I, Vibhor Chaswal, hereby submit this original work as part of the requirements for the degree of Doctor of Philosophy in Materials Science.

It is entitled:
A study of Laser Shock Peening on Fatigue behavior of IN718Plus Superalloy: Simulations and Experiments

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This work and its defense approved by:

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A study of Laser Shock Peening on Fatigue behavior of IN718Plus Superalloy: Simulations and Experiments

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by

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Abstract

Laser shock peening (LSP) for improving fatigue life of IN718Plus superalloy is investigated. Fatigue geometry and LSP parameters were optimized using finite element method (FEM). Residual stress distributions estimated by FEM were validated using Synchrotron XRD and line focus lab XRD, and correlated with microhardness. An eigenstrain analysis of LSP induced edge deflections (measured with optical interferometry) was also conducted. Transmission electron microscopy (TEM) of single-spot LSP coupons shows sudden increase in dislocation density under LSP treated region.

Total life fatigue was conducted at R=0.1 at 298K and 923K, with and without LSP. S-N curve endurance limit increases at both temperatures with FEM optimized LSP samples. Based on TEM of fatigue microstructure and LSP coupons, mechanistic description of observed fatigue improvement is attempted.

Often need arises to weld components, and weld heat-affected-zone reaches near-solvus temperatures. To simulate this treatment, sub-solvus hot-rolled IN718Plus is aged at 923K. We observe precipitation of thin $\eta$-Ni$_3$(Al, Ti) plates after 1000 hours, making the material susceptible to cracks, and lowering fatigue life.
Effect of LSP on fatigue crack growth (FCG) is studied following ASTM guidelines on M(T) geometry at R=0.1. Acceleration in FCG rate with LSP is observed for this geometry and LSP condition. Prior FEM optimization was not conducted for FCG tests, and may account for lower FCG resistance after LSP. FCG results were corroborated with COD compliance based analysis. Crack measurements were done using potential drop method, and crack closure was analyzed.

Effect of LSP on overload FCG was investigated by single-cycle 100% overload followed by single-spot LSP on the crack-tip plastic zone. Crack retardation occurs after application of overload+LSP. Effective contribution of overload+LSP to crack retardation is estimated.

Fractographic analysis of LSP treated fatigue samples suggests sub-surface crack nucleation, and is analyzed based on stress concentration behavior of small cracks.
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Table Caption

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5.1 Chemical compositions of IN718Plus alloy used in our tests, reprinted from manufacturer’s online product specifications [courtesy: http://www.atimetals.com/products/718plus-alloy]

6.1 Effect of crack tip LSP on FCG rates
## Abbreviations

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tr>
<td>γ'</td>
<td>Gamma prime: $\text{Ni}<em>3\text{Al} \ L</em>{12}$ crystal structure</td>
</tr>
<tr>
<td>γ''</td>
<td>Gamma double prime: $\text{Ni}_3\text{Nb} \ D_0^{22}$ crystal structure</td>
</tr>
<tr>
<td>δ</td>
<td>Delta phase: $D_0^a$ crystal structure</td>
</tr>
<tr>
<td>η</td>
<td>Eta phase: $\text{Ni}_3(\text{Ti, Al, Nb}) \ hcp \ D_0^{24}$ crystal structure</td>
</tr>
<tr>
<td>ν</td>
<td>Poisson's ratio</td>
</tr>
<tr>
<td>σ_y</td>
<td>Yield stress</td>
</tr>
<tr>
<td>ΔK</td>
<td>Stress intensity Factor range</td>
</tr>
<tr>
<td>3PB</td>
<td>3 Point Bend</td>
</tr>
<tr>
<td>ACPD</td>
<td>Alternating current potential drop</td>
</tr>
<tr>
<td>AFGROW</td>
<td>Air Force Fatigue Crack Growth simulation program</td>
</tr>
<tr>
<td>APS</td>
<td>Advanced Photon Source at Argonne National Lab</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing of Materials</td>
</tr>
<tr>
<td>EBSD</td>
<td>Electron back scatter diffraction</td>
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<tr>
<td>FCG</td>
<td>Fatigue crack growth</td>
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<tr>
<td>FEM</td>
<td>Finite element method</td>
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<tr>
<td>HEL</td>
<td>Hugoniot elastic limit</td>
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<tr>
<td>Hypermesh</td>
<td>FEM software used for mesh generation</td>
</tr>
<tr>
<td>IN718</td>
<td>a γ’ strengthened Ni-based superalloy</td>
</tr>
<tr>
<td>IN718Plus</td>
<td>a γ’ strengthened Ni-based superalloy</td>
</tr>
<tr>
<td>LSDYNA</td>
<td>FEM software used for dynamic impact analysis</td>
</tr>
<tr>
<td>LSP</td>
<td>Laser shock peening</td>
</tr>
<tr>
<td>LXRD</td>
<td>Laboratory XRD machine for residual stress</td>
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<tr>
<td>SEM</td>
<td>Scanning electron microscopy</td>
</tr>
<tr>
<td>SXRD</td>
<td>Synchrotron XRD</td>
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<tr>
<td>TEM</td>
<td>Transmission electron microscopy</td>
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<tr>
<td>TTT</td>
<td>Time temperature transformation</td>
</tr>
<tr>
<td>XRD</td>
<td>Conventional XRD</td>
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<tr>
<td>WCR</td>
<td>Water confinement regime</td>
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Chapter 1. Introduction

This work addresses two main aspects of value to society.

A. It builds confidence in a new technique called laser shock peening (LSP) for improving fatigue life of components subject to cyclic loads. LSP has shown promise of greater improvement in fatigue life compared to similar conventional technique of shot peening, with greater control and programmability. B. It builds confidence in a new high temperature superalloy called IN718Plus designed for aero-engine applications. IN718Plus has shown promise of up to 50 °C (122 F) improvement in high temperature capability over similar conventional superalloy IN718, at a comparable cost. Both the aspects above are aimed at economically providing a better material that can last longer under high temperature service and cyclic loads than what is in use today. While the cost of IN718Plus superalloy is comparable to the conventional IN718 alloy, capital cost for LSP treatment is more expensive than conventional shot peening. But the flexibility and control offered by LSP as well as the advantages of deeper residual stresses that give high cycle fatigue life improvements up to 10 times prove it economical in the long run. In a complex and expensive multi-component machine like jet turbine, not all components experience the same stresses. Often there are critical components that are under severe stresses and are more susceptible to failure, limiting the life of the whole assembly. Having the added benefit of being able to extend the life of such critical load bearing component beyond what is possible by current industrial practice of shot peening, makes LSP treatment economical in the overall scheme of things.
LSP being a highly customizable treatment, there are several parameters that need to be finalized before LSP treatment can be applied to a sample. Optimizing these parameters for each geometry, material and treatment is vital towards ensuring that LSP effects are beneficial. We find FEM simulations to be of great help in this regard. Laser time & material can be saved by simulating the stress distributions using FEM. We use LS-DYNA FEM software corroborated with residual stress analysis. Details of FEM simulations on optimizing the LSP procedure to suit of our samples and testing needs are presented in section 6.3.

The primary outcome of LSP of interest to us is the generation of compressive residual stresses. Synchrotron XRD, conventional XRD and line focus XRD were used to determine the residual stresses experimentally, and were corroborated with XRD measurements. We also validate our observations with an eigenstrain analysis based on precise deformations of the sample measured with optical interferometry. This analytical technique can be an inexpensive alternative for industrial validations in absence of X-Ray measurements technique. Eigenstrain analysis is presented in section 6.4.5. The results are in good agreement with XRD measurement of residual stress profile. With our analysis we derive the relative importance of the different shots in a multiple shot LSP sequence.
We found encouraging results in the total life fatigue response of IN718Plus to LSP and show life improvement in the S-N curves endurance limits for both service (923K) and room temperature tests upon optimized LSP treatment. Total life fatigue results are discussed in Chapter 6, section 6.5. TEM investigations of fatigued microstructure as well as LSP-treated coupons, and a discussion of mechanistic description for fatigue life improvement upon application of LSP based on the observations is provided in section 6.5.5.

Often, while using a material we try to extend the life of a damaged component containing cracks detected during routine inspection, by repair welding. Such situations result in the material being subjected to a non-standard heating and cooling cycle. During welding, temperatures in the weld heat affected zone (HAZ) reach close to solvus temperatures. Since, repair welding is common in aircraft engine components; we wanted to check if it was safe to expose our material to such a non-standard heat treatment. Hence, a material that was rolled at near solvus temperatures similar to those that may occur during welding, was aged directly at 923 K, corresponding to service temperatures, without subsequent hardening treatment (since age hardening treatment is normally not carried out on a small section of the component after repair welding). We found brittle plate shaped $\eta$-Ni$_3$(Al, Ti) precipitates with an adjacent soft precipitate free zone. The material was susceptible to cracking and lower fatigue life, an important finding with respect to the industrial application. Repair-welding guidelines need to be carefully established and implemented when using this new alloy in critical components to minimize the probability of sudden unexpected failure due to a notch sensitive weld HAZ. Further discussions on our observations are in Chapter 6, section 6.5.3.
We also investigate if LSP could be used to slow-down the failure of a material that has already started cracking. We do this by conducting LSP treatment on pre-cracked and fatigued samples. We examine subsequent fatigue crack growth behavior following ASTM guidelines for fatigue crack growth testing [ASTM e-647 00]. Though we tread with caution in over-predicting the benefits of LSP. Conventional techniques like shot peening have also shown little benefit on pre-cracked samples. Normally repair welding, hole-drilling or alternate joining techniques are required for slowing/arresting crack growth in pre-cracked material. The results for our fatigue crack growth investigations on standard M(T) samples are presented in section 6.6.1 We observe acceleration in fatigue crack growth and an improvement was not seen in this case. The crack length measurements were carried out with an optically calibrated alternating current potential drop (ACPD) method. We also conduct a crack opening displacement (COD) based compliance analysis of the crack growth data in section 6.6.2 and found the results to be in agreement with increased acceleration observed in growth rates. To understand the underlying mechanism of fatigue crack growth response, we present a crack closure analysis in 6.6.3.
Since real life components are subject to non-uniform loadings unlike the constant amplitude cycling conducted in our lab experiments, it is important to see the effect of LSP on overload fatigue crack growth. We conducted a 100% single cycle overload on a growing fatigue crack and subjected the crack tip plastic zone to single spot LSP, and monitored the fatigue crack growth rates before and after the overload by resuming the cycle at same loads. Encouraging results were observed in this case, with crack retardation taking place after the application of overload and LSP. Though, the results of overload on crack retardation are mixed with the effect of LSP we ensure the sample is under plain strain, and estimate the effective contribution of LSP+overload to crack retardation. Results are discussed in Chapter 6 section 6.6.4

Finally, we conduct a fractographic analysis of our fatigued samples and find evidence of subsurface crack nucleation in LSP treated samples. Based on this evidence we provide a mechanistic explanation of the improvement in endurance limit observed in our fatigue results based theory of stress concentration behavior of small notches [Timoshenko and Goodier, 1970]. Results and analysis are presented in section 6.6.5.
Chapter 2. Objectives

This research integrates three distinct areas of materials research. Corresponding objectives categorized by the important issues being addressed in each topic are listed below:

A) End Goal: Fatigue

1) Total life fatigue: Generate fatigue S-N curves of IN718Plus superalloy at room and high temperature. Replicate the improvement with laser shock peening in fatigue life reported in other alloys at service temperatures, and establish a reasonable procedure to improve the consistency of fatigue life enhancement using LSP. Identify the limitations of the process for efficient production practice and a conservative design code.

2) Fatigue crack growth: Investigate the general dilemma in literature on the beneficial vs detrimental effects of LSP on fatigue crack growth, and conduct a dedicated study on IN718Plus alloy. Use a conservative approach to report the effect of LSP, considering critical applications of the alloy in aero engine design (esp. the possibility of fast crack advance or crack tunneling due to LSP induced modification of the crack tip stress field). Investigate the effect of overloads and crack closure in LSP fatigue.

B) Means to an end: LSP

Optimize LSP process parameters for fatigue life improvement in IN718Plus using FEM simulations, experiments, literature. This involves the following components:

1) LSP procedure

   1) identify effects of shot energy
2) identify effect of shot sequence

3) identify effect of shot overlap

4) identify effect of number of layers of LSP treatment

2) LSP Specimen

1) determine an optimized specimen geometry for LSP fatigue life

2) identify effect of specimen thickness

3) identify effect of specimen misalignment on LSP residual stresses

4) identify effect of heat treatment on LSP fatigue life

3) Residual stresses

1) establish a procedure for LSP induced residual stress measurement from Laboratory XRD (LXRD), conventional XRD and synchrotron XRD while using finite element method (FEM) simulations as a guiding tool.

2) identify the thermal response and relaxation of residual stresses at service temperature (thermal relaxation).

