn with the FDD drawing practice; however, for the HDD wire due to the involvement of contrasting deformation mechanisms, the SRS behavior was found to be different. The SRS of the HDD drawn wire at an SR of 10^{-6}s^{-1} had an m value of 0.0142, this was 65% lower than FDD drawn wire, the reason for such a low SRS value can be attributed to the increased localized strain concentration in the material, leading to the formation of shear bands in the material as shown in Figure 7.11(c). The formation of shear bands in the material reduces the capability of the material to resist necking [100, 131, 297, 302, 353] during the tensile tests, therefore, contributing to lower ductility and decreased m . The correlation of ductility with m between the FDD and HDD drawn wires can be noticed by comparing Figure 7.9 and Figure 7.3, where a higher ductility and higher m was noticed in the FDD drawn wire at a lower SR, whereas a lower m and lower ductility is observed in the HDD drawn wire. With the increase of SR to 10^{-5}s^{-1} , the m value in the HDD drawn wire increased to 0.0022 and then linearly decreased as in the FDD wire. So, the increased m with the increase of SR to 10^{-5}s^{-1} can be associated with the reduced shear bands in the structure and an increased hardening ability in the material. With no twins observed in the microstructure of the HDD wire as seen in Figure 7.13, the reduced m with the increased SR can be ascribed to the reduced GB activities with the increasing SR. The overall lower m for the HDD drawn wire can also be associated with its coarser grain size when compared to the finer grain size of the FDD drawn wire as seen in Figure 7.6 and Figure 7.7, and the microstructure results corresponded well with the observations from prior studies [100, 101, 126, 141, 145] that larger grain size in the material contributes to lower SRS in the material.

Figure 7.16 and Figure 7.17 shows the SEM morphological images of the FDD and HDD drawn wires subjected to different SR after the tensile test. All the samples exhibited a ductile mode of fracture, but the size of the necking zone and area fraction of the dimples varied with different strain rates. There was a noticeable difference in the distribution of the dimpled region between the FDD and HDD wires. It can be perceived from Figure 7.16, that the size of the necking zone decreased with the increase of SR, and the samples tested at a lower SR had a larger dimple size, and the dimples looked equiaxial, when compared to the dimple region at higher SR, where the dimple size and the area fraction of the dimpled region reduced. Also, the distribution of the dimples between the center and the surface of the wire was homogenous in the FDD wire. The prodigious dimple size observed at a low SR of 10^{-6}s^{-1} in the FDD drawn wire correlates well with the results of higher m and higher ductility observed in the FDD drawn wire.

Figure 7.16: Fracture samples of the FDD drawn wires deformed to various SR (a) 10^{-6}s^{-1} (b) 10^{-5}s^{-1} (c) 10^{-4}s^{-1} (d) 10^{-3}s^{-1} (e) 10^{-2}s^{-1}

Figure 7.17: Fracture samples of the HDD drawn wires deformed to various SR (a) 10^{-6}s^{-1} (b) 10^{-5}s^{-1} (c) 10^{-4}s^{-1} (d) 10^{-3}s^{-1} (e) 10^{-2}s^{-1}

As the SR increased to 10^{-2}s^{-1} , the dimpled area and size progressively decreased as shown by the high magnification insets. However, with the HDD drawn wire, the fracture for the sample deformed at a low SR, exhibited a mixed-mode of fracture displaying both shear and ductile regions. The dimple size and fracture area of the samples were comparatively smaller than the sample deformed at an SR of 10^{-5}s^{-1} . This confirms that the fracture-resistant necking area was lower and the sample fractured early due to early onset of necking. The dimpled region and size increased at an SR of 10^{-5}s^{-1} and then lowered with the decrease of SR to 10^{-2}s^{-1} . It can also be

observed in the HDD samples that the center region exhibited larger dimple size when compared to the dimpled size near the surface of the sample, which confirms that the deformation is rather inhomogeneous [354] in the HDD drawn wire when compared to the FDD drawn wire as observed from the true fracture strain plot in Figure 7.9(b). From the SEM images shown in Figure 7.16 and Figure 7.17, it can be observed that the central dimpled region essentially shows the final region before catastrophe in a cup-cone type failure and the outer regions are shear fractures.

7.5 Summary

Based on the above results and discussions, the following conclusions can be outlined:

- There is a prominent influence of the Strain Rate (SR) on the Strain Rate Work Hardening (SRWH) and Strain Rate Sensitivity (SRS) on the wires drawn with different drawing practices. The Full Die Drawing (FDD) wires had higher strength, ductility, higher SRWH, and SRS when compared to the HDD drawn wire subjected to a similar strain rate deformation.
- The strength and the SRWH of the FDD drawn wires increased with the increase of SR. A single-stage I hardening curve was observed for the wire deformed at an SR of 10^{-6}s^{-1} . The wires deformed to other SR, exhibited a stage II hardening curve with the stage II peak, broadening with the increase of the SR. This was attributed to the increased dislocation density, reduced twin thickness, and a reduction in the Grain Boundary (GB) mechanisms such as GB sliding and shearing with the increased SR, due to decreased dislocation cell sizes and reduced pile up the GB. The Strain Rate Sensitivity (SRS), or m value of the FDD drawn wire decreased with the increase of SR, due to the reduction in the dislocation-based activities and increased GB shearing and diffusion in the deformed wire. The twin spacing increased with the increase of SR for the FDD drawn wire, thus contributing to a lower m .
- The HDD drawn wire exhibited a different deformation mechanism with varying SR. At an SR of 10^{-6}s^{-1} , the strength and hardening of the material was significantly lower than the samples tested at other SR. TEM investigations, confirmed that in the low SR deformed sample, due to the absence of prior dislocations in the microstructure, the dislocation conciliated plastic deformation activities were absent in the material, which made the deformation intergranular due to the enhanced GB activities such as GB sliding. This limited the ability of the material to resist the large strains and thus promoting an extremely

localized deformation, resulting in the formation of shear bands in the microstructure which made the deformation highly inhomogeneous and contributing to severe plastic instability in the material. This reduced the work hardening and load-bearing capacity of the material, thus contributing to lower strength, ductility and a reduced SRS.

- An abnormal hardening effect was observed in the HDD drawn subjected to an SR of 10^{-2}s^{-1} , the stage II hardening peak originated at a very high strain, instead of its usual commencement at low SR, after the dynamic recovery. This effect was accredited to the solute segregation of the Mo atoms to the GB, as observed by the increased concentration of the Mo concentration in the deformed structure, which increased the strength and the hardening capacity of the wire.
- The fracture morphology of the FDD samples looked different when compared to the HDD samples, with the low SR deformed FDD drawn wires exhibiting a higher fraction of the dimpled area and bigger dimple size and vice-versa. The dimpled region between the surface and the center of the wire looked homogeneous in size and shape. The fractography observations correlated well with the mechanical results of higher m and increased ductility seen in the FDD wire at a lower SR, which confirms the FDD drawn material to have higher resistant to necking, because of its higher SRS and higher hardening rate than the HDD wire.
- However, with the HDD drawn wire, the fractography observation of the low SR samples exhibited smaller dimple size and a reduced fractured area, the fractured area also displayed a combination of shear and ductile mode fracture. The dimple size at the surface of the wire was smaller when compared to the center, and the deformation looked inhomogeneous.

Chapter 8 Fatigue behavior in Co-Cr-Ni-Mo alloy wires drawn with different drawing practices

8.1 Introduction

Accessibility to the difficult to critical sites during the invasive surgeries such as Cardiac Rhythmic Management (CRM), neurostimulation or Deep Brain Stimulation (DBS), have led to the miniaturization of devices in the medical industry [355, 356], such as pacemakers and neurostimulator leads. This also impacted the geometrical attributes of the wires and cables used to manufacture the leads, with the wire dimensions [357, 358] been reduced from fine to ultra-fine, in the range of tenths of a micron. The leads [3] form the critical part of the pacing system, and they ensure that the rhythmic management of the heart activities or transfer of the pacing pulses and shocks to the implanted site are done with utmost efficiency. These challenges compound the assessment of the mechanical reliability [359] of the device before implanting into patients. A lead failure [360] can be very catastrophic, may result in sudden death, and also the extraction procedures are considered to be of high risk.

