Effect of residual stress and cold work on fatigue of inconel 718

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**EFFECT OF RESIDUAL STRESS AND
COLD WORK ON FATIGUE OF INCONEL 718**

CHIN KAI SIANG

SCHOOL OF MECHANICAL AND AEROSPACE ENGINEERING

2019

**EFFECT OF RESIDUAL STRESS AND
COLD WORK ON FATIGUE OF INCONEL 718**

CHIN KAI SIANG

SCHOOL OF MECHANICAL AND AEROSPACE ENGINEERING

**A thesis submitted to the Nanyang Technological University
in partial fulfillments of the requirements 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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Author Attribution Statement

This thesis contains materials from two papers published in the following peer-reviewed journals and from papers accepted at conferences in which I am listed as an author. The thesis also contains materials from two papers under review in the following peer-reviewed journals where I am listed as an author.

Chapter 4 is published as K. S. Chin, S. Idapalapati, D. T. Ardi, “Fatigue Crack Initiation on Surface Treated Nickel Based Superalloy at High Temperature”, Journal of Materials Engineering and Performance (2019)

The contributions of the co-authors are as follows:

- K. S. Chin designed the experiments in the guidance of Ph.D. supervisor S. Idapalapati and co-supervisor D. T. Ardi.
- K. S. Chin analyzed the data, prepared the manuscript and the response to the reviewers.
- K. S. Chin conducted the experimental work i.e. surface mechanical treatments, electron microscopy, sample preparation, stress measurement at Materials Lab 1, School of Mechanical and Aerospace Engineering, Nanyang Technological University and Advanced Remanufacturing and Technology Centre (ARTC).
- S. Idapalapati and D. T. Ardi provided the comments on the manuscript and edited the manuscript drafts.
- S. Idapalapati and D. T. Ardi provided guidance on the project direction and scientific inputs to the paper.

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The contributions of the co-authors are as follows:

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- A. Paradowska and M. Reid provided the stress measurement strategy using neutron diffraction at KOWARI beam at Australia Centre of Neutron Scattering, Australia Nuclear Science and Technology Organization.
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- K. S. Chin analyzed the data and proposed the model for thermal stress relaxation.
- K. S. Chin prepared the manuscript and the response to the reviewers.
- K. S. Chin conducted the experimental work i.e. surface mechanical treatments, electron microscopy, sample preparation, stress

measurement at Materials Lab 1, School of Mechanical and Aerospace Engineering, Nanyang Technological University and Advanced Remanufacturing and Technology Centre (ARTC).

- S. Idapalapati and D. T. Ardi provided the comments on the manuscript and edited the manuscript drafts.
- S. Idapalapati and D. T. Ardi provided guidance on the project direction and scientific inputs to the paper.

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Abstract

Fatigue is defined as failure under repetitive loading and is considered as one of the most common types of failure in engineering components. Fatigue failure could be fatal as it can occur not only under abusive service conditions, but also under normal conditions, with no available warning until the final catastrophic failure. Critical components are often subjected to rigorous fatigue testing to prevent failure during service to avoid catastrophic consequences.

Over the years, researchers have made significant contributions to fatigue study spanning a wide variety of factors and materials. From the early interest concerning mechanical and materials behavior, factors such as heat, corrosion, surface finish have also been taken into consideration. Not only that, fatigue life is related to the interaction between loading history, material characteristics and deformation process by manufacturing or surface treatment processes.

In this study, factors that are of concern are residual stress and cold work produced by surface treatments. The motivation of this study could be attributed to the existing controversy to ascribe the beneficial effects to either compressive residual stress or cold work. Despite the controversy present, current literature commonly suggests that fatigue enhancement as a result of complex interaction between residual stress, cold work and microstructure alteration [1–3].

Residual stress is defined as the stress presents in equilibrium in a component after external processes (thermal or mechanical) have been completed. Residual stress is

linked to fatigue enhancement by inducing delay in crack initiation and crack closure effect. In this study, the origin of residual stress is introduced through the utilization of surface treatment. Compressive residual stress could be produced onto the surface and sub-surface layer of a component by local plastic deformation through surface treatment processes such as shot peening and cold rolling.

On the other hand, cold work is generally defined as the strengthening of a metal by plastic deformation. The effects of cold work are normally hard to discern to that of residual stress, where cold work is inevitably produced simultaneously with compressive residual stress in surface treatment. Therefore, the desirability and beneficial effects of cold work on fatigue life is questioned or even debated among researchers. From the available literature, there are contradictory observations in the desirability of cold work. Some researchers view cold work as desirable due to its effect on fatigue life enhancement [4]. On the contrary, cold work is viewed as undesirable as the high value of cold work is often associated with greater stress relaxation which diminish the original effects of residual stress on fatigue[5].

In addition, stress relaxation increases the complexity of the interaction between residual stress and cold work. Mechanical stress relaxation or thermal stress relaxation is a certain event during fatigue depending on the condition. At room temperature cyclic loading, the stress relaxation is normally attributed to plasticity induced yielding. On the other hand, thermal exposure itself would cause stress relaxation due to reduction of misfit, thermal recovery and dislocation movement.

This thesis pays particular attention in investigating the interaction between residual stress and cold work to determine their influence on fatigue life of Inconel 718. Emphasis will be given to mechanical cyclic relaxation and thermal relaxation as stress relaxation is an inevitable phenomenon during fatigue. The first part of the experimentation covers the characterization of the material after the surface treatment on Inconel 718. Residual stress was characterized using X-ray diffraction while the cold work was measured using EBSD, FWHM and microhardness methods. The effects of residual stress and cold work are realized in the fatigue test conducted where the enhancement in fatigue life was observed. The effects on residual stress and cold work were also observed from the fractography.

The next part of the experimentation focused on the mechanical relaxation of the shot peened and laser peened specimens. The stress measurement was conducted in-situ using the KOWARI beamline at Australian Centre of Neutron Scattering. An alternative set up using reflective method was adopted to achieve optimum temporal and spatial resolution where the residual stress was measured at predetermined cycle. The use of neutron diffraction to resolve the steep residual stress gradient was demonstrated. The deep penetration ability allowed the acquisition of tensile residual stress in the bulk of the material. It was observed that the stress relaxation is the greatest in the first cycle. The role of work hardening in cyclic relaxation was also discussed in this section.

The final part of this dissertation covers the thermal relaxation of laser peened and shot peened specimens. Different temperature and period of exposure were set to

investigate the thermal stress relaxation. It was observed that the role of cold work is more dominant in thermal relaxation when compared to mechanical relaxation. Different thermal stress relaxation behaviours were observed for shot peened and laser peened specimens with distinct residual stress and cold work profiles. A modified Zener-Wart-Avrami function was used to fit the thermal stress relaxation behaviour of the surface treated specimens. Good correlation was observed between the numerical simulation and the experimental data. The last section is dedicated to conclusion and future work where some of the key statements and possible work in the future are highlighted.

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Abbreviations

CW	- Cold work
EBS	- Electron backscattered diffraction
EDX	- Energy dispersive X-ray
DCR	- Deep cold rolling
ESPI	- Electronic speckle pattern interferometry
FCC	- Face centered cubic
FWHM	- Full width half maximum
GOS	- Grain orientation spread
GPa	- Gigapascal
HCF	- High cycle fatigue
HV	- Vickers hardness
IPF	- Inverse pole figure
LCF	- Low cycle fatigue
LP	- Laser peening
MPa	- Megapascal
Nd:YAG	- Neodymium-doped Yttrium Aluminium Garnet
Ni	- Nickel
PSB	- Persistent slip bands

RT	- Room temperature
SEM	- Secondary electron microscope
SP	- Shot peening
TEM	- Transmission electron microscope
Ti	- Titanium
UTS	- Ultimate tensile strength
XRD	- X-ray diffraction
ZWA	- Zener-Wert-Avrami

Symbols

k	- Boltzmann's constant
K	- Kelvin
t	- time
T	- Temperature
T_m	- Melting temperature
a	- crack size
N	- Number of cycles
$\frac{da}{dN}$	- crack growth rate per load cycle
K_{eff}	- Effective stress intensity factor
K_{min}	- Minimum stress intensity factor
K_{max}	- Maximum stress intensity factor
R	- loading ratio
E	- Young's modulus
ν	- Poisson's ratio
ϵ	- Strain
σ_ϕ	- Transverse stress
σ_{ys}	- Yield stress
ΔH_A	- Activation enthalpy

λ	- Wavelength
θ	- Scattering angle
σ^{RS}	- Relaxed residual stress
σ_0^{RS}	- Initial residual stress
d	- interplanar spacing
d_0	- interplanar spacing of unaffected region
α	- Gradient of relaxation dependency on cold work
γ	- Primary gamma matrix
γ'	- Primary gamma precipitate
γ''	- Secondary gamma precipitate
δ	- Delta precipitate

Chapter 1 Introduction

1.1 Background

Fatigue failure is defined as failure under repetitive loading. Fatigue is one of the most common types of failure in engineering components. Fatigue failure can occur not only under abusive service conditions but also under normal conditions, with no available warning that a crack has already developed until the final catastrophic failure. Critical components are often subjected to rigorous fatigue lifing to prevent failure during service which may lead to catastrophic consequences. Initial studies on fatigue were mainly motivated by axle failures due to a major railroad disaster caused by fatigue without visible signs or distortion prior to the incident to warn of the impending failure [6]. Since then, engineers and scientists have made significant contributions to fatigue study spanning a wide variety of materials.

Wöhler made a significant contribution in the area of fatigue through his systematic investigation of fatigue failures during the mid-1800s resulting in the characterisation of fatigue behavior using the stress amplitude-life (S-N) curves and the introduction to the concept of fatigue ‘endurance limit’. As the study on fatigue advances, relationships describing fatigue behavior such as Coffin-Manson relationship, Goodman relationship were also developed and modified empirically[6].

As the research on fatigue progresses over time, the variety and number of factors contributing to fatigue have developed as well. From the early interest concerning mechanical and materials behavior, factors such as temperature and surface finish [7] have also been taken into consideration. Fatigue life is also influenced by load history, material properties and deformations introduced via manufacturing or surface treatment processes.

In this study, the factors that are of concern are residual stress and cold work. The motivation of this study is fueled by the need to correlate the relationship between cold work and residual stress during fatigue. This study also aims to increase the certainty in attributing the beneficial effects of surface treatment process such as shot peening or cold rolling to either the compressive residual stress or cold work generated. Despite the uncertainty present, current literature commonly suggests that the enhancement of fatigue life as a result of a complex interaction between residual stress, cold work and microstructure alteration [8–12].

Residual stress is defined as stress present in equilibrium in a component after all external process (thermal or mechanical) has been completed. Residual stresses have been shown to have a significant effect on fatigue life. In general, compressive residual stress improves the fatigue performance as it induces the crack closure effect and delays crack initiation. Compressive residual stress improves fatigue performance by locally reducing the applied mean stress, thus resisting crack initiation and inducing crack closure effects during the earliest stage of crack propagation [2]. Residual stress could be introduced by various means such as heat energy, mechanical energy, phase transformation, etc. In this study, residual stresses were introduced by mechanical surface treatments. Compressive residual stresses could be produced onto the surface and sub-surface layer of a component by inducing local plastic deformation through surface treatment processes such as shot peening and cold rolling. Compressive residual stress when superimposed with the applied fatigue loads could alter the mean level of the fatigue cycle and the crack nucleation phase [13]. On the contrary, tensile residual stress is deemed detrimental as it adds on to the applied fatigue loads which will decrease the fatigue life.

The term cold work is commonly used interchangeably with work hardening or strain hardening in the literature. Cold work is generally defined as the strengthening of a metal through plastic deformation. It is introduced together with residual stress from a specific surface treatment process. However, the extent of

beneficial effects of cold work on fatigue life remained a question. Another concern regarding cold work is the consistency in the measurement and characterisation. Unlike stress measurement, cold work measurement does not have a standardized system for measurement across methods. This makes it difficult for researchers to directly quantify cold work or compare their results with other researchers.

The research on the effects of residual stress and cold work on fatigue is a challenging and demanding work that is already of the concern of many researchers [1,14–17]. The challenges in this area of fatigue emerge as the effects of residual stress are normally hard to discern to that of cold work, where cold work is inevitably produced in the process of introduction of compressive residual stress by mechanical means. Thus, in this context, the question of the desirability of cold work becomes apparent. From the available literature, it appears that there is a lack of agreement in the desirability in cold work. Some researchers view cold work as desirable due to its effect on fatigue life enhancement from the increase of fatigue crack resistance. On the contrary, cold work is viewed as undesirable as the high value of cold work is often associated with greater stress relaxation which tends to diminish the original effects of residual stress on fatigue.

In addition, the investigation of the interaction between residual stress and cold work is further complicated by residual stress relaxation (either mechanical or thermal). The revolving state of residual stress and cold work under mechanical and thermal influence could eventually dictate the fatigue performance of a component. Mechanical stress relaxation is attributed to plasticity induced yielding. On the other hand, thermally activated stress relaxation occurs due to the reduction of misfit, recovery and dislocation movement. At high temperature fatigue, the combined effects of mechanical and thermal relaxation are known to be synergistic [18,19]. Cold work is observed to play a significant role during stress relaxation especially at elevated temperature due to the recovery of the high density of dislocation.

1.2 Objective

This study aims to understand the interaction and interplay between residual stress and cold work during fatigue. Through the investigation on the interaction between residual stress and cold work, this study could provide an understanding in the determination of the predominant factor in fatigue. The effects of residual stress and cold work in fatigue would also be clarified and discerned.

As stated in the Introduction, intricate interaction between residual stress and cold work are hard to discern. In addition, residual stress, cold work and fatigue each encompasses a huge scope and area which is hard to be covered in a single study. In order to attain the objective of this study, it is imperative to divide this complex matter into phases. Therefore, this study is divided into three sections which focus on materials characterization, mechanical relaxation and thermal relaxation respectively. The alteration in material properties induced by various surface treatment will first be evaluated. Significant changes in the residual stress and cold work will be addressed and considered in subsequent stress relaxation studies. The scope of this study is illustrated in detail below:

Scope

a. Materials characterisation

A nickel-based superalloy, Inconel 718 is subjected to various surface treatments such as shot peening, deep cold rolling and laser peening in this study. The material properties after surface treatment are characterized using different methods and characterisation tools in terms of surface roughness, residual stresses, cold work and fatigue. The difference in their respective residual stress and cold work profiles would also be evaluated. Fractography is also adopted to provide a different perspective on the effects of residual stress and cold work on fatigue.

b. Stress relaxation

The stability of residual stress is important in fatigue life. Significant redistribution of residual stress while performing functional requirements could affect the fatigue performance of a component. Investigation of stress relaxation under the mechanical and thermal influence could provide a better understanding of the interplay between residual stress and cold work and thus their effects in fatigue performance. Current difficulties in the research of residual stress and cold work are being addressed using alternative methodology such as neutron diffraction. Conventional lab-based diffraction method such as XRD is also utilized to evaluate the redistribution of residual stress during thermal stress relaxation.

It is of interest for this Ph.D. study to further distinguish and correlate the relationship between residual stress and cold work during fatigue. Figure 1-1 illustrates the scope covered in this study. A comprehensive literature review in the following chapter will discuss the current state in research and the gaps identified for the investigation of this thesis.

Figure 1-1 - Scope of investigations includes materials characterisation followed by investigations on cyclic and thermal relaxation

1.3 Dissertation overview

This thesis focuses particularly on the correlation between residual stresses and cold work and their respective influence on fatigue performance. Investigation of the stress relaxation behaviour will be the focal point to discern the interaction and effects of residual stress and cold work on fatigue. Therefore, the emphasis will be given to the understanding of the stress relaxation behaviour under mechanical and thermal influence. This thesis is organised into seven distinct chapters covering: literature review, materials and methods, results presentation, discussions, conclusions and future work.

The introduction, objectives and scopes of the study are presented in Chapter 1. Chapter 2 presents the literature review which covers the origin of residual stress and cold work and a review on the role of cold work and residual stresses in fatigue. A section in Chapter 2 is also dedicated to illustrating the focus of this dissertation which includes mechanical relaxation, thermal relaxation and thermo-mechanical relaxation. A summary of the challenges faced and gaps identified at the current state of research is also provided in the end of Chapter 2. The methodology adopted and materials used is discussed in Chapter 3. Next, the following three chapters will present the results of the investigations conducted. Chapter 4 begins with the characterisation of the materials after various surface treatments using several characterisation tools such as X-ray diffraction method, electron backscattered diffraction, secondary electron microscopy etc. In Chapter 5, the mechanical relaxation was investigated using neutron diffraction to evaluate the revolving state of residual state during fatigue cycling. In Chapter 6, the investigation of thermal relaxation using shot peened and laser peened specimens exposed at different temperature and period of exposure is presented. Chapter 7 is dedicated to the conclusion and future work where some of the key findings and possible work in the future are highlighted.

Chapter 2 Literature Review

This chapter aims to provide an understanding on the interaction between residual stress and cold work and the way they manifest their effects during fatigue. This chapter begins with the introduction on the origin of residual stress and cold work. Only residual stress and cold work introduced by mechanical means, ie surface treatment, are focused in this chapter. The roles of residual stress and cold work in fatigue are also being illustrated. A summary of the previous work conducted by other researchers relevant to residual stress and cold work is presented. Emphasis is given on the stress relaxation phenomena where the interaction between residual stress and cold work is the most profound. A section is dedicated to describing the stress relaxation phenomena under mechanical means, thermal exposure and also thermo-mechanical loading. The quantification methods available for residual stress and cold work are also briefly described. An introduction to typical fatigue fracture relevant fractography is also included. A brief description on the type of material adopted (Inconel 718, nickel based superalloy) in this study is included. In the final section of this chapter, a summary of the gaps and challenges identified are being highlighted and addressed.

2.1 Residual stress

2.1.1 Origin of residual stress

Residual stress is defined as the stress remained in a body when no external force is applied [20]. Residual stress normally arises due to misfit within the material or between components. It is often a result of the mechanical and metallurgical history of each point in the component and the component as a whole. In engineering components, residual stress can be introduced mainly through the source of thermal, chemical or plastic deformation [13]. Residual stress is normally divided into three types, depending on the scale on which it is observed. Type I stress is the continuum level stresses or macroscopic residual stress that is present in a body of a component on a scale which is larger than the grain size of the materials. Type II stress is the microstructurally related stress on the scale of an individual grain to a few grain diameters. Type III stress, which exists within a grain, or at a finer scale of crystallite size is often related to the presence of crystalline defects or dislocation.