3) identify stress relaxation observed after an overload in fatigue crack growth samples (fatigue relaxation).
C) Material on which to apply the means: IN718Plus Superalloy

1) identify microstructural detriments peculiar to this alloy towards application of high-energy laser impacts with the intent of improving fatigue life.

2) identify microstructural advantages peculiar to this alloy towards application of high-energy laser impacts on the surface with the intent of improving fatigue life.
Chapter 3. Literature Survey

As described, the problem entails three important aspects of materials science. The aim is to improve fatigue behavior by using laser impacts, which introduce compressive residual stresses and test it on a newly developed aero-engine superalloy. We start with reviewing 1) this new alloy followed by 2) fatigue and fatigue crack growth and finally 3) effects of laser shock peening and the analysis of its resultant residual stresses.

A) Understanding the Material: IN718Plus Superalloy

IN718Plus is the youngest in a long line of high temperature Ni-base alloy development. To understand this alloy, we need to review the origin of superalloys that form the basis of IN718Plus. We start with an overview of superalloys and then compare IN718Plus with its nearest service counterpart, IN718, which has been the choice of industry for applications up to 923K. In this comparison we bring out the characteristics of IN718Plus that make it a potential improvement on IN718 for the same cost. Finally, we review the thermal and mechanical stability of this alloy vis-à-vis the possible microstructural advantages and detriments with respect to high-energy laser impacts with the intent of improving fatigue life.

3.1 Superalloys

As the demand for easily formable and weldable alloys that retain good high temperature creep, fatigue and corrosion resistance increased with the advent of gas turbine technology, jet engines,
modern power plants, petrochemical and furnace industry during the middle of the 20th century; candidate materials that provide these remarkable properties came to be known as superalloys. Superalloys are used typically above 823 K. There are three main categories of superalloys: iron-nickel, nickel and cobalt based. Precipitates usually strengthen superalloys. While thermodynamic stability of these precipitates determines the maximum operating temperature of a superalloy, their coarsening kinetics determines service life and creep strength. Most popular nickel-based superalloys are primarily γ’ or γ’’ strengthened [Donachie 2002]. IN718Plus is a γ’ strengthened nickel-based superalloy.

As the open structure of body centered cubic (bcc) lattice has higher diffusion, it is more prone to creep than close packed face centered cubic (fcc) structure. Fcc also provides better ductility than hcp due to greater available slip systems. Hence, the superalloys are generally fcc.

Incoherent hardening is limited by the stability and coarsening of precipitates. To impart greater stability and high temperature strength, a range of finely distributed inter-metallic precipitate phases are formed using reactive elements like Ti and Al. There are three main intermetallics in Ni-Al-Nb phase diagram, Ni₃Ti, Ni₃Al and Ni₃Nb. Crystal structures of the few of the several intermetallics seen in nickel base superalloys are shown (Figure 3.1). All of these have the unique property of enhanced strength at higher temperatures, thus making them attractive for high temperature applications. Further, they exhibit excellent chemical stability along with good corrosion resistance. The gamma prime (γ’) Ni₃Al, was first such inter-metallic extensively studied. It has a L₁₂ crystal structure [Nunomura 2004] offering superior strengthening with excellent high temperature capability. On the other hand γ’’ (Ni₃Nb) has a DO₂₂ crystal structure which is a bct type structure [Kasubiraki 1995]. γ’’ strengthened iron-nickel based alloys have
better weldability and ease of processing, even though they do not reach as high temperatures as γ’-strengthened alloys. The IN718 invented in 1960s [Eiselstein 1965] is a D022 γ’’ strengthened superalloy finding remarkable success from cryogenic use to the aerospace industry (General Electric, Pratt & Whitney) for elevated temperature service up to 923K. While, its excellent formability allows wrought and welded designs, it also finds extensive application in castings for modern aero-engine gas turbines by investment casting technology. While IN718 also forms coherent γ’ cubic L12 precipitates, the main hardening of IN718 is by virtue of γ’’ precipitates finely dispersed throughout the fcc γ grains as nanometer sized ellipsoidal particles causing the main strengthening. These ordered precipitates ensure good toughness, slow coarsening kinetics as well as uniform strengthening by offering resistance to dislocation motion due to coherency strains and formation of anti-phase boundaries when dislocations cut through them. The c-axis of D022 structure has a mismatch with the fcc lattice, hence bct γ’’ precipitates transform to delta (δ) Ni3Nb phase above 923K [Azadian 2004], which is a thermodynamically more stable orthorhombic D0a crystal structure with a kinetic a maxima around 1173K. However, since the transformation is sluggish, the γ’’ always precedes the formation of δ-phase up to 1173K beyond which γ’’ doesn’t occur in the phase diagram. δ-phase forms at grain boundaries initially and is useful as a grain refiner during thermo-mechanical processing [Desvallees 1994]. Upon longer ageing at higher temperatures δ-Ni3Nb phase δ-Ni3Nb phase starts forming in Widmanstätten type pattern within the grain interiors [Kasubiraki 1995], and tends to reduce the strength of the alloy by using up the Nb required for forming γ’’ Ni3Nb strengthening phase. The γ’ Ni3(Al,Ti) on the other hand transforms to hcp eta (η) Ni3Ti upon longer ageing [Brown 1987; Oblak 1969; Brooks 1988; Ordei-Basile 1991]. The η-phase has a plate-shaped morphology and can act
as a stress concentrator reducing the fracture toughness and strength. Ni and Fe-Ni based superalloys are susceptible to forming topologically close packed phases at larger supersaturations, which are often sharply faceted and can act as harmful stress concentrators.

3.2 General properties

**Single crystal alloys:** It is important to mention the use of these alloys in single crystal blades. While aeroengine blades started being made as single crystals around 1960’s, initial superalloys used directional solidification chemistry that resulted in high grain boundary precipitates. These materials often failed due to crack initiation at these secondary precipitates giving poor fatigue life [Gell 1968, Leverant 1970, 73, 75].

**Rafting:** Dynamic strain ageing of γ’ strengthened alloys was found to grow the γ’ precipitates into a raft like structure. This structure is reported to improve the creep rupture life [Anton 1985] as well as deteriorate it in certain single crystal configurations [Legros 2002] due to modification of dislocation motion. The rafted microstructure can act as a dislocation source or sink, and depending upon temperature range, lead to γ’ precipitate shearing or looping. Normally γ’ is stronger compared to the γ matrix, hence dislocations motion is slower when moving from γ to γ’ than vice versa.

Higher temperature applications up to 1050C have been possible with improved alloy chemistry and single crystal fabrication by directional solidification. At high temperature dislocation climb becomes the main deformation mechanism and dislocations can loop around precipitates. However at intermediate temperatures (1023K), which are the target for this study, dislocation glide is more prevalent and γ’ precipitate shearing is observed. Perfect dislocations in γ’ are
called super-dislocations due to their larger Burger vectors. These high-energy dislocations dissociate into stacking faults and super-stacking faults at intermediate temperatures, with anti-phase boundaries at high temperatures.

A unique shearing mechanism of ordered precipitates (like γ’) by dislocation pairs temperature gives superalloys them their ambient temperature mechanical strength. The crystal structure of the γ -matrix as well as γ’-particles is fcc, and two matrix dislocations of the type 1/2(1 1 0) are observed to glide together in pairs. When a γ’ precipitate with L1₂ superlattice structure is sheared by the leading 1/2(1 1 0) dislocation of the pair, it creates an anti-phase boundary. This boundary being an area of lattice mismatch, has higher energy. The passage of the subsequent trailing 1/2(1 1 0) dislocation of the pair reverts the matrix back to lattice registry, and eliminating the anti-phase boundary.

The yield strength of superalloys is higher at intermediate temperatures than room temperature. This yield anomaly arises out of the lowering of (100) anti-phase boundary energy and the cross slip of the leading partial in the dislocation pair to the (100) plane, where it gets locked since (100) is not a glide plane. This dislocation locking leads to anomalous hardening effect. As the temperatures are raised still higher to about 1050K, most superalloys see a rapid decrease in hardness due to the thermal activation of (100) slip, which allows them to overcome the γ’-precipitates without shearing [Nembach 2003]. This leads to lowering of hardening behavior as in the absence of shearing of γ’-precipitates, as the dislocations do not need to move in pairs, and the (100) antiphase boundary (APB) locking becomes less common. Further, at higher temperatures the locked dislocations in (100) plane can also free themselves by climb and thermal activation.
3.3. **IN718Plus vs. IN718**

Table 3.1 gives the Chemical compositions of IN718 and IN718Plus alloys. Nickel forms the austenitic matrix, iron stabilizes $\gamma''$ though at the cost of lowering its solvus temperature. The main solid solution strengthening comes from Cr, Mo, Ti, Al, Nb, Co, Zr, V and C [Muzyka 1987]. Nb forms $\gamma''$ ($\text{Ni}_3\text{Nb}$) and Ti and Al form $\gamma'$. Higher Al contents increase the solvus temperature and reduce $\delta$-phase formation. Most of the above elements also form complex carbides that precipitate at the grain boundaries. Cr forms a stable protective oxide layer for corrosion resistance, while B and Zr minimize Laves phase, which is often found to increase with Ti, Nb, Si and Al. Formation of $\delta$-phase is enhanced with greater Nb and Si, while Mg scavenges impurities like oxygen, sulphur and nitrogen that may form inclusions and brittle phases. Further details of phase stability and strengthening in nickel-based alloys is available in literature [Sabol & Stickler 1969; Decker & Sims 1972; Bridges 1974; Collier 1988; Gao 1995; Arya 2002].

IN718 has been the popular choice at intermediate temperatures application and accounted for up to 35% of industrial use of superalloys [Collier, 1988] in last 25 years. Recent advances in thermal stability of strengthening phases and the role of chemical modifications as well and coarsening kinetics have opened up the field to improved alloys with better high temperature capabilities. Amongst them, IN718Plus offers superior high temperature improvements with a stable $\gamma'$ strengthened matrix similar to several other successful Ni-base superalloys.
Both IN718 and IN718Plus strengthen by coherency strains and associated APB strengthening. FCC-$\gamma'$ is the main strengthening precipitate in IN718Plus while in IN718, bct-$\gamma''$ nucleates on $\gamma'$ and is the main strengthening phase. Based on this understanding new variants of IN718 were tried early on to retain the compact structure of $\gamma''$ [Cozar and Pineau, 1973]. Similar alloys were proposed later by modifying the nucleation behavior of $\gamma''$ on $\gamma'$ interface, which slowed the coarsening of the precipitates [He 1998] and improved the thermal stability of alloys.

Around the same time, it was observed that increasing the Al-Ti content and the Al/Ti ratio increased the thermal stability of $\gamma''$ in IN718 while reducing the tendency of brittle $\delta$-phase formation [Collier 1988] making IN718 more stable at high temperatures for longer durations.

From and examination of the available phases in the Ni-Al-Ti-Nb phase space, it was proposed [Tomihisa 2002] that a combined alloy of Ni$_3$Al, Ni$_3$Nb and Ni$_3$Ti could offer greater freedom and flexibility in high temperature applications by overcoming specific limitations like poor creep or corrosion resistance associated with having a single dominant precipitate. Such multi-component intermetallic-hardened alloys with L1$_2$, D0$_{24}$ and D0$_a$ precipitates exhibit increasing strength and stability of D0$_{24}$ and D0$_a$ phases with temperature, offer excellent high temperature oxidation resistance, as well as good chemical stability. Hence, as a part of aircraft industry initiative and Government’s encouragement to economically enhance the range of operation of IN718 to higher temperatures, IN718Plus was developed by ATI Allvac, USA. The chemical composition of IN718Plus alloy (Table 3.1) is close to IN718 but is different in the following aspects [Cao & Kennedy 2006]:

1. Composition is modified by increase in Al+Ti, Al/Ti ratio and W. Fe has been partially replaced by Co and a small increase in P content.
2. It is primarily $\gamma'$ strengthened.

3. Ordered phases/ precipitates $\eta$-Ni$_3$Ti, $\delta$-Ni$_3$Nb and a new hexagonal phase Ni$_3$Al$_{0.5}$Nb$_{0.5}$ are reported to form with thermal ageing in addition to $\gamma'$.

Varying the Al/Ti ratio and Fe content improves the high temperature stability [Cao, Kennedy 2006]. IN718Plus is reported as having at least 50 °C improvement in operating temperatures (1073 K), good corrosion resistance and creep rupture strength. Subsequently, other variants of IN718 have also been proposed based varying Al-Ti content and higher Al/Ti ratio [Du, 2007], though IN718Plus offers better high temperature capability.

### 3.4 IN718Plus

Since the rapid coarsening kinetics of $\gamma'$ precipitates is a concern compared with $\gamma''$, which exhibits slower coarsening due to a lower lattice mismatch [Slama 1997; Azadian 2004], it is important to study the dynamic ageing behavior of these alloys to establish the stability of $\gamma'$, the main strengthening phase over long service exposures.