The implantable leads should not only sustain the stress and strain foisted by the surrounding tissues but should also possess sufficient fatigue strength and durability during its life period [361]. On top of the strength requirements, the material should be biocompatible and not susceptible to any corrosion. Many biocompatible metals such as stainless steel, platinum-based alloys, tantalum are used in the design of pacemakers. However, cobalt-based alloys [362] have gained significant importance in the medical industry for the last few decades due to its high strength and excellent corrosion resistance. The cobalt-based alloys are alloyed to different elements such as Cr, Ni, Mo, Pt, Ag, Ta, and with different compositions to obtain the desired properties in the material. One such high-performance cobalt alloy is MP35N with a chemical composition of 35Ni-35Co-20Cr-10Mo; has been used in the medical industry for the past 30 years, in the manufacture of pacemakers and neurostimulation leads. This material has been the best choice for all the medical designers because of its high fatigue strength [54, 363], excellent corrosion resistance and good chemical passivation. [364] Due to the increased criterion for enhancing the endurance limit of the product, there has been a significant improvement in the melt refining and processing techniques of MP35N alloy [213], to reduce the Ti content in the material, which lowered the TiN inclusion formation and thus improving the fatigue life. The new alloy was designated as 35NLT® (Low Titanium-Trademark of FWM metals) or otherwise known as

MP35NLT; this surged the demand to use MP35NLT material and its alloys for the manufacture of leads replacing the existing MP35N.

Recent years have witnessed a surge in the usage of the alloy for medical applications. As a result, there were many fatigue studies [212, 365, 366] conducted on MP35NLT and its alloys in the wire [367, 368] and cable forms [233, 369, 370]. Prior studies performed on MP35NLT reported the cyclic behavior of the material tested under different applied stress and strain amplitudes [371], or with distinct test methods (tension-compression vs. tension-tension) [164], contributed to a diverse fatigue response in the material. SEM analysis was done on the fatigue specimens, to visualize and determine the mode of fracture [165]. However, there were no studies done on MP35NLT fine wires, investigating the influence of the manufacturing process and its relevant microstructure on the fatigue performance of the material. It is well known that the fatigue is a microstructure phenomenon [174, 179] and a robust understanding of the processing methods and its interrelationship to the intrinsic and extrinsic microstructural parameters and their contribution to the fatigue mechanisms and variability need to be well defined.

In the previous chapters, the influence of drawing practices on the mechanical, work hardening, SRS, microstructural, properties of the MP35NLT wires were studied, and it was concluded that there was a significant difference in the material's behavior processed with different drawing methods. The deformation homogeneity and plastic instability of the material with their relationship to the intrinsic microstructural parameters were characterized, and it was observed that the processing method had a compelling effect on its hardening behavior and their deformation mechanisms. So, the purpose of the current study is to characterize the influence of the drawing practices on the cyclic response or fatigue behavior of the MP35NLT wires, drawn to varying cold work (CW) reductions and correlate a link between the process, microstructure, and fatigue.

8.2 Materials and methods

The materials and the experimental methods, for wire drawing, tensile and fatigue tests, TEM, EBSD, and SEM characterization are discussed in Chapter 3. The starting diameters of the material used for the fatigue characterization was 0.62mm and 0.141mm respectively, and the material was prepared in a fully annealed condition. Three different samples namely 50%, 75% and 95% CW are manufactured by varying the total area reduction. The 0.141mm annealed wire was drawn to a diameter of 0.101mm to obtain the 50% CW samples; however, for the 75% and 95% CW samples, the 0.62mm wire was drawn to a diameter of 0.318 and 0.141mm respectively. Table 8-1 shows

the mechanical properties of the as-received annealed wire at 0.141mm and its comparison to the 0.62mm. The disparity in the strength of the material perceived between the two wires is because of the size effect, which led to a difference in the load-bearing capacity of the tested sample. The EL% of both the wires are noticed to be close to each other as the material has been annealed to a similar temperature between 1025 to 1050°C.

Table 8-1: Mechanical properties of the annealed wire at different diameters.

Wire Diameter	Yield stress	UTS	EL	RA	ϵ_f
(mm)	(MPa)	(MPa)	(%)	(%)	mm ²
0.62	872.86	1,427.07	42.69	66.74	1.10
0.141	723.13	1,198.04	40.55	66.98	1.10

The reason for the selection of the 0.101mm wire as the 50% CW sample for the fatigue characterization instead of the 0.448mm diameter wire, is due to the inadequacy of the fatigue machine to test in the lower stress amplitudes, for its limitations in the bush to chuck distance of the equipment, which exceeds the current test condition. The Rotary Beam Fatigue Tester (RBFT) used for the current experiments is shown in Figure 8.1, it can be seen the wire is bent to the desired radius based on the applied stress amplitude, and the wire is fixed in between the rotating chuck and the bush. With the decrease of the stress amplitude, the chuck to bush distance increases, and with 0.448mm wire, it exceeded the maximum distance of the equipment.

Figure 8.1: RBFT fatigue test setup used for the current experiments.

Five specimens for each stress amplitude are tested between 500-1500MPa with a stress ratio of $R=-1$. Six lots (three from FDD and three from HDD) are tested for the 50% CW condition, and four lots (two from FDD and two from FDD) are tested for the 75 and 95% CW conditions. The runout or the endurance limit for the particular condition is determined only if all the five samples reach the 30 million (30M) cycles. The reason behind the selection of 30M cycles as the endurance limit is based on the accelerated testing on the reliability of the wires which are manufactured to leads and their survivability without fracture after implantation for one year. The fractured specimens are collected and appropriately identified for further fractography analysis.

8.3 Results

8.3.1 Microstructure and tensile results

The microstructure, grain size, and texture distributions of the 0.62mm annealed wire were discussed in chapter 4, and the TEM images of the as drawn 75% and 95% CW FDD and HDD wires are shown in section 7.3.2. The EBSD-IPF map for the 0.141mm annealed wire and the 0.101 HDD and FDD as-drawn wires are shown in Figure 8.2.

Figure 8.2: Inverse pole figure maps (a) 0.141 annealed wire (b) 0.101 as-drawn HDD wire (c) 0.101 as-drawn FDD wire.

It can be observed from Figure 8.2(a), that the microstructure of the 0.141mm annealed wire is, recrystallized with a mixture of coarse and fine grains and the average grain size of the wire was around 4.5 μ m. There were several twins embedded inside the grains, and the wire had 54% volume fraction of <111>, 28% of <100> and 18% of <113>. The annealed wire was drawn to a diameter of 0.101mm with the HDD and FDD drawing practice, and Figure 8.2(b) and Figure 8.2(c) shows the IPF comparison between the wires. The HDD drawn wire displays a less distorted structure and the grains can be visible in the microstructure even though not to the best resolution. However, with the FDD drawn wire the microstructure is heavily distorted. The microstructure results correlate well with the 50% CW wire discussed in chapter 4. From the observed results, it can be confirmed that the FDD drawing practice induced high plastic strain to the material when compared to the HDD drawn wire, which increased the number of lattice defects in the material, thus contributing to extremely disoriented grain structure. The average grain size of the HDD drawn wire was around 1.2 μ m, whereas the average grain size of the FDD drawn wire was around 0.3 μ m.

Figure 8.3: Tensile properties comparison between the FDD and HDD drawn wires drawn to different CW% (a) UTS (b) RA and EL%.

Figure 8.3 shows the tensile properties comparison between the HDD and FDD drawn wires used in the current study. For the FDD drawn wire, as seen from Figure 8.3(a), the tensile strength of the material increased with the increase of CW% until 75% CW and then decreased when deformed to a CW of 95%. However, for the HDD drawn wire, the UTS was initially lower than the FDD drawn wire up to 75% CW, and at 95% CW, the HDD drawn wire had a higher UTS when compared to the FDD drawn wire. For the HDD drawn wire, the increase of strength with the increase of deformation was linear, unlike the FDD wire. The ductility or EL% follows a

similar trend as UTS for the FDD drawn wire as shown in Figure 8.3(b), with the FDD wire having a higher ductility than the HDD wire until 75% CW and then decreasing with the increased strain. The reason for the abnormal strength variation has been interpreted in chapter 4, to the grain softening or the “Inverse Hall Petch effect” in the material. Moreover, the rationale for the decreased ductility in the FDD wire has been described in chapter 6, to the formation of shear bands in the material which contributed to strain localization and a reduced ductility in the material.

8.3.2 Cyclic Performance (S-N curve) of MP35N wires

The cyclic data collected from the RBFT tests are plotted in an S-N (Wohler) curve as shown in Figure 8.4, where the S represents the stress amplitude, and N represents the no of cycles to failure. It can be observed from Figure 8.4(a), that the fatigue performance of the 50% CW FDD drawn wires was significantly higher than the HDD wires in both the low cycle fatigue (LCF) and high cycle fatigue (HCF) regimes. The runout (30 million cycles) achieved for a particular stress amplitude was indicated for the tested batch of wire, with the respective color-coded arrow. It can be noticed that in FDD drawn wires, the runout stress amplitude in the HCF region was around 670-780MPa, whereas the runout stress level for the HDD drawn wire was around 525-585MPa. The LCF fatigue performance of the FDD drawn wires was exceptionally higher than the HDD drawn wires with many specimens able to withstand over 1 million (1M) cycles, when tested between 1170-850MPa stress amplitudes, however for the HDD drawn wires, the wire specimens could reach a fatigue endurance of 1M cycles only at a stress amplitude of 670MPa.