Residual stresses could influence the mean stress during cyclic loading which will affect the fatigue life. Tensile and compressive residual stress could have a significant effect in altering the fatigue life of a component [6,21]. Tensile residual stress is deemed as detrimental as it adds on to the value of applied stress increasing the value of mean stress during the fatigue cycling which decreases fatigue life. On the contrary, compressive residual stress is usually desirable and is being exploited in the design or surface processing of engineering components. Compressive residual stress could enhance fatigue life by the crack closure effects induced combined with the decrease in the magnitude of applied stress. Nonetheless, the magnitude of the effects of residual stress on fatigue is hard to be determined and quantified as they are often mixed with or masked by other modification such as surface roughness and microstructural alteration [5,6].

The study of residual stress could cover a wide scope depending on its origin and application. In order to meet the objectives of this study, it is important to mention that the primary attention of this study is on the residual stress caused by mechanical means and its effect on fatigue.

2.1.2 Source of residual stress

Residual stress is normally introduced through mechanical means by plastic deformation or misfit between components. When a component experiences different thermal processes, the geometry and heat coefficient of different materials could also produce residual stress. Also, phase transformation in a material could also produce micro residual stress [20]. In this section, only residual stress introduced by mechanical means is focused.

In surface treatment processes, a cold worked layer is formed upon the mechanical deformation method, such as high velocity shots in shot peening, high pressure roller in deep cold rolling and high energy laser plasma in laser peening. The cold worked layer need to expand upon the tensile plastic deformation but is constrained by the bulk volume of the material below the surface. As a result of this restriction, the compressive residual stress is formed near the surface and tensile residual stress is formed in the bulk. Figure 2-1 illustrates the basic characteristics of different surface treatments and how the residual stress profile is formed.

Figure 2-1 - Characteristics of different surface treatment

Different surface enhancement processes are sometimes utilized to produce specific types of residual stress profiles. Figure 2-2 shows the features of interest in a typical residual stress profile which includes the value of surface residual stress, maximum compressive residual stress, the depth of transition to tensile and the depth of the maximum compressive residual stress. These features or residual stress distribution could be generated by different surface enhancement processes and parameters modification within the processes themselves. A brief description of shot peening, cold rolling and laser peening employed in this study could be found in the subsequent subsection.

Figure 2-2 - Typical features in residual stress depth profile

2.1.2.1 Shot peening

Shot peening is a process where a small shot of hard material are blasted at high velocity onto the surface of engineering components to introduce compressive residual stress by plastic deformation [7]. The residual stress introduced normally span onto the subsurface layer depending on the parameters used. Local plastic deformation occurs as the result of indentation at the point of impact. Compressive stress field is produced as the expansion of the plastically stretched surface layer is being restrained by the adjacent underlying elastic mass.

Shot peening is a cold work process that imposes significant changes to the surface and subsurface of a component. Examples of the alteration includes microstructure, microhardness, dislocation density and surface roughness [22]. This process is typically employed to enhance fatigue life and could be used on components of various geometries. However, a rougher surface finish which proved to be detrimental to fatigue life is inevitable [6]. The basic mechanism of shot peening and illustration of the introduction of compressive residual stress on the surface of a component is shown in Figure 2-3.

Figure 2-3 - Mechanism of shot peening

2.1.2.2 Deep cold rolling

Deep cold rolling involves rolling on the surface of the components using rollers with high pressure with the purpose to induce plastic deformation and compressive residual stress in near surface layers [24–26]. It is relatively a simpler process when compared to shot peening. This process uses a hydrostatically controlled ball roller which is applied on the material surface using a controlled pressure. Deep cold rolling normally introduces considerable work hardening depth and compressive residual stress up to 1mm with better surface finish. Care needs to be exerted when rolling force is exerted on the component as an increase in rolling force could shift the area of compression into greater depth but a further increase of rolling force could result in tensile residual stress on the surface [26]. However, the drawback of this process is the lack of flexibility as it could not be applied to components of various and complex geometries.

2.1.2.3 Laser peening

Laser peening is an established process adopted to induce great depth of residual stress and work hardening onto a wide variety of components and materials including nickel and titanium based alloy for aerospace application [10,27,28]. Laser peening involves a high energy density of short duration laser pulse which results in a rapidly expanding plasma burst on the surface of the material. This will result in a shockwave that travels through the material. The work hardening in the material is induced by the shockwave which surpasses the material yield strength. As the shockwave travels through the material, the shockwave loses energy and thus a cold work gradient is observed over the depth of the material. Due to the presence of the plastically deformed layer, residual stresses arise to accommodate for the misfit between the work hardened layer and the elastic bulk. Generally, the shock loading is achieved by using an ablation layer or coating on the surface. The laser peening process can also be enhanced using a transparent fluid layer for momentum transfer.

2.1.3 Methods of residual stress quantification

Stress measurement often serves as a quantitative assessment for the judgement of magnitude and standard of a surface treatment process. Information obtained from residual stress measurement are often used in fatigue life investigation. In order to make a valid and sensible deduction on fatigue life based on residual stress measurement, the researcher needs to be concern of the existing issues and difficulties. The existing technical challenges present in the measurement of residual stress on surface and subsurface has always been a tough hurdle to overcome. This is due to the fact that most of the measurement techniques are destructive which makes the coupon unrecoverable for verification or subsequent measurement after the residual stress is relaxed or redistributed in fatigue loading. Also, the insufficiency and lack of promising non-destructive technique when measuring residual stress relaxation during the service of the component have made

it difficult for the tracking of the magnitude of relaxation and subsequently the assessments of effects of residual stress on fatigue life [3,29].

Efforts have been made into developing a more accurate and reliable residual stress measurement from destructive mechanical release method to non-destructive neutron diffraction method. Table 2-1 and Figure 2-6 at the end of this section provides a quick summary of the common measurement methods. Table 2-1 provides a brief comparison of the advantages and disadvantages of the mentioned methods whereas Figure 2-6 compares the measurement methods in terms of penetration depth and spatial resolution. Generally, mechanical release methods which involve the removal of materials and the destruction of the coupon are still preferred due to its simplicity and versatility. This method often combined the use of either strain gauges, holography and various interferometry for residual stress quantification [30,31]. Below are some of the measurement techniques available:

2.1.3.1 X-Ray diffraction

X-Ray Diffraction, XRD is one of the most popular methods but is only non-destructive for surface measurement. Due to its limited penetration depth, material removal is still required if subsurface residual stress value is to be obtained. Electropolishing is often the preferred material removal method as it induced only minimal or negligible residual stress redistribution which would not affect the subsequent measurement. XRD method is still time-consuming to a certain extent when compared to the mechanical release method if an accurate and reliable of subsurface residual stress distribution is to be obtained.

X-Ray Diffraction technique utilizes the change in atomic plane spacing in the crystal structure when a material is under applied stress or residual stress. The change in interplanar atomic spacing will result in the change in elastic strain which is subsequently used for the determination of stress [32]. In this way, the crystallographic lattice is used as a strain gauge for the determination of macroscopic residual strain and stress.

Theory of $\sin^2 \phi$ method

Interference patterns are formed when a crystalline material is irradiated by X-rays. The determination of stress using X-ray radiation utilizes Bragg's Law for the constructive interference, see Eq. (1).

$$n\lambda = 2d \sin\theta \quad (1)$$

where n = integer
 λ = Wavelength of electron
 d = Interplanar spacing
 θ = Scattering angle

The interplanar spacing of a material will be altered when it is subjected to strain or plasticity, causing a shift in the interference pattern. Residual stress can be evaluated by the measurement of the shift. Laboratory X-rays normally has small depth of penetration which allows for plane stress assumption in the $\sin^2 \phi$ technique [29].

Considering the biaxial stress states in the plane stress condition and taking angles ϕ and ψ with axis x and z respectively, see Figure 2-4, the transverse stress could be obtained by Eq (2).

$$\sigma_\phi = (\epsilon_\phi - \epsilon_z) \frac{E}{(1 + \nu)\sin^2\phi} \quad (2)$$

where E = Young's Modulus
 ν = Poisson's Ratio
 ϵ = Strain
 σ_ϕ = Transverse Stress

2.1.3.2 Neutron diffraction

Neutron diffraction has similar principle as of X-Ray diffraction which relies on changes in the spacing of the lattice plane caused by elastic deformations within a polycrystalline material. The advantage of this technique over X-Ray diffraction is that it has a larger penetration depth deep into the materials, up to 100mm for aluminium or 25mm in steel [34]. The penetration depth of neutron diffraction depends on the material and the power of the beam. Hardware configurations such as detector configuration, sampling gauge volumes, diffracting angles could also affect the strength of the signal [35]. Also, the large penetration depth allows fully non-destructive measurement unlike the need for material removal for depth measurement using XRD. However, the downside of this method is a relatively higher cost when compared to XRD due to its high equipment cost and availability, which makes it not suitable for routine measurement and process control.

2.1.3.3 Hole drilling

Hole drilling is a destructive method commonly used for its reliability and precision. Depth profile can be obtained in a shorter time when compared to XRD technique. The working principle is through the determination of corresponding strains caused by the hole drilling. Figure 2-5 illustrates the schematic representation of center hole drilling with the use of three strain gauges. Strain gauges are attached in the vicinity of the hole aligned with the specific axis of interest to get relative longitudinal and transverse strain [36,37].

Figure 2-5 - Schematic representation of hole drilling technique using strain gauges

2.1.3.4 Optical interferometry (ESPI)

This method normally utilized the strain relief produced by the drilled hole. Electronic Speckle Pattern Interferometry (ESPI) involves the use of interference created by laser for stress measurement. The surface of a material deforms as stress is applied, causing a change in the displacement and thus changing the interference fringe pattern. Stress analysis is done by comparing the change in interference in the fringe pattern to the reference pattern. Strain relief caused by hole drilling could be measured and the stress distribution of the component could be derived [38,39]. Eventually, the residual stress could be calculated from the value of strains using the appropriate calibration constants derived for the particular strain gauge.

2.1.3.5 Magnetic methods and Barkhausen Noise

This magnetic method of Barkhausen analysis can only be used on ferromagnetic materials. The use of magnetic Barkhausen noise has slowly gained attention for residual stress measurement and the potential to detect other microstructural parameters. In a ferromagnetic material, due to domain wall movements, the magnetization of sample changes when an external magnetic field is applied. During the magnetization, domains with alignments parallel or nearly parallel to the applied field vector will expand at the expense of those less favorably oriented. The induced changes are discontinuous on a micro scale and these changes can be studied when an electrical pulse induced on the coil is placed near to it. A noise signal named as Barkhausen Noise is generated when all electrical pulses produced by all domain movements are added together [40].

Table 2-1 - Comparison of residual stress measurement techniques

Technique	Advantage	Disadvantage
X-ray Diffraction	<ul style="list-style-type: none"> - Wide range of systems - Not destructive (for surface measurement) - Versatile - Generally available 	<ul style="list-style-type: none"> - Only basic measurement - Slightly time-consuming
Hole Drilling	<ul style="list-style-type: none"> - Wide range of materials - Quick - Simple - Generally available 	<ul style="list-style-type: none"> - Limited strain and resolution - Destructive - Variance in interpretation of data
Optical Interferometry	<ul style="list-style-type: none"> - Wide range of materials - Generally available 	<ul style="list-style-type: none"> - Limited resolution - Destructive (if couple with hole drilling technique)
Magnetic	<ul style="list-style-type: none"> - Fast - Wide variety of magnetic materials - Portable 	<ul style="list-style-type: none"> - Limited to magnetic materials - Limited resolution
Ultrasound	<ul style="list-style-type: none"> - Portable - Fast - Low cost - Generally available 	<ul style="list-style-type: none"> - Limited resolution - Bulk measurement over the whole volume
Neutron	<ul style="list-style-type: none"> - Excellent penetration and resolution - Macro and micro residual stress 	<ul style="list-style-type: none"> - Expensive - Lab based system - Specialized facility only

Figure 2-6 - Comparison of penetration and spatial resolution of residual stress measurement techniques [41].

2.1.4 Role of compressive residual stress in fatigue

The beneficial effects of compressive residual stress have already been highlighted by many researchers [4,15,42,43]. In a noteworthy study conducted by Wagner [4], the effects of residual stress, cold work and surface roughness were systematically compared by using shot peening, stress relieve and electropolishing. It was demonstrated that the effects of shot peening and the effects of shot peening plus electropolishing give the best performance in terms of fatigue behavior. It was also observed that the fatigue life decreased significantly when the component undergone stress relieve process after shot peening due to the surface roughness produced by shot peening. This could serve as an indicator of the significance of residual stress in fatigue life enhancement. On the other hand, another intriguing observation made is that a subsequent electropolishing step after shot peening and stress relieve still results in a net increase in fatigue life. This increment in fatigue life was attributed to the cold work layers produced on the surface by the shot peening process. However, the increment of fatigue life of these three subsequent processes was not as beneficial as shot peening alone.

2.1.4.1 Effects on crack initiation and crack growth

The beneficial effects of compressive residual stress are being highlighted in some researches with evidence of delay in crack initiation and suppression in crack growth. Hack and Leverant [43] with their study have suggested that the effects of compressive residual stress on crack opening and growth rate behaviors of surface fatigue cracks are more pronounced when compared to fatigue crack nucleation.

It has also been observed from various studies that the presence of compressive residual stress could alter the crack initiation site and crack initiation mechanism of a component [44–46]. For example, the conventional semi-elliptical shapes for crack are distorted under the presence of residual stress fields [47]. A change in crack initiation site is more pronounced in HCF regime, where it has been observed that crack initiation site has shifted from surface to subsurface layer after the introduction of compressive residual stress by shot peening. In the same study,

Torres and Voorwald [16] suggested that the greater the depth of the crack source, the greater the number of cycles until failure.

Regardless of the uncertainty of the effects of residual stress on crack initiation or crack nucleation, researchers tend to agree on the effects of residual stress on fatigue crack growth rate. Over the years, tons of studies have been reported to illustrate the beneficial effects of compressive residual stress on fatigue crack growth rate [43,47,48].

Figure 2-7 - Typical fatigue crack growth curve [15]

A typical fatigue crack propagation curve, shown in Figure 2-7, usually comprises of three regions. Region B is also known as Paris regime where it is relatively insensitive to load ratio, R (K_{min}/K_{max}) and is usually described using Paris Law, the rate of crack growth per number of cycles in relation to the stress intensity factor, Eq. (4)

$$\frac{da}{dN} = C \Delta K^m \quad (4)$$

where a = Crack size
 N = Number of cycles
 C, m = material parameters

The principle of superposition could be used to explain the alteration in load ratio and stress intensity factor range by the presence of residual stress, Eq. (5).

$$R_{eff} = \frac{K_{min} + K_{res}}{K_{max} + K_{res}} \quad (5)$$

where K_{res} = Stress intensity factor due to the residual stress distribution

In a crack with tensile residual stress field, the value of K_{res} is positive leading to $R_{eff} > R$ and vice versa for a crack with compressive residual stress. The alteration in R could lead to change in ΔK values and difference between K_{min} and K_{max} . An increase in value of R could move Regions A and C to lower ΔK values.

When compression is transmitted across the crack faces, it is assumed that $K = 0$ when as there is no fatigue damage. Consequently, for $K_{min} + K_{res} > 0$, R will change according but $\Delta K_{eff} = \Delta K$ remained, where the stress intensity factor is unchanged by tensile residual stress. However, when $K_{min} + K_{res} < 0$, R will also change according to the above equation but ΔK_{eff} is now

$$\Delta K_{eff} = K_{max} + K_{res} \quad (6)$$

With this approach, the equation now becomes,

$$\frac{da}{dN} = C \Delta K_{eff}^m \quad (7)$$

Eq. (7) works for material with fatigue crack growth properties which are insensitive to R in region B. In this circumstance, at low R and low ΔK , compressive residual stress should have the most influence on fatigue crack growth when $K_{min} < |K_{res}|$.

2.2 Cold work

2.2.1 Origin of cold work

Work hardening is rather a relative term and is often linked to cold working, strain hardening and plastic strain. It could be defined as the strengthening of a metal by plastic deformation. Ductile metals often undergo such process to increase the yield strength. The yield strength is increased as a result of the presence of dislocation generation and movements within the crystal structure of the materials [49]. The presence of new dislocations by work hardening will increase to an extent until it is saturated and nucleation of further new dislocation is prevented. As a result of this, the materials experience an increase in strength but a decrease in ductility. The strengthening that is observed in the material after work hardening is the resistance to further plastic deformation due to the prior dislocation formation. To reverse the effects of work hardening, the work hardened material could be annealed at high temperature to reduce the dislocation density by recovery and recrystallization.

As work hardening is a consequence of plastic deformation, the material retains the change in shape or size experienced. When plastic deformation in a material occurs, the inter-atomic bonds will break and a rearrangement of atoms in a solid material occurs. In a non-cold worked material, plastic deformation will occur when a stress beyond the yield strength of is applied. However, a cold worked material will only undergo elastic deformation due to the plastic deformation that has already occurred.

Depending on the context, work hardening may or may not be desirable. An example of undesirable work hardening is the unintended plastic deformation introduced onto the materials when they are processed or machined. On the other hand, desirable work hardening refers to the intended plastic deformation with the purpose to increase the hardness, tensile strength and yield strength. Surface treatments such as shot peening and cold rolling are some examples of the desirable work hardening processes.

Bauschinger effect is another concept that is associated with cold work. After a certain amount of tensile plastic deformation, the yielding of the material can occur at a lower stress of reversed loading direction [6]. For example, the compressive yield strength of the material will decrease with the increase of tensile cold working. The effects of lower compressive yield strength after tensile cold working in mechanical stress relaxation will be discussed in the later section.

2.2.2 Methods of cold work quantification and estimation

2.2.2.1 Full Width at half Maximum (FWHM)

FWHM is defined as the width of the diffraction peak at half of the height of the maximum amplitude, see Figure 2-8. Many researchers have been using FWHM to quantify cold work due to its simplicity and availability [5,11,50]. In a cold worked component, the increased dislocation density caused the decrease in the size of the perfectly crystalline regions and increase of the microstrain in the crystal lattice. As a result of that, the diffraction peak is broadened [51]. Nevertheless, the information on the mode of deformation (tension or compression) could not be determined from the diffraction peak width as the degree of cold work is accumulative and independent of the mode of deformation [51].

Figure 2-8 - FWHM and the effects of strain, instrumental broadening and crystallite size

The effects of other factors such as instrumental broadening, temperature effect and the nature of polycrystalline itself in peak broadening should also be considered [52]. The effects of these individual factors on peak broadening, peak shifting, peak symmetry have been categorized by Singh [53] and is illustrated in Table 2-2 . Regarding this issue, a study conducted by Warren and Avenbach [54] has focused on separating the distortion produced by cold work and particle size broadening. However, it is not the main concern if the comparison of cold work by peak breadth were to be made in components of the same materials.