#### 3.4.1. $\gamma'$ Precipitation during ageing:

Strengthening of $\gamma'$ superalloys normally involves a two step heat treatment. First is a solutionizing treatment just above or below the solvus, followed by a low temperature age to get the peak hardness. In the former, if the alloy is heated and held just below the solvus temperature for $\gamma'$ dissolution so that some of $\gamma'$ remains at grain boundaries while rest goes into solution. This grain boundary $\gamma'$ helps in preventing excessive grain growth, serving as a grain refiner. On
the other prior precipitation reduces the amount of $\gamma'$ available for precipitation during ageing, thus lowering the strength. Hence, the heat treatments could be selected to arrive at a desired grain size and strength combination. The precipitation of large number of finely dispersed $\gamma'$ precipitates during second step of heat treatment results in a large interfacial area. Reduction in interfacial surface energy is the driving force for $\gamma'$ coarsening. Over extended thermal exposures, larger $\gamma'$ particles coarsen at the expense of smaller ones while conserving the overall volume fraction. This process is known as **Ostwald ripening.** Studies on coarsening behavior were conducted in an earlier research by the author on the same alloy [V Chaswal, MS Thesis 2011]

### 3.4.2. $\gamma'$ - $\eta$ transformation during Ageing:

Over longer thermal exposure, L1$_2$ $\gamma'$ strengthened nickel-based superalloys are found to transform to following stable precipitates through a superlattice faulting mechanisms involving shear along \{111\}$_{1/3}$\langle 112 \rangle or climb along \{111\}$_{1/3}$\langle 111 \rangle [Kear 1970]:

i) Superlattice Extrinsic Fault - \textbf{DO19} structure

ii) Superlattice Intrinsic Fault - \textbf{DO24} structure

iii) Superlattice Extrinsic+Intrinsic Fault - \textbf{VCo$_3$} type structure

As has been reported, corresponding precipitates seen in IN718Plus are the Ni$_3$Ti$_{0.7}$Nb$_{0.3}$ (D019) and Ni$_3$Ti (DO24) phases [Cao, 2005]
Crystallography of $\eta$ transformation from $\gamma'$ by a stacking fault mechanism is well known. As Figure 3.2 and Table 3.2 from [Kear 1970] explain, the transformation by introduction of intrinsic stacking fault associated with a Frank sessile in the L1$_2$ structure to give rise to abc/abc D0$_{24}$ Ni$_3$Ti. The mechanism for creation of stacking fault is known to be segregation of solute atoms at the Frank sessile. Since the matrix is enhanced in Ti as Al migration takes place to $\gamma'$ during coarsening based on the phase diagram (Figure 3.3), there exists a viable mechanism for $\eta$-transformation, which is thermodynamically more stable compared to $\gamma'$ at longer durations and higher temperatures. A detailed study of the possible transformations in the Ni-Al-Nb pseudo-ternary system indicates phase stability of different intermetallics possible. The main intermetallics observed with their crystal structures, electronic concentration and atomic radius ratio are listed in (Table 3.3). Both electronic concentration and atomic radii ratio suggest the transformation proceeds from L1$_2$ (Ni$_3$Al) to Ni$_3$Ti (D024) to Ni$_3$Ti$_{0.7}$Nb$_{0.3}$ (D019) to Ni$_3$Nb(D0a) [Tomihisa 2002]. Further, thermodynamic ternary phase diagram of IN718Plus predicted by [Cao 2006] also indicates that Ni$_3$Ti precipitation can occur. The authors have also noted the presence of an hcp Ni$_3$(Nb, Al) phase in the alloy. In our earlier investigations on IN718Plus [V Chaswal, MS Thesis, 2011], we observed the transformation of $\eta$ from $\gamma'$ takes place by the faulting mechanism described above. The faulting tendency was found to depend on the size of precipitates, with coarse precipitates showing faulting (Figure 3.4) while fine precipitates did not show faults (Figure 3.5) as the coherency strains associated with them were smaller.
Crystal structure of (a) Ni$_3$Al and (b) Ni$_3$Ti, reprinted with permission [Nunomura 2004]

Crystal structure of (c) Ni$_3$Ti$_{0.7}$Nb$_{0.3}$ (D0$_{19}$) (d) Ni$_3$Nb (D0$_a$)

Crystal structure of (c) Ni$_3$Ti$_{0.7}$Nb$_{0.3}$ (d) Ni$_3$Nb reprinted with permission [Tomihisa 2004]

Figure 3.1 Structure of few ordered precipitates in Ni superalloys
<table>
<thead>
<tr>
<th>Precipitate</th>
<th>Designation</th>
<th>Crystal structure</th>
<th>Stacking</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni$_3$Al</td>
<td>γ'</td>
<td>FCC – L1$_2$</td>
<td>ABC/ABC</td>
</tr>
<tr>
<td>Ni$_3$Ti</td>
<td>η</td>
<td>Hexagonal – D0$_{24}$</td>
<td>ABAC/ABAC</td>
</tr>
</tbody>
</table>

Table 3.2: Stacking arrangements in γ' and η precipitate phases of IN718Plus
Table 3.3: Electron concentration and atomic radius ratio of Ni$_3$X intermetallic phases

<table>
<thead>
<tr>
<th>Intermetallic phases</th>
<th>Crystal structure</th>
<th>Electron concentration ($e/a$)</th>
<th>Atomic radius ratio ($R_x/R_{Ni}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni$_3$Al</td>
<td>L1$_2$</td>
<td>8.25</td>
<td>1.149</td>
</tr>
<tr>
<td>Ni$_3$Ti</td>
<td>D0$_{24}$</td>
<td>8.5</td>
<td>1.173</td>
</tr>
<tr>
<td>Ni$_3$Sn</td>
<td>D0$_{19}$</td>
<td>8.5</td>
<td>1.378</td>
</tr>
<tr>
<td>Ni$<em>3$Ti$</em>{0.7}$Nb$_{0.3}$</td>
<td>D0$_{19}$</td>
<td>8.575</td>
<td>1.177</td>
</tr>
<tr>
<td>Ni$_3$Nb</td>
<td>D0$_a$</td>
<td>8.75</td>
<td>1.185</td>
</tr>
<tr>
<td>Ni$_3$V</td>
<td>D0$_{22}$</td>
<td>8.75</td>
<td>1.084</td>
</tr>
</tbody>
</table>

Figure 3.2: D0$_{24}$ Ni$_3$Ti Stacking sequence
Among the different precipitates mentioned above, the hexagonal D0$_{24}$ $\eta$-Ni$_3$Ti transition (Figure 3.2, Table 3.2) takes place with the introduction of a stacking fault in the FCC Ni$_3$Al (L1$_2$) precipitates [Kear 1970]. This stacking fault based mechanism gives the $\eta$-precipitates their plate shaped morphology, which is important for fatigue and fracture resistance, since sharp plate like precipitates can act as a stress concentrators and initiate a crack. The Ni$_3$Al-Ni$_3$Ti binary phase diagram (Figure 3.3) indicates the possibility of up to 8% Al in Ni$_3$Ti D0$_{24}$ structure [Nunomura 2004]. Thus, the transformation does not need sudden diffusion of large amounts of Al from the lattice. While $\eta$-phase is observed in other nickel-based superalloys [Wu, Vasudevan 2008; McGurran 1981], our prior studies on IN718Plus investigated $\eta$-phase in detail [Vibhor Chaswal, MS thesis, UC 2011]. We also investigated coarsening of the main strengthening precipitate $\gamma'$, that follows LSW kinetics [Zener 1944; Greenwood 1956; Lifshitz, I.M. and V.V. Slyozov 1961; Wagner 1961] and is important for overall strength considerations including residual stress evolution, dynamic precipitation and in service temperature fatigue.
Figure 3.3: Ni$_3$Al-Ni$_3$Ti Phase diagram, reprinted with permission [Nunomura 2004]
Figure 3.4: Coarse $\gamma'$ transform to $\eta$ by faulting (red arrow)  
[V Chaswal, MS Thesis, 2011]

Figure 3.5: No transformation seen in fine $\gamma'$  
[V Chaswal, MS Thesis, 2011]
B) Means to an end: LSP

Laser Shock Peening (LSP) as a technique for surface processing has been in existence for over 25 years. LSP being a new implementation of an old technique of introducing surface compressive residual stresses, has its unique advantages over conventional shot peening. While the extensive literature on shot peening helps identify potential areas where LSP would be most beneficial, LSP generates a plasma that leads to the shock wave and creates deep compressive stresses. Hence, literature data from other similar techniques cannot be adopted categorically to LSP. Since LSP is new on an industrial scale, the details of optimizing this process are not common in literature. Literature survey was conducted keeping in mind our objectives of optimizing the process with eventual target of improving fatigue performance:

3.5 Laser Shock Peening

In LSP, high energy laser pulses of very short durations are impacted on a material surface. The sample surface is coated with an ablative absorbent polymer that evaporates and creates a plasma that is confined by a laser-transparent outer layer, usually water (Figure 3.6). This confinement generates pressures up to 10 GPa and leads to plastic deformation to a depth at which the peak pressure no longer exceeds the metal’s Hugoniot elastic limit (HEL). HEL is related to the dynamic yield strength ($\sigma_{y, dyn}$) at high strain rates and the Poisson’s ratio ($\nu$) according to [Johnson 1971] as:
\[ HEL = \left( \frac{1 - \nu}{1 - 2\nu} \right) \sigma_y \text{ dyn} \]  \hspace{1cm} (1)

Along with plastic deformation, this process results in a deep compressive residual stress layer extending from the surface to depths up to 1 mm, depending on the energy density and material. Typically a pulsed Nd:Glass laser ($\lambda=1.07 \, \mu m$) providing energy in the range of 1 to 100 J per pulse and pulse durations from 5-50 ns is used.
Figure 3.6: Double side LSP Process Schematic
Compared to other techniques like shot peening (SP), ball-burnishing (BB) and ultrasonic shot peening (USP), LSP is reported to produce lowest work hardening close to the surface [Maawad 2011]. With a protective environment and good surface strengthening, laser peening is suitable for fatigue life improvement of fastening holes. LSP also has improved the crack growth resistance post foreign object damage (FOD) with delayed crack initiation [Spanrad 2011] valuable for aircraft gas turbine engine compressor and fan blades [See 2002]. We have found rigid spinal implant Ti rods improve in flexibility by LSP for a given fatigue strength [Mannava 2011]. For Ti alloys suitable for high-speed motor rotors used in centrifugal compressors, LSP decreases the fatigue crack growth rate [Balyts'kyi 2002]. Improvements in laser technology have enabled high throughput production [Dane, Brent 2000] using new femtosecond lasers, though the extent of work hardening is similar for both femtosecond lasers and older nanosecond laser based peening [Nakano 2010].

During this research we optimize LSP process parameters for fatigue life improvement in IN718Plus alloy for the test geometries using FEM simulations, experiments, literature as discussed in section 6.3

3.5.1 LSP Parameters

A number of process variables, including laser shock intensity (energy/power density or fluence), spot size, multiple laser shots, overlapping of laser spots, etc., are available to control the depth of residual stress, surface roughness and distortion [Clauer and Lahrman 2001]. Intensity of LSP is mainly controlled by the power density (power per unit area) applied to the laser treated region and is proportional to the magnitude and depth of the compressive residual stress. The depth of compression can also be increased at the same energy by applying multiple impacts. Basic theory
of deformation dynamics of shock compression has been investigated by several notable researchers starting from shear bands [Zener and Hollomon 1944], constitutive behavior and thermally activated mechanism of dislocation motion [Harper, Shepard, Dorn 1958], high-speed dislocations and modeling of shock front by dislocation movement [Weertman 1986] and constitutive modeling [Kocks 2003] of plastic deformation.

3.5.2 Residual stress

Cyclic softening: In response to fatigue loading, the material strength may increase, stay constant or decrease depending on its prior state [Kettunen & Kocks 1972]. Cyclic hardening is seen for annealed materials, while cold worked materials show cyclic softening. Since LSP results in shock induced plastic strain, it is expected to show fatigue softening. While cyclic softening in disordered alloys is normally associated with prior cold-work, in case of ordered alloys like most superalloys, cyclic softening can arise due to shearing of precipitates by movement of dislocations along slip bands [Sundararaman 1990]. Cyclic softening was observed during fatigue of IN718 due to shearing of γ' precipitates dislocations moving on planar slip bands [Xiao 2008]. Fatigue induced relaxation is also reported for LSP [Nalla 2003] treated steels to a partial extent, though complete relaxation is also possible in certain cases [McClintock 1982]. Our results presented in section 6.4.5 show up to 50% reduction in residual stresses after fatigue cycling, in agreement with literature.
3.5.2.1 Thermal relaxation

Residual stress relaxation studies carried out are essentially empirical. As LSP is a relatively new technique, the reasons behind introduction of deeper compressive residual have not been analytically explained, though numerical results, simulations and empirical results do provide help in modeling residual stress relaxation.