(a)

(b)

(c)

Figure 8.4: S-N curve comparison between FDD and HDD wires drawn to (a) 50% CW (b) 75%CW (c) 95%CW.

With the increase of CW to 75% as shown in Figure 8.4(b), the performance gap between the two drawn wires reduced, the FDD drawn wires could reach the runout at a stress amplitude

530-760MPa, and the HDD drawn wires reached runout between 460 to 685MPa. The LCF performance of the HDD drawn wires also improved remarkably when compared to the 50% CW conditions, there were multiple specimens which were capable of withstanding 1M cycles, in the stress range of 850 to 970MPa. However, with the FDD drawn wires; nearly all the samples could reach 1M cycles, and a few of them could even reach the runout of 30M cycles at such high stress. Further increase of CW to 95%, improved the fatigue behavior of the HDD drawn wires both in the HCF and LCF regions when compared to the FDD drawn wires. The FDD wires reached the runout at a stress level of 1036MPa whereas the HDD drawn wire could withstand the fatigue life of 30M cycles at a stress amplitude of 1110MPa. The LCF performance of the HDD drawn wire was also observed to be significantly higher than the FDD drawn wire, with the HDD wires capable of withstanding the 1M cycles at a stress amplitude of 1200MPa, with an average fatigue life of 16-23M. However, the FDD drawn wire had an average fatigue life of 2.7-9M only. The LCF performance of the HDD wire between the stress amplitude of 1400-1550MPa was also on an identical note, with the HDD drawn wire outperforming the FDD drawn wire as shown in Figure 8.4(c).

8.4 Discussions

8.4.1 Influence of drawing practice on the cyclic behavior of 50%CW wires.

It is acknowledged that the prior deformation history or the method used to process the material to its finished size, influence the fatigue properties of the material. In the current study, by keeping the total area reduction or the total plastic strain constant, the amount of deformation reduction per pass has been differed to study the influence of the drawing method on the cyclic response of the MP35NLT wires. Figure 8.4(a), shows the comparison of the cyclic behavior of the FDD and HDD drawn wires, subjected to a plastic strain of 0.67 or 50% CW. It can be seen that the fatigue performance of the FDD drawn wires were comparatively higher than the HDD drawn wires, in both the LCF and HCF regimes. The LCF and HCF behavior of the materials subjected to cyclic loading is dependent on the properties of the wire such as tensile strength and ductility, which are interrelated to the intrinsic microstructural parameters such as grain size, dislocation density, twin density, and the slip behavior [372]. As seen in Figure 8.2(c), the grain size (GS) of the FDD drawn wire was in the range of 100-500nm, whereas the HDD wire GS was in the range of 700-1500nm as in Figure 8.2(b).

Table 8-2: Comparison of the grain boundary characteristics between FDD and HDD 50%CW wire

Sample	HAGB (%)	LAGB (%)	$\Sigma 3$ Twins (%)	Average GS (nm)
FDD	56.4	26.2	17.4	300
HDD	32.8	61.9	5.3	1200

Table 8-2 shows the grain boundary characteristics comparison between the FDD and HDD drawn wires, the FDD drawn wires had a 41% higher volume fraction of HAGB and 70% higher $\Sigma 3$ twins in the wire, whereas the HDD drawn wire had a 57% higher percentage of LAGB. In the HCF regime, for the three batches of wires tested, FDD-2 and FDD-3 could reach the runout requirement of 30M cycles at a stress amplitude of 720MPa, and the FDD-1 wire reached the runout at an amplitude of 670MPa. However, with the HDD drawn wire, the three batches of wire could reach runout only at a stress amplitude of 550MPa. In the HCF fatigue test where low stress is applied to the material, the mechanics of fatigue, i.e., crack initiation and crack growth is governed by the strength of the material, which is dependent on the GS of the material. Based on the observations in Figure 8.2 and Table 8-2, the superior performance of the FDD wires in the HCF regime when compared to the HDD wires, can be attributed to the smaller grain size and high dislocation density of the FDD drawn wire. The smaller GS contributed to the increased strength of the material, as shown in Figure 8.3(a), where the FDD drawn wire had higher strength than the HDD wire. Since it is well acknowledged [172, 373] that in the HCF region, where the applied stress amplitudes are low, the fatigue behavior of the material is dominated by the crack initiation and most of the time is spent in initiating a crack, rather than propagating a crack which accounts for the total fatigue life. So, with smaller grain size, the residual stress distribution is more homogenous and spread over many grains in the FDD wire, which increased the fatigue resistance in the HCF region.

The LCF behavior of the FDD drawn wire was also better than the HDD wire, even though the GS is smaller for the FDD drawn wire, as the LCF performance is noticed to be enhanced with coarser grain size [182, 374]. At an applied stress amplitude of 1170MPa, the average no of cycles to failure (N_f) for the three batches of FDD wire were, 3×10^4 , 8×10^5 and 4×10^6 cycles, whereas it was only 1×10^4 cycles for all the three batches of HDD wire tested. Even with the decrease of the stress amplitude to 853MPa, which is considered as lower specification of the applied stress

amplitude in the LCF region, the no of cycles to failure slightly improved for the HDD drawn wire with the N_f , being 4×10^4 , 4.8×10^4 and 7×10^4 , and 1×10^5 , 7×10^5 , and 1×10^6 for the FDD drawn wire.

The materials ductility [191, 375] controls the LCF performance or its resistance to crack growth during the fatigue tests. Because of the higher amplitude of applied stress, the crack initiations are absent in the material, and the fatigue limit is controlled by the resistance of the material to crack propagation. It can be noted from Figure 8.3(b), that the ductility of the FDD drawn wire is higher than the HDD drawn wire, and it can also be observed from Table 8-2, that the number of HAGB and coherent twin boundaries (CTB) is higher in the FDD wire, when compared to the HDD wire. HAGB are known to be effective obstacles to the dislocation motion [174, 376], thus increasing the strength of the material, but they also act as preferential sites for crack nucleation with the increased stress amplitude due to the rise in the stress incompatibility of the material. However, the LAGB [377, 378], allow the dislocations to pass through them thus decreasing the stress concentration in the material due to a reduced pile-up of dislocations, thus reducing its strength and increasing the resistant to crack growth. The CTB also plays an essential role in increasing the fatigue endurance, by allowing the dislocations to penetrate and slip through them [379-381], thus contributing to increased strength and ductility of the material. It is acclaimed that higher number of twins [382-384] contributes to improved plasticity and toughness of the material. So higher LCF observed in the FDD drawn wires can be associated with its higher twin density, achieved by the deformation process which bestowed higher strength and higher ductility to the material.

Figure 8.5 and Figure 8.6 show the BF TEM comparison of the post-fatigue FDD and HDD LCF samples; the microstructure of the samples was significantly different. In the FDD wire, there were severe dislocation pile-ups at the GB as shown in Figure 8.5(a), and the sample exhibited a dislocation cell structure as in Figure 8.5(b). There were also several nano twins observed at the interface between the GB and the dislocation pileup as shown in Figure 8.5(c) with the FFT inset. However, with the HDD drawn wire, several slip bands were observed in the deformed structure as in Figure 8.6(a). The dislocations piled up along the slip band forming a ladder structure which is the characteristic of the persistent slip band (PSB) formation as seen in Figure 8.6(b), there were no nano twins observed at the interface between the slip bands and the dislocation pile-up as seen in Figure 8.6(c).

(a)

(b)

(c)

Figure 8.5: TEM post fatigued FDD LCF sample (a) Microstructure of the fatigued sample (b) Deformation cells formed in the microstructure (c) Nano twins observed at the area between the dislocation pileup and the GB.

(a)

(b)

(c)

Figure 8.6: TEM post fatigued HDD LCF sample (a) Microstructure of the fatigued sample (b) Dislocation pile-up along the slip band (c) Absence of nano twins at the interface between the dislocation-pile up and the GB.

Figure 8.7: TEM post fatigued FDD HCF sample (a) Microstructure of the fatigued sample (b) Deformation twins in the microstructure (c) Dislocations being obstructed at the TB (d) Dislocation pileup at the TB.