Table 2-2 - Peak alterations and their respective sources [53]

	Peak shift	Peak Broadening	Peak Asymmetry	Anisotropic Peak Broadening	Peak Shape
Dislocation		+	+	+	+
Stacking faults	+	+	+	+	+
Twinning	+	+	+	+	+
Microstresses		+			
Grain boundaries	+	+			
Sub- boundaries	+	+			
Internal stresses	+				
Point defects					+
Precipitates and inclusions			+		+

2.2.2.2 *Electron Backscatter Diffraction (EBSD)*

EBSD is a tool incorporated in SEM to investigate the microstructure of a material. A focused electron beam is directed onto the material surface and diffracted by the crystalline layer. Kikuchi bands are formed and detected using a phosphor screen detector. Information such as texture, phase distribution, crystalline orientation could be obtained from the processed images [55].

Researchers have highlighted the use of EBSD to measure the strain in the deformed polycrystalline material [56,57]. The quantification of plastic deformation in metals and alloys could be done from the EBSD pattern quality maps where the quality correlates inversely with the defects density. The strain will cause the distortion of the crystal lattice and result in degradation of the diffraction pattern, see Figure 2-9. A new EBSD tool has also been demonstrated by Child et al. [58] to obtain a quantitative evaluation of the depth of the work hardening effects by statistical analysis of the grain orientation spread.

Figure 2-9 - Unstrained and strained crystal and their corresponding diffraction [55]

For the case of plastic strain, EBSD pattern is the combination of two effects. Firstly, when a dislocation has a corresponding opposing edge dislocation, the difference in the diffracting lattice planes will result in an incoherent scattering of electrons, leading to a degraded scan pattern. Secondly, in regions where opposing dislocations are not present (with net burgers vector), a change in the crystal orientation is normally observed and a subgrain boundary is formed. The changes in crystal lattice orientation is reflected with a degraded contrast which corresponds to the amount of distortion present in the crystal lattice [55], see Figure 2-10.

Figure 2-10 - a) Two opposing edge dislocations, b) subgrain boundary, c) effect of subgrain boundary on EBSD pattern

Therefore, EBSD can be used to identify grain boundaries and mapping of the crystalline structure. Work hardening could be measured using a parameter named Grain Orientation Spread, GOS. GOS is defined as the average divergence in the orientation of each point in the grain and the total average orientation in the grain. A higher amount of work hardening would result in a higher GOS which indicates a higher number of dislocations. However, the drawback of this method is that it serves only as an indicative value. Unlike FWHM, GOS cannot be used to describe the absolute amount of dislocations or cold work present in the material.

2.2.2.3 Hardness measurement

Plenty of studies correlated the amount of cold work to hardness empirically [59–61]. The change in microhardness is often associated with the change in yield strength. The localized plastic deformation by surface treatment onto the subsurface layer will result in the increase of localized yield strength. The increase in the localized yield strength could be manifested in the microhardness reading where the hardness value is normally proportional to the amount of cold work. However, there is a possible error in cold work quantification using hardness as the introduction of compressive residual stress increases the value of hardness as well. Unlike cold work quantification using FWHM or EBSD profiles, the hardness value obtained might not just take into account the effect of cold work, but also compressive residual stress as well [62].

2.2.3 Role of cold work in fatigue

Different surface treatments such as shot peening, laser shock peening and cold rolling produce different magnitude of cold work. Generally, it is observed that shot peening imposed relatively a higher value of cold work when compared to deep rolling or laser shock peening [63]. Cold work is introduced simultaneously with residual stress in mechanical surface treatment process. Nonetheless, the effects of cold work seemed to be convoluted and the desirability of cold work on fatigue remained a question.

It has been observed that the depth of the work-hardening effects is reduced by thermal exposure [61]. In terms of stress relaxation, a higher value of cold work is often linked to greater stress relaxation which is deemed detrimental to fatigue life [61,64]. It is often observed that greater stress relaxation occurs at the surface where the maximum value of cold work is observed [65]. Despite that, some researchers still attributed the enhancement of fatigue life to the work hardening rather than compressive residual stress [60,66]. For instance, Altenberger et al. [60] observed improvement in fatigue life with almost complete stress relaxation on the surface. Wagner [4] also observed a similar enhancement in fatigue life even when the residual stress introduced by the shot peening process is relieved.

As a matter of fact, due to the lack of conclusive strategies and techniques, the difficulties in discerning and separating the role of cold work and residual stress in fatigue have been a consistent challenge to many researchers. However, at the current state of research, it is postulated that it is the stability of work hardening combined with residual stress after relaxation that plays a predominant role in fatigue life [63].

2.3 Fatigue

Fatigue is defined as the failure of a component under cyclic loading. It is one of the major types of failures commonly found in structural components which should be avoided to prevent devastating consequences from occurring. Initial studies on fatigue were mainly motivated by axle failures [67] and the effort spend to understand fatigue has never decreased throughout the years. Many rigorous fatigue lifing methods and relationships have been developed to estimate the fatigue life of a component subjected to different environment and application.

Fatigue could be divided into two regimes, namely low cycle fatigue (LCF) which occurs at less than 10^5 cycles and high cycle fatigue (HCF) for failure that occurs at more than 10^5 cycles [6]. The illustration and evaluation of the fatigue life of a component are normally done using an S-N curve, or known as Wöhler Curve to demonstrate the relationship between fatigue life and stress amplitude [6], see Figure 2-11. Apart from that, the separation of the fatigue process into the crack initiation phase and crack propagation phase proposed initially by Langer [68] has definitely affected the definition and scope of fatigue research conducted ever since.

Figure 2-11 - Typical S-N curve

In crack initiation, the mechanism of crack formation could be attributed to the formation of slip bands, the presence of inclusion and pores, etc. Different mechanisms of crack initiation could influence the fatigue life of a component. Generally, low magnitude loading would have no apparent effect on the material in a single cycle. However, if this low magnitude load is repeated for thousands to millions of cycles, changes will be made onto the material on the microscopic scale where dislocations are produced. These dislocations are far too small to be considered as a crack but are sufficient to be considered as the focal point of stress concentration in microscopic scale which leads to crack initiation. Furthermore, it is of interest to further study the changes in fatigue crack initiation mechanism under the presence of other factors such as changes in crack initiation site from the surface to subsurface layer after the introduction of compressive residual stress.

As soon as a fatigue crack initiates, the crack advances further with each stress cycle. This process, known as crack growth or crack propagation will continue as long as the material is subjected to cyclic loading or stress. The crack will grow depending on the frequency and amplitude of loading until it finally reaches a point where the remaining intact cross section is unable to sustain the load in the subsequent cycle. Finally, rapid crack growth and final fracture of the material occurs.

In the crack propagation phase, Paris Law is usually applied where engineering components are assumed to contain inherent cracks. It is of interest to determine the number of cycles for one of these cracks to grow to the extent of catastrophic failure. To illustrate fatigue crack growth data, a graph having an axis of crack growth rate, da/dN versus the stress intensity factor range ΔK , which is the difference between the maximum stress intensity factor, $K_{\max}$ and minimum stress intensity $K_{\min}$ factors applied is normally plotted, see Figure 2-7.

Despite the fact that different scope is defined for the study of crack nucleation and crack propagation phase, it is practically difficult to define the boundary between

these two stages [6]. This difficulty mainly arises due to the capability of the detection method and the interpretation of the terms. In practice, the nucleation stage is mostly defined as the crack initiation plus small crack growth event that happens below the sensitivity of the non-destructive measurement technique.

Researchers have been trying to determine and study the factors to increase the accuracy of fatigue life prediction by numerical and experimental means. One particularly interesting factor affecting fatigue life is residual stress. Residual stress is one of the factors considered by fatigue researchers, manifested by the critical need to enhance fatigue life in aviation and civil industries. Processes like shot peening and cold rolling have been proved to enhance fatigue life by the introduction of compressive residual stress induced by plastic deformation.

It has been mentioned earlier that the focus of this literature review is to provide a comprehensive understanding of the effects of residual stress and cold work on fatigue. In this section, the researches that have been conducted are being categorized according to the materials and surface treatment, see Table 2-3 and Table 2-4. In Table 2-3, a summary of the view of authors on with the significant observation made is provided. Table 2-4 categorizes the researches into materials and surface treatment adopted and the remark column highlights the main focus of the respective research. It could be observed from this section that the most common surface treatment process adopted is shot peening due to its availability and reliability. From the summary, it could be observed that a variety of materials have been researched using different surface treatments. Also, most of the researchers generally agree on the commonly held notion that compressive residual stress enhances fatigue life.

Table 2-3 - Summary of research on the effects of residual stress and cold work on fatigue

Authors	Summary of Research	Remark
Altenberger et al.[60]	Combination of compressive residual stress, cold work and microstructure enhance fatigue life.	Deep rolled and laser shock peened Ti-6Al-4V
Cammett[50]	Compressive residual stress enhances fatigue life. High value of cold work promotes higher magnitude of stress relaxation.	Shot peened Inconel 718
Prev��y[69]	Compressive residual stress enhances fatigue life. High value of cold work promotes higher magnitude of stress relaxation.	Shot peened Ni-Cr-Mo Alloy steel
Abikchi et al.[70]	Microstructure modification enhances fatigue life.	Inconel 718 subjected to different heat treatment
Torres & Voorwald[16]	Compressive residual stress enhances fatigue life. Compressive residual stress alters the crack source to subsurface layer.	Shot peened AISI 4340 steel.
Kim et al.[65]	Compressive residual stress enhances fatigue life.	Shot peened medium carbon steel
Dalaei et al.[71]	Compressive residual stress enhances fatigue life.	Shot peened micro alloyed pearlitic steel
Soady et al.[72]	Combination of compressive residual stress and cold work.	Shot peened tempered martensitic steel
Klotz et al. [11]	Compressive residual stress enhances fatigue life.	Shot peened Inconel 718

Table 2-4 - Surface treatment and materials adopted by various researchers

Materials	Treatment	Authors	Remark
RR1000	Shot Peening	Foss et al.[61]	Residual stress relaxation.
Inconel 718	Shot Peening	Camett et al.[50]	Effects of shot peening coverage on cold work, residual stress and fatigue.
Inconel 718	Low Plasticity Burnishing	Prevéy[64]	Cold work and thermal stability of residual stress.
Waspaloy	Shot Peening	Turnbull et al.[73]	Fatigue crack resistance.
Inconel 100	Shot Peening	John et al.[74]	Stability of residual stress under creep and thermal loading.
Udimet 720	Shot Peening	Evans et al.[75]	Residual stress relaxation under high isothermal fatigue.
Ti-6Al-4V	Deep Rolled & Laser Shock Peened	Altenberger et al.[60]	Fatigue behavior at ambient and elevated temperature.
Ni-Cr-Mo Alloy Steel	Shot Peening	Prevéy & Camett[69]	Effects of shot peening coverage on cold work, residual stress and fatigue.
AISI 4340 Steel	Shot Peening	Torres & Voorwald[16]	Evaluation of shot peening and residual stress relaxation.
Medium Carbon Steel	Shot Peening	Kim et al.[65]	Residual stress relaxation and fatigue.
7075-T6 Al Alloy	Shot Peening	Zhu & Shaw [48]	Correlation of fatigue crack growth with crack closure.

2.4 Interplay of residual stress and cold work during fatigue

Figure 2-12 illustrates a simplified interaction of the three areas of concern which are residual stress, cold work and fatigue. Both residual stress and cold work contribute to fatigue life enhancement by playing a role in crack initiation and fatigue crack resistance. However, the interplay between residual stress and cold work under different circumstances adds complexity into this matter and should not be neglected. Regardless of the introduction of residual stress and cold work by surface treatment such as shot peening and cold rolling, the presence of stress relaxation appears to diminish the beneficial effects.

Figure 2-12 - Simplified interaction between fatigue life, residual stress and cold work

Stress relaxation is an inevitable phenomenon in fatigue cycling and the interplay between residual stress and cold work plays a significant role. The residual stress can be redistributed and relaxed by various mechanisms. One of the mechanisms occurs via yielding in a localized region where the yield strength of the material is exceeded due to the superposition of residual stress and the applied loading [76]. Another mechanism is often linked to high stress amplitude service condition which favors more dislocation movement for the recovery process that promotes initiation and higher crack growth driving forces [77]. The presence of crack could also induce the redistribution of residual stress. Thermal exposure can also affect the stress redistribution and play a significant role in thermo-mechanical cyclic loading.

Wagner [78] demonstrated the complexity of the interplay between residual stress and cold work. Specimens are shot peened (SP), stress relieved (SR) and electropolished (EP) to systematically discern the effects of residual stress and cold work. SP alone was observed to have the best fatigue performance where the combination of SP + SR steps demonstrated the poorest fatigue life. A combination of SP + SR + EP steps resulted in an increase of the fatigue life similar to the fatigue performance of SP alone even when there is no residual stress that remained. Such an inconsistent role of residual stress observed in this study resonates with the notion that it is difficult to pinpoint the fatigue enhancement solely due to residual stress.

Such variance in the behavior of residual stress relaxation needs to be considered in many aspects, especially when a specific value of residual stress needs to be chosen for fatigue lifing or prediction. The potential for residual stress relaxation should be considered in fatigue studies as it is one of the factors causing the different relative effect on LCF and HCF. Instead of using the initial residual stress value, the stabilized residual stress after relaxation should be considered in the calculation to increase the accuracy of fatigue life estimation. However, the evolving residual stress state throughout the fatigue cycling or thermal exposure is hard to be

captured using current measurement methods. Due to this measurement difficulty and the presence of stress relaxation, it is hard to attribute the enhancement in fatigue life to residual stress solely with a high level of confidence.

This section provides a summary of the studies that have been conducted on the stability of residual stress in terms of their origins and nature. The role of cold work under these stress relaxation circumstances is also explored and discussed. This section is imperative as it serves as a foundation to explain the current understanding of the interplay between residual stress and cold work. By conducting a thorough investigation on stress relaxation, the failure to discern the effects of residual stress and cold work which would lead to confusion or farfetched conclusion could be avoided.

2.4.1 Interaction of residual stress and cold work during cyclic loading

In cyclic or mechanical loading, Zhuang and Halford [79] attributed the relaxation to three factors, namely 1) initial magnitude and gradient of the residual stress field and degree of cold working, 2) fatigue stress amplitude, mean stress ratio, number of cycles, 3) material cyclic stress-strain response and degree of cyclic work hardening/softening. The residual stress will relax if the sum of the applied and residual stress exceeds the material yield strength [80]. Another explanation is that stress relaxation occurs via the inhomogeneous yielding where the bulk deformed plastically while the work-hardened surface remains elastic. Consequently, the misfit is reduced by the difference in plasticity between the plastically deformed layer and the bulk which eventually caused stress relaxation [74]. James [81] has also suggested another possible mechanism of mechanical stress relaxation that could take place at late fatigue life or HCF at $10^5 - 10^7$ cycles. The stress relaxation mechanism is suggested to be related to the accumulation of defects generated in the material due to the cyclic loading.

The general finding of many papers showed that under large cyclic stress and strain amplitudes, stress relaxation is more pronounced [79,82]. As opposed to large

cyclic stress and strain amplitudes, small fatigue stress amplitudes has caused little or no stress relaxation [74,79,80]. The residual stress relaxation was observed when the sample is loaded beyond the apparent yield stress [83]. As a result of this, the enhancement in fatigue life is more pronounced in the HCF regime due to less stress relaxation with the decrease of the mean stress amplitude or cyclic amplitude. In contrast, the cyclic strain introduced in large amplitude loading may be enough to reduce the effects of initial compressive residual stress introduced. Enhancement in fatigue life is often observed under most of the stress amplitudes except at stress amplitude close to the yield strength of the material [65].

Stress relaxation does not occur in every loading conditions and has been claimed to only occur in the presence of work hardening [64]. This statement is also supported by Prevey and Foss et al. [51,61] where relaxation is only observed under the presence of work hardening. It is also observed that a higher mean stress amplitude and higher cold work could lead to greater stress relaxation. It has also been reported that the stress relaxation is proportional to the stored energy [61]. On the other hand, an increase in the rate of stress relaxation is observed in the component which has the steepest residual stress gradient normal to the surface[81].

In cyclic loading, components may cyclically harden or soften which result in the redistribution of residual stresses [15]. In extreme cases, residual stress has been observed to relax entirely in the first few load cycles [82]. Nonetheless, this extreme behavior has only been suggested to be limited only in the first few cycles [79,84]. In most of the cases, the significant relaxation is only observed to occur in the first few cycles [76,85,86]. Further relaxation is observed to correlate with the logarithm of the number of cycles [65,76,87]. Mattson and Coleman [42] were some of the first to notice the presence of cyclic stress relaxation. In their study, even though the compressive residual stress is partially relaxed, the fatigue life is still enhanced. Conversely, at high stress amplitude of 80% above the yield strength, Torres and Voorwald [16] showed that stress relaxation occurs and the

compressive residual stress introduced has no positive effects on the fatigue life. Such discrepancies in observation signify that stress relaxation is an occurrence that should not be neglected in the estimation of the fatigue life.

Another factor that could come into play is the Bauschinger effect. Bauschinger effect could be understood as the decrease of compressive yield strength due to the tensile cold working and vice versa. Higher work hardening normally indicates a greater extent of Bauschinger effect where the compressive yield strength is depreciated at the expense of the increment of tensile yield strength. The Bauschinger effect influences the stress relaxation where the reduced compressive yield strength of the material could be exceeded relatively easily by the superposition of applied compressive loading and compressive residual stress [76].

In a literature review on deep rolling conducted by Altenberger [26], the effects of cold work and compressive residual stress are summarized. It was suggested that near surface work hardening is responsible for fatigue enhancement in materials exhibiting low 0.2% yield stress whereas compressive residual stress has the most effects in fatigue enhancements in materials having high 0.2% yield stress. It was also speculated that work hardening has the most effects on the smooth surface produced by deep rolling in crack initiation dominated fatigue life.

From the literature review conducted on mechanical relaxation, it is found that the role of cold work is more speculative than quantitative. Generally, the governing stress relaxation mechanism in cyclic loading could be explained by the mechanics model attributed to quantifiable material properties such as yield strength and residual stress. Yielding and the need to reduce misfit between the plastically deformed layer and the elastic bulk could be viewed as the main factors causing the mechanical stress relaxation. It is worthy to note that mechanical stress relaxation is mostly observed at the beginning of the cyclic loading where further cyclic loading seems to play little significance. Under this circumstance, the mechanical stress relaxation could be considered as material properties driven (irrespective

with time) where the focus should be given to the initial residual stress distribution and magnitude of cold work.