Residual Stress measurements are often subject to calibration errors. Since stresses are generated from internal strains, which are not visible from the outside and get relaxed at the free surface, accurate absolute values of the residual stresses are quite difficult to determine. Most of the techniques compare the residual stress in the material with another material of known residual stress. However, since the internal microstructure varies from one sample to another, there is a large variation in the value reported in literature. Finally, no set methodology exists about using same calibration standard across materials processed through different techniques. A residual stress state measured using LSP and LPB may give different results for same calibrant when probed by XRD compared to synchrotron XRD due to a different depth of penetration and drastically different surface cold work in the two cases, unless detailed depth gradient corrections are made.

Our results on SXRD analysis show a reduction in LSP induced stresses by around 45% of their original value upon thermal exposure near the service temperatures of 973K and 1023K for 1 h each (Figure 3.8). This stress relaxation is in agreement with literature, where reduction in residual stresses was reported with thermal treatment [Clauer 2001, Buchanan 2011]. Further details are presented in section 6.4.
Near surface compressive Residual stresses relax during Thermal Ageing of IN718 Plus alloy

- LSP + Aged at 700 °C/1h, 750 °C/1h
- LSP, Unaged

5mm thick DS LSP

% Reduction in residual stress = 100\%((\sigma_i-\sigma_o)/\sigma_o)

= 44.6 \% for this case

\( \varepsilon_i = 0.5553 \varepsilon_o \)

\( \sigma_i = \varepsilon_i E \)

Figure 3.7: SXRD residual strain relaxation on thermal exposure

[V Chaswal, MS Thesis, 2011]


C. End Goal: Fatigue

Origin of all plastic deformation of metals lies in the dislocation slip of a row of atoms along shear direction, discovered by Taylor, Orowan and Polanyi [Orowan 1933, Taylor 1934, and Polanyi 1934]. Superalloys are often employed at the limits of material performance. In order to ensure that the effect of a small improvement in one property does not lead to an undesirable effect on the other, it is important to understand the underlying basis of overall mechanical behavior.

3.6 Fatigue

Fatigue accounts for failure of 75% of all machines and structures. Fatigue started being observed scientifically around the 20th century when the products of industrial revolution accumulated enough service cycles to start failing mysteriously at low loads. In 1830, Woehler documented the number of cycles (N) to failure as a function of mean stress (S) in railways axles. His S-N curve approach is used to date. It was observed that the flat plateau occurring at high cycles called endurance limit, may not be well defined in most non-ferrous alloys (except Al-Mg) where solute migration doesn’t occur. Also, since load history may vary, Miner’s rule has been traditionally used to estimate the remaining life of a component as:

\[ \sum \left( \frac{n_i}{N_i} \right) = C \]  \hspace{1cm} (2)

where \( n_i \) = number of cycles at stress (i), \( N_i \) = number of cycles to failure at stress (i), \( C \) = cumulative damage (empirically determined: 0.7 to 2.2). The identification of fatigue as a material problem was made in the beginning of the 20th century by Ewing and Humfrey [Ewing and Humfrey 1903], when they carried out a microscopic investigation that showed that fatigue
crack nuclei start as micro-cracks in slip bands. Fatigue begins as micro-crack initiation at a region of high stress intensity, a structurally weak spot or a region of tensile stress, followed by crack growth leading to final failure when the material cross section can no longer bear the load [Richard W. Hertzberg 1983].

Origin of fatigue crack lies in the Bauschinger effect and the formation of extrusions and intrusions by a dislocation based mechanism. The intrusions and extrusions act as stress concentrators and initiate a crack on the surface [Hull 1957, Forsyth 1953, Greenfield 1971]. Early work [Mughrabi 1978, Laird 1976] successfully identified the formation of persistent slip bands (PSB) by dislocations during fatigue loading, to be the basis of intrusions and extrusions formation. Later, the origins of Bauschinger effect were modeled in terms of the back stress originating due to dislocation pile up and hardening [Kocks and Mecking 2003].

Introduction of compressive residual stresses improves fatigue resistance by preventing crack initiation and growth. One of the most popular methods of improving fatigue life is by impacting the surface with hard shots or balls, called shot peening (SP), which introduces plastic hardening of the surface layer and gives rise to compressive residual stresses. LSP works on the same principle by utilizing high-energy lasers to generate a high-energy plasma associated with a shock wave. This to shock wave compresses the material and induces residual stresses. While at room temperature, both techniques are quite successful, for superalloy application, high temperature operation has its own particular demands. Apart from residual stress relaxation, thermo-mechanical activation of alternative slip systems makes the problem more complex.
3.6.1 High temperature fatigue in superalloys

Superalloys were primarily designed for high temperature service; hence minimizing creep was the main concern. Directionally solidified single crystal components offered superior creep performance by eliminating the grain boundaries. Though, this improves creep life substantially, eliminating grain boundaries makes the alloy more susceptible to fatigue since grain boundaries act as obstacles to a fatigue crack. Further, fatigue being crystallographic in origin, anisotropy of single crystals leads to a reduction of life in the direction with lowest modulus i.e. <100>. A solution to anisotropy was found by dynamic strain ageing (DSA) of superalloys. DSA leads to coarsening of γ’ substructure and creation of a rafted structure. The rafted structure lowers the anisotropy of single crystals fatigue (Anton 1984), but in certain temperature-precipitate combinations, it may reduce the creep life due to a change in dislocation precipitate interaction. Our room and high temperature fatigue tests on IN718Plus superalloy did not show any incidence of rafting.

At high temperatures several superalloys show brittle inter-granular failure by dynamic embrittlement [Ghonem 1993, Molins 1997], primarily due to susceptibility of grain boundaries. IN718 is susceptible to oxygen induced dynamic embrittlement at operating temperatures of 650 °C [Krupp 2004]. Our high temperature fatigue tests on IN718Plus superalloy did not show inter-granular failure and the fracture surface was observed to be trans-granular in both fatigue and fast fracture regions.
3.6.2 Low cycle fatigue:

Low cycle fatigue corresponds to fatigue failure occurring at high plastic strains and low number of cycles which may arise often at high temperature or loads. Laird observed that power-law cyclic stress-strain behaviors of pure metals and simple alloy systems correspond to the shear stress required to form PSBs [Liard 1976]. Lower plastic strain amplitude limit for formation of a single PSB was found to be 6 x 10^{-5} for copper [Mughrabi 1979] and 2 x 10^{-5} for γ’ strengthened Ni-base superalloy Nimonic 115 [Fritzemeier 1988]. The fact that PSBs do not form below the minimum strain was postulated to be the basis of an endurance limit. A three-stage description of cyclic stress-strain curve, described by a power law equation similar to monotonic stress-strain curve of fcc crystals was given by Mughrabi [Mughrabi 1978]. In the central plateau, the plastic strain is independent of the stress indicating the little amount of stress required for subsequent deformation within the PSBs making them a soft region compared to the matrix in the strain range. The interaction of long range internal stresses with nucleation and growth of PSB’s was related to the subsequent microstructure of the PSBs. PSBs tend to form under a symmetrical loading and only up to intermediate temperatures [Lukas 2001]. For Ni, fatigue cycled under asymmetrical loading, cell structure was found to be dominant with the absence of PSB’s [Holste 1994]. Irrespective of the underlying origin of extrusions and intrusions being PSB’s, Ni exhibits slip localization and an endurance limit under asymmetrical loading. Our tests fatigue were conducted under asymmetric tension-tension loading with R = 0.1.
Ni-base superalloys exhibit tension-compression asymmetry. At 298K cyclic stress-strain curve shows 3-stages similar to f.c.c. polycrystals while at elevated temperatures the curve follows a power law [Fritzemeier 1988].

**Thermal barrier coatings:** Superalloys are often coated with thermal barrier coatings when being used at very high temperatures. This improves the oxidation resistance, which is known to cause stage-I fatigue cracking [Greenfield 1971]. These coatings also improve high temperature fatigue performance. Coatings have been since long known to improve the fatigue life at lower temperatures as well. Coating nickel and gold on copper improves fatigue resistance by limiting the intrusion-extrusion mechanism [Dover 1969]. Our tests being primarily conducted at intermediate temperatures, did not require thermal barrier coatings.

**Compressive stress and Fatigue:**

Introduction of deep compressive stresses dramatically enhances fatigue life in components compared to conventional shot peening. Ti-6Al-4V turbine engine fan blades show much higher fatigue life after LSP compared to shot peening [Clauer and Lahrman 2001]. This is the main driving force behind choosing LSP compared to conventional shot peening. LSP is reported to have deeper compressive residual stresses.

**Dynamic precipitation at high temperatures:** $\gamma'$ strengthened alloys undergo fragmentation and dissolution at lower temperatures. This give rise to fatigue softening and re-precipitation of fine $\gamma'$ precipitates [Sundararaman 1990]. We did not observe the fragmentation of the $\gamma'$ precipitates, though slip bands seen similar to those observed by [Sundaraman 1990] were seen
in our samples. Further, since $\gamma^\prime$ precipitates can be very fine, there is a possibility that they were not resolved during our TEM investigations. Since our tests are asymmetric tension-tension loading, as suggested by [Lukas 2001] the formation of well defined slip bands may not have occurred which seem to be the primary regions where precipitate fragmentation occurs.

### 3.7 Fatigue crack growth & Fracture

Most fatigue data available on LSP are S-N curves, but such results are generated by testing smooth specimens, which have large statistical variability due to surface conditions. However, most engineering structures/components contain a variety of defects. Hence, the defect-tolerant, fracture-mechanics-based fatigue crack growth approach with each cycle defines a rate, which can be measured in independent tests as a function of the stress intensity range. The stress intensity factor (K) is a fracture mechanics parameter that can be evaluated analytically for standard geometries [ASTM e-399] or by finite element method for non-standard geometries using the load and crack length data. Fatigue crack growth (FCG) rate, $da/dN$, is plotted against the $\Delta K$ values with the double logarithmic axes to represent material behavior. Central linear portion of this curve represents steady state FCG in a polycrystalline metal and follows a power law called Paris Equation:

$$da/dn = C(\Delta K)^m$$

where $\Delta K = K_{max} - K_{min}$, $a = crack \ length$, $n = number \ of \ cycles$, $m = Paris \ exponent \ (2-3 \ for \ metals)$.
Paris equation can be integrated to get number of cycles to grow the crack to failure, and estimating the design life.

**Crack length measurements**: can be measured optically, by using strain gauges, compliance measurements and potential drop techniques as per ASTM recommendations [ASTM e647].

Since the initiation of micro-cracks and initial growth of fatigue cracks after LSP treatment depends highly on the conditions of the material surface, cold work, retained residual stress, environmental conditions etc., it is important to arrive at a reliable and quantifiable fatigue design in both constant and variable amplitude loading. This is often done by using closed-loop servo-hydraulics for simulating complex load cycles, fracture mechanics, conducting stress analysis to obtain values of the stress concentration factor (K_t) and the stress intensity factor (K), electron microscopy for post test sub-structural damage analysis, improved measurement of in-service load history coupled with crack closure estimation and employing new computational and statistical techniques. Most of these issues are addressed for conventional fatigue crack growth, but LSP being a new technique introduces newer dimensions. While in principle LSP is beneficial compared to other treatments, a conservative approach to fatigue design for critical components like aircraft engines would include looking for the flaws of a technique first before looking for its benefits.

Fatigue crack growth in IN718 was studied at service temperatures of 923K in air and vacuum and inter-granular failure was reported in Air compared to trans-granular mode in vacuum. Longer exposures at higher temperature in air tend to increase the tendency for inter-granular
failure. This was attributed to susceptibility of oxygen induced weakening of grain boundaries [Osinkolu 2003]

Fatigue crack growth behavior of IN718Plus has been studied for service temperatures of 823K [Xingbo 2005]. Fatigue resistance of IN718Plus was found comparable to Waspaloy and better than IN718. While failure was predominantly crystallographic and trans-granular, at longer hold times at high temperatures, there were indications of inter-granular failure. In cases where there was greater delta phase precipitation on the grain boundaries, better performance with dwell times was observed. Their room temperature test shows similar fracture morphology as observed in our FCG tests (section 6.6).
Chapter 4. Theory and Simulations

4.1 Introduction

Theoretical analysis is developed and simulations conducted for LSP process to develop a basis for the practices to be followed in experiments. LSP being non-stochastic, customizable and programmable technique, several parameters need to be decided depending upon test requirements to get good LSP results. While a judicious choice of parameters can enhance fatigue life, an improper choice can lead to the localization of tensile stresses at failure prone sites and stress concentrations inside the material causing a reduction in fatigue life. While we often used reported literature on LSP and derived an understanding of theory from similar surface compression techniques like SP; LSP having been recently adopted for industrial scale production, the literature is scarce in this field. Further, LSP is similar to other techniques like shot peening, low plasticity burnishing, water jet peening etc. in principle, as all of them try to achieve a compressive surface layer, but it is also unique in the way it generates the shock wave by a plasma by bombarding the material with high energy photons. Accordingly, care has to be taken while adopting practices followed in from techniques to LSP. We take the help of simulations and residual stress analysis of test samples to fill in the gaps of understanding and to provide valuable directions in optimizing LSP wherever other means of information are insufficient. Following is a detailed description of the steps we took towards optimizing the LSP procedure with the intent of improving fatigue and fatigue crack growth life, and validating the effect of LSP on IN718Plus material during the course of this research.