Figure 8.8: TEM post fatigued HDD HCF sample (a) Microstructure of the fatigued sample (b) Fewer dislocations observed in the microstructure (c) Fewer TB with no dislocation pile-up (d) Surface crack blocked at the GB.

Figure 8.7 and Figure 8.8 shows the comparison of the post-fatigued samples for the HCF condition. Similar to the LCF condition, the microstructure of the FDD drawn wire was considerably deformed when compared to HDD wire, the FDD drawn wire had a vein structure as shown in Figure 8.7(a), whereas the HDD drawn wire had an irregular dislocation structure as in Figure 8.8(a). The twin density in the FDD drawn wire was also observed to be higher, as seen in

Figure 8.7(b) when compared to the HDD drawn wire in Figure 8.8(c). The pileup of the dislocations at the GB was also noted to be higher in the FDD drawn wire, and the dislocations were observed plying the TB as shown in Figure 8.7(c) and Figure 8.7(d), which is the quality of the coherent twin boundary (CTB). This explains why the HCF performance of the FDD drawn was substantially better than the HDD drawn wire, owing to the higher number of twins in the microstructure which contributed to enhanced strength, because of the increased cyclic hardening. The enhanced hardening improves the fatigue resistance in the material, and also stress concentrations accrued in the material are reduced because of the increased density of CTB. However, in the HDD drawn wire, there were a fewer number of twins as shown in Figure 8.8(c) and fewer dislocations piled up at the GB. The GB controls the crack initiation and propagation in the material, as shown in Figure 8.8(d), where the crack after initiating at the surface traveled a sufficient distance before being arrested by one of the GB.

The significant microstructure difference observed in the post-fatigue samples for the LCF and HCF conditions, confirms that the variation in the deformation process by the two different drawing methods brought a disparity in operation of the slip systems due to the dissimilarity in the resolved shear stress applied on the wire by the distinct drawing practices. The appearance of cell structure and the vein structure in the LCF and HCF FDD wires, represent the low energy dislocation configuration of the material which is related to the slip length of the dislocations and depend on the number of active slip systems available in the material. The operation of different or multiple slip systems on the wire deformed with different drawing methods contributed to the different fatigue performance of the material in both the LCF and HCF condition.

8.4.2 Influence of drawing practice on the cyclic behavior of 75 and 95%CW wires.

The wires were subjected to a total plastic strain of 1.34 and 2.96 to obtain the 75% and 95% CW wires. An increase of plastic deformation induces grain refinement in the material, and the GS comparison between the FDD and HDD drawn wires are shown in chapter 6. The average grain size for the FDD 75% CW wire was around 47nm, with the distribution of grains between 30-65nm, and the average grain size for the 95% FDD wire was 35nm with the distribution of grains being in the range of 20-50nm. The HDD drawn wire exhibited an average grain size of 75nm and 58nm for the 75 and 95% CW conditions. The GS distributions were 40-160nm for the 75% CW, and 45-70nm for the 95% condition. The GS distributions corroborated well with the observed UTS in Figure 8.3(a), where an increase of strength was noticed with the reduction of grain size

for the 75% CW FDD, HDD and 95% CW HDD wires. However, the UTS and EL% of the FDD wire decreased with the increase of CW to 95% CW, and this was described in section 4.4.1 and 6.4.3, where the reduced strength in the wire has been attributed to the inverse hall patch effect and plastic strain localization due to the formation of shear bands.

The as-drawn wires are subjected to cyclic fatigue tests in the stress amplitudes of 430-1340MPa for the 75% CW wires, and 740-1550MPa for the 95% CW condition. Figure 8.4(b) and Figure 8.4(c), show the S-N curve comparison for both the wires. The LCF performance of the 75% FDD wire was relatively better than the HDD wire, at a stress amplitude of 1340MPa. The average no of cycles to failure (N_f) for the FDD-1 and FDD-2 batches of wire, were 5×10^4 and 6×10^4 cycles, and the N_f for the HDD-1 and HDD-2 wire were 2×10^4 and 3×10^4 cycles. With the reduction of stress amplitude to 850MPa, the LCF performance of the HDD wire improved with the average N_f being 7M and 10M cycles for the HDD-1 and HDD-2 batches of wire, however, for the FDD drawn wires, the N_f was around 19M and 18M cycles for the two batches of wire. The observed enhanced behavior was ineluctably better than the 50% CW wire condition. The HDD-1 batch of wire reached the runout at a stress amplitude of 492MPa and the HDD-2 batch at an amplitude of 685MPa, whereas the FDD batches could reach the runout at a stress amplitude of 760MPa.

Figure 8.9: TEM post fatigued FDD 75%CW LCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1350MPa (b) Magnified image of the dislocation cell structure highlighted in red box (c) Microstructure of the fatigued sample put to a stress amplitude of 850MPa (d) Magnified image of the dislocation cell structure in 8.9(c)

Figure 8.10: TEM post fatigued HDD 75% CW LCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1350MPa (b) Magnified image of the dislocation cell structure having cross-slip in 8.10(a) (c) Microstructure of the fatigued sample put to a stress amplitude of 850MPa (d) Dislocations crossing the TB, whereas the PSB terminating at the TB .

Figure 8.9 and Figure 8.10, show the comparison between the FDD and HDD post-fatigued 75% CW samples subjected to two different stress amplitudes of 1350 and 850MPa, it can be detected from Figure 8.9(a) and Figure 8.9(c), that the microstructure of the FDD drawn wire,

appeared identical for both the applied stress amplitudes. A dislocation cell structure was observed for both the stress amplitude samples, with the cell size being smaller for the 1350MPa sample when compared to the 850MPa sample. It can also be noticed that the dislocation density of the 1350Mpa stress sample was higher than the 850MPa. This could be due to the increase of the cyclic hardening with the surge in the applied stress, which contributed to increased dislocation density and smaller cell size. However, for the HDD drawn wire there was a compelling difference in the microstructure between the samples subjected to different stress levels, the wire subjected to a 1350MPa stress exhibited a PSB ladder structure, with the slip bands (SB) moving across the GB, by the cross-slip mechanism as shown in Figure 8.10(a). The SB angled or tilted at the interface of the GB, before straightening out again and continuing with their motion. The SB thickness was observed to be smaller, and the dislocations piled along the SB, forming the PSB ladder structure. This explains as for why the HDD LCF wire, exhibited a lower fatigue life when compared to the FDD wire, due to the formation of PSB in the microstructure, which are preferential sites for crack nucleation [385, 386]. There was a slight improvement in the LCF performance of the HDD wire when compared to the 50%CW LCF wire, this can be attributed to the lower thickness of the PSB, and due to the cross-slip of the PSB at the GB which reduced the stress concentration at the pileups.

However, for the 850MPa, stress level, an incoherent dislocation structure (IDC) was observed in the microstructure of the fatigued HDD sample. There was no definite structure observed, and the dislocations were loosely connected as shown in Figure 8.10(c), it can also be observed that the dislocations were able to cross the TB without any hindrance, but the SB were terminated at the TB as shown in Figure 8.10(d). This explains as for why the fatigue performance of the HDD LCF wire improved at this stress level, due to the reduction in the number of PSB, and the ability for the dislocations to cross the TB and GB. However, the fatigue life of the HDD wire was comparatively lower than the FDD drawn wire; this can be ascribed to the PSB ladder structure inherent in the microstructure of the HDD wire which is potentially a preferred site for crack nucleation due to their limited mobility as seen in Figure 8.10(d).

(a)

(b)

(c)

Figure 8.11: TEM post fatigued FDD 75% CW HCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 685MPa (b) Higher twin density in the sample subjected to a stress amplitude of 685MPa (c) Lower twin spacing and thickness in the sample subjected to a stress amplitude of 430MPa

(a)

(b)

(c)

Figure 8.12: TEM post fatigued HDD 75% CW HCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 685MPa (b) Higher twin density in the sample subjected to a stress amplitude of 685MPa (c) Lower twin spacing and thickness in the sample subjected to a stress amplitude of 430MPa

Figure 8.11 and Figure 8.12, compares the FDD and HDD post fatigued HCF samples for the 75% CW, it can be perceived that the microstructures of both the wire were similar, with several twins embedded in the microstructure. The samples exposed to higher stress amplitude of

685MPa, had a higher density of twins with less spacing in between them, whereas the 430MPa samples, had less twin density, but with lower twin thickness. This could justify as to why there was a less notable difference in the HCF performance observed in the 75% CW FDD and HDD drawn wires. The runout stress level for the FDD drawn wire was only slightly higher than the HDD wire, the FDD wire samples attained the runout at a stress level of 760MPa, whereas with the HDD drawn wires, the HDD-2 batch of wire reached the runout at 685MPa and the HDD-1 batch of wire at 460MPa.