2.4.2 Interaction of residual stress and cold work during thermal exposure

Under thermal exposure, the stability of residual stress is crucial, especially in the application of gas turbine engine. Therefore, it is crucial to understand the behavior and pattern of stress relaxation under the presence of the thermal source. At elevated temperature, it is observed that the residual stress is greatly relaxed [80,84]. In one of the earlier studies by Vohringer et al. [88], it is observed that stress relaxation does not occur at 300°C. However, stress relaxation was observed with the increase in temperature and period of thermal exposure in many studies [5,18,89,90]. The stress relaxation caused by thermal exposure could be explained by the recovery of dislocations which reduces the misfit in plastic strain. It is also suggested that stress relaxation is caused by the thermal exposure which results in the diffusive or dislocation movement of atoms which reduces the misfit between the bulk and plastic deformed layer [91].

At early stages, the thermal relaxation is the most significant due to rapid annihilation or reorganisation of defects by short-range diffusion [92]. This significant thermal relaxation was highlighted in a study conducted by Nalla et al. [59] where ~50% reduction of residual stress was observed in just 45 minutes of thermal exposure. Abhishek et. al [93] also observed various degree of relaxation (~15-30% depending on the surface treatments) just after 5-10 minutes exposure at 550°C. Regardless of the significant relaxation during the initial period, many studies have reported observing stability in stress relaxation with slight changes over a long period of time [74,94]. However, not all component relax in the same manner as were observed by Prévy [5] and Cammett [50] in two different studies. They have attributed the difference in stress relaxation to the value of cold work percentage where they observed the higher value of cold work was associated with more significant stress relaxation. Surface treatment such as shot peening with a high level of cold work was linked to greater stress relaxation when compared to

surface treatments with lower value of cold work such as laser peening or gravitational peening. The high level of cold work signifies the presence of greater defect density which would increase the propensity of thermal stress relaxation[95].

In the stress relaxation study conducted by Masmoudi and Castex [96], it is observed that relaxation varied from 50% at lower temperature to almost 100% at higher temperature. It is also observed that all the changes in the residual stress occur within the first 10 hours. Buchanan [97] in his study observed different dynamics of surface residual stress and subsurface residual stress relaxation. The surface residual stress relaxed quickly in the first hour and remained stable after further exposure while the subsurface continued to relax slowly with increased exposure time.

Few numerical approaches of thermal stress relaxation have been found to be accurate to a certain extent [98,99]. Generally, the model is based on the Zener-Wert Avrami (ZWA) function, Eq. (8),

$$\frac{\sigma^{RS}}{\sigma_0^{RS}} = \exp[-(At_a)^m], A = B \exp\left(-\frac{\Delta H_A}{kT_a}\right) \quad (8)$$

where A , B , m are fitting parameters and ΔH_A is the activation energy.

Nikitin and Besel [98] attempted to explain higher stability of work hardening when compared to residual stress at high temperature by using the ZWA method. It is found that the activation energy for stress relaxation is lower than the one of work hardening relaxation. The difference in the activation energy reflects the higher thermal stability of cold work when compared to residual stress. From the activation energy obtained, they suggested that the favorable mechanism for stress relaxation is controlled by the thermally activated gliding of dislocations. From another study by Foss et al. [95], residual stress and work hardening relaxation were also observed. Significant residual stress relaxation observed in the early

stage was attributed to the rapid annihilation or reorganisation of defects via short range diffusion.

From the literature review conducted on thermal relaxation, factors such as the selection of temperature and duration of exposure were observed to play the most significant role. While the stress relaxation dynamics differ across materials, it is worth to note that the onset temperature for stress relaxation to occur was observed in some of the studies. On the other hand, the role of cold work percentage is also crucial as mentioned by several studies. The magnitude of cold work is observed to have a direct correlation to the magnitude of stress relaxation. Cold work relaxation is also observed but in a smaller extent when compared to the residual stress relaxation. It is postulated that cold work possesses a higher thermal stability when compared to residual stress.

While the information on subsurface residual stress is considerably scarcer than surface residual stress, it is generally noticed that the dynamics of subsurface stress relaxation are much slower and more stable when compared to the surface residual stress relaxation. Such observation could be attributed to the difference in the level of work hardening often obtained after surface treatment. The treated surface normally possesses the highest magnitude of work hardening with decreasing gradient onto the subsurface and eventually to the bulk of the material. The stress relaxation model based on ZWA function were also observed to describe the stress relaxation behavior accurately to a certain extent.

2.4.3 Interaction of residual stress and cold work during high temperature fatigue

During high temperature fatigue (or known as thermo-mechanical fatigue), the combined effects of mechanical relaxation and thermal relaxation are observed. Generally, it is observed that the mechanical and thermal relaxation are synergistic under such circumstance [3,59,100]. It is an established fact that an increase in temperature will result in the decrease of the material yield strength. The decrease of the material yield strength will increase the likelihood of yielding caused by the

superposition of the applied loading and the residual stress of the same sign. This will result in the increase of the propensity for stress relaxation when combined with the thermal exposure of the material happening at the same time. Therefore, under thermo-mechanical loading, the effects of mechanical relaxation and thermal relaxation are often observed to be synergistic. Also, it is worth noting that localized or short range microplastic deformation during cyclic loading can also induce stress relaxation below the macroscopic yield stress of the material used[100].

Nalla et al. [59] have also observed various degree of stress relaxation on the fatigue cycling at different temperature on Ti-6Al-4V. It was observed that the magnitude of room temperature cyclic relaxation is only minimal for laser peened specimen and moderate for deep rolled sample. However, the residual stress relaxation was observed to be extensive when the temperature is increased. A similar observation was also obtained by Tufft [101], where the relaxation observed at high temperature is more severe than the effects of fatigue cycling alone. However, complete or near complete relaxation of residual stress is rarely observed [3]. Severe cycling and the additional influence from the elevated temperature are required for such complete relaxation to occur as observed in the study by Zeller [102] and Holzapfel et al. [103].

While it is generally accepted that the stress relaxation in thermo-mechanical cycling is synergistic of mechanical and thermal relaxation, Kim et al. [90] observed that the stress relaxation in the first cycle is dominated by mechanical relaxation. It was also observed in that study that the extent of residual stress relaxation after one cycle is similar despite the use of different temperature. In the study conducted by Meguid et al. [100], it is postulated that the effects of thermo-mechanical cyclic is similar to that of cyclic loading with an increase of applied tensile stress due to the reduction in the yield strength of the material. Cao et al. [18] also categorized the separate effects of thermal and mechanical stress relaxation from the material and stress-strain perspectives, see Figure 2-13.

Figure 2-13 - Thermal and mechanical stress relaxation [18]

In mechanical relaxation, only the residual stress in the loading direction is relaxed. On the other hand, the residual stresses relax homogeneously under thermal condition. Thermo-mechanical relaxation sees the combination of both thermal and mechanical relaxation but with decreasing difference between the longitudinal and transverse directions as the fatigue cycle progresses [90,103]. With an increasing number of cycles, the residual stress between longitudinal and transverse directions converge. This observation suggests that the reduction in misfit at elevated temperature occurs primarily by a thermally enhanced mechanism [90].

Work hardening is claimed to be more stable than residual stress under either mechanical or thermal relaxation. The relaxation of work hardening under mechanical loading or thermal exposure is insignificant when compared to residual stress. However, under thermo-mechanical cycling, the occurrence of work hardening relaxation increases [61,75,90]. The synergistic effects of thermal and cyclic relaxation reduce the stability of work hardening albeit in a lesser extent when compared to residual stress. Despite the reduction in the magnitude of work hardening, the depth of the work hardened layer remained unchanged.

Table 2-5 - Summary of type of stress relaxation and their respective characteristics

Relaxation type	Characteristics
Mechanical relaxation	<ul style="list-style-type: none"> - Time independent. - Material properties driven. - Localized yielding and reduction of misfit as main mechanisms. - Macro scale properties. - Role of cold work is relatively insignificant. - Relaxation in the direction of loading only. - Relaxation can occur beyond the depth of work hardening.
Thermal relaxation	<ul style="list-style-type: none"> - Time and temperature dependent. - Diffusion, dislocation movements and annihilation as main mechanisms. - Micro scale properties. - Role of cold work is significant. - Homogeneous relaxation.
Thermo-mechanical relaxation	<ul style="list-style-type: none"> - Synergistic of mechanical and thermal stress relaxation. - First cycle dominated by mechanical relaxation. - High temperature decreases the material yield strength. - Increase propensity of relaxation due to reduction of yield strength caused by the increase in temperature.

2.5 Fractography

Fractography refers to the study of fractured surfaces of materials involving features interpretation on the fractured surface. Fractography is employed to determine the cause of failure and to understand the origin of fracture, i.e. to understand how and why it fails. An accurate identification of the failure mode is crucial to understand the reason to why a component fails [104]. Some common failure modes include fatigue, corrosion, ductile or brittle overload, creep and etc. The cause of failure could be determined by studying the characteristics present on a fractured surface. An essential part of fractography is the understanding of how cracks nucleate and grow microscopically to cause macroscale fracture. Therefore, the establishment and examination of crack origin are one of the most important aims in fractography [105].

In some of the cases, if the loading pattern is known, optical microscopy at microscale is enough to determine the nature of failure and causes of crack initiation. Otherwise, SEM is employed to give more information at a finer scale and also with higher confidence. It is also typical to begin an examination of fractured surface starting with low magnification and increase it subsequently to examine features of interest. When combined with Energy Dispersive X-ray Spectroscopy, EDX, useful information regarding elemental composition around crack origin could also be revealed. The use of EDX around crack origin could be utilized if the crack is suspected to originate from brittle phase, inclusion or precipitate.

In fractography related to fatigue, common terms associated include beach marks or concentric rings, striations, ratchet marks and secondary crack, see Figure 2-14. These distinct features observed could give different information regarding the fracture surface. Positions of the crack position could be represented by the presence of beach marks or concentric rings which are usually crescent shaped macroscopic marks. Beach marks are formed due to changes in the environment

when the crack grew such as the change in crack growth mechanism due to bursts of fast fracture intersperse with fatigue. Ratchet marks are macroscopic step marks or ridges that occur when the initiation of multiple cracks at slightly different planes is observed. These cracks will combine into a single fracture plane as they progress. However, the presence of ratchet marks does not exclusively confirm fatigue failure mode.

Formation of striations, see Figure 2-14 - Beach marks and striations, is a classic feature related to fatigue fracture. Striations normally appear as relatively evenly spaced parallel lines that show the propagation of the crack front and give information on fatigue crack growth. It represents the crack growth increment in one load cycle or it could be viewed as a microscopic crack that results from a single load or cycle. It is also possible for irregular striations to form if the load amplitude and condition are varied during the cyclic loading. Repetition of these cycles will result in progression of shallow crack and this repetition is termed as fatigue crack propagation. One important thing to take note is that not all appearance of striations conclusively proves the occurrence of fatigue as striations-like markings are also observed in stress corrosion cracking.

A systematic study was also conducted by Hershko et al. [106] to correlate the fatigue striations to the fatigue cycle. Good correlation was obtained where the fatigue life could be estimated by looking at the striations from a few regions. A few criterias were also mentioned to increase the accuracy to estimate the fatigue cycle from the striations counting. For example, few areas must be used in counting striations where the area used for the counting must be planar and normal to the electron beam.

Figure 2-14 - Beach marks and striations

Crack initiation is often the first site of fractography as many details can be obtained from it. The observation of crack initiation site is significant as the site of crack initiation has been correlated to fatigue performance [107]. It is also crucial to observe whether the cracks have propagated in transgranular or intergranular manner to distinguish the phases during fatigue. For example, a fatigue crack initiation site could be identified if the persistent slip bands are observed on the free surface of the material. Then, the crack site can be further investigated to determine if the crack propagates under mode I or mode II mechanism, or in a transgranular or intergranular manner, see Figure 2-15.

Generally, three crack growth mechanisms are possible in fast fracture – brittle fracture via cleavage along crystallographic planes or through pearlite lamellae, transgranular ductile fracture via microvoid coalescence, and interdendritic fracture or intergranular along grain boundaries. Fatigue, in general, involves transgranular fracture but intergranular fatigue is possible under special circumstances. A general observation is that the fatigue region is normally smoother than the fast fracture zone with a lighter shade of grey. It may sometimes be difficult in fractography to differentiate between fatigue and fast fracture under certain circumstances, but a thorough consideration of all the observations and facts generally leads to the correct interpretation.

Figure 2-15 - Schematics of fatigue crack initiation and propagation [108]

2.6 Nickel based superalloys

Nickel-based alloy has additions of chromium, titanium, cobalt, molybdenum and other elements in a different composition to give a specific performance. Generally, the nickel based alloy is well known for its outstanding high temperature strength and oxidation resistance at elevated temperature in excess of 550°C[109]. One commonly used nickel based alloy is Inconel 718, which is a typically wrought nickel based alloys. It is one of the most widely used alloys that has been studied extensively in terms of machinability, surface integrity and residual stress [110–112].

2.6.1 Application and motivation

The development of nickel-based superalloy has been driven by its primary application in high temperature environment, especially in gas turbine engines. It is also commonly used in space vehicles, nuclear reactors and other high temperature application. The complexity and flexibility in their composition allow a wide range of optimization of alloys to be developed for a specific application. However, nickel based alloy is also known for its poor machinability for reasons such as severe surface abuse of machined workpiece and short tool life which is damaged by the work hardening that occurs on the workpiece rapidly during machining[113]. The changes that are induced during machining on the surface such as residual stress could have an adverse effect on the mechanical and stress corrosion properties[113]. Surface integrity of the component is one of the issues that should not be neglected during machining. Due to the rapid work hardening characteristics of nickel-based superalloy, it is preferable for this material to be machined using as few passes as possible. Some major changes that could occur during the machining process that is of concern are residual stress, plastic deformation due to hot or cold work and metallurgical transformation caused by high temperature machining. The adverse effects of the machining process could be

minimized if most of the machining processes are done before the age hardening are carried out.

2.6.2 Typical microstructure

Nickel-base alloys have developed progressively due to the continuous study of alloying elements and its microstructure. The strengthening by some alloying elements and microstructure or phase are generally described below [114]:

Alloy matrix (γ) –Contains high percentage of solid solution elements such as cobalt, molybdenum and tungsten, cobalt, iron, titanium and aluminium in the continuous matrix with a continuous matrix of FCC nickel-based austenitic phase.

Gamma prime (γ') – Normally precipitated in austenitic nickel superalloys by precipitation hardening. The addition of titanium and aluminium to precipitate high volume fractions of FCC γ' coherently with the austenite. It is a coherent precipitate phase that could be found homogeneously throughout the γ matrix due to the similar lattice parameter to the γ matrix and high chemical compatibility. Also, the generation of film of γ' along the grain boundaries by heat treatment is believed to optimize rupture properties.

Gamma double prime (γ'') – A metastable coherent precipitate with a body-centered tetragonal (BCT) lattice structure. At high temperature, the γ'' will transform to delta (δ) phase resulting in a loss of hardness.

Carbides – Carbon is often added in the range of 0.05-0.2% by wt to chemically interact with the reactive and refractory elements to form primary MC carbides. The alloys high temperature strength, ductility and creep properties are dependable on the distribution of carbides along the grain boundaries.

Topologically close packed phase – Plate-like phases such as σ , γ and Laves are usually formed at elevated temperature. Their brittle and hard properties make it liable to fracture and thus causing a lower rupture strength and ductility.

2.7 Summary

This chapter presents an overview of the roles of residual stress and cold work in fatigue with emphasize on stress relaxation. The scope of this study is designed to address some of the mentioned challenges highlighted in the literature review. Based on the literature review conducted, the following issues will be addressed in the following chapters:

- i. The effects of surface integrity on fatigue life have been mentioned by many researchers. The role of surface integrity, particularly residual stress and cold work have been shown to have profound effects on fatigue life. Nonetheless, their effects are difficult to discern and often convoluted with other factors such as surface roughness. These factors are often in mutual competition to dictate the fatigue performance. Therefore, preferred features in the residual stress and cold work profiles for fatigue enhancement would need to be identified. Methods such as fractography could be utilized to determine the predominant factor in dictating the fatigue life.
- ii. Mechanical stress relaxation involves the redistribution of residual stress through plastic yielding. The magnitude of relaxation differs based on the applied loading condition, initial residual stress and cold work profiles. Researchers have previously mentioned the challenges present in tracking the redistribution of residual stress during cyclic loading. The destructive nature and incapability of many measurement methods to perform in-situ residual stress tracking presents a certain degree of uncertainty. The use of multiple coupons and the subsequent inconsistency in material removal (either by compliance methods or electropolishing for depth measurement using XRD) could also introduce variance and errors in data analysis leading to results with low

confidence. The use of non-destructive residual stress measurement such as neutron diffraction could reduce such concern and increase the confidence in the data obtained.

- iii. Materials or component experience thermal stress relaxation to reduce the misfit within themselves when exposed to elevated temperature. The magnitude of relaxation mainly depends on the magnitude of work hardening and cold work, temperature and duration of exposure. Correlation between work hardening and magnitude of relaxation has been observed by many researchers. Such correlation has been utilized in the modelling of thermal stress relaxation and is generally in good agreement with experimental data. However, such modelling effort is often suitable only for the specific surface treatment where the correlation is based on. For example, a thermal relaxation model developed based on shot peening would be in poor agreement with the experimental data obtained from laser peening. An exact quantification suitable for the description of thermal stress relaxation across different surface treatment processes is still lacking. In order to address this concern, this study attempts to look into the development of a suitable model to describe the thermal relaxation process across different surface treatment processes.

Chapter 3 Materials and Methods

3.1 Overview

From the literature review, the difficulty in distinguishing the effects of cold work and residual stress during fatigue has been identified. This study approached this complication by first characterising the materials after surface treatment. Then, emphasis is given to mechanical and thermal stress relaxation as stress relaxation is a definite event to occur during fatigue loading. From the scope of investigation, it is anticipated that the role of residual stress and cold work under different circumstances during fatigue could be clarified.

The extent of research that have been done on residual stress and cold work covers a large domain. The huge domain encompasses the origin of residual stress and cold work, measurement of residual stress and cold work, their respective effects on mechanical properties etc. In addition, the intricate and complex interaction between residual stress and cold work adds difficulty to this study. Therefore, it is imperative to split the investigation into parts to further dissect the effects of residual stress and cold work on fatigue. This study focused only on the residual stress and cold work which are generated through plastic deformation, i.e. surface treatment.

The first part of this study begins with materials characterisation which covers the quantification of residual stress and cold work. Fatigue test and fractography are adopted to investigate the extent of the effects of residual stress and cold work. The second part of this study focused on the mechanical relaxation after surface treatment. The final part of this study explores the thermal relaxation at different temperature and period of exposure. From all these sections, a comprehensive understanding on the interaction between residual stress and cold work during fatigue could be obtained.