4.2 Confining media: LSP under a water confinement regime (WCR) is reported to produce plasma pressures on the target surface four times higher and two to three times longer than those
under unconfined configurations [Wu 2005]. Hence, for our experiments water confinement is used rather than having the geometry directly exposed to laser beam.

4.3 Spot size: Can be controlled using the beam delivery system consisting of an array of lenses and mirrors. The spot size can vary from 1.8 mm to around 3 mm for single side peening and between 1.8 mm to 3 mm for double side peening. Reports in literature on two lasers in water-confined regime: a Continuum Powerlite Plus laser, operating at 0.532 mm with 9 ns laser pulses, and near 1.5 mm spot diameters; a new generation Gaia-R Thales laser delivering 10 J - 10 ns impacts, with 4-6 mm homogeneous laser spots at 1.06 mm, indicate similar surface deformations and work-hardening levels, but lower residual stresses were obtained with 4-6 mm impact configuration. This reduction of residual stresses with increase of spot size was attributed to a reduced number of local cyclic loadings in the larger spot compared with the small impact configuration for laser treating the same surface area. Additionally, more anisotropic stresses were obtained with small impacts [Peyre 2011]. Since we are interested in more homogeneity as well as high energy density, we chose an intermediate value of 2 mm laser spot size for most of our testing.

4.4 Laser matter interaction

The LSP process being thermo-mechanical is reported to affect the surface layer in different zones: external - oxidized with cracks and porosity, central – transformed, and internal - deformed with high dislocation density level [Rozmus 2009]. In IN718Plus, we do not see a transformed zone as in martensite-forming materials, but considering the significance of surface cracks towards limiting fatigue life, surface was examined using scanning electron microscope.
LSP induces a supersonic shock wave in the material that is followed by slower waves. The X-ray diffraction results [Liss 2009] indicate a strong dependence of wave behavior on sample geometry. This suggests that the sequencing and location of successive shots in a multiple shot LSP treatment of the surface is going to be critical. Since establishing analytic solutions to the shock wave propagation for each geometry under possible sequencing schemes is not pragmatic, numerical simulations are employed using FEM code LSDYNA for the predictions.

Stress relaxation becomes more important since the dynamic yield strength for the material is reported to have up to 50% - 100% increases compared to static quasi-static conditions [Peyre 1998], and the accompanying cold work is lower compared to alternate techniques introducing comparable deep residual stresses like LPB or water jet peening. Further, results indicate that the magnitude of plastic deformation increases non-linearly with the energy density of laser, and also increases with the increasing numbers of laser shocks. Since higher cold work is associated with greater damage in the material, we choose the maximum possible thickness while ensuring through thickness compression, thus cold work was minimized while retaining good overall compression.

4.5 Materials

Basic microstructure, chemistry and mechanical properties of IN718Plus alloy are evaluated and compared with those reported by the manufacturer, as well as available literature to establish a reliable baseline. Results are covered in our prior work on aging kinetics [Chaswal, 2011] and are found to be in agreement with literature.
**Aging kinetics:** is vital towards long-term service reliability of this IN718Plus. An activation-energy-based kinetic analysis was conducted for γ’ precipitates, and found to be in close agreement with literature at longer durations. For shorter durations, the coarsening time of γ’ followed radius square dependence with γ’ size. A brittle plate type η-precipitate was characterized in detail considering its potential effect on decreasing overall fracture and fatigue strength [Chaswal, 2011]

**B. Simulations**

Finite element analysis techniques have been applied in literature successfully to predict the residual stress induced from laser shock peening [Braisted 1999]. While several packages are available, most commonly ABAQUS [Lee 2009] and LSDYNA [Hu 2006; Carney 2011; Arif 2009] softwares have been employed by researchers.

**4.6 Desktop Microscopist:** Desktop Microscopist software was used for electron diffraction simulations, convergent beam electron diffraction and dislocation analysis of the metal substructure to characterize the important precipitates, their orientation relationships, symmetry, microscopic strains and defect structures.

**4.7 Hypermesh and LS-DYNA:** Hypermesh software was used to model the different specimen geometries used for LSP, and LSDYNA software is used to run the laser-impact simulations. LSDYNA is designed for dynamic impact loading simulations, has been extensively used in crash and impact testing of automobiles etc, and provides one of the best platforms for LSP simulations by FEM.
The laser loading is characterized by using a repetitive Gaussian increment pressure applied uniformly on a circular impacted zone. The behavior of the subjected material is coupled with damage using the Johnson Cook model. The proposed model leads to (i) residual stress profile, (ii) plastic strains profile. A comparison of residual stresses, obtained by X-ray diffraction and by finite element calculations, has been reported to show a good correlation [Frija 2010]. Our results do not aim at obtaining absolute match with the experimentally measured residual stress values, but the trends are the more important and valuable guidelines to us for selecting laser peening conditions giving highest compression. Simulations are used to establish good laser shot sequencing as well as optimize sample geometry to minimize effects of shot-to-shot variation in impacts.

4.8 Residual Stress analysis

Residual stress is a function of multiple parameters, all of which were not practical to be modeled together analytically. Hence, LSP simulations were used for residual stress prediction and the results were compared with reliable XRD measurements. Also since laser shock peening has little or no influence on work-hardening [Cellard 2012], results of FEM simulations were not extended to predicting microscopic substructure evolution upon peening. Microstructural effects were characterized experimentally and mechanistic explanation based on fundamental material behavior literature was attempted.
Chapter 5. Experimental Plans and Procedures

Experimental plans and procedures employed are given as under. We followed the following general methodology in chronological order during the course of this research:

1) Pre-LSP characterization and preparation

2) LSP treatment and procedures

3) Post-LSP and pre-fatigue characterization

4) Fatigue & Fatigue crack growth tests, samples and standard procedures

5) Post-Fatigue characterization and analysis

5.1: Pre-LSP characterization and preparation

The steps taken to prepare samples suitable for fatigue and LSP testing are given below listed in nominal chronological order. At times experimental needs required us to go back and forth between the steps:

5.1.1 Materials: Two sets of plates were received from ATI-Allvac©.

i) Set 1: consisted of 0.1” thick sheet from heat #9983J-2 and 0.188” thick sheets from heat #998J-2 of IN718Plus obtained from ATI-Allvac© of chemical composition given in Table 3.1. Sheets were hot rolled and heat treated to 1226 K for 0.5 h followed by air cooling. Part of this
set was used for our experiments. Later, some additional material was received from ATI-Allvac of same batch.

**ii) Set 2:** consisted of 0.042”, 0.065” hot rolled sheet material of heat 993J and 0.5” thick hot rolled plates of IN718Plus obtained from ATI-Allvac® of chemical composition given in Table 5.1. Heat 993J was vacuum induction melted (VIM), then vacuum arc re-melted (VAR) for homogeneity and forged for sheet rolling. Sheets material was hot rolled and heat-treated at 1750 - 1775°F for a time less than 1 hour by ATI Allvac, The average billet chemistry measured for heat 993J in Table 5.1a is compared to the nominal 718Plus alloy sheet chemistry (Table 5.1b). The light gauge sheet material was a part of another study by ATI-Allvac. Further details of heat treatment and chemical composition are also available [Bergstorm, 2005]. The material was given a standard age [Cao & Kennedy 2006] by heat treating in a Materials Research vacuum furnace at 1450 F (788 °C) for 8 hours followed by furnace cooling at rate faster than 100 F (56 °C) to 1300 F (704 °C) and holding at 1300 F (704 °C) for 8 hours, followed by cooling with Argon purging at a pressure slightly greater than 1 atm to simulate air cooling.

All the processing heat treatments and nominal compositions fall within the composition ranges specified by ATI-Allvac for IN718Plus sheet [Table 5.1b] and plate [Table 5.1c].
<table>
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<th>Mo</th>
<th>Al</th>
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**a. Chemical composition of heat 993J used in our experiments and by [Bergstorm 2005]**

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<td>1.40</td>
<td>0.02</td>
<td>0.05</td>
<td>0.008</td>
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</table>

**b. Chemical composition of IN718Plus sheet material [courtesy: ATI-Allvac]**

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<td>0.02</td>
<td>0.05</td>
<td>0.008</td>
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</table>

**c. Chemical composition of IN718Plus plate material [courtesy: ATI-Allvac]**

Table 5.1: Chemical compositions of IN718Plus alloy used in our tests, reprinted from manufacturer ATI-ALLVAC’s online product specifications [courtesy: http://www.atimetals.com/products/718plus-alloy]
5.1.2 **Metallography:** Samples were cut from the as-received material using a Beuhler Isomet 2000 saw liquid cooling at a speed of 1800 rpm under auto-cut control with Beuhler grinding wheel. Smaller size samples were mounted using a Beuhler cold mount resin solution. Samples were ground on LECO or Beuhler emery cloth from 180, 320, 600, 800 and 1200 emery grit paper manually. Samples were then fine polished on a lapping cloth, using Beuhler Mastermet colloidal silica solution to a mirror finish.

**Grain size:** Metallography was conducted on polished samples for grain size measurements. Two etchants were found to be individually effective in revealing the microstructure

(a) 5% HCl + 1% H$_2$O$_2$ in C$_2$H$_5$OH

(b) 95% HCl + 5% H$_2$O$_2$

at room temperature. Samples were etched for 30 seconds to 1 minute in either of the above freshly prepared solutions, and then examined using a Keyence VHX optical microscope. As-received material was found to have an ASTM grain size of 9.

5.1.3 **Microhardness:** Prior to microhardness measurements, samples were polished to mirror finish using Buehler colloidal silica solution. Microhardness measurements were made using Knoop as well as Vicker’s indenters on a LECO microhardness tester at 500 gm load for 15 seconds on the polished surface.

5.1.4 **Scanning Electron microscopy:** Samples were polished for 30 minutes on lapping cloth with colloidal silica to get better surface finish and minimize the disturbed layer since electron back scattered diffraction is quite sensitive and picks up sub-surface deformation due to any deeper scratches from the prior polishing steps. Absence of texture and preferred orientation was
confirmed through electron back scatter detector (EBSD) mapping and mis-orientation analysis of grain boundaries of as received materials using an FEI XL-30 FEG ESEM with EDAX Pegagus 4040 EDS/EBSD System.

5.1.5 Transmission Electron Microscopy: TEM samples were prepared after cutting thin slices using a diamond saw followed by mechanical grinding to 100 microns and twin jet electrolytic thinning using 10% perchloric acid and methanol solution at 278 K. Transmission electron microscopy was carried out on an analytical Philips CM20 TEM with Gatan CCD, EDAX and nanoprobe mode operating at 200 kV. TEM microstructure is characterized in as received microstructure as well age hardened condition. Since the material is susceptible to notch sensitivity if heat treatment condition is modified, a direct ageing treatment on unhardened material was simulated to examine the microstructure. As this condition could occur during welding when the system is subject to re-solutionizing followed by in service ageing. In particular, the coarsening behavior of the main strengthening precipitates, the γ’ precipitates, is examined to ensure that strength of the system is retained. Further, formation of any new precipitates including the brittle plate shaped η-precipitates by dissolution of γ’ are also studied in detail considering the reports on formation of an hcp phase in the alloy [Cao 2005], and the tendency of any material to be notch sensitive with such sharp precipitates. Finally, nature of dislocations arising in as-received, unaged material is verified against published literature in γ’ strengthened Ni-base superalloys [Kear 1970].
5.2 LSP Treatment and procedures

**# Laser Shock Peening:** A Gen-1 GE LSP system shown in (Figure 5.1) was used with tape as an ablative medium and water as the confining media. Power density employed was 3–12 GW/cm² with a pulse width of 25-30 ns and rise time of 3-5 ns. Beam energy measurements were calibrated using a Scientech laser calorimeter. Pulse waveforms as shown in (Figure 5.2) were acquired using a Tektronix TDS 640, 2 Ghz oscilloscope. The time between two laser shots was 10 seconds. LSP samples were mounted using a MOTOMAN robotic arm that was programmed to move to the next position after each shot. Firing of the laser and the movement of the Motoman robot were controlled by a common program run on the Motoman controller. Programs written for the LSP are provided in Appendix.

**Laser Generation:** A Q-switched high energy Infra-red Nd-Glass laser was used in our tests. The glass rods were water cooled all the time the lasing action is performed due to the high energy of the laser.