Based on the above discussions for the 75% CW wire, it can be inferred that there was a significant difference in the LCF performance of the FDD and HDD drawn wire, because of different microstructural features developed in the wire, during the course of its cyclic deformation by virtue of its operation of different slip systems. The HCF performance, on the other hand, was similar for both the wires, which can be accredited to identical microstructures, strength, and GS in the wires.

The cyclic behavior of the 95% CW FDD and HDD wires is shown in Figure 8.4(c), it can be observed that there was a drop in LCF performance of the FDD wires in the stress range of 1195 MPa to 1550MPa. The average number of cycles to failure N_f for the FDD batches wire at a stress amplitude of 1550MPa, was 3×10^3 cycles, while the HDD batch wires had an N_f of 6×10^3 and 7×10^3 respectively. With the decrease of stress to 1195MPa, the HDD drawn wires had an N_f of 16M and 23M cycles, and the FDD drawn wire had an N_f of only 2M and 10M cycles. Figure 8.13 and Figure 8.14, shows the comparison of the FDD and HDD LCF samples, it can be noticed from Figure 8.13(b), that there were shear bands (SB) observed in the microstructure of the FDD fatigued sample, and as discussed in section 6.4.3 and as shown in Figure 6.12 and Figure 6.13, the shear bands are present in the 95% CW FDD wire even prior to their cyclic tests.

(a)

(b)

(c)

(d)

Figure 8.13: TEM post fatigued FDD 95% CW LCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1550MPa (b) Shear bands observed in the fatigued microstructure (c) High magnification image of the shear bands intersecting with the TB and the dislocations piling up at the SB d) DF image of the shear bands.

(a)

(b)

(c)

Figure 8.14: TEM post fatigued HDD 95% CW LCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1550MPa (b) Dislocation Cell (DC) structure with a high pile-up of the dislocations at the walls of the DC (c) Nanotwinned structure and twins hindering the movement of the dislocations. .

Unlike, the post drawn microstructure, the shear bands in the post fatigued samples are surrounded by a high density of dislocations as seen in Figure 8.13(b) and the shear bands are seen intersecting the TB, with the TB blocking the movement of the SB as shown in Figure 8.13(c). The FFT inset also confirms the parallel markings observed to be the SB as the diffraction pattern exhibits a texture profile with a small misorientation which could be due to the intersection with the TB. The DF TEM image shown in Figure 8.13(d), also demonstrates the several pile-ups of the dislocations along the SB and the movement of the dislocations being impeded. As discussed before in Chapter 6, the presence of shear bands in the microstructure causes strain localization in the material leading to the reduced strength and ductility in the wire, as displayed in Figure 8.3. During the cyclic fatigue tests in the LCF region, the interaction site between the shear band and the TB acts as a potential site for crack initiation [172, 387-392]. This is because of the increased stress concentration at the interface and the pile-up's due to the enhanced mobility of the dislocations, and increased blockage of the shear bands by the TB [393]. This explains as for why the FDD drawn wire had a lower LCF performance than the HDD wire, due to the absence of shear bands noticed in the HDD microstructure as seen in Figure 8.14. The microstructure of the HDD wire exhibited a well-defined dislocation cell structure with severe pile up at the GB as shown in Figure 8.14(b), which activates the intergranular mode of crack propagation, where the mobility and deflection of the crack along the GB is high which increases the tortuosity of the crack path when compared to the transgranular cracking. There were also many nano twins noticed at the interface between the GB and the TB, the presence of nano twins in the microstructure impede the crack advancement [394-397], due to the reduced slip irreversibility [398, 399] and increased slip-twin interactions.

The HCF performance of the HDD drawn wires also improved slightly than the FDD drawn wire, with the runout being attained at a remarkably high-stress amplitude between 1050-1110 MPa, unlike the 50% and 75% CW wires, where the runout stress is low. This could be the extensive plastic deformation applied on the 95% CW wires, which reduced the grain size to a nanometer-scale thus contributing to an overall increase in strength of the wire, which increased the resistance to the crack initiation. Figure 8.15 and Figure 8.16 shows the comparison of the FDD and HDD HCF samples, subjected to a stress amplitude of 1110MPa.

(a)

(b)

(c)

(d)

Figure 8.15: TEM post fatigued FDD 95% CW HCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1110MPa (b) Deformation twins observed in the fatigued microstructure (c) Tangled dislocation structure and several pile-up of dislocations at the GB d) Dislocations moving from the grain exterior to the interior.

Figure 8.16: TEM post fatigued HDD 95% CW HCF samples (a) Microstructure of the fatigued sample subjected to a stress amplitude of 1110MPa (b) IDS and several pile-up of dislocations at the GB (c) Deformation twins observed in the fatigued microstructure with a reduced twin thickness (d) High magnification image of the dislocation debris accumulated at the GB shown in (b).

It can be noted from Figure 8.15(a), that the dislocation cell size of the HCF FDD wire increased, and the number of dislocation cell structures in the wire reduced, the dislocation structure looked tangled and irregular. It can also be observed that the density of dislocation twins

relatively remains unchanged as compared to the 75% CW FDD HCF wire, but the spacing between the twins reduced as seen in Figure 8.15(b). There was an increase of the mobility of the dislocations, and the dislocations cross from the exterior of the GB to the interior of the grain as shown in Figure 8.15(c) and Figure 8.15(d). However, with the HDD drawn wire, the microstructure of the fatigued sample looks akin to the FDD wire, with a combination of tangled and vein dislocation structure as shown in Figure 8.16(a), there was severe dislocation debris accumulated at the GB as shown in Figure 8.16(b) and Figure 8.16(d), because of the increased dislocation activities such as annihilation and interaction. It could also be seen from Figure 8.16(c), that the twin thickness of the HDD wire further lowered when compared to HDD 75%CW wire.

The microstructural differences observed between the two drawn wires could explain why the HCF of the FDD drawn wire slightly lowered when compared to the HDD wire. The reduced pile up at the GB because of the reduction in the grain size, contributed to a lowered dislocation density. It can also be observed that the movement of the dislocations changed its path from the grain exteriors to the interiors which brought about a change in the crack propagation path from intergranular to transgranular. The appearance of the dislocation debris seen in Figure 8.16(d), also justifies the reason behind the increased HCF performance of the HDD wire. As the magnitude of the frictional stress increases, the existing dislocations or the cracks need to apply higher flow stress to cross the debris, thus increasing the threshold intensity in the material. This contributed to a reduced crack propagation rate and increased fatigue resistance in the wire [400, 401].

8.4.3 Fatigue fractography and fracture surface analysis

Post fatigued fractured samples are examined under SEM and HRSEM, to analyze and compare the mode of failure between the FDD and HDD wires and to examine the fracture path and morphology of the crack. Figure 8.17 to Figure 8.22 shows the fractography of the wire samples and their fracture and crack morphology in both LCF and HCF tests.

(a)

(b)

(c)

(d)

Figure 8.17: Fracture surfaces of the as-drawn FDD wire subjected to a 50% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1170MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 670MPa (c) High magnification image of the striations in the LCF sample. (d) High magnification image of the HCF sample showing the striation and steps formation and the crack deflection mechanisms.

(a)

(b)

(c)

(d)

Figure 8.18: Fracture surfaces of the as-drawn HDD wire subjected to a 50% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1170MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 670MPa (c) High magnification image of the LCF wire exhibiting a cleavage fracture (d) High magnification image of the HCF wire showing the microcracks and the cracks propagating as a transgranular through the grains .

(a)

(b)

(c)

(d)

(e)

Figure 8.19: Fracture surfaces of the as-drawn FDD wire subjected to a 75% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1350MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 430MPa (c) High magnification image of the LCF sample with visible striations (d) High magnification image of the HCF sample showing the formation of tongue steps. (e) High magnification image of the inset in (b) showing the shear lips formation at the circumference of the wire.

(a)

(b)

(c)

(d)

(e)

Figure 8.20:: Fracture surfaces of the as-drawn HDD wire subjected to a 75% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1350MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 430MPa (c) High magnification image of the LCF sample showing a visible crack extending from region 1 to region 2 (d) High magnification image of the LCF sample showing cleavage fracture mechanisms and formation of extensive microcracks in the sample . (e) High magnification image of the HCF sample exhibiting the step formed in the sample.

(a)

(b)

(c)

(d)

(e)

Figure 8.21: Fracture surfaces of the as-drawn FDD wire subjected to a 95% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1550MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 1110MPa (c) High magnification image of the LCF sample exhibiting a shear mode fracture with many brittle facets (d) High magnification image of (c) exhibiting transgranular micro cracks fracture within the cracks initiating from multiple sites (e) High magnification image of the HCF sample exhibiting no striations and a micro cracked structure.