3.2 Materials - Inconel 718 and heat treatment

A nickel-based alloy, Inconel 718 was adopted in this study. Inconel 718 is a well-known nickel-based alloy with additions of chromium, titanium, molybdenum to give a specific high temperature performance. The typical chemical composition of Inconel 718 is shown in Table 3-1. It is generally used in the aerospace industry for its outstanding high temperature performance and high oxidation resistance at high temperature. The common heat treatment adopted for Inconel 718 is the double precipitation aging treatment. According to AMS 5663 standard, this heat treatment would require the material solution to be solution annealed at 954°C for 2 hours, undergone precipitation heat treatment at 718°C for 8 hours and furnace cooled to 621°C followed by air cooling for 8 hours. For fatigue testing, the specimens are designed according to the ASTM standard, E466, see Figure 3-1.

Figure 3-1 - Flat and cylindrical coupons geometry design according to standard E466

Table 3-1 - Typical composition of Inconel 718 according to AMS 5663 standard

Elements	Min (wt%)	Max (wt%)
Carbon	--	0.08
Manganese	--	0.35
Silicon	--	0.35
Phosphorus	--	0.015
Sulfur	--	0.015
Chromium	17.00	21.00
Nickel	50.00	55.00
Molybdenum	2.80	3.30
Niobium (Columbium)	4.75	5.50
Titanium	0.65	1.15
Aluminium	0.20	0.80
Cobalt	--	1.00
Boron	--	0.006
Copper	--	0.30
Lead	--	0.0005
Bismuth	--	0.00003
Selenium	--	0.0003
Iron	remainder	

3.3 Surface treatments

In this study, different surface treatments were adopted to induce significant difference in the profiles of residual stress and cold work. For example, shot peened specimen is normally characterised with shallow compressive residual stress and high cold work on surface whereas laser peening is generally characterised with greater depth of residual stress and cold work. Such distinct difference in the profiles could help in the discerning of their respective effects in the following investigations. Similar surface treatments could not be adopted throughout these separate investigations as these investigations were carried out at different testing facilities such as Nanyang Technological University, Advanced Remanufacturing and Technology Centre and Australia Centre for Neutron Scattering. The design of specimen geometry (flat or cylindrical geometry) was compromised and thus affecting the selection of surface treatments. The selection of surface treatments was summarized in the table below.

Table 3-2 – Surface treatment adopted in different investigations

Investigation	SP	LP	DCR	Remarks
Characterisation	✓		✓	Cylindrical specimens
Mechanical relaxation	✓	✓		Flat specimens
Thermal Relaxation	✓	✓		Flat specimens

3.3.1 Shot peening

Shot peening involves impacting the workpiece with shots of small and hard materials at high velocity to introduce compressive residual stress by plastic deformation [23,115]. In the characterisation work, the shot peening process on the specimen was carried out on the test section of the cylindrical specimen. The shot peening process utilized cast steel shot of 0.6mm diameter at a flow rate of 3kg/min, resulting in Almen intensity of 0.308mmA. In the mechanical and thermal stress relaxation studies, the specimens were peened on the top and bottom surfaces with cast steel shots of 0.6mm diameter at a flow rate of 3kg/min, resulting in Almen intensity of 0.200mmA.

3.3.2 Deep cold rolling

Deep cold rolling is a cold work process where the normal force produced by the supporting pressurized fluid is applied on the workpiece by the planetary rotation of hardened rollers. The irregular peaks and valleys on the machined surface are plastically deformed by the roller where the peaks are displaced into the valley in the subsequent rolling pass [116,117]. In the characterisation work, the rolling direction is perpendicular to the longitudinal axis (loading axis) of the cylindrical specimen. The ball size adopted was 6mm diameter hardened steel ball, rolled at a pressure of 200 bar.

3.3.3 Laser peening

Laser peening produces compressive residual stress with considerable depth through the shockwave generated by the high energy laser plasma. Laser peening at Advanced Remanufacturing and Technology Centre was utilized in the investigations of mechanical and thermal stress relaxation. Nd:YAG laser with wavelength 1064nm at a frequency of 5 Hz was utilized. 2mm beam spot size and 5J of beam energy was used for the process.

3.4 Part I - Materials characterisation

This study begins with the materials characterisation to identify the effects of residual stress and cold work on fatigue performance.

3.4.1 Sample preparation

Table 3-3 presents the chemical composition determined by X-ray fluorescence method. The bars were solution annealed at 1,030°C and furnace cooled before precipitation heat treatment at 780°C for 6.5 hours followed by air cooling. The surface roughness profiles were obtained using a Taylor Hobson's CCI HD non-contact 3D optical profiler.

Table 3-3 – Composition (wt%) of Inconel 718 adopted in materials characterisation study

C	Mn	Fe	S	Si	Cu	Ni	Cr
.03	.08	17.65	.002	.06	.19	53.46	18.46
Al	Ti	Co	Mo	Ta	B	Nb	P
.56	.94	.33	2.90	.003	.001	5.19	.008

3.4.2 Residual stress measurement using XRD

Residual stress measurement was carried out using X-Ray diffraction method in full compliance to BS EN15305:2008. The measurements were completed using Stresstech Xstress 3000 G3 X-ray Stress Analyser by employing Mn $K\alpha$ -radiation at 30kV voltage and 6.6 mA current. $\sin^2 \psi$ method was adopted on {311} planes. The measurement on the {311} planes was adopted as it is more representative of the bulk elastic constant and less sensitive to intergranular strain when compared to other planes [118,119]. Also, high 2θ angle has greater sensitivity for peak broadening with influencing factor and provides a good option for Full Width Half-Max (FWHM) analysis. The ψ angle was varied between -38° and $+38^\circ$ in 13 oscillations and cross correlation method was used to determine the 2θ peak shift. The residual stress depth profiles were obtained by measurement in between layers

removed via electro polishing technique. The error bars were determined based on ISO/IEC Guide 98-3:2008 which accounts for the uncertainty in test procedure such as instrument alignment, operator bias and materials specification.

3.4.3 Cold work measurement using XRD, microhardness, EBSD

Full Width at Half Maximum (FWHM) using X-Ray diffraction method has been used to estimate the level of cold work by the broadening of diffraction peak due to reduced crystallite size and increased microstrain [51]. FWHM values were obtained concurrently with the XRD stress measurement and the error bars were determined via the same uncertainty of measurement method.

Microhardness values were obtained by Vickers Hardness Test on polished cross sectional area using 500g load and a dwell time of 15s. Error bars were derived from the calculation of average and standard deviation of three indentations made at each depth at a separation more than 2.5 times the length of the indent diagonal.

A JEOL JSM 7600F Field Emission Scanning Electron Microscope equipped with Oxford Instruments' backscattered electron detector was used. The EBSD tool (Aztec Software) was employed to assess the depth of work hardening by analyzing the grain orientation spread (GOS). Prior to EBSD assessment, the specimens were prepared using 0.5 μm colloidal silica for final polishing.

3.4.4 Fatigue testing

Fatigue tests were conducted in the longitudinal direction of the specimens on an MTS Landmark servo hydraulic fatigue tester under load control at a load ratio of $R=0.1$ with sinusoidal wave profile at 20Hz. The as-machined samples were tested at room temperature, whereas the surface treated specimens were tested at 400°. For high temperature test, the specimens were soaked at 400° C for 2 hours with displacement adjustment on zero balancing load prior to the beginning of fatigue test to prevent the building of thermal strain in the specimens. At each test condition, one specimen is tested. The cylindrical dog bone fatigue specimens were

machined in accordance to ASTM E466 standard and the treated surface area is illustrated in Figure 3-2.

3.4.5 Fractography

The fractography was conducted on the optical microscope and secondary electron microscope. The fractured specimens were preserved after failure from fatigue testing and were cleaned using ultrasonic bath prior to microscopy examination.

Figure 3-2 - Fatigue specimen design in accordance with ASTM E466. Shaded area illustrates the surface area treated

3.5 Part II – Mechanical stress relaxation

In this investigation, neutron diffraction was employed to measure the residual stress at predetermined cycles. The deep penetration capability of neutron diffraction enables the acquisition of tensile residual stress at the centre of the bulk.

3.5.1 Sample preparation

Flat bars of IN718 produced via vacuum induction melting were used in this study, see Figure 3-3. See Table 3-4 for the chemical composition determined via X-ray fluorescence method. The material was solution annealed at 954°C for 2 hours, undergone precipitation heat treatment at 718°C for 8 hours and furnace cooled to 621°C followed by air cooling for 8 hours. The flat bars were then machined into fatigue specimens according to the standard ASTM E466.

Table 3-4 – Composition (wt%) of Inconel 718 adopted in mechanical stress relaxation study

C	Mn	Fe	S	Si	Cu	Ni	Cr
.03	.05	18.35	.001	.06	.03	53.46	18.46
Al	Ti	Co	Mo	Ta	B	Nb	P
.51	.98	.13	3.03	.003	.004	5.33	.007

Figure 3-3 - Specimen design adopted in mechanical stress relaxation study

3.5.2 Ex-situ residual stress measurement using neutron diffraction

The diffraction measurements were conducted at ANSTO using KOWARI strain scanner. Ex-situ residual stress was conducted with the specimens placed on the neutron diffraction stage. Ex-situ residual stress measurements were first performed prior the in-situ measurement to determine d_0 spacing from a reference specimen without surface treatment. Next, using the d_0 spacing obtained, subsequent ex-situ residual stress mapping was performed on five specimens, which included the pristine, shot peened, laser peened, shot peened fatigued at 10,000 cycles and laser peened fatigued at 10,000 cycles. The gauge volume used was 0.2mm x 0.2mm x 10mm. The depth penetration per gauge volume is 0.282mm and 25 overlapping gauge volume steps were conducted across the thickness of the specimens.

3.5.3 In-situ residual stress measurement using neutron diffraction

The non-destructive nature of neutron diffraction enables the in-situ residual stress depth measurement on one specimen. The fatigue cycling was conducted on an INSTRON stress rig at 10Hz using R ratio = 0.1 at a peak stress of 900MPa. The residual stress was measured at the 0th, 1st, 10th, 10000th and 50,000th cycles while the same specimen remained attached on the stress rig during the in-situ measurement.

A modified reflective mode setup was used for the measurement to optimize the spatial resolution and measurement time, see Figure 3-4. In this setup, the spatial resolution is determined by the gauge volume. Gauge volume was designed to be parallel with the top and bottom surface (the x_2x_3 plane) to resolve the steep residual stress gradient near surface. The measurements were done at specific steps of depth using gauge volume of 3mm x 3mm x 0.15mm for residual stress depth profiling. The first in-situ measurement was targeted on the surface and each subsequent depth-specific data is shifted by steps of depth from the surface. This dimension of gauge volume was selected to achieve a spatial resolution for the measurement of the steep residual stress gradient of a typical surface treated

material while not sacrificing a great amount of time for measurement at each predetermined cycle. Measurements were taken across the thickness of the specimen to obtain a full residual stress depth profiling.

Figure 3-4 - Schematic diagram of alternative reflective method set-up for in-situ diffraction measurement.

3.5.3 Cold work measurement using EBSD

A JEOL JSM 7600F Field Emission Scanning Electron Microscope equipped with Oxford Instruments' backscattered electron detector was used. The EBSD tool (Aztec Software) was employed to assess the depth of work hardening by analyzing the grain orientation spread (GOS). Prior to EBSD assessment, the specimens were prepared using 0.5 µm colloidal silica for final polishing.

3.6 Part III – Thermal stress relaxation

For the study of thermal stress relaxation, shot peened and laser peened specimens were subjected to different temperature and period of exposure. The residual stresses of the specimens were measured using XRD method. FWHM and EBSD methods were also utilized for the comparative assessment of cold work.

3.6.1 Sample preparation

Flat bars of Inconel 718 produced via vacuum induction melting were used in this study. See Table 3-5 for the chemical composition determined via X-ray fluorescence method. The material was solution annealed at 954°C for 2 hours, undergone precipitation heat treatment at 718°C for 8 hours and furnace cooled to 621°C followed by air cooling for 8 hours.

For the thermal exposure, the specimens were cut into 2cm x 2cm square and were placed into Elite Thermal Systems BRF14/5-2416 furnace at various temperature. The selection of temperatures was based on the application temperature of typical nickel based superalloy [120] and expected recovery temperature suggested by Khadhraoui et al. [99] at 393°C. Madariaga [121] in his thermal annealing study also observed minimal stress relaxation at 400°C. Therefore, the temperatures of 250°C, 400°C and 600°C were chosen for this experimentation. Please see Table 3-6 for the experimentation matrix for the thermal relaxation study. The furnace was pre-heated to respective temperature before the specimens were put into the furnace. Upon the completion of thermal exposure, the specimens were removed from the furnace and left to cool at room temperature.

Table 3-5 – Composition (wt%) of Inconel 718 adopted in thermal stress relaxation study

C	Mn	Fe	S	Si	Cu	Ni	Cr
.03	.05	18.35	.001	.06	.03	53.46	18.46
Al	Ti	Co	Mo	Ta	B	Nb	P
.51	.98	.13	3.03	.003	.004	5.33	.007

Table 3-6 - Exposure temperature and duration of thermal exposure

Exposure Temperature, °C	Thermal Exposure Duration, h
250	1, 10, 20
400	1, 5, 10
600	1, 10

3.6.2 Residual stress measurement using XRD

The residual stress depth profiles of the specimens exposed under different temperatures and different period of exposures were obtained. The residual stress measurement was also carried out on laser peened and shot peened specimens without thermal exposure. A similar residual stress measurement methodology to section 3.4.2 was carried out using Stresstech Xstress 3000 G3 X-ray Stress Analyser at Advanced Remanufacturing and Technology Centre.

3.6.3 Cold work measurement using XRD and conversion to cold work percentage

FWHM values were obtained concurrently with the XRD stress measurement and the error bars were determined via the same uncertainty of measurement method. Then, the obtained FWHM values were converted to cold work percentage.

As demonstrated by Klotz et. al. [11] and Prevey [51], the true plastic strain of the material could be deduced by the value of the peak broadening. The conversion from peak broadening to the true plastic strain normally involves the measurement on material with known cold work percentage. Otherwise, the true plastic strain could be calculated using the changes in the dimensions of the specimens from a series of tensile test. Then, the plastic strains could be correlated to FWHM using the equation,

$$FWHM = H[1 - e^{-I\epsilon_p}] + J\epsilon_p + K$$

where ϵ_p is the plastic strain of the specimen and H, I, J, K are the fitting parameters.

As the materials and measurement parameters utilized in this study were the same as Klotz et al. [11], the fitting FWHM fitting parameters were adopted in this study, see Table 3-7. The values obtained were used to determine the cold work distribution across the depth of the laser peened and shot peened specimens.

Table 3-7 - FWHM fitting parameters for Inconel 718 treated according to AMS 5663

H	I	J	K
0.9622	0.4808	0.0410	2.65

Chapter 4 Materials Characterisation of Surface Treated IN718[#]

This chapter describes the materials characterisation work on surface treated Inconel 718 specimens. The complete materials and methods adopted for the characterisation work is described in Chapter 3. Nickel based superalloy IN718 specimens were subjected to two surface treatments, namely shot peening (SP) and deep cold rolling (DCR) to investigate the effects of surface integrity (in particular residual stresses and cold work) on fatigue performance. Shot peened and deep cold rolled specimens recorded different residual stress profiles using X-ray diffraction stress measurement. Microhardness profiles were also obtained from the samples as a comparison against full-width at half maximum (FWHM) from XRD in evaluation of cold work. Deep cold rolled specimen recorded a greater depth of compressive residual stress and work hardening. A load controlled fatigue test was then conducted to determine the extent of beneficial effects of residual stress and cold work. Surface roughness effects were found to be insignificant, evident from the surface initiated fatigue cracks in both SP and DCR specimens. Fatigue life improvements exhibited by SP and DCR specimens over the as-machined equivalent can be attributed to crack retardation due to work hardened layer as well as compressive residual stresses.

[#] the content is published as “Fatigue of Surface Treated Nickel Based Superalloy at High Temperature” in Journal of Materials Engineering and Performance.

4.1 Results

a) Surface roughness

The as machined surface roughness has Ra value of $0.406\mu\text{m}$ and Rz value of $5.193\mu\text{m}$. Deep cold rolling provides comparable surface finishing whereas the shot peening increases the surface roughness parameter significantly, see Figure 4-1. However, in deep cold rolled specimen, distinct series of peaks and valleys are observed along the direction of rolling. Surface roughness of shot peened specimen has a higher Ra and Rz value, at $1.635\mu\text{m}$ and $13.579\mu\text{m}$ respectively whereas Ra and Rz value of deep cold rolled specimen are $0.992\mu\text{m}$ and $5.338\mu\text{m}$ respectively.

Figure 4-1 - Optical surface roughness profile, a) as machined, b) shot peened, c) deep cold rolled

b) Residual stress profiles variation

Conventional machining such as cutting, turning and grinding produces tensile residual stress on the surface [113]. Such feature is observed in the as machined specimen where the near surface residual stress is tensile, see Figure 4-2. However, to maintain stress equilibrium in the material, the residual stress beyond 50 μm is compressive but in a much lower magnitude when compared to shot peened and laser peened specimens.

The shot peened and laser peened specimens show compressive residual stress on the surface which increases until 50 μm depth and gradually decreases across the thickness. Shot peened specimen shows a consistent value in both longitudinal and transverse directions with maximum compressive residual stress at -800MPa at 50 μm . The directionality nature of deep cold rolling is not only evident in terms of surface roughness but also in the residual stress value of longitudinal and transverse direction. Deep cold rolled specimens are normally characterized with large compressive depth due to the mechanism where the plastic deformation is produced by a single application of large force at a relatively low strain rate [26]. The stress measurement obtained in this study also shows a greater depth of compressive region for deep cold rolled specimen. Unlike the steep gradient of compressive residual stress in shot peened specimen, the deep cold rolled specimen's maximum compressive residual stress remains high at -500MPa at 500 μm below the surface.

Figure 4-2 - Residual stress profiles, a) as-machined, b) shot peened, c) deep cold rolled

c) Cold work distribution

The determination of cold work deformed region using FWHM and hardness values appears to be sufficiently consistent, see Figure 4-3. Both methods indicate similar trend and reduction of values with distance from the surface. Both FWHM and hardness profiles showed that shot peened specimen has a higher value of cold work on the surface but decrease with a steeper gradient.