**Stage I:** The Laser consists of two initial stages of low energy laser pulse generation of around 0.25J by the Q-switch. Two flashers generate the photons to excite Nd- Glass atoms to higher energy state followed by a population inversion resulting in the lasing action. The q-switch is an electronic switch that changes the polarity at the right moment after the generation of the laser beam in synchronization with the initial flashing of the mirrors to allow only the 20 -30 ns burst of the laser pulse with a sharp rise time (2-6 ns) to pass through to the second stage. Rest of the photon energy is rejected as heat and absorbed by the heat sink (chiller).
Stage II: Before entering the second stage, the original laser beam is split into two identical beams with a 45° - 50% mirror to create two coherent beams. Each of the beams forms one of the sides of the double-side laser peening. Both beams pass to an identical arrangement of second stage photon amplification. The second stage consists of thicker Nd-Glass rods connected to the PFNs. PFNs are energy storage capacitor banks where the electrical energy is stored for conversion to light and to amplify the photon intensity. These banks charge in between every laser pulse and store the energy to be passed to the laser by an electric discharge. The banks are synchronized with the flashing frequency to time the energy discharge at the same moment as laser generation. Since the PFN’s in Gen-1 laser were of the earlier design, the capacitors needed longer time between two charge - discharge cycles. Accordingly, there is a minimum time of 10s between two laser shots. In subsequent laser designs with faster electronics and better cooling, higher frequencies could be achieved. The PFN’s were operated up to 16 J. A discussion on optimum choice of energy is presented in the simulations section later. Best results in LSP were achieved at low energies between 2J – 3J.

Beam delivery system: The laser beams exit the two Nd-glass rods that are charged by the PFNs. These two lasers are directed through identical set of high-energy-rated focusing lenses and deflecting mirrors to give the laser beam a sharp circular profile and focus it to the desired spot size on the sample from opposite sides.

Since the original laser pulse without switching on the PFN’s is of low energy ( \( \approx 0.25 \) J), but of the same overall profile, the low energy stage-I laser output can be used for beam alignment and focusing of the beam delivery system.
Alignment of Laser beam: Laser beams were aligned so that the focus of the beams is at the sample surface on both the sides of the sample. The spot size was chosen between 2.5 mm - 3 mm by taking several shots with photographic laser paper. The beam delivery system was oriented to prevent back reflection, minimize losses and maximize the energy delivery using a combination of optics and deflecting mirrors.

Beam eccentricity: The beam profile was checked for good circularity and uniform energy spread by taking several the low energy shots on the laser sensitive photo-paper and observing uniformity of the burnout pattern. Misalignment and eccentricity were corrected by adjusting the final focusing lens.

Energy calibration: Energy of the beam was calibrated using a Scientech laser calorimeter. The calorimeter was re-calibrated from the factory to check if the readings were correct. The calorimeter was placed in the beam path just after the final beam focusing lens, and in the path of the sample. A 90% attenuator plate was used for energies exceeding the capacity of the calorimeter. For lower energies, the laser beam was allowed to enter the calorimeter directly. The readings from digital readout of the calorimeter were recorded as absolute energy readings.

Waveform acquisition for laser pulse width and rise time: Two Moleclectrons were placed inside on both the sides of left and right beam delivery systems, close to the first deflecting mirrors (but not in their path) as the main laser entered the beam guidance array of mirrors and lenses. These low power moleclectrons are solid state devices capable of picking the laser waveform from the reflected laser light from the first beam deflecting mirrors. This waveform is identical to the final laser profile. This signal was fed to a 1GHz storage oscilloscope (Tektronix TDS 540). Since the total width of the laser pulse is of the order of 25-30 ns and the rise time of
the order of 1-5 ns, a high frequency oscilloscope of minimum 500 MHz is required for displaying the waveform. The oscilloscope was able to acquire 2-channels and display the pulse width and rise time for both the left and right laser beams. Waveforms for every LSP shot could not be recorded due to storage memory limitations on the oscilloscope, but representative waveforms were randomly sampled and stored.

**Energy for every shot:** Since the energy calibration requires the laser to hit the calorimeter, it cannot be carried out along with peening of the sample. But the Molecrtons acquire a reading from the reflected energy from every pulse. Hence, in order to be able to monitor shot-to-shot energy while peening, the molecrtons were connected to a digital readout and were calibrated with the Scientech calorimeter before the start of peening. After the Molecrtons were calibrated, the Scientech calorimeter was removed and sample could be peened.

The data from Molecrtons was acquired via a National Instruments GPIB card set up with Labview by our LSP engineer. Initial set up and bringing the Generation - 1 G.E. laser to functioning status was done by very careful work by a brilliant engineer who was a part of the installation team of original machine at General Electric (GE) before it was brought to UC, Mr. Pat Keeney. Most of the subsequent LSP machine setting and calibrations were carried out by our enthusiastic and cheerful LSP engineer, Mr. James Guines. Programming of the LSP sequence and actual peening on individual samples was carried out by the author with occasional help from our LSP engineer.

**Laser Safety:** Safety is paramount in high energy laser environment. Under no circumstance, the testing was allowed without wearing laser safety glasses from Thorlabs and noise isolating earphones similar to those used in shooting range were used, since each laser shot makes a sound
similar to a high caliber gunshot. Care was taken that only authorized persons were allowed to be in the laser room while laser was in operation. Being an infra-red laser, the laser beam is invisible to the human eye making it nearly impossible to detect. Care was taken to ensure that the beam path was closed, isolated, and the operator protected from stray radiation being reflected off the sample while peening the sample edge by means of shielding during the operation.

**Sample preparation:** Different types of samples were LSP treated during the course of this research. General sample preparation for each type of samples is listed below:

**a. Surface preparation:** LSP coupons were polished prior to LSP treatment to remove any oxide layer generated during prior heat treatment, provide a clean fresh surface for LSP treatment and reduce scatter in post LSP microhardness and XRD measurements that may arise due to unclean and rough surfaces. The samples were washed with acetone and dried. They were then mounted on sample holders using a double-side adhesive tape. The region to be LSP treated was polished using 320, 600, 800 and 1200 grit emery paper under decreasing load from 180 to 30 gm.

**Manual polishing:** Since almost all the samples to be fatigue and fatigue crack growth tested were around 120 - 200 mm long, they could not be mounted on automatic polisher. Hence, they were manually polished as follows:
i) Tensile fatigue samples: were manually polished using 320, 600, 800 and 1200 grit emery paper in the complete gauge section including the shoulders for all the fatigue samples on both sides.

ii) 3PB fatigue crack growth samples: Whole sample surface was manually polished using 320, 600, 800 and 1200 grit emery paper followed by lapping on sloth using Beuhler colloidal silica on both sides to a mirror finish to enable measurements of cracklength using optical microscope. One of the sides was left as polished while the other was etched using either of the two solutions:

(a) 5% HCl + 1% H₂O₂ in C₂H₅OH

(b) 95% HCl + 5% H₂O₂

at room temperature. Samples were etched for 30 seconds to 1 minute in either of the above freshly prepared solutions, to highlight the grain boundaries. Etching enabled us to later image and identify the path followed by fatigue crack with respect to microstructure.

iii) M(T) fatigue crack growth samples: A strip around 30 mm wide in the center of the sample ahead of the central notch was manually polished on both sides of the samples up to 800 grit emery, which was adequate to enable reliable optical verification of crack length.

Automatic polishing: Smaller coupons 15-25 mm long were used to study the effect of LSP energy and residual stresses on the material for optimizing the process to be used in fatigue and fatigue crack growth samples. These samples were small enough to be mountable in the automatic polisher. Along with the coarse polishing mentioned above, samples were polished to final mirror finish using a lapping cloth with Beuhler colloidal silica solution. All steps for the smaller coupons were carried out on a Beuhler automatic polisher with head rotation in counter-
clockwise direction to base rotation with water-cooling at all times. Polisher base and head rotations were between 50 – 200 rpm and 20 - 30 rpm respectively during polishing.

# LSP Samples: Samples were laser peened with different spot sequences, number of shots and energy level depending upon application. LSP treatment was carried out both at UC and at LSP technologies, but all the materials came from the same plates supplied by ATI-Allvac in order to maintain consistency and for comparisons.

# Coupons for LSP optimization: An important concern in LSP is the achievement of through thickness compression. Through thickness compression is important to ensure that crack initiation does not take place in the center of the sample. Also, since the net force on a stationary sample in the absence of external stress is zero, force balance requires that for the amount of compressive residual stresses generated, equal amount of tensile stresses exits outside the LSP-treated region distributed over the remaining sample volume. Accordingly, if LSP compressive residual stresses are not able to penetrate through the sample section, there is a high probability that the mid section will have accumulation of tensile stresses. This tensile stress may not affect surface crack initiation, since it lies far below the surface, but it can aid crack propagation. Once a long crack has been generated, these tensile stresses may lead to unusually fast crack growth. This fast crack growth in the central section of the sample, while the outer surfaces remain slow growing, is called crack tunneling. To prevent tunneling, it is necessary to ensure through thickness compression is achieved in LSP treatment. Since every material has a different response and yield strength, we needed to characterize the depth profile of LSP residual stresses. For depth profile measurement, small coupons of 20 mm x 20 mm are prepared and LSP treated at different energies and different LSP patterns. Subsequently depth profile of LSP stresses is
determined by both destructive (conventional XRD) and non-destructive (Synchrotron XRD, Eigenstrain analysis) described in next chapter. Here we describe the coupons and their LSP treatment:

**a. Single spot LSP coupons:** Effect of double side single spot LSP of energies 4, 6, 8 and 10J was studied on 10 mm x 20 mm x 1.69 mm coupon. The sample was polished to mirror finish as described above and single shot LSP was conducted using a 3M tape overlay as absorbing media and water as confining media. The LSP beam hit the sample from both sides to prevent bending. Sample was held in position by a restraining from one side while keeping the other end free, like a cantilever, in the Motoman robotic arm. Each spot was hit with two laser shots in the same location, the burnt out tape overlay was changed manually between the two shots without changing the sample position.

**# Multiple spot overlap pattern LSP:** Since the samples to be peened in industrial setup are bigger in size, a multiple spot laser patter is used to cover the area to be peened. Hence, it is important to identify the depth profile of the residual stresses using the multiple spot pattern compared to the single spot. LSP was conducted on representative patch patterns with 50% overlap. 25 mm x 25 mm square, 5 mm thick coupons were cut from IN718P plates by Wire electro-discharge machine (an Accutex Wire-EDM). Double side laser peening was done in a 10 x 10 mm region with 50% overlap. After the peening, depth profilometry of the peened surface was mapped using VEECO optical interferometric profilometer (Figure 5.3) and residual stress measurements carried out using conventional XRD described in the next section.
Fatigue and Fatigue crack growth test Samples: Several geometries of fatigue and fatigue crack growth samples were attempted. The detailed specifications are given section 6.4 on fatigue. The LSP treatment is described here:

I) Tensile fatigue samples: flat tensile samples of two geometries were LSP treated and tested.

A) Design 1: The first design involved a flat tensile sample with a uniform gauge section of 10 mm x 90 mm x 1.69 mm. The total sample length was 120 mm. Samples were peened double side multiple spot LSP treatment from both the sides along the length of the gauge section. Samples were tested at 923K, but some of them failed in the region outside the furnace. This was attributed to the yield point strengthening of superalloys and possible grip effects near the ends. Out of furnace failure caused difficulty in being able to compare the high temperature tests with low temperature. Further, the number of good LSP samples obtained with overall surface compression was low in this patch design. Hence, this design was not continued further.

B) Design 2: Second design was used in most of the study. It consisted of flat tensile samples 110 mm long with a reduced LSP treated gauge length of 10 mm long a uniform section of 1.69 mm x 1 mm that allowed complete coverage by a single row of multiple LSP shots with 50% overlap. Smaller gauge length ensured that complete gauge section remained inside the 30mm furnace hot zone of the MTS 659 furnace, as some samples failed outside the hot zone as described above due to yield anomaly of this alloy and probable grip effects. Based on experience on the first design, time taken to peen, and the need to be able to get a reasonable number of good samples for subsequent fatigue tests despite shot-to-shot scatter in LSP energy being observed in the laser, it was decided to reduce the patch size. Better success was achieved in getting high percentage of usable samples with overall compression with these samples. With
indications of improvement from first fatigue tests of these samples, detailed LSDYNA simulations were carried out on this geometry, to optimize the LSP conditions and peen multiple samples required to generate the full S-N curve both at room and high temperature in LSP treated and untreated materials.

C) 3PB fatigue samples: Small rectangular bar geometries (6.62-7.02) mm high x (2.70-2.75) mm thick x (32.0-32.05) mm long of 3PB samples were polished, as described above and tested at room temperature after double side multiple spot LSP treatment from both the sides. LSP treatment involved mounting the samples in a single side restraint while keeping the other end free as a cantilever and allowing the laser to hit from both sides simultaneously. The Motoman robot moved the sample between shots to the next position. The shot-to-shot overlap was 50% but the shots were staggered in multiple sequences to allow distance between two successive laser shots on the same tape to be 3 mm apart. This ensured that un-burnt tape was available for the next shot. After one sequence of shots was over, the tape was changed and the whole sequence shifted by 50% spot diameter and repeated. This sequence was repeated, until the overlap reached the next spot position ensuring complete coverage.