(a)

(b)

(c)

(d)

(e)

Figure 8.22: Fracture surfaces of the as-drawn HDD wire subjected to a 95% CW (a) SEM image of the LCF sample inflicted to a stress amplitude of 1550MPa (b) SEM image of the HCF sample inflicted to a stress amplitude of 1110MPa (c) High magnification image of the LCF sample (a) near the yellow box showing the crack propagation through the GB (d) High magnification image of crack in image (c) showing fracture (e) High magnification image of the HCF sample exhibiting striations.

Figure 8.17(a), shows the cross-section of the 50% CW FDD LCF sample subjected to a stress amplitude of 1170MPa, and Figure 8.17(b) displays the cross-section of the FDD HCF sample, subjected to a strain of 670MPa. From the SEM images, it can be observed that there were two distinct zones in the wire cross-section as illustrated by 1 and 2, respectively. Zone 1 represents the fatigue fracture zone, and zone 2 specifies the overload or the fast fracture zone. It can be viewed that the LCF sample had a greater area of zone 2 and lesser zone 1 area whereas the HCF sample exhibited a contrary effect as pointed out by the white arrow bisecting between the two zones. The difference in zone areas could be ascribed to the variation in the applied stress, thus contributing to different fracture zones [54, 164]. High magnification images shown in Figure 8.17(c) and (d), compares the striation spacing between the LCF and HCF samples. It can be noticed that the striation spacing of the LCF wire, was more substantial and a dimpled microstructure was observed, however with the HCF wire, the striations spacing decreased, and several steps (elevated boundaries) were observed in the sample. As described by Kocanda [402], steps are generated owing to the difference in the orientation of the GB and are perceived to retard the crack growth, by deflecting the crack to run along multiple planes and causing the crack tip to bow out. The river-like patterns viewed in Figure 8.17(d), is also the characteristic terrain of steps. The variation in the striation spacing observed between the LCF and HCF explains as for why the LCF sample cannot withstand a higher number of cycles, due to enhanced crack growth rate with the increased striation spacing [403, 404].

However, with the HDD wires, the morphology of the crack mechanics were different, even though the wire exhibited two regions as in FDD samples as in Figure 8.18(a) and (b), the striation spacing in the LCF HDD wire was relatively higher than the FDD wire, as shown in Figure 8.18(c). A cleavage type fracture [177, 405, 406] was observed in the LCF wire, with all the striations aligned along a single plane with no crack deflections along the cleavage planes. For, the HCF sample, there were many micro and macro-cracks propagating along the GB transgranularly as seen in Figure 8.18(d), at a higher speed, thus leading to a catastrophic failure. From the above observations, it could be demonstrated as for why the FDD HCF sample was able to reach the runout of 30M cycles at a stress amplitude of 670MPa, whereas the HDD wires could not. The higher LCF performance examined in the FDD wire could also be reflected upon the fracture characteristic, with a brittle cleavage intergranular fracture noticed in the HDD wire, and a ductile fracture in the FDD wire.

With the increase of the CW, to 75% the number of striations decreased in the HCF FDD and HDD wires as shown in Figure 8.19(b) and Figure 8.20(b), however, the LCF samples had visible striations of reduced width as in Figure 8.19(a) and Figure 8.20(a). There were two zones of fractures, and the spread of the zone areas are similar to the 50% CW condition. Shear lips were observed on the outer circumference of the FDD HCF wire as shown in the inset of Figure 8.19(b), however the lips are not seen on all the HCF wires tested, which suggests that the formation of the lips is dependent on the mode of deformation and the stress conditions at the surface of the specimen. Figure 8.19(b) and (c) display the high magnification images of the LCF and HCF FDD samples. As shown in Figure 8.19(c), the striations formed in the LCF FDD sample were irregular in different crystallographic planes, and the sample manifested a fibrous structure, with the demonstration of a plastic-ductile fracture mode. The HCF FDD sample subjected to a stress amplitude of 430MPa exhibited a tongue step structure, as seen in Figure 8.19(d). Kocanda [402], in his study described that the tongues are developed in the material when the propagating crack interacts with the existing twins or TB in the structure, which contributes to the crack deflecting from its original path upon interacting with the twin plane and retarding the crack growth. In the case of 75% CW FDD HCF wire, with a higher number of twins in the microstructure as shown in Figure 8.11, would have enhanced the crack deflection in the wire, therefore contributing to an increased HCF performance. The formation of shear lips also indicates a mixed-mode of fracture with the cracks not always propagating in a plane perpendicular to the direction to the applied stress [407]. Ling, M. R., and Schijve, J, et al. [408], in their work demonstrated that the crack growth rate decreases with the increase of the shear lip width. The shear lips on the HCF sample shown in Figure 8.19(e), were developed near to the crack initiation site.

However, with the HDD drawn wire, the SEM images shown in Figure 8.20(a) and (b) appeared similar to FDD drawn wire, but the fracture surfaces were different. The LCF HDD wire, had many micro-cracks formed along the cleavage planes forming a quasi-cleavage or mixed fracture as shown in Figure 8.20(d). There were many microcracks noticed in one plane which coalesced with each other thus forming a macrocrack of sufficient crack length or a tear ridge as shown in Figure 8.20(c) which propagated along the cleavage planes. Because of the limited plasticity in the wake of the crack, the elastic anisotropy in the material increased because of the rise in non-homogenous stress distribution. This contributed to an increased SB and GB cracking, with the cracks propagating in planes very close to each other. The HCF wire contrarily exhibited a channel or a stepped wall structure with many cleavage facets perpendicular to the stepped surface. The formation of such a channel structure can be attributed to reduction in the number of

slip planes in the material with the increased deformation. This leads to a reduced cross slip in the material due to increased dislocation annihilation. It was described by [402], that when two screw or edge dislocations of the same sign intersect the cleavage planes, the step height increases, which is again a function of the dislocation density. Because of the formation of this channel structure, the crack jumps between the cleavage planes, thus deviating the crack path and retarding the overall crack growth. The difference in the fracture surfaces acutely define as why the HCF performance between the FDD and HDD were similar, and the LCF behavior was different, due to different topography of the deformation surface.

The LCF and HCF performance of the FDD drawn wire decreased at 95% CW when compared to the other CW reductions. The post fatigued TEM study, concludes that the drop-in performance of the LCF behavior of the FDD drawn wire is ascribed to the formation of shear bands in the structure, which increased the plastic strain localization and reduced cyclic performance. The fracture surfaces of the FDD and HDD fatigue samples are shown in Figure 8.21 and Figure 8.22. It can be noticed from Figure 8.21(a) that the fracture surface of the LCF 95% CW FDD wire was remarkably different from the other FDD wires drawn to lower reductions. The surface displayed a shear mode of fracture, with the zone 1 higher than the zone 2, and no visible striations seen in zone 1. The high magnification images are shown in Figure 8.21(c) and (d), which exhibited a brittle cleavage fracture, with the material decohering from the matrix along the crystallographic planes, yielding a cleavage trans-granular facet structure. This increased the crack propagation rate in the material, and also lead to the formation of multiple secondary cracks, which coalesced and promoted the crack's propagation. However, with the HCF sample shown in Figure 8.21(e), there were no visible striations observed in the fracture zone 1, and very few striations are observed in zone 2. There were many cracks observed on the fracture zone, with the cracks propagating along different planes, and with the crack path being less torturous. It was illustrated by Hanlon [374] that excessive grain refinement in the material, accelerates the Fatigue Crack Propagation Rate (FCPR) due to the reduced crack path tortuosity.

The low cycle fatigue HDD fracture surface sample shown in Figure 8.22(a), had a typical fatigue fracture, with the crack initiating from the fracture zone and terminating at the overload zone, there were regular and well-defined striations observed in Figure 8.22(c), and the crack path looked tortuous and blocked by the GB as shown in Figure 8.22(d). A similar structure was also observed for the HDD high cycle fatigue sample, unlike the FDD HCF sample, there wear many regular and well-defined striations noticed in the structure as shown in Figure 8.22(e). There was

a mixture of tongue and step structure, with micro-cracks traveling inter-granularly along the GB and their movement being hindered along the tongues and steps. The different fracture morphology observed between the FDD and HDD 95% CW wires in the LCF and HCF region contributed to a difference in fatigue performance.