Figure 4-3 - Comparison of FWHM and hardness at different depth, a) shot peened, b) deep cold rolled

d) Fatigue performance

Significant improvement in fatigue lives were observed in surface treated specimens despite the tests being carried out at elevated temperature, see Figure 4-4. The difference in fatigue life is more significant at lower stress load. At higher fatigue stress level, the beneficial effects of surface treatment processes are not significant as all the specimens possess similar fatigue life. At high stress level regime, the superposition of the tensile residual stress and the tensile cyclic load could cause yielding and results in stress relaxation. Deep cold rolled specimen possessed the longest fatigue life where a run out specimen is observed at 600MPa. The longer fatigue life of deep cold rolled specimen could be due to the greater depth and relatively higher magnitude of compressive residual stress. Also, deep cold rolled specimen with lower cold work level near the surface would experience less stress relaxation and thus have a more stable residual stress relaxation profile which contributes to a longer fatigue life [64].

Figure 4-4 - Fatigue cycle of shot peened and deep cold rolled specimens at high temperature and as machined specimens at room temperature.

e) Fractography of fractured fatigue specimens

Fractography analysis indicated that specimens tested at higher stress have a rougher and fibrous fractured surface with multiple ratchet marks whereas specimens tested at lower stress show a smoother fractured surface. At higher stress level, multiple crack initiations on surface are more easily observed on as machined specimens, see Figure 4-5. Ratchet marks indicate the presence of cracks originated from different planes which coincides as one. Fibrous fractured surface indicates the fast fracture of fatigue while the bright facet area indicates a slower and more steady fatigue crack propagation. The bright facet area increases when the number of cycles to failure increases which indicates that a larger portion of the crack is propagating in a steady manner when the load level decreases, see Figure 4-5 - Figure 4-7. Another interesting observation is that concentric rings are only observed in shot peened and deep cold rolled specimens at high temperature where the number of concentric rings increases together with the number of cycles, see Figure 4-6 - Figure 4-7.

Figure 4-5 - a) As machined specimen - 650MPa, 162025 cycles, ratchet marks indicating multiple crack initiation sites. b) As machined specimen - 550MPa, 1014287 cycles, large bright facet area.

Figure 4-6 - a) Deep cold rolled specimen - 700MPa at 400°C, 84190 cycles. b) Deep cold rolled specimen – 650MPa 400°C, 129983 cycles. Concentric rings increased with number of cycles.

Figure 4-7 - a) Shot peened specimen - 700MPa at 400°C, 50940 cycles. b) Shot peened specimen – 550MPa 400°C, 10474349 cycles. Concentric rings increased with number of cycles.

4.2 Discussion

Considering the stress-life data collected from the study as presented in Figure 4-4, fatigue life improvements were found to be insignificant at LCF regime ($<10^5$ cycles). Such observations are consistent with the findings by Prevey and Klotz [11,69] where the fatigue life of as machined sample is in the same order of shot peened specimen at LCF regime. The benefits of residual stress and cold work from surface treatments usually supersede any detrimental effects on initiation life from surface roughening, especially in propagation dominated fatigue fracture mechanism where crack initiation due to surface roughness is relatively insignificant [77]. Nonetheless, it is still the best practice to keep the surface roughness as low as possible because the role of surface roughness, residual stress and cold work might varies depending on the nature of the component operation[122].

From the fatigue test, no run-out was observed as amongst the as-machined specimens. The effects of surface treatment become more pronounced at lower applied stress level. Tested at the same stress levels, deep cold rolled specimens exhibited a run-out specimen at 600MPa suggesting fatigue life improvements. Trendlines were superimposed on the stress-life plot to illustrate fatigue lives under different stress levels. Further tests coupled with staircase method can be conducted in the future to quantify the fatigue life improvement through the endurance limit.

The absence of multiple crack initiation in the surface treated specimens can be linked to the presence of work hardened near surface layer. When multiple crack initiations are present, different cracks propagate at different planes before coalescing. The convergence of these cracks often accelerate failure resulting in life debit [105]. In this study, the propensity for multiple cracks initiation was reduced as a consequence of the work hardened layer that is often associated with high dislocation densities and increased yield strength. High dislocation density provides the resistance for further plastic deformation attributed to dislocation

motion. As a result, only a single dominant crack could propagate to failure. The enhancement in fatigue life is still prominent even though all surface treated specimens exhibited surface crack initiation site. This could suggest that the role of residual stress and cold work is best played in the crack propagation regime by crack growth retardation. On the other hand, the role of surface roughness in this study is consistent with findings published by Klotz et al [11] where the role of surface roughness in fatigue is significant only when the comparison is done within the same surface treatment.

Residual stress profiles obtained from shot peened and deep cold rolled surfaces vary in terms of their values at the free surface, peak and depth of compressive stresses. The role of surface compressive residual stress was determined to be insignificant because all initiating cracks were found at the free surface. The peak as well as depth of these compressive residual stresses were, on the other hand, play significant role as observed from the fatigue test. This is consistent with the results reported in the literature (by Nalla et al, Mutoh et al. and Cammett et al. [50,59,123]) where reduced crack propagation rate is observed up to the depth corresponding to the maximum compressive residual stress. Greater depth of compressive residual stress also allows a more consistent approximation of compressive mean stress leading to evaluation of longer fatigue life in conventional fatigue approaches, such as Gerber, Goodman and Morrow stress life approaches[6].

The lack of improvements in fatigue life at low cycle regime can be attributed to the rapid residual stress relaxation due associated with high applied stresses, causing local plastic yielding [77,84]. While stress relaxation can happen under compressive or tensile load, compressive loading requires a lower applied stress for relaxation as a result of Bauschinger effect and superposition with the residual compressive residual stress [79,84,124]. The near yield strength compressive residual stress of deep cold rolled specimen could be a concern if high compressive loading is used as greater relaxation could occur due to the Bauschinger effect.

Concentric rings appear when bursts of fast fracture alternate with fatigue cycling. The observation of concentric rings on surface treated specimen could suggest the crack growth retardation at irregular interval. The number of concentric rings was observed to increase with fatigue life (see Figure 4-6 and Figure 4-7), indicating crack growth retardation sites. It could be seen that at the region of concentric rings (see Figure 4-8), a less defined and larger striation pattern was observed at the fast fracture area when compared to the area of steady crack propagation [106].

The observation of concentric rings could be explained from the perspective of work hardening. Generally, plastic zone developed at the crack tip blunts the crack resulting in a steady crack propagation. However, the cold rolled and shot peened surfaces exhibit a decreasing amount of work hardening (and therefore yield strength) with distance from the free surface. This increase in yield strength is accompanied by the loss of ductility reducing maximum allowable strain prior to failure. When the crack front propagates to a localized region where the plasticity is unable to withstand the local stress, fast fracture occurs but is quickly suppressed by region with more ductility. The repetitive occurrences of steady crack propagation followed by fast fracture and suppression of fast fracture resulted in the concentric rings. In the absence of varying local yield strength associated with the peening processes, steady crack propagation develops until rapid failure as observed in as-machined specimens (see Figure 4-5).

Figure 4-8 - Striations images of shot peened specimen – 550MPa 400 °C, 10474349 cycles.

At high temperatures, low amount of cold work is desirable for compressive residual stresses retention (i.e. reduced thermal stress relaxation) [79,125,126]. large amount of cold work promotes stress relaxation through thermally activated processes such as dislocation annihilation due to the high level of stored energy [61,64]. The compressive residual stress of deep cold rolled specimen relaxed to a lesser extent compared to shot peened specimens due to the variation in cold work. Long fatigue lives exhibited by deep cold rolled specimens relative to that of shot peened can also be attributed to the retention of compressive residual stresses associated with low level of cold work.

In contrast with the well-defined development in stress measurement, cold work or strain hardening quantification appears to be less developed and still has room to improve. The main concern that needs to be addressed in cold work quantification is the lack of agreement and consistency across different methods [77]. Another issue to address due to the lack of consistency in results between methods is the decision as to which method is the most appropriate. As a matter of fact, none of the methods mentioned here has enough consistency and reliability to characterize cold work independently. The reason could be mainly due to the difficulty in

comparing or verifying the values obtained as each of these methods adopts different units in data collection. It is best that the scientific community could come up with a more standardized measurement system to allow expression of the effects of cold work in fatigue investigation to be free of doubt.

Chapter 5 Investigation of Mechanical Relaxation of Shot Peened and Laser Peened IN718 Using Neutron Diffraction[#]

This chapter describes the investigation of mechanical relaxation conducted on surface treated Inconel 718 specimens. The neutron diffraction work was conducted at Australian Centre for Neutron Scattering, Australia Nuclear Science and Technology Organisation. The materials and methods adopted for the is described in Chapter 3. Shot peening and laser peening were employed to modify the surface and subsurface of Inconel 718. The residual stress and work hardening were characterized using neutron diffraction method and electron backscattered diffraction (EBSD) respectively. A modified set up in reflective mode was utilized during in-situ neutron diffraction to optimize the spatial and temporal resolution for residual stress measurements at pre-determined cycle. The modified set up demonstrated the capability to resolve the steep residual gradient of surface treated specimen throughout the specimen's thickness. Laser peened specimen recorded a very high tensile residual stress in the bulk of the specimen. Residual stress relaxation was only observed in the direction of loading while the residual stress in the transverse direction remained at a similar magnitude. Residual stress relaxation was observed to be most prominent in the first cycle of fatigue at R ratio = 0.1 with little stress relaxation in subsequent fatigue load cycles. Stress relaxation occurs when the superposition of tensile residual stress and applied loading exceeds the localized yield strength of the material. Stress relaxation was found to be well correlated with the magnitude of work hardening. Residual stress relaxation as a function of depth and number of cycles were also recorded to illustrate the changes in residual stress during the cyclic loading.

[#] partial content of this chapter is published as "Mechanical stress relaxation of a laser peened and shot peened Ni-based superalloy" in the INCASE 2019, LNME Proceedings (2019).

5.1 Results

a) Work hardening variations

GOS is a parameter in EBSD defined by the average deviation in orientation between each point in a grain and the average orientation of the grain. It is used in this study as an indication of work hardening as it could be correlated to dislocation density. When dislocations are produced in the material, the residual or plastic strain is manifested as local variations in lattice orientation [55]. In material subjected to work hardening or work hardening, local orientation differences are observed within deformed grains where individual grains with zero strain will largely display a constant orientation.

Laser peened specimen possessed higher value GOS that are still scattered to around 2° even at the depth of $500\mu\text{m}$, see Figure 5-1. For shot peened specimen, depth of work hardening is only evident up to $50\mu\text{m}$. However, from the surface to $50\mu\text{m}$ depth, shot peened specimen recorded a higher average value of GOS at 0.80° as opposed to 0.44° possessed by laser peened specimen.

The distinct difference in the GOS maps and plots illustrate the characteristics of work hardening experienced by shot peened and laser peened specimens. In shot peened specimen, the repeated impacts by high volume of shots generates a great amount of dislocation but is only limited to the near surface ($50\mu\text{m}$). In laser peened specimen, the considerably lesser number of impacts by plasma shockwave induces a lower GOS value near surface. However, the high value of GOS observed at $500\mu\text{m}$ is indicative of the extent of work hardening induced by the generation of plasma shockwave.

Figure 5-1 - GOS graph and illustration, a) laser peening, b) shot peening.

b) Residual stress profiles and relaxation

The ex-situ residual stress measurements were performed after the determination of d_0 . The capability of the neutron diffraction method to resolve the steep residual stress gradient was evaluated. The residual stress states after surface treatment are shown in Figure 5-2, the magnitudes of residual stresses in laser peened specimen are relatively high compared to shot peened specimen (-700 vs -400 MPa for peak compressive and 400 vs 50 for peak tensile). The compressive residual stress layer in laser peened specimen extends to a depth of 1mm, approximately double that of shot peened specimen. After 10,000 cycles of fatigue, relaxation of residual stresses was only observed in the loading direction (transverse), see Figure 5-3.

At the 50 μ m near surface, laser peened specimen experienced lesser stress relaxation of around 100MPa when compared to 200MPa in shot peened specimen. Significant stress relaxation was observed to occur at shot peened specimen up to depth of 800 μ m in contrast to laser peened specimen with relaxation only up to 400 μ m.

Based on the observation from the ex-situ measurement, the initial residual stresses of laser peened and shot peened specimens are homogeneous and significant relaxation is only observed in the direction of the loading. Therefore, the in-situ residual stress measurement was conducted in the direction of loading only to reduce the collection time. For the in-situ measurement, stress relaxation was significant but is only limited to the first cycle for both shot peened and laser peened specimens, see Figure 5-4. Further cycling observed insignificant stress relaxation, see Figure 5-5.

Figure 5-2 - Ex-situ residual stress profiles, a) - laser peening, b) - shot peening.

Figure 5-3 - Ex-situ residual stress profiles at 10000 cycles, a) - laser peening, b) - shot peening.

Figure 5-4 - In-situ residual stress profile at 0th and 1st cycle, a) - laser peening, b) - shot peening

Figure 5-5 - In-situ residual stress profile at 1st, 10th, 10000th, and 50000th cycles, a) - laser peening, b) - shot peening.

5.2 Discussion

The ability of neutron diffraction in resolving the steep residual stress gradient and tracking of residual stress redistribution was demonstrated. The ex-situ measurements were necessary for the determination of d_0 spacing and verification of stress relaxation behaviour. The calculation of residual stress using the d_0 spacing obtained is included in the Appendix B. After that, the in-situ measurements adopting the modified reflective set up is shown to be adequate to illustrate the stress relaxation of surface treated specimens at positive R ratio loading. Such measurement strategy combined with the non-destructive nature of neutron diffraction provides a higher confidence in the data analysis, eliminating the error and discrepancy that could be caused by the specimen, operator or environmental variation.

Residual stress relaxation was observed at positive R ratio from both ex-situ and in-situ experiments. In contrast to stress relaxation described by Bauschinger effect (often observed in negative R ratio fatigue cycling due to reduction of compressive yield strength caused by tensile work hardening), the stress relaxation in this study could be attributed to the superposition of tensile applied and residual stresses resulting in local plastic yielding in tension. Also, the mechanical stress relaxation can be explained by necessity for reducing misfits between the deformed layer (region affected by work hardening) and elastic bulk (region unaffected by work hardening) [76]. Significant stress relaxation was observed along the direction of loading only. Correlation was observed between work hardening and residual stress relaxation.

In spite of the greater depth and magnitude of compressive residual stresses, laser peened specimens retained compressive residual stresses better than shot peened specimens. It was suggested that stress relaxation correlates better with the magnitude rather than depth of work hardening. The magnitude of work hardening is higher in shot peened specimen as observed from the high value of GOS on the

surface (0 μ m depth) while laser peened recorded a greater depth of work hardening. The stress relaxation behaviour corresponds better with the magnitude of work hardening where the stress relaxation in shot peened specimens was more significant when compared to laser peened specimen.

Also, higher strain misfit is observed between the work hardened layer and the bulk layer (unaffected by work hardening) of shot peened specimen. Shot peened specimen has a thin (only 50 μ m) but highly work hardened layer when compared laser peened specimen. This large misfit which occurs over a narrow plastically deformed layer will increase the propensity for mechanical stress relaxation. As a result of the large misfit, the highly work hardened layer and the elastic bulk will undergo different magnitude of yielding [76]. Thus, the elastic bulk layer will be affected and result in a greater stress relaxation even at a depth where the effect of the work hardening is not observed.

The high tensile residual stress observed in laser peened specimen could be attributed to the necessity to balance the compressive residual stress in a relatively thin specimen (4mm). The significant depth of work hardening and compressive residual stress in laser peened specimen leave only a limited region in the bulk for the specimen to achieve equilibrium in terms of residual stress. The detrimental effects that could be associated by the high tensile residual stress observed in laser peened specimen should be considered for further testing. From the author's knowledge, such high tensile residual stress has not been highlighted in the research of surface treatments, ie shot peening, laser peening, deep cold rolling. Therefore, further fatigue test could be performed to determine the influence of such high tensile residual stress present in the bulk of a specimen (please refer to Appendix for preliminary data presentation). It would be compelling to determine if either the compressive residual stress, tensile residual stress or work hardening would be the predominant factor in fatigue performance.

Chapter 6 Thermal Relaxation of Shot Peened and Laser Peened IN718[#]

This chapter describes the investigation of thermal relaxation conducted on surface treated Inconel 718 specimen. The materials and methods adopted for the characterisation work is described in section 3.5. Shot peening and laser peening were employed to modify the surface and subsurface of Inconel 718. The shot peened and laser peened specimens were exposed at different temperature and duration of exposure. Stress measurement using X-ray diffraction was then conducted on the surface and subsurface layer. FWHM values obtained concurrently during X-ray diffraction were converted into cold work percentage. Shot peening with higher level of work hardening observed greater magnitude of stress relaxation. A correlation between stress relaxation and work hardening was observed. The correlation was quantified and being utilized in further modelling effort to describe the thermal stress relaxation. A parameter describing the dependency of relaxation on cold work is introduced into a modified Zener-Wert-Avrami function. Despite having slight discrepancy at higher temperature, the predicted residual stress profiles using the modified Zener-Wert-Avrami function showed good agreement with experimental data.

[#] the content is under review (1st revision) as “Fatigue of Surface Treated Nickel Based Superalloy at High Temperature” for publication in Journal of Materials Science & Technology.

6.1 Results

a) Residual stress profiles

Shot peened specimen recorded a higher maximum compressive residual stress at approximately -1000MPa at approximately 70 μ m depth whereas laser peened specimen's maximum compressive residual stress was at -800MPa, see Figure 6-1. Both laser peened and shot peened specimen possessed surface residual stress at similar magnitude at -800MPa and -700MPa respectively. However, laser peened specimen possessed a greater depth of compressive residual stress with magnitude around -700MPa even at a depth of 600 μ m. Upon the depth of maximum compressive residual stress at 100 μ m depth, the compressive residual stress of shot peened specimen decreased steeply to -200MPa at 200 μ m depth.

Figure 6-1 - Residual stress profiles of shot peened and laser peened specimens

Under thermal exposure, shot peened specimens consistently exhibit more stress relaxation than laser peened specimens at different temperatures. The relaxation was most obvious on the free surface for both shot peened and laser peened specimen where the highest magnitude of work hardening was observed. At 600°C, almost 45% of the compressive residual stress on the surface of shot peened specimen was relaxed even only after 1 hour of exposure, see Figure 6-2. Significant relaxation of compressive residual stress was still observed after extended exposure up to 10 hours at 600°C. The maximum compressive residual stress of shot peened specimen was also reduced from -1000MPa to 700MPa after 10 hours of exposure at 600°C. A shift in the depth of the maximum compressive residual stress from 80μm to 100μm was also observed as a result of uneven stress relaxation. For laser peened specimen, only 10% and 20% of stress relaxation was observed on the surface after 1 hour and 10 hours of exposure at 600°C respectively. The subsurface residual stress (<0μm depth) only experienced minimal stress relaxation.