These samples were the first batch of IN718Plus sheet material, were heat treated to a notch sensitive condition by heat treating at 923 K – 1023 K up to 500 h without a hardening treatment, to check the response of non-standard heat treatment on LSP which may be encountered during repair welding and or thermal operation transients of superalloy components. Fatigue life was found to be lower for these tests than the untreated material.

II) 3PB fatigue crack growth samples: Two geometries of 3PB samples were tested. The first geometry was similar to the small 3PB bar geometry used with non-standard age as described
above, the other was a longer span 3PB integral notch geometry used for overload study with single spot LSP.

A) **Small 3PB fatigue crack growth samples:** Small rectangular bar geometries 8 mm high x (2.70-2.75) mm thick x (32.0-32.05) mm long of 3PB samples with a 2 mm notch created with a diamond saw were polished as described above, and tested at room temperature after double side multiple spot LSP treatment from both the sides. LSP treatment involved mounting the samples in a single side restraint and keeping the other end free, as a cantilever and allowing the laser to hit from both sides simultaneously. The Motoman robot moved the sample between two shots to the next position. The shot-to-shot overlap was 50% but the shots were staggered in multiple sequences to allow distance between two successive laser shots on the same tape to be 3 mm apart. This ensured that un-burnt tape was available for the next shot. After one sequence of shots was over, the tape was changed and the whole sequence shifted by 50% spot diameter and repeated. This was sequence was repeated until the overlap reached the next spot position ensuring complete coverage.

A) **Long span 3PB overload fatigue crack growth samples:** Longer rectangular bar geometries 12 mm high x (2.70-2.75) mm thick x (68-68.1) mm long of 3PB samples with a (3.0-3.1) mm wire cut notch were polished as described above and tested at room temperature in fatigue overload. Samples were double side single spot double layer LSP treated from both the sides at the crack tip overload plastic zone. LSP treatment involved mounting the samples in a single side restraint and keeping the other end free as a cantilever and allowing the laser to hit from both sides simultaneously. The Motoman robot held the sample stationary between two overlapping
shots on the same next position. The shot-to-shot overlap was 100% and the tape overlay was changed between the shots to ensure that un-burnt tape was available for the next shot.

III) M(T) fatigue crack growth samples: M(T) samples 25.5 mm wide x 120 mm long and 1.6 mm thick with a 4 mm fatigue crack in the center of the sample were LSP treated on polished section as described above on both sides. A 10 mm x10 mm 50% overlap LSP patch used in the depth profile coupon described earlier was used in this treatment as well.

LSP treatment involved mounting the samples in a single side restraint and keeping the other end free as a cantilever and allowing the laser to hit from both sides simultaneously. The motorman robot moved the sample between shots to the next position. The shot-to-shot overlap was 50% but the shots were staggered in multiple sequences to allow distance between two successive laser shots on the same tape to be 3 mm apart. After one sequence was over, the tape was changed and the whole sequence shifted by 50% spot diameter and repeated. This was done till the overlap reached the next shot ensuring complete coverage. The process was repeated twice to get double layer coverage at 6J energy.
Figure 5.1: LSP machine showing the sample being treated in inset
Figure 5.2: Laser pulse waveform monitoring by oscilloscope
Figure 5.3: Depth profile of LSP patch
5.3 Post-LSP and pre-fatigue characterization:

The primary parameter defining the success of an LSP treatment are the residual compressive stresses and their magnitude and depth from the sample surface. Hence, residual stress characterization was carried out by different techniques. Apart from residual stress, it is important to see the condition of the LSP surface and the surface deformation, for this VEECO interferometric profilometry was employed to measure the depth profile of surface topography, as well as the edge distortions for select coupons. Profilometry was complimented by examining the surface with scanning electron microscopy. Finally, it is important to be able to find a quick estimate of the effect of LSP on mechanical behavior of the alloy. Hence, microhardness measurements were carried out on select samples to map the change in hardness across the LSP treated and adjacent regions. These techniques followed are described below in detail:

I) Residual stress measurements: both destructive and non-destructive residual stress measurements were carried out using XRD.

**Conventional X-Ray:** As XRD sources in lab like Cu Kα1 (1.52 A), Mn Kα1 (2.1 A), Cr Kα1 (2.28A) are of lower intensities of the order of $10^{10}$ photons/mrad$^2$/mm$^2$/0.1% on axis brilliance as well lower energy of around 10 KeV [Cullity & Stock, 2001], their penetration depth in the material is limited to a few microns. Hence, any measurements of residual stress are limited to a few microns near the surface. Accordingly, to carry out a depth profile measurement using the conventional X-rays one needs to remove the material physically layer by layer from the sample surface and take X–ray measurements at each depth. Layer removal makes the technique destructive in nature.
**Synchrotron X-Ray:** On the other hand using a powerful high intensity, high energy source like the synchrotron X-ray allows one to penetrate even a 5 mm thick sample to get a Laue transmission type of pattern for single crystals and diffraction rings for polycrystals. Since transmission method does not require layer removal, it is a more representative and in-situ measure of residual stress distribution inside the material, and is also non-destructive in nature. We used the brightest X-ray source in the Northern hemisphere, the Advanced Photon Source (APS) at Argonne National Lab, Chicago to characterize our samples using this method. The intensity at APS is of the order of $10^{15}$ photons/mrad²/mm²/0.1% on axis brilliance [Cullity & Stock, 2001] and the photon energy is 114-115 KeV.

**# APS Synchrotron samples:**

Residual strain characterization using synchrotron X-ray diffraction (SXRD) on 5 mm and 1 mm thick samples of 30 x 10 mm area double side peened by LSP Technologies was conducted, at the Advanced Photon Source, Argonne National Lab. These samples were characterized at APS in as-peened condition and then the same samples were heat treated for 0.1 hour at 923K followed by 1h at 923K followed by 1h at 1023K. SXRD measurements were made after each heat treatment to identify the effect of heat treatment on residual lattice strains. The SXRD data acquired was analyzed using Matlab codes for residual stress analysis written by Dr. Uhlrich Lienert and Dr. John Almer. X-ray Beamlines under Dr. Leinert at Sector - 1 and Dr. Yang Ren at Sector – 11 at 114 KeV were used for SXRD characterization in two visits.
**LXRD measurements:** Since getting access to APS beam time is at a premium, finding a quick alternative to reliable measurements of residual stress in-lab using conventional X-rays was important. Residual stress validation is required since our laser had a high shot-to-shot variation in energy, and it was important to ensure if this variation did not create any regions of tensile stress on the surface. Hence, following the LSP treatment all samples were characterized for surface residual stress to ensure that surface compression was achieved. Residuals stress measurement step was consistently used as a quality control check before running the fatigue tests since any presence of tensile stresses enhanced crack initiation and reduced the life from unpeened condition. A dedicated line focus XRD (LXRD) by Proto that gave quick results on residual stress measurement was used for this purpose. Bulk of residual stress measurements were carried out using the dedicated Proto LXRD X-Ray system. The system is based on careful peak profiling and peak shift measurements for the most strain sensitive diffraction peaks in the target material. For IN718+ the (311) peak is known to be sensitive for residual stress detection. The measurements were carried out using Cu-Kα (1.54 Å) and Mn-Kα (2.1 Å). Mn radiation was found to be more sensitive for residual stress measurements using L-XRD compared to Cu-Kα radiation. Prior to measurements, calibration of system was done with manufacturer supplied coupons of known residual stress values.

# Post LSP Heat Treatment and Residual stress relaxation

LSP treated coupons used for residual stress relaxation were covered with sacrificial Ta- foil and vacuum sealed in quartz tube with Ar purging. These samples were then heat treated at 973K for 1 hr and 24 hr followed by air cooling.
# Nanoindentation microhardness: nanoindentation microhardness was carried out on LSP treated samples single dimple peened at 4, 6, 8 and 10 J LSP energy to detect any changes in surface-hardness across the region of impact. Vicker’s microhardness was measured using a Berkovich indentor on a CSM instruments nanoindentor. A line scan of successive programmed indents was carried out across the region of impact. The indent was optically visualized and found to be around 5 µm diameter on top surface. Spacing between two successive indentations was 100 µm to exclude the effects of residual elastic strains from preceding indentation. The results are discussed in section 6.2.

5.4 Fatigue and fatigue crack growth test samples and characterization:

Both fatigue and fatigue crack growth tests were conducted on the LSP treated samples of different geometries described above. Since LSP machine had a slow peening rate and high rejection of samples due to shot to shot variations, initially select tests were conducted as a pilot study to see if LSP was improving the fatigue or fatigue crack growth behavior for each geometry. In case an improvement was seen in these initial tests, further samples were peened and tests were carried out to validate the improvements and generate the full fatigue curve at both room temperature as well as high temperature. Tests were conducted on an MTS 810 servo-hydraulic machine with computer control and equipped with an MTS 653 high temperature furnace (Figure 5.4). Some of the typical samples tested are shown (Figure 5.5).
5.4.1. S-N Curve fatigue:

A) Fatigue: 298K

Constant stress tests asymmetric tension-tension at R=0.1 were carried out in 3-point bend geometry as well as flat tensile geometry at room temperature on an MTS 810 test system with computerized data acquisition and control at frequency from 15 to 30 Hz in both peened and unpeened conditions. Testing frequency was 25 -30 Hz with a sinusoidal waveform.

B) Fatigue: 923K

High temperature fatigue rack growth (FCG) tests were carried out on M(T) geometry with an MTS 653 furnace equipped with Eurotherm 1240 controller. Elastic unloading compliance was measured in select cases using MTS high temperature extensometer along with the potential drop signals using a lock-in amplifier based ACPD system. Tests were carried out at 10-25 Hz frequency with a sinusoidal loading. Load ratio was kept constant at R = 0.1

Monotonic yield strength was evaluated before starting the fatigue tests. Run-out was considered between 1 to 10 million cycles depending upon the test.

5.4.2 Fatigue Crack growth

Constant amplitude fatigue crack growth tests were carried on MTS 810 servo-hydraulic using a lock in amplifier based alternating current potential drop (ACPD) system for crack length measurements. The crack length was also physically calibrated before and after the test with optical measurements under a metallurgical microscope.
A. Pre test: Room temperature FCG tests were carried on 3-PB as well as M(T) geometries in peened and unpeened conditions. Elastic unloading compliance was measured in select cases using MTS 620E room temperature extensometer concurrent with the ACPD measurement. Following specimen geometries were used:

i) M(T) specimen: Samples were prepared as per ASTM e647-00 specification (Figure 5.6).

Pre-cracking: was conducted using a stress intensity factor (K)-decreasing technique to obtain sharp pre-crack with low K range (ΔK). Pre-crack size on the front and back sides of the specimen were within 0.025 times Width (W). Pre-crack size was measured optically using a metallurgical microscope. Average of crack-length on both sides of the sample was used in all calculations of growth rate and K. ΔK was calculated as per the following equation [ASTM e647-00]:

\[
R = 0.1 \\
\Delta P = P_{\text{max}} - P_{\text{min}} \\
\Delta K = \frac{\Delta P}{B} \sqrt{\frac{\pi \alpha}{W}} \sec \frac{\pi \alpha}{2} \\
R = \frac{P_{\text{min}}}{P_{\text{max}}}
\]  

Where \(\alpha = 2a/W\). Above expression was used for up to \(\alpha < 0.95\).

\(a = \text{the crack size}\) \(B = \text{the specimen thickness}\)

\(W = \text{the specimen width}\) \(P_{\text{max}} = \text{maximum load}\) \(P_{\text{min}} = \text{minimum load}\)

\(R = \text{stress ratio}\) \(\Delta K = \text{stress intensity factor range}\)
**Grips:** samples were gripped using MTS hydraulic grips. Loads on the grips were kept adequate to ensure there was no slipping of the samples. This was confirmed by ensuring there were no slip marks in the gripped section of the sample after test completion. The gripping force was not kept excessively high to prevent failure at the grips.

*Figure 5.4: MTS810 HT testing*
Figure 5.5: Some Fatigue and FCG samples
Figure 5.6: M(T) sample geometry, reprinted with permission [ASTM E647-00]
B. FCG Test

Compliance: measurements were carried out using an MTS crack opening displacement (COD) gauges both at room (MTS 632.02e) and high temperature (MTS 632.53). The compliance calculations were not used in crack length estimation. They were used to detect crack closure and measure elastic unloading response of the specimen.

ii) 3-point Bend: compression-compression FCG was carried out on 3-PB samples prepared as per ASTM E399 (Figure 5.7). To ensure specimen meets plain strain conditions, it was verified:

\[
B > \frac{2.5(K_{lc})^2}{(\sigma_{ys})^2}
\]  

(5)

Where \( B = \text{specimen thickness} \); \( K_{lc} = \text{plain strain fracture toughness} \); \( \sigma_{ys} = \text{yield stress} \)

Crack starter notch: a straight through crack starter notch was prepared by wire electro-discharge machining.