There were some outliers observed during the fatigue testing of the wires drawn with different drawing practices, as shown in Table 8-3. For example, for the 50% CW HDD-1 condition, four out of five wires subjected to a stress level of 586MPa, could reach the runout of 30M cycles, but one sample failed intermittently at 473K cycles. A similar phenomenon was also noticed in the other CW conditions with one or two samples unable to reach the runout stress condition among the wires tested. To confirm for this observed anomaly, the wires were analyzed by SEM/EDX to identify for any inclusions in the wires as MP35NLT material is prone to embed inclusions in the material during its melting process. Figure 8.23 shows the SEM images of the fractured outlier samples highlighted in Table 8-3. It can be noticed that the fractured samples displayed Al_2O_3 and MgO inclusions at the fractured areas, as shown in Figure 8.23(a) and (b). There were also sites of fallout inclusions observed in the fractured areas as shown in Figure 8.23(c),(f) and (h), where the inclusion might have displayed from its original position during the fatigue testing of wires, creating a potential pit like appearance at the fractured area. There were also some fractures observed because of the defects generated by the wire drawing process such as in Figure 8.23(d) and (e), where longitudinal splits and deep drawing lines were noticed at the fracture location.

Table 8-3: List of outliers observed during the cyclic fatigue testing of the wires drawn with different drawing practices and CW conditions

Applied Stress (MPa)	Wire and lot condition					
	50% CW-HDD-1	50% CW-FDD-1	75% CW-HDD-2	75% CW-FDD-1	95% CW-HDD-1	95% CW-FDD-2
1110.0	NA	NA	NA	NA	30,746,561	NA
1110.0	NA	NA	NA	NA	1,529,617	NA
1110.0	NA	NA	NA	NA	30,097,907	NA
1110.0	NA	NA	NA	NA	30,785,673	NA
1110.0	NA	NA	NA	NA	30,674,327	NA
759.5	NA	NA	30,378,941	NA	NA	NA
759.5	NA	NA	30,486,743	NA	NA	NA
759.5	NA	NA	17,421,376	NA	NA	NA
759.5	NA	NA	30,467,489	NA	NA	NA
759.5	NA	NA	30,846,321	NA	NA	NA
740.0	NA	NA	NA	NA	NA	30,656,319
740.0	NA	NA	NA	NA	NA	30,139,160
740.0	NA	NA	NA	NA	NA	5,164,549
740.0	NA	NA	NA	NA	NA	30,189,524
740.0	NA	NA	NA	NA	NA	30,422,670
721.7	NA	349,599	NA	NA	NA	NA
721.7	NA	30,987,665	NA	NA	NA	NA
721.7	NA	30,797,296	NA	NA	NA	NA
721.7	NA	30,654,749	NA	NA	NA	NA
721.7	NA	333,849	NA	NA	NA	NA
685.1	NA	NA	NA	27,017,684	NA	NA
685.1	NA	NA	NA	30,123,888	NA	NA
685.1	NA	NA	NA	30,903,027	NA	NA
685.1	NA	NA	NA	30,508,732	NA	NA
685.1	NA	NA	NA	30,178,435	NA	NA
586.4	30,076,992	NA	NA	NA	NA	NA
586.4	473,726	NA	NA	NA	NA	NA
586.4	30,365,132	NA	NA	NA	NA	NA
586.4	30,451,598	NA	NA	NA	NA	NA
586.4	26,120,282	NA	NA	NA	NA	NA

(a)

(b)

(c)

(d)

(e)

(f)

(g)

Figure 8.23: SEM analysis of the fractured outlier specimens drawn with different drawing practices (a) 50% CW HDD wire subjected to a stress amplitude of 586MPa displaying the presence of Al_2O_3 inclusion at the fractured area. (b) 50% CW FDD wire (Sample 1-349,599 cycles) subjected to a stress amplitude of 721MPa, displaying the presence of MgO inclusion at the fractured area. (c) 50% CW FDD wire (Sample 5-333,849 cycles) subjected to a stress amplitude of 721MPa, shows a possibility of fallout inclusion at the fractured area. (d) 75% CW HDD wire subjected to a stress amplitude of 760MPa displayed a longitudinal split at the fractured area. (e) 75% CW FDD wire subjected to a stress amplitude of 685MPa fractured at 27M cycles due to crack originating from deep drawing lines on the wire surface. (f) 95% CW HDD wire (Sample 2-1,529,617 cycles) subjected to a stress amplitude of 1111MPa, shows a possibility of fallout inclusion at the fractured area. (g). 95% CW FDD wire (Sample 3-5,164,549 cycles) subjected to a stress amplitude of 1111MPa, shows a possibility of fallout inclusion at the fractured area.

8.5 Summary

From the above results and discussions, the following points can be summarized, concluding the influence of the drawing practices on the cyclic behavior of MP35NLT wires drawn to different plastic strains.

- The 50% CW FDD wires, demonstrated an enhanced LCF and HCF fatigue performance when compared to the HDD wires. The improved LCF behavior was attributed to the formation of nano twins and dislocation cell structures in the FDD drawn wire, which improved the ductility and increased resistance to FCP rate when compared to HDD wire, which displayed a PSB structure, which increased the stress concentration and an increased FCP. However, the increase in HCF performance was ascribed to the smaller GS, and higher density of CTB noticed in the FDD wire, which contributed to increased strength and improved resistance to fatigue crack initiation. The fatigue results were supported by the fractography analysis, which demonstrated a ductile mode of fracture for the FDD wires, with the observation of ductile striations with smaller spacings in between, for the LCF region and a stepped structure in the HCF region. On the other hand, the HDD wire displayed a cleavage mode of fracture in the LCF region and developing a transgranular micro and macro-cracks in the HCF region which increased the crack propagation and lead to a decreased fatigue life.
- The LCF performance of the 75% CW HDD wires, slightly improved with the increase of the plastic strain, and the HCF behavior was almost comparable to each other. The increased dislocation mobility, reduced the pile-up at the slip bands, thus decreasing the width and formation of the PSB and an increased LCF life. The number of cleavage facets reduced, and there were dimple striations noticed in the fractured surface of the HDD wire, the mode of crack propagation change to intergranular and the tortuosity of the crack path increased. The FDD wires displayed a dislocation cell structure with reduced

cell size and decreased striation spacing. However, the HCF performance of both the wires was comparable, and this has been attributed to the similar strength observed in both the wires. The FDD wires, exhibited a tongue structure, whereas the HDD displayed a channel structure, which are both contributors to increased resistance to FCP.

- There was a substantial decrease in the LCF and HCF performance of the FDD wires, upon the increase of CW to 95%, there were shear bands observed in the microstructure of the FDD drawn wire, which increased the strain localization and reduced fatigue endurance. There were no striations observed in the FDD drawn wire in both the LCF and HCF wires, this is because of the reduced ductility in the wire, due to the formation of shear bands. The fractured samples exhibited a brittle cleavage fracture, whereas the HDD wire displayed a dimple striation fracture with a dislocation cell structure with nano twins embedded at the interface of the GB. The HCF HDD wires, displayed a irregular dislocation structure (IDC) with increased dislocation pileup and dislocation debris at the interface of the GB, whereas in the FDD wires a tangled dislocation structure was noticed and with the dislocations seeping through the GB to the interior of the grains thus changing the mode of crack propagation from intergranular to transgranular. In the HDD wire, a mixture of tongue and stepped structure was observed, which increased the HCF performance of the wires.
- It can be concluded that the FDD practice is suitable for MP35NLT wires until 75% CW, as it contributed to increased strength, ductility and LCF, and HCF fatigue performance when compared to the wire drawn with the HDD practice. For an increased reduction or plastic strain above 75%, it is best to avoid the FDD drawing practice, due to exorbitant microstructural changes, which impacted the mechanical and fatigue performance of the wires.

Chapter 9 Conclusions and Recommendations for further study

9.1 Conclusions

The aim of the current study is to elucidate the influence of the drawing practices i.e. Full Die Drawing (FDD) and Half Die Drawing (HDD) on the mechanical, electrical, fatigue properties and microstructure of MP35NLT (35% Ni-35% Co-20% Cr-10% Mo) fine wires, which are used in the manufacture of many medical devices and components. The work focused primarily on developing the interrelationship between the processing method to its microstructure. A robust understanding of the effect of the processing method on the fatigue behavior of the wires was also done by associating the mechanical properties of the wire to its microstructural attributes. Based on the above studies, the following conclusions can be drawn:

1. The wires drawn with FDD drawing technique contributed to a higher strength, lower inhomogeneity, and increased work hardening in the material when compared to the HDD wires for a similar CW reduction. It was also observed that the FDD drawn wires had a smaller grain size, uniform grain size distribution, homogenous texture and higher volume of lattice defects such as twins and dislocation density in the material. This can be attributed to the higher reduction per pass in the FDD drawing process, which reduces the deformation factor (Δ), and thus reducing the amount of redundant work subjected to the material by the drawing process. The increased contact length of the wire during the FDD drawing process induces higher plastic strain or deformation in the wires which conferred to the observed phenomenon.
2. A softening effect in the material was observed in the wires drawn with FDD practice between 75-90% CW, which lead to a reduction of material strength and increased ductility. This phenomenon was attributed to the “Inverse Hall Petch Effect due to the variation of slip mechanisms in the material with the increased deformation.