Figure 6-2 - Stress profiles of shot peened and laser peened specimens at 600°C.

At lower temperature of 400°C and 250°C, the magnitude of relaxation of shot peened specimen was lesser than of 600°C even for longer period of exposure such as 10 hours or 20 hours, see Figure 6-3. After 10 hours exposure at 250°C, the maximum compressive residual stress at around -1000MPa was still retained. However, the maximum compressive residual stress was reduced to -900MPa after further exposure of 20 hours at 250°C. Also, the compressive residual stress on the surface of shot peened specimen was relaxed by 10% and 20% after 10 hours and 20 hours exposure at 250°C respectively. For laser peened specimen, only noticeable stress relaxation at 20% was observed at 400°C.

Figure 6-3 - Stress profiles of shot peened and laser peened specimens at 400°C and 250°C

b) Work hardening profiles and variation

The FWHM profiles shows that shot peened specimen possessed higher value on the surface at 5.5° when compared to laser peening at around 4.5° , see Figure 6-4. The FWHM value of shot peened specimen reduced steeply to 3° at approximately $200\mu\text{m}$ whereas the FWHM value of laser peened specimen remained at 3.5° at $500\mu\text{m}$. GOS maps obtained by EBSD with a dimension of $800\mu\text{m} \times 200\mu\text{m}$ also illustrate the magnitude of work hardening in shot peened and laser peened specimens, see Figure 6-5. From the GOS maps, it could also be observed that shot peened specimen possessed a higher magnitude of work hardening from the surface to approximately $50\mu\text{m}$ depth when compared to laser peened specimen.

Figure 6-4 - FWHM graph, a) shot peening, b) laser peening

Figure 6-5 - GOS maps of shot peened and laser peened specimens

Under thermal exposure, the relaxation of the FWHM was the most obvious on the surface for both shot peened and laser peened specimen. The surface FWHM of shot peened specimen was reduced from 5.8° to 4.8° and 4.6° after 1 hour and 10 hours of exposure at 600°C respectively, see Figure 6-6. FWHM of laser peened specimen was relatively stable when compared to shot peened specimen. For laser peened specimen, only significant FWHM relaxation was observed at 600°C exposure. For shot peened specimen, FWHM was relaxed all temperatures except for 250°C . However, the difference in the level of work hardening after thermal exposure is only reflected narrowly in terms of GOS plots and maps. The reasons for such occurrence will be discussed in the Discussion section. Please refer to Appendix for the GOS maps and plots of shot peened and laser peened specimen at other thermal conditions.

Figure 6-6 - Surface FWHM at various temperature and duration of exposure

6.2 Discussion

a) Cold work quantification and level of work hardening

FWHM and GOS have been utilized to assess the level of work hardening. However, FWHM and GOS could only indicate the relative magnitude of work hardening in the material. Misinterpretation could arise from such use of relative quantification. Based on the FWHM and GOS graphs (see Figure 6-4 and Figure 6-5), it is expected to perceive that the level and depth of work hardening in laser peened specimen are greater than shot peening to a significant extent. In contrary, when the FWHM values are converted to cold work percentage, the magnitude of work hardening beyond 100 μ m in laser peened specimen is only approximately 5%. The difference of 0.5° in FWHM between shot peened and laser peened specimen beyond 100 μ m depth is not manifested as great difference in terms of cold work percentage, see Figure 6-7. This is because of the non-linear relationship between the conversion of FWHM to cold work percentage [11,127]. Such magnitude of work hardening (~5%) is relatively insignificant when compared to the ~50% work hardening recorded on the surface of shot peened specimen.

Despite the difference in magnitude between the conversion from FWHM to cold work percentage, FWHM is a more established method to quantify cold work when compared to GOS. The factors that could affect the FWHM values includes instrumental broadening, interaction between crystallite size, strain in the material etc. The effects of these factors on FWHM values are well understood from the research conducted (see Table 2-2), where some researchers have successfully correlated FWHM to cold work percentage to a certain extent [11,51]. To the author's knowledge, a methodology to correlate and quantify the relationship between GOS and cold work percentage has yet to be developed. Despite the fact that GOS could provide information and illustration of the strain experienced by each grain, there are many variables in this method of analysis that could be varied and result in inconsistent conversion to cold work percentage. Examples of the

sources of variance includes hardware (type of detector, detector distance, SEM setting), software (scan step size and bin size, analysis software) and human or operator error (polishing methods, surface finishing)

Figure 6-7 – Conversion of cold work percentage from FWHM of, a) shot peening, b) laser peening

b) Stress relaxation

Shot peened specimen with the highest magnitude of work hardening experienced stress relaxation even at 250°C, which is lower than the thermal recovery temperature suggested by Khadhraoui [89]. It is suggested the high magnitude of work hardening (50% cold work percentage) introduced by shot peening increases the number of defects in the near surface of the material. The high density of dislocation increases the stored energy which could promote the recovery or recrystallization stage. At high temperature, the capability of dislocation movement, the rates of dislocation rearrangement and annihilation increases. As a result of the increase in stored energy and the rates of dislocation movements, the propensity and magnitude of stress relaxation increases. In contrary to shot peened specimen with significant stress relaxation up to 100µm depth, stress relaxation was only noticeable on the surface of laser peened specimen where the highest cold work percentage (~20%) was recorded.

A significant shift in the depth of the maximum compressive residual stress was observed in shot peened specimen. This could be explained by the uneven stress relaxation across depth due to the variation in work hardening. When the duration of the thermal exposure increases, the steep gradient of work hardening causes a great difference in the magnitude of stress relaxation between the highly work hardened layer and less work hardened layer. This difference in the stress relaxation result in the shifting of maximum compressive residual stress to greater depth. The shifting of maximum compressive residual stress was not noticeable in laser peened specimen due to the relatively mild gradient of work hardening in laser peened specimen.

c) Work hardening and stress relaxation correlation

It was postulated that the magnitude of stress relaxation is proportional to the magnitude of work hardening [3,74,127]. The high magnitude of work hardening on the shot peened surface could explain why stress relaxation is observed even at exposure temperature which is lower than the suggested recovery temperature by Khadhraoui [89].

In this study, it was observed that the magnitude of stress relaxation corresponds linearly to the magnitude of work hardening. All the fitted lines describing the relationship between residual stress and cold work percentage possessed R^2 value > 0.85 . The stress relaxation dependency on work hardening increases with the increase of temperature as observed from the increase in the steepness of the gradient in Figure 6-8. On the other hand, an increase in the duration of exposure will shift the slope further below with similar gradient, see Figure 6-9.

Figure 6-8 - Correlation between residual stress retained and cold work percentage at various temperature for shot peened specimens

Figure 6-9 - Correlation between residual stress retained and cold work percentage at various temperature and duration of exposure for shot peened specimens

The large range of cold work percentage of shot peened specimen allows a better representation of the stress relaxation behavior corresponding to the magnitude of work hardening. Similar fitting was attempted on laser peened specimen but poor fitting was obtained. As observed from Figure 6-2 and Figure 6-3, the stress relaxation experienced by laser peened specimen is lower when compared to the shot peened specimen. The lower stress relaxation resulted in the scattering of data points for the fitting. The lower stress relaxation combine with the limited data from the smaller range of cold work percentage is suggested to result in the poor fitting, please refer to Appendix for the graph of the fitting.

c) Modelling of stress relaxation using modified Zener-Wert-Avrami function

To the author's knowledge, such linear correlation (see Figure 6-8 and Figure 6-9), has not been observed from previous research. Previous research mostly highlighted the observation of increased stress relaxation with an increase in cold work without further quantification. Therefore, in this section, the effort to the model stress relaxation incorporating observed linear correlation is presented.

In order to provide a description of the thermal residual stress relaxation, a stress relaxation model based on the Zener-Wert-Avrami function is proposed. A coefficient, C describing the stress relaxation dependency on cold work is introduced. Many researchers have used the Zener-Wert-Avrami method for the determination of the activation enthalpy, ΔH_A to describe the propensity of stress relaxation under the presence of cold work [91,128–130]. The determination of a lower activation enthalpy signifies a greater propensity for stress relaxation. Nonetheless, there is no specific term in Eq. (8) that describes the effects of cold work on stress relaxation. Therefore, this proposed model, Eq. (9) attempts to incorporate the dependency of stress relaxation on cold work.

$$\frac{\sigma^{RS}}{\sigma_0^{RS}} = C e^{-(At)^m}, \quad 393K < T < 1600K \quad (9)$$

$$A = B e^{\left(-\frac{\Delta H_A}{kT}\right)},$$

$$C = [1 - (\alpha)(CW)], \quad 0 < \alpha < 1 \quad (10)$$

where

σ^{RS} = residual stress after thermal exposure

σ_0^{RS} = residual stress before thermal exposure

t = duration of thermal exposure

T = temperature of thermal exposure

ΔH_A = activation enthalpy

k = Boltzmann's constant

A, B, C, α, m = empirical parameters

CW = cold work percentage

First, the term $e^{-(At)^m}$ which describes the residual stress relaxation without the presence of cold work need to be defined. The self diffusion enthalpy of Ni at 2.88eV is being adopted for ΔH_A where the main micro-structural process responsible for the thermal stress relaxation is achieved by diffusion-controlled mechanism [129,131]. The recovery temperature is assumed to be at 400°C as postulated by Khadhraoui et al [99].

Then, from Eq. (8), a graph of $\log \ln (\sigma^{RS}/\sigma_0^{RS})$ versus $\log t$ at a given temperature T gives a straight line with slope m and intercept $m \log A$. The values of m at different depth associated with different value of cold work were determined at 400°C, see Figure 6-10. From the set of values of m , the value of m associated to 0% of cold work could be extrapolated. The value of B associated with 0% cold work could also be obtained by the same extrapolation from the intercept which is equivalent to $m \log A$.

Figure 6-10 - Influence of cold work and exposure time on the thermal relaxation of shot peened IN718 specimen.

Equation (10) illustrate how the effects of cold work is being incorporated into the stress relaxation behaviour. The linear dependency of stress relaxation on cold work percentage at different temperature could be described by the gradient α , see Figure 6-8 and Figure 6-9. The value of α at various temperature could be derived from the plot of gradient of stress relaxation against the temperature. The boundary condition for α is defined by the melting temperature of IN718, 1600K where immediate stress relaxation is assumed. The threshold temperature for the stress relaxation dependency on cold work to take place is determined to be at 393K where the value of the gradient of stress relaxation, $\alpha=0$. The term CW considers the cold work percentage present in the material. When cold work is absent in the material, ie $CW=0$, the terms $[1 - (\alpha)(CW)]$ will give a value of 1 rendering the stress relaxation to be governed only by the term $e^{-(At)^m}$. The parameter α in the terms $[1 - (\alpha)(CW)]$ is normalized to ensure that the term $Ce^{-(At)^m}$ would produce a value of 0 to 1 for a valid description of the residual stress retained, $\frac{\sigma^{RS}}{\sigma_0^{RS}}$.

The proposed model is able to illustrate the stress relaxation of shot peened and laser peened specimens accurately to a certain extent. The experimental data of shot peened and laser peened specimens at 400°C and 250°C show good agreement with the proposed model, see Figure 6-11 and Figure 6-12. However, the model appears to be less accurate at higher temperatures. The model overestimates the relaxation by -200MPa for the maximum compressive residual stress at 10 hours of 600°C exposure for shot peened specimen and -300MPa for laser peened specimen, see Figure 6-13. This observation suggests that the model might not able to reflect the stress relaxation at high temperature represented by the assumed diffusion dominated mechanism only. At higher temperature, the dependency of relaxation behaviour could be dominated by thermal activated dislocations motion as observed by the significant reduction in FWHM at 600°C, see Figure 6-6, which suggests the reduction of dislocation density. Such thermal activated dislocations motion with a different kinetic is not represented by the proposed model. Further experiment could be conducted at various time scale to verify the reliability of the

model at higher temperature. Also, such discrepancy between the experimental data and the model could arise from the extrapolation of the parameters from the term $e^{-(At)^m}$. In order to improve the reliability and accuracy of the model, it would be preferable to obtain the parameters by conducting stress relaxation experiment on specimen without cold work. However, such case might not be practical and theoretically impossible given that significant residual stress is often introduced with considerable amount of cold work.

Figure 6-11 - Comparison between experimental and modelled profile of residual stress for shot peened and laser peened specimen at 400°C.

Figure 6-12 - Comparison between experimental and modelled profile of residual stress for shot peened and laser peened specimen at 250°C.

Figure 6-13 - Comparison between experimental and modelled profile of residual stress for shot peened and laser peened specimen at 600°C.

In this study, the activation enthalpy is defined as the self diffusion enthalpy of Ni. In order to improve the reliability of the model, the activation enthalpy for the specific batch of material should be determined experimentally. As Eq. (10) illustrates, the value of residual stress retained is very much dependent on the activation enthalpy. The possible case of other stress relaxation mechanism such as thermal activation of dislocations combined with diffusion creep should be considered and integrated to the current model.

The model also poses some limitations such as the inability to account for the transformation from compressive residual stress to tensile residual stress. The model can only provide the value on the residual stress retained of the same sign which does not consider the possibility for the compressive residual stress to transform into tensile residual stress during the process of relaxation. During stress relaxation, especially at region under the surface, compressive residual stress with smaller magnitude could transform into tensile residual stress to attain equilibrium within the material. Nonetheless, such magnitude of compressive residual stress often occurs at considerable depth below the surface where their role in fatigue life application is often trivial when compared to the surface compressive residual stress or maximum compressive residual stress.

The proposed model has been able to predict the residual stress relaxation under the influence of cold work to a certain extent. Following research on thermal relaxation could adopt such method based on the current model developed. More experimental data could be collected to reduce the uncertainty in the empirical parameters obtained. Other than that, it would be desirable to further validate the modelling framework under different circumstances, such as longer durations, different temperature, different surface treatment and etc. The current proposed model has only focused on thermal stress relaxation. Future work could be expanded to thermo-mechanical relaxation which is more complex due to the change in material properties such as yield strength and the coupling mechanism between the mechanical and thermal relaxation.

Chapter 7 Conclusions and Future Work

a) Summary and conclusions

Residual stress and work hardening have been commonly utilized to improve the mechanical properties and thus fatigue performance of many critical components, especially in the application of aerospace industry. Nonetheless, the simultaneous nature of the introduction of residual stress and work hardening through surface treatments has raised some uncertainty of their respective roles during fatigue. This study had undertaken this matter by first looking into the characterisation of the material after various surface treatments on Inconel 718. Next, the investigation on the effects of residual stress and work hardening were extended to the stress relaxation phenomena. From this study, the extent of the effects of residual stress and cold work on fatigue had been identified. The dominance of the role of residual stress and cold work under different circumstances had also been clarified. The role of cold work in fatigue has been illustrated in terms of crack initiation. The redistribution of residual stresses under mechanical and thermal relaxation had been investigated. The interaction between cold work and residual stress on Inconel 718 has been assessed and quantified in the study of thermal relaxation. The significant findings and major contributions of the investigations are described below.

i) Materials Characterisation

Surface integrity of a part encompassing its topography, residual stresses and microstructure, plays a significant role in fatigue performance. The influence of the different aspects of surface integrity on the fatigue performance of a Nickel-based superalloy following shot peening and deep cold rolling has been presented. The two processes were found to generate distinct residual stress and cold work distribution where shot peened surfaces were found with high cold work and shallow compressive stressed layer relative to the cold rolled surfaces.

Effects of surface roughness in fatigue lives were not found to be significant as fatigue crack initiation sites were observed at the free surface regardless of the roughness. Stress-life data collected from the study demonstrates the beneficial effects of residual stress and cold work, especially at low applied stresses. Greater depth of residual stress and cold work contributed to fatigue life extension. The absence of multiple crack initiations in surface treated specimen suggests the role of cold work in crack retardation. Observation of concentric ring and absence of multiple crack initiation site suggest that the fatigue fracture mechanism have been altered by the compressive residual stress and cold work. Based on the above observation, such alteration in the fracture mechanism is beneficial as fatigue life enhancement is observed. The low value of cold work in deep cold rolled specimen also reduces the propensity of stress relaxation, leading to a greater enhancement in fatigue life when compared to shot peened specimen.

The characterisation work of surface treatment processes in this study could help in the building of knowledge for selection of appropriate surface treatment. Fatigue is very complex phenomenon and the authors acknowledge limitations in developing precise relationship between residual stresses and cold work in determining fatigue performance. This can be further complicated by exposure to high temperatures at which the alloy is designed to operate. Therefore, further studies focusing on thermally induced stress relaxation can be carried out through in-situ experiments.

ii) Mechanical relaxation

A modified reflective mode setup was used for the neutron diffraction measurement to achieve a balance between spatial and temporal resolution. The deep penetration ability and non-destructive nature of neutron diffraction allow the acquisition of stress relaxation data from one specimen, increasing the confidence in the data acquired. The use of single specimen eliminates the random artefacts and inconsistencies due to the use of multiple coupons and relatively small probing area by other conventional methods.

Stress relaxation was most significant in the first cycle and limited to the direction of the loading. Since Bauschinger effect is insignificant under positive R ratio, stress relaxation under tensile-tensile loading can be attributed to the superposition of applied tensile loading and tensile residual stress.

From the EBSD characterisation, it was observed that laser peening and shot peening induced different magnitude and depth of work hardening. The difference in the work hardening affected the stress relaxation behaviour. Shot peening specimens exhibited more stress relaxation than laser shock peened specimens. The stress relaxation was most significant in the first cycle. It is also postulated that the magnitude of work hardening plays a more significant role in the stress relaxation of mechanical loading rather than the depth of work hardening. It is also suggested that more stress relaxation will occur when the difference in the misfit between the deformed layer and the bulk is larger.

iii) Thermal relaxation

The FWHM values of shot peened and laser peened specimens were converted to cold work percentage. Shot peened specimen possessed near to 50% of cold work percentage on the surface as compared to ~25% observed on the surface of laser peened specimen. The 0.5° difference in FWHM at the bulk of the material between the laser peened and shot peened only manifested a difference less than 5% in terms of cold work percentage. Shot peened specimen with greater magnitude of work hardening observed greater extent of stress relaxation generally when compared to laser peened specimen. Shot peened specimen even observed stress relaxation at a lower temperature of 250°C. On the other hand, the FWHM values were relatively stable when exposed to elevated temperature. Significant relaxation was only observed at 600°C for shot peened and laser peened specimens.

A linear dependency between the cold work percentage and residual stress retained was observed. At higher temperature, the dependency of stress relaxation on the magnitude of cold work increases. The linear dependency between thermal stress

relaxation and cold work percentage was being utilized to describe the stress relaxation behaviour under the presence of cold work.