3. The variation in the electrical conductivity between the FDD and HDD drawn wires was observed only until 50% CW, after which the two curves merged, irrespective of the higher amount of dislocations and lattice defects observed in the FDD microstructure. This can be explained by the size effect in the wires, where the surface volume of the wire is reduced with the increase of CW% due to the lowered thickness of the wire, therefore contributing to reduced density of grains. The electrical conductivity of the HDD wires was higher than the FDD drawn wires, but the relative increase of strength to the loss of electrical performance was minimal.
4. The Plastic Instability (PI) which is defined by the $(\gamma+m)$ parameter and was comparatively lower in the FDD wires when compared to HDD drawn wires until 75% CW, and increased for a reduction of 95% CW, with the $(\gamma+m)$ value being higher for the FDD wire. The increased PI with the increased deformation for the FDD drawn wires, can be ascribed to exhaustion of the dislocation density with the reduced grain size, which restricts the dislocation motion and localizes the applied plastic strain in the material leading to the formation of shear bands in the material, which contribute to a reduced strain rate sensitivity (SRS) and strain rate work hardening (SRWH) in the material. However, the lowering of $(\gamma+m)$ for the HDD wires can be ascribed to increased dislocation annihilation which led to an increased dynamic recovery in the material due to the increased grain boundary shearing and sliding mechanisms, thus lowering the dislocation density and the hardening rate.
5. There is a pronounced influence of the strain rate (SR) on the SRWH and SRS on the MP35NLT wires drawn with different drawing practices. Different hardening regimes were observed in the wires deformed with increasing SR, and the SRWH increased with the increase of SR; this was attributed to the increased dislocation density, reduced twin thickness, and a reduction in the grain boundary (GB) mechanisms such as GB sliding and shearing with the increased SR. This also led to a reduction in the dislocation cell size and a decreased pile-up of dislocations at the GB, which decreased the activation

enthalpy of the material due to the reduction in the mean free path of the dislocations and reducing the overall dislocation annihilation rate, and reducing the dynamic recovery rate in the material.

6. The Strain Rate Sensitivity (SRS) or the m value, of the FDD drawn wire decreased with the increase of SR, due to the reduction in the dislocation-based activities and increased GB shearing and diffusion in the deformed wire. An increased SR also lowered the twinning rate in the material, thus reducing the twin density and increasing the twin spacing, which contributed to a lower m . However, with the HDD drawn wire, at an SR of 10^{-6}s^{-1} , the value of m , was 65% lower than the FDD value. The reason for such a low SRS value has been attributed to the increased localized strain concentration in the material, leading to the formation of shear bands in the material which reduces the capability of the material to resist necking and thus reducing the ductility of the material.
7. An abnormal hardening effect was observed in the HDD drawn subjected to an SR of 10^{-2}s^{-1} , the stage II hardening peak originated at a high strain, instead of its usual commencement at low SR, after the dynamic recovery. This effect was due to the solute segregation of the Mo atoms to the GB, as observed by the increased concentration of Mo in the deformed structure, which increased the strength and the hardening capacity of the wire.
8. The Low Cycle Fatigue (LCF) and High Cycle Fatigue (HCF) performance of the FDD drawn wire was remarkably higher than the HDD drawn wire at 50% CW. The higher strength and ductility observed in the FDD wires justifies the reason behind the observed phenomenon. This has been verified and supported by the post-fatigued microstructure of the wires, where a dislocation cell and vein structure with several nano twins embedded between the grain boundary (GB) was observed in the FDD wires, and a persistent slip band (PSB) structure was observed in the HDD wires. The fatigue results were supported by the fractography analysis, which demonstrated a ductile mode of fracture for the FDD wires, with the observation of ductile striations with smaller spacings in

between, for the LCF region and a stepped structure in the HCF region. On the other hand, the HDD wire displayed a cleavage mode of fracture in the LCF region and developing a transgranular micro and macro-cracks in the HCF region which increased the crack propagation and lead to a decreased fatigue life.

9. At 75% CW the HCF fatigue behavior of both the FDD and HDD wires were very much alike, because of the similar strength and analogous microstructure with many twin bands in them. The HCF FDD wires, exhibited a tongue structure, whereas the HDD displayed a channel structure, which are both contributors to increased resistance to FCP. The LCF performance of the 75% CW HDD wires, improved with the increase of the plastic strain. The number of cleavage facets reduced, and there were dimple striations noticed in the fractured surface of the HDD wire, the mode of crack propagation change to intergranular and the tortuosity of the crack path increased.
10. There was a substantial decrease in the LCF and HCF performance of the FDD wires, upon an increase of CW to 95%, there were shear bands observed in the microstructure of the FDD drawn wire, which increased the strain localization and reduced fatigue endurance. There were no striations observed in the FDD drawn wire in both the LCF and HCF wires, this is because of the reduced ductility in the wire, due to the formation of shear bands. The fractured samples exhibited a brittle cleavage fracture, whereas the HDD wire displayed a dimple striation fracture with a dislocation cell structure with nano twins embedded at the interface of the GB. The HCF HDD wires, displayed an irregular dislocation structure (IDC) with increased dislocation pileup and dislocation debris at the interface of the GB, whereas in the FDD wires a tangled dislocation structure was noticed and with the dislocations seeping through the GB to the interior of the grains thus changing the mode of crack propagation from intergranular to transgranular. In the HDD wire, a mixture of tongue and stepped structure was observed, which increased the HCF performance of the wires.

11. FDD drawing practice is suitable for processing of MP35NLT wires for CW reduction below 95% and intermediate heat treatment, needs to be applied on the material to recover from the microstructural characteristics contributing to a lowered performance in the material. However, the HDD drawing practice is suitable for reductions above 50% due to the increased deformation homogeneity, strength, and cyclic performance. The FDD drawing practice can also be cost-effective as it involves fewer dies for drawing operation and a reduced machine and set-up times

9.2 Recommendations for Future Work

The development of residual stresses in the wire, during the wire drawing operation, is because of the deformation inhomogeneity, induced on the wire when the wire is pulled through a series of dies. The residual stresses can be tensile or compressive depending upon the mode of the deformation method, and they can have a detrimental effect on the mechanical and fatigue performance of the wires. It is good to have a knowledge and a better understanding of the distribution of the residual stress profile on the wires drawn with different drawing practices and correlate them to the microscopic attributes.

From the studies conducted, it was observed that there were significant differences observed in the strain hardening and the strain sensitivity of the wires drawn with different drawing practices. It should be understood that the distribution of stress and strain in the microstructure is highly non-uniform and can vary from grain to grain based on the applied load and the lattice rotations in the material. So, the measurement of elastic and plastic strain across the grains using EBSD digital image correlation techniques would be very beneficial in understanding the localized stress and strain distributions in the wire which could be valuable in understanding the fatigue crack mechanics.

Finally, it is observed that when the material is plastically deformed, most of the energy is dissipated as heat, but much energy is stored inside the material, increasing its internal

energy and affecting the material stability. The method to recover the instability is by subjecting the material to recovery or recrystallization process, which releases the internally stored energy of the material, but this depends upon the prior deformation and the energy state of the material. Because of the complexity of the deformation structures observed in the wire drawn with different practices, it would be useful to understand the recrystallization and recovery mechanisms of the FDD and HDD wires, subjected to different temperatures.

In the current work, the test methodology used to evaluate the cyclic performance of the wire samples drawn with FDD and HDD drawing process was rotary beam fatigue test (RBFT) where an alternate tension-compression force is applied on the samples during their cycling, with the stress ratio being $R=-1$. So, in the future work, it would be useful in examining the influence of different fatigue tests such as push-pull fatigue, flex bend fatigue, ultrasonic fatigue on the performance of the wires drawn with different drawing practices. It would also be beneficial to collect more data points and develop a microstructural model to determine the influence of the various parameters on the fatigue life. It would also be beneficial to understand the performance of the wires in the very high cycle fatigue regime (VHCF) (*i.e.*, $>10^9$ cycles), which predicts more accurately the final device implantation life in the human body for 25 years.

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