A modified Zener-Wert-Avrami function has been proposed and a coefficient describing the dependency of stress relaxation on cold work has been established. The coefficient is derived from the linear dependency of stress relaxation on cold work percentage. Good agreement is observed between the experimental and modelled profile of residual stress, especially at 400°C and 250°C. However, more experimental data could be obtained to improve the reliability of the model. The proposed model has been able to predict the residual stress relaxation under the influence of cold work accurately to a certain extent. Future research on thermal relaxation could adopt such method based on the current model developed.

b) Recommendations and future works

Future work can be expanded to thermo-mechanical loading to investigate how the role of work hardening affects the stress relaxation mechanism. It would be crucial to understand the interaction between work hardening and residual stress under the combined effects of thermal exposure and mechanical loading.

The difference in the kinetics between mechanical and thermal stress relaxation has been highlighted in this investigation. In this study, thermal relaxation has shown a certain degree of time and temperature dependence while mechanical relaxation has been shown to be relatively time independence and is more material properties driven. Thermal relaxation was observed to occur at region where work hardening is present. On the other hand, even though mechanical relaxation could be correlated to the magnitude of work hardening, stress relaxation was also observed at region where work hardening is irrelevant. Apart from that, mechanical relaxation has shown to be directional where relaxation was only observed in the direction of loading while thermal relaxation has shown to be homogeneous.

It is of interest to build on this current understanding where the effects of mechanical and thermal relaxation combined. It would be imperative to determine the extent of the directionality, time and temperature dependence of relaxation under different residual stress and cold work profiles. In-situ experiments employing non-destructive techniques such as neutron and synchrotron diffraction could be employed to capture the transient state of residual stress during thermo-mechanical cycling.

In terms of replicability, further fatigue tests should also consider the need for conducting replicates test to reduce the uncertainty level. Same condition should also be applied on to thermal relaxation when fitting and modelling based on empirical data are employed. More thermal conditions such as the increase in the duration of the exposure and selection of the temperature should be considered to increase the confidence in the empirical parameters obtained. Apart from that, specimens with various shot peening and laser peening parameters with different residual stress and cold work profiles could be used to validate the robustness of the model developed. Further work could also consider further expansion of the model to different surface treatments and material system.

With regards to the microstructure and dislocation analysis, EBSD quantification of work hardening allows only the assessment of macro-scale effects. The evaluation of deformation, stress relaxation mechanism and the role of the precipitate and phases is lacking. Although EBSD can be utilized in the evaluation of crystal rotation and correlation of dislocation density, it could not be used to measure the true dislocation density. As highlighted in a research by Laine et al [10], different dislocation types were observed in shot peened and laser peened specimen. Rather than looking at work hardening level solely and correlating them to the stress relaxation behaviour, stress relaxation mechanism could be better assessed if the inherent difference in dislocation type and behaviour are being investigated. Therefore, transmission electron microscopy (TEM) analysis could be considered for further study to dissect the role of work hardening in stress

relaxation as TEM allows a more detailed evaluation of microstructure and dislocation density (see Appendix for preliminary TEM observation). However, the concern that the scale of the sample measured might not accurately reflect the materials properties should be considered.

TEM analysis could also be conducted to complement the understanding of stress relaxation mechanism from microstructural perspective. TEM observation can also be used in conjunction with the information obtained by EBSD to give a more thorough and comprehensive explanation on the microstructural changes induced by various surface treatments. Different surface treatments have been shown to introduce a particular type of dislocation with distinct characteristics [10]. TEM analysis could be used to inspect the role of dislocation type and the nature of stress relaxation behaviour from microstructural perspective.

Appendix A : Materials characterisation

Figure i - Stresstech Xstress 3000 G3 X-ray Stress Analyser

Figure ii - Centre hole drilling

Category	Centre hole drilling	X-ray diffraction
Spatial resolution (μm)	<p>$\varnothing 5\text{mm}$ Collimator</p> <p>$16 - 128\mu\text{m}$</p> <p>$5\mu\text{m}$</p> <p>$\varnothing 1.8\text{mm}$ Hole diameter</p> <p>Legend</p> <ul style="list-style-type: none"> Surface Sub-surface XRD CHD <p>* Drawing is not to scale</p>	

Figure iii – Comparison between centre hole drilling and X-ray diffraction

Figure iv – MTS fatigue tester at Advanced Remanufacturing and Technology Centre

Figure v – Surface roughness profiles

Appendix B : Mechanical relaxation

Figure vi – Layout of KOWARI beam

Figure vii – Position and levelling calibration of KOWARI beam with INSTRON fatigue tester

Figure viii – Alignment of KOWARI beam with the specimen 1

Figure ix – Alignment of KOWARI beam with the specimen 2

Figure x - Alignment of KOWARI beam with the specimen 3

Figure xi – MTS fatigue tester at Nanyang Technological University

Stress measurement methodology using neutron diffraction, KOWARI

Neutron strain scanning provides the average elastic strain within the gauge volume defined by the intersection of incident and diffracted beams. The elastic strain, ε_{xx} , along a direction 'x', (say) is determined from the change in the lattice spacing, d_{xx} , of the crystalline material referred to the unstressed value, d_0 , thus;

$$\varepsilon_{xx} = \frac{(d_{xx} - d_0)}{d_0} \quad (i)$$

where in this instance d_0 is assumed independent of the direction.

Essentially, in all diffraction investigations, stress calculations are based on continuum mechanics using Hooke's law. The stress is calculated from the elastic strains in the gauge volume measured along three mutually orthogonal directions, i.e.

$$\sigma_{xx} = \frac{E}{(1 + \nu)(1 - 2\nu)} [(1 - \nu)\varepsilon_{xx} + \nu(\varepsilon_{yy} + \varepsilon_{zz})] \quad (ii)$$

$$\sigma_{yy} = \frac{E}{(1 + \nu)(1 - 2\nu)} [(1 - \nu)\varepsilon_{yy} + \nu(\varepsilon_{xx} + \varepsilon_{zz})] \quad (iii)$$

$$\sigma_{zz} = \frac{E}{(1 + \nu)(1 - 2\nu)} [(1 - \nu)\varepsilon_{zz} + \nu(\varepsilon_{xx} + \varepsilon_{yy})] \quad (iv)$$

where E is the Young's modulus and ν is the Poisson ratio of the material.

All lattice parameters were calculated using in-house software QKOWARI in which single peaks are fitted over a user-defined range.

Furthermore, one may want to look at hydrostatic stresses which can be calculated from Equation (v), and calculate effective Von Mises stresses (Equation vi), which are independent of the d_0 because only stress differences are involved.

$$S_H = \frac{1}{3}(S_{xx} + S_{yy} + S_{zz}) \quad (v)$$

$$S_M = \frac{1}{\sqrt{2}} \sqrt{(S_{xx} - S_{yy})^2 + (S_{xx} - S_{zz})^2 + (S_{yy} - S_{zz})^2} \quad (vi)$$

DATA ANALYSIS, with no reference value

The following outline the method for determining stresses in three orthogonal directions from neutron diffraction measurements where no reference sample is available. This is done via assuming one of the directions has zero stress (plane stress). This method is applicable to situations where one of the sample directions is thin and as such has little or no constraint and thus the stresses in that direction can be considered negligible. An example of this situation is in welds involving thin plates where the normal direction can be considered to be stress free.

As with previously we have our three orthogonal strain measurements:

$$\varepsilon_{xx} = \frac{(d_{xx} - d_0)}{d_0} \quad (vii)$$

$$\varepsilon_{yy} = \frac{(d_{yy} - d_0)}{d_0} \quad (viii)$$

$$\varepsilon_{zz} = \frac{(d_{zz} - d_0)}{d_0} \quad (ix)$$

In the following we assume ε_{xx} is the normal direction and applying our assumption to Hooke's law we obtain the following:

$$0 = \frac{E}{(1 + \nu)(1 - 2\nu)} [(1 - \nu)\varepsilon_{xx} + \nu(\varepsilon_{yy} + \varepsilon_{zz})] \quad (x)$$

Thus we eliminate the term in front of the brackets by dividing giving:

$$0 = [(1 - \nu)\varepsilon_{xx} + \nu(\varepsilon_{yy} + \varepsilon_{zz})] \quad (\text{x i})$$

Substituting in equations 1 – 3 gives:

$$0 = [(1 - \nu)\frac{(d_{xx} - d_0)}{d_0} + \nu(\frac{(d_{yy} - d_0)}{d_0} + \frac{(d_{zz} - d_0)}{d_0})] \quad (\text{x ii})$$

Expanding all of the terms and multiplying by d_0 gives:

$$0 = (1 - \nu)d_{xx} - (1 - \nu)d_0 + \nu.d_{yy} - \nu.d_0 + \nu.d_{zz} - \nu.d_0 \quad (\text{x iii})$$

Grouping like terms on either side of the equation and dividing by the factor for d_0 (and cleaning up the algebra) gives:

$$d_0 = \frac{(1 - \nu)d_{xx} + \nu(d_{yy} + d_{zz})}{(1 + \nu)} \quad (\text{x iv})$$

Thus, our reference lattice spacing can be determined via the three orthogonal measured lattice spacings.

Uncertainty in neutron diffraction results

The experimental uncertainties presented in this report are derived using an error propagation method. This approach calculates the effect of uncertainties in the measured quantities on the calculated stress; the stressed and un-stressed lattice spacing, (Δd and Δd_0). There are a number of possible contributions to Δd and Δd_0 . The most easily quantified is the error due to the uncertainty in the peak position. This source of uncertainty is dependent on the counting time of the measurement and is present in all lattice spacing measurements. Other sources of experimental uncertainty may also be present, including thermal, grain size and texture effects.

The error propagation method was used in the calculation of the strains and stresses. The experimental uncertainties presented in this report are derived using an error propagation method of the form:

$$\Delta z = \sqrt{\left(\frac{\partial z}{\partial x} \Delta x\right)^2 + \left(\frac{\partial z}{\partial y} \Delta y\right)^2} \quad (\text{xv})$$

where z is a function of x and y ; and Δx and Δy are the uncertainties in x and y . The uncertainty in a strain component ε_{xx} (say) is therefore

$$De_{xx} = \sqrt{\left(\frac{\partial e_{xx}}{\partial d_{xx}} Dd_{xx}\right)^2 + \left(\frac{\partial e_{xx}}{\partial d_0} Dd_0\right)^2} \quad (\text{xvi})$$

which, by Equation (1) may be written;

$$De_{xx} = \frac{d_{xx}}{d_0} \sqrt{\left(\frac{Dd_{xx}}{d_{xx}}\right)^2 + \left(\frac{Dd_0}{d_0}\right)^2} \quad (\text{xvii})$$

The uncertainty in a stress component σ_{xx} (say) is similarly derived, i.e.

$$\Delta \sigma_{xx} = \sqrt{\left(\frac{\partial \sigma_{xx}}{\partial \varepsilon_{xx}} \Delta \varepsilon_{xx}\right)^2 + \left(\frac{\partial \sigma_{xx}}{\partial \varepsilon_{yy}} \Delta \varepsilon_{yy}\right)^2 + \left(\frac{\partial \sigma_{xx}}{\partial \varepsilon_{zz}} \Delta \varepsilon_{zz}\right)^2} \quad (\text{xviii})$$

which, by Equation (2) gives;

$$\Delta \sigma_{xx} = C \sqrt{(1-\nu)^2 \Delta^2 \varepsilon_{xx} + \nu^2 \Delta^2 \varepsilon_{yy} + \nu^2 \Delta^2 \varepsilon_{zz}} \quad (\text{xix})$$

where the constant $C=E/[(1+\nu)(1-2\nu)]$.

Equations (xv) to (xix) relate the uncertainty in the strain, stress and hydrostatic stress calculations to the uncertainty in the original stressed and un-stressed lattice parameter spacings, Δd and Δd_0 , respectively.

Fatigue Test

Fatigue test conducted at 900MPa at R ratio = 0.1 (tensile-tensile) revealed that shot peened specimens possessed the longest fatigue life at an average of 185196 cycles, see Figure xii. It was observed that the shot peening doubled the fatigue life when compared to the as machined specimen which averaged at 118734 cycles. Laser peened specimens only recorded similar fatigue life to as machined specimens averaging at 93570 cycles. Also, it could be observed that the laser peening and shot peening introduced a large scatter to the fatigue life. When compared to the fatigue life scatter of the as machined specimens at 17327 cycles, the scatter in the fatigue life of shot peened and laser peened specimens were higher at a value of 27631 and 28474 cycles respectively.

Figure xii - Fatigue performance of as machined, laser peened and shot peened specimen at 900MPa

Fractography

From the fractography, the as machined specimen observed multiple crack initiation sites. The observation of ratchet mark which only occurs when multiple crack planes coalesce as one confirms that multiple crack initiation occurred, see Figure xiii. Two crack sites can be observed from the top of the fractured surface where the ratchet mark lies in between the middle of both crack sites. From the SEM image, it could be observed that the crack initiated from the surface around the edge of the specimen with the observation of slip bands around the crack site.

Another secondary crack was also observed around 2mm from the fractured surface, see Figure xiv. However, the crack was only able to propagate to a length of around 600 μ m as the final fracture did not occur on that plane.

Figure xiii - Crack initiation of as-machined specimen, 103743 cycles, a) optical microscopy image, b) SEM image

Figure xiv – Secondary crack initiation away from the fracture surface of as-machined specimen

For shot peened specimen, only one subsurface crack initiation site was observed, see Figure xv. Instead of surface crack initiation, a subsurface crack initiation site was observed to occur around 50 μ m from the surface and away from the edge of the specimen. A subsurface intergranular crack initiation was perceived to occur with the observation of fine striations originating from the intersection of three grain boundaries.

Figure xv - Crack initiation of shot peened specimen, 221017 cycles, a) optical microscopy image, b) SEM image

For laser peened specimen, a subsurface crack initiation was also observed, see Figure xvi. A subsurface crack initiation site was observed around 40μm from the surface and away from the edge of the specimen. A similar subsurface intergranular crack initiation site was also observed from the laser peened specimen from the evidence of fine striations originating from the intersection of multiple grain boundaries.

Figure xvi - Crack initiation of laser peened specimen, 135530 cycles, a) optical microscopy image, b) SEM image.

It is postulated that the detrimental effects of high tensile residual stress overshadow the beneficial effects of compressive residual stress in fatigue performance. The high tensile residual stress when superimposed with the tensile loading induces local plastic yielding and causes stress relaxation. Therefore, in the occurrence of high tensile residual stress, the role of cold work becomes less dominant in dictating the fatigue performance.

The high degree of scattering in the fatigue life of surface treated specimens suggests that there are several competing fracture mechanisms. Factors such as tensile residual stress, depth and magnitude of compressive residual stress, magnitude and depth of cold work are in mutual competition and will dictate the fatigue performance. Further fractography revealed that as machined specimens experienced multiple crack initiations which coalesce into one dominant crack. The cracks initiated from the free surface via the persistent slip bands induced fracture which is commonly observed from fatigue fracture. The multiple crack initiation sites were not observed in the shot peened and laser peened specimens.

In contrary to the multiple surface crack initiation from the surface of the edge of the as machined specimen, surface treated specimens only observed single subsurface crack initiation. The absence of the multiple crack initiation site and the observation of subsurface crack initiation could be attributed to the work hardening. Shot peening and laser peening introduced work hardening on the top and bottom surfaces which hinder the crack from originating at the edge of the specimen where stress is normally concentrated. Work hardening is a result of plastic deformation which increases the localized yield strength near the surface of the material. As a result of the increased yield strength, crack resistance on the surface is increased which causes subsurface crack initiation to be more favourable.

Despite having subsurface crack initiation, laser peened specimens did not observe enhancement in fatigue performance. This could be due to the high tensile residual stress present in the bulk of the laser peened specimen. Residual stress is often related to the crack propagation rate where compressive residual stress is often linked to the reduction of crack propagation rate [45,106]. However, in laser peened specimen, it is possible that the tensile residual stress could minimize or even eliminate the reduction in crack propagation rate. The effects of tensile residual stress on crack propagation rate combined with the aforementioned stress relaxation caused by tensile residual stress could explain why the fatigue enhancement is not significant in laser peened specimen.

From the fatigue test, only shot peened specimens showed enhancement in fatigue life. Lasers peened specimen despite having greater depth of compressive residual stress and work hardening only recorded a similar fatigue life when compared to as machined specimen. Also, laser peened showed a larger scatter in the fatigue life indicating a higher degree of uncertainty which is not favourable in real life application. It is postulated that the high tensile residual stress has a more dominant effect than the compressive residual stress. First, the high tensile residual stress can induce relaxation through localized plastic yielding. The high tensile residual stress when superposition with the applied loading could exceed the yield strength at a localized region in the material. When a localized yield strength is exceeded, the residual stress in the material will be redistributed. As a result of that, stress relaxation occurs and the compressive residual stress is decreased.

From the fractography, the use of laser peening and shot peening reduced the crack initiation sites. As machined specimen without work hardening and residual stress experienced multiple crack initiation and secondary crack was observed. Subsurface crack initiation observed in laser peened and shot peened specimens could be attributed to the work hardening. The plastic deformation by work hardening increases the yield strength on the surface and near surface of the material which makes subsurface crack initiation more favorable. Even though subsurface crack initiation was observed for both laser peened and shot peened specimens, only shot peened specimens showed enhancement in fatigue life. It is suggested that the high tensile residual stress in laser specimen eliminates the beneficial effects of compressive residual stress in crack propagation rate. From this observation, the introduction of a great depth of compressive residual stress at the expense of high tensile residual stress should be avoided as it brings detrimental effects on fatigue performance. Under the presence of high tensile residual stress, the role of cold work in dictating the fatigue performance is less dominant even though correlation has been made to stress relaxation.

Appendix C : Thermal relaxation

Figure xvii – Conversion of cold work percentage from FWHM for Inconel 718 heat treated according to AMS 5663

Figure xviii – EBSD map for laser peened specimen at various duration and temperature of exposure

Figure xix – EBSD plot for shot peened specimen at various duration and temperature of exposure

Figure xx – Poor fitting for laser peened specimen in ZWA fitting

Appendix D : Preliminary TEM investigation

Figure xxi – Focused ion beam milling for TEM lamella preparation

Figure xxii – Dislocation tangles and dislocation segregation at grain boundary

Figure xxiii – Dislocation tangles observed on shot peened specimen

Figure xxiv – Dislocation lines and slip bands observed on laser peened specimen

Figure xxv – Dislocation observed at the same region from different zone axis

Figure xxvi – Observations of precipitates and dislocation tangles

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