Grips: samples were gripped using MTS 3-point bend grips. Slight compressive loads on the grips were maintained to prevent slipping of the samples during fatigue.

Pre-cracking: was conducted using a K-decreasing technique to obtain sharp pre-crack with low \( \Delta K \). Pre-crack size on the front and back side of the specimen were within 0.025W. Pre-crack size was measured optically using a metallurgical microscope. Average of crack-length on both sides of the sample was used in all calculations of growth rate and K.
Compliance: measurements were carried out using an MTS COD gauge both at room with a travel from 5 mm – 7.5 mm.

Fatigue crack growth: tests were performed on an MTS 810 servo-hydraulic system at room temperature in the air at 15-30 Hz cycling frequency with a sine wave loading. Load ratio $R = \frac{P_{\text{min}}}{P_{\text{max}}}$ was maintained at $R = 0.1$. Crack size was measured optically on both sides of the samples at beginning and end of testing, using a metallurgical microscope and was found to be within 10% of each other. Average of crack length on both sides of the sample is used.

A montage of several optical images was joined to get the total crack length as regular intervals. Samples were etched and polished prior to testing to be able to find the crack path. Typical long crack montage in IN718Plus for room temperature test is shown (Figure 5.8).
Figure 5.8: Long crack image created by montage
ΔK was calculated as per the following equation [ASTM e1820]:

\[
K = \frac{PS}{BW^{3/2}} f\left(\frac{a}{W}\right)
\]

\[
f\left(\frac{a}{w}\right) = \frac{3(a/W)^{1/2}[1.99 - (a/W)(1-a/W)(2.15 - 3.93a/W + 2.7(a/W)^2)]}{2(1 + 2a/W)(1-a/W)^{3/2}}
\]

Where \(P\) = load, kN

\(a\) = the crack size \(B\) = the specimen thickness \(W\) = the specimen width \(S\) = the specimen span between loading pins = \(4W \pm 0.2W\)

C) **Fatigue crack growth: crack closure**

Crack closure is important to find the stress intensity factor range that determines the fatigue crack growth behavior of an alloy [Elber 1970 Budiansky & Hutchinson 1978, Suresh & Ritchie 1984]. Crack closure was measured in select samples with and without LSP.

**5.4.3 Fatigue crack growth: overload effect**

Real-life loading, is not identical to controlled amplitude tests conducted in lab, and often consists of overloads and underloads in a spectrum of loading. Such loads are common in aircraft fatigue especially in turbulent flying conditions or hard landings and slow takeoffs. Hence, overloads characterization is important. Single 100% overload tests were conducted on the fatigue samples to identify the effect on fatigue. LXR measurement of residual stress relaxation
in the overload samples was also conducted to find the role of LSP and overload on crack tip residual stresses.

5.5 Post Fatigue fractography and analysis:

A. SEM fractography: SEM fractography was conducted in samples after testing. It provides information to validate that there is no tunneling of the crack front requiring curvature correction. Slight curvature at high $K_{\text{max}}$ values is considered acceptable. Crack initiation sites and fatigue striations were identified as well as the extent of damage zone below the LSP surface is examined. Crystallographic nature of fatigue at low temperature and indications of oxidation at high temperatures were also observed to be in agreement with those reported in literature.

B. TEM characterization: samples were prepared from the fatigue tested materials from the gauge section of the wider gauge samples, by cutting the fatigued portion of the section with a Beuhler Isomet 2000 wheel saw with water cooling and autocut control at 1800-1950 rpm. Cut sections were carefully hand ground using 180 and 320 grit emery paper with water cooling around 100 $\mu$m thickness. The thin foils were cleaned of any grinding debris and mounting glue residue using acetone and air dried. 3 mm discs were punched using a mechanical punch from these thin foils. The thin foils were then electro-polished in Fischione electrojet polisher with 10% perchloric acid and 90% Ethanol solution at sub-zero temperatures. After electrojet thinning the foils were cleaned in methanol and water and dried. They were then examined in the Philips CM20 electron microscope for the underlying substructure.
A) **Single dimple LSP:** Thin foils were prepared just below the single shot LSP sample and thinned from one side only to reach as close as possible to the LSP treated surface. Observations of sudden dislocation density increase in the LSP treated region were made. Results are discussed in section (6.5.4)

B) **High temperature fatigue:** Thin foils were prepared from the un-failed section of the fatigued sample at tested at 923K. Observations of multiple slip bands in fatigue were made, results are discussed in section (6.5.4).

C) **Room temperature fatigue:** Thin foils prepared from room temperature fatigue tested samples as well as the region of failure in high temperature fatigue samples lying outside the furnace were characterized by TEM. Observations of slip bands were made; results are discussed in section (6.5.4).

D) **Unpeened, unfatigued, age hardened alloy:** thin foils were prepared from manufacturer recommended age hardened samples, without any fatigue and LSP treatment. Observations of lower dislocation density and uniformly distributed γ’ substructure were made, results are discussed in section (6.5.4).
E) **Unpeened, unfatigued, non-standard aged samples:** thin foils were prepared from non-standard direct aged samples, without any fatigue and LSP treatment. Observations of lower dislocation density, coarsening of $\gamma'$ substructure and formation of sharp $\eta$-$\text{Ni}_3(\text{Ti, Al})$ plates were made, results are discussed in section (6.5).

F) **Unpeened, unfatigued, as received samples:** thin foils were prepared from as received hot rolled plates supplied by the manufacturer, without any fatigue and LSP treatment. Observations of intermediate dislocation density, few prior $\gamma'$ substructure and grain boundary $\delta$-phase were made, results are discussed in section (6.5).
Chapter 6. Results and Discussion

6.1 IN718Plus superalloy: The results on IN718Plus superalloy microstructure are presented. The as received plates were characterized for grain size and texture using optical microscopy and electron back scatter diffraction (EBSD) technique. Surface of as rolled plates was also examined since fatigue cracks are quite sensitive to surface conditions. Underlying microstructure of as received, non-standard aged and standard aged microstructure was characterized using TEM and microhardness.

6.1.1 As Received: Microstructure

Since the IN718P plates were subjected to sub-solvus hot rolling, the microstructure based on TTT curves [Cao and Kennedy 2006] is expected to contain varying amounts of δ-Ni$_3$Nb phase particles at grain boundaries. Our observations of as received material show equiaxed grains. EBSD orientation microscopy confirms the material is isotropic and has a grain size of 24 microns (Figure 6.1).

Figure 6.2 shows TEM images of a typical as received microstructure with δ-phase that is often observed at grain boundaries in plate shaped morphology, and which acts as a grain refiner during hot forming. Figure 6.3 shows the γ matrix containing significant dislocation density in as received unaged alloy as well as presence of prior coarse γ’.

6.1.2 As Received: Mechanical properties: As received material was in hot rolled condition as described. Prior grain boundary δ-phase is seen in the microstructure as well as some
dislocations intersecting the prior coarse $\gamma'$. The pairing of dislocations is found to be similar to those observed in ordered superalloys.

6.1.2.1 Dislocation and ageing: TEM, SEM

Dislocations in IN718Plus typically occur in pairs where single dislocation movement destroys the ordering and creates a high energy anti-phase boundary, that gets eliminated with the passage of second dislocation which restores the order. Strengthening in IN718Plus arises from intersection of these dislocation pairs with $\gamma'$ precipitates in the matrix. $\gamma'$ being coherent with the fcc $\gamma$ matrix, require the dislocations to pass through them offering lattice resistance due to associated coherency strains. This strengthening is further compounded by creation of disorder and formation of antiphase boundaries mentioned above. Figure 6.3 shows dislocation pairs in as received matrix. The blue arrows show the dislocation pairs and the red arrow show the $\gamma'$ precipitates. Figure 6.4 show the pair dislocations shearing through a $\gamma'$ precipitate (marked with arrow). While one of the dislocation has passed through the precipitate and sheared it, the other dislocation is beginning to intersect the precipitate to restore the order. This behavior is in agreement with $\gamma'$ strengthened Ni-base-superalloys [Shen 2007] where the plastic deformation takes place by the shearing of $\gamma'$ by two $\frac{1}{2}[110]$ dislocations with the introduction and annihilation of anti-phase domain boundaries. In some instances and introduction of superlattice stacking fault has also been observed and follows the mechanisms proposed by [Kear 1974]. The change in mechanism is based on temperature as planar glide on (111) of paired $1/2 < 110 >$ dislocations at low temperatures and $\gamma'$ cutting by paired $1/2 < 110 >$ in climb configurations at high temperature.
The \( \frac{a}{3}<112> \) stacking fault modes of shear are of secondary importance, except in primary creep at intermediate temperatures where the motion of superlattice intrinsic/extrinsic stacking fault pairs is rate controlling [Kear 1974]. We find our observations to confirm the cutting of precipitates as proposed in the above mechanism.
Figure 6.2: TEM Bright field of unaged IN718Plus showing delta precipitates
Figure 6.3: Dislocation pairs in as-received 718Plus and $\gamma'$ precipitates
Figure 6.4: Dislocation pair shearing a γ' precipitate
6.1.3 Dislocation dissociation: Dislocation imaging has been done under two-beam imaging condition in the TEM. The dislocations slip systems are well studied in these ordered systems, and typical two beam images used in Burgers vector analysis are presented in Figure 6.5. The evolution of dislocation density and dislocation cell size has been reported in LSP treated metals with expected dislocation density of the order of $10^{14}$ m$^{-2}$ for LSP [Ding 2012]. Our observations show reduction in dislocation density upon service ageing in virgin samples. This dislocation recovery is important for dynamic precipitation and increase in strength observed at higher temperatures seen in our fatigue results.

Reduction in dislocation density at higher temperatures also contributes to understanding the novel practice of warm LSP that involves pre-heating the target resulting in dynamic aging of the microstructure giving precipitates surrounded by dislocations.

TEM images of hot rolled IN718Plus (Figure 6.6) show dislocation dissociation in $\gamma'$ precipitates. The $\gamma'$ precipitates seen were formed since the hot rolling for IN718Plus is done sub-solvus. This allows of $\gamma'$ precipitates to nucleate and grow that can act as grain refiners along with the g.b. $\delta$-phase. The confirmation of presence of $\gamma'$ is done by (001 zone) (Figure 6.7) and (011 zone) (Figure 6.8) selected area diffraction patterns of $\gamma'$, corresponding to the images containing the spherical precipitates in (Figures 6.5) and (Figure 6.9) respectively. The diffraction patterns clearly show the $\gamma'$ superlattice reflections marked with an arrow in the Figures. The (011) pattern is seen to match well with the simulated diffraction pattern using Desktop Microscopist (Figure 6.10).
Dislocations are generated and dynamically recovered during the hot rolling, but due to the ordered L1_2 structure, they dissociate into partials to minimize the energy inside the γ’ precipitates (Figure 6.6). Another noticeable point in the dislocation precipitate interaction is that most of the dislocations can be seen to pass through the γ’ and not around them. This indicates that precipitate shearing is the primary mechanism operative rather than precipitate looping.
Figure 6.5: 2-beam dislocation images for different scattering vectors in hot rolled IN718Plus
Figure 6.6: Dislocation dissociation into partials in a υ' precipitate
Figure 6.7: 100 γ // 100 γ' precipitate Orientation relationship
Figure 6.8: SADP from figure of round precipitate, arrow points to superlattice reflections

Figure 6.9: Dislocation pairs and γ' precipitate in IN718Plus
Figure 6.10: simulated diffraction pattern of 011 γ'// 011 γ showing identical pattern
6.1.4 Detriment of brittle precipitates: another important feature of IN718Plus precipitation, are the plate shaped precipitates that can form upon non-standard thermal ageing for long durations (Figure 6.11). These precipitates were identified to be of $\eta$-Ni$_3$(Ti, Al, Nb) type and form by a faulting mechanism from $\gamma'$ described as earlier. They are associated with precipitate-free zones adjacent to them [Chaswal, 2010]. Lack of precipitates, makes the region adjacent to them weaker compared to the matrix. Since they are also brittle, they can fracture under laser shock impact, creating potential stress concentration regions and reducing the fracture and fatigue resistance of the alloy. Since these precipitates are not seen under manufacturers recommended heat treatment [Cao and Kennedy 2006], and occur only over longer ageing durations, it is important to follow the correct heat treatment under LSP application. Our fatigue results to this effect follow in section 6.5

Effect of thermal ageing: Effect of thermal ageing and coarsening kinetics of $\gamma'$ is important towards high temperature applications of IN718Plus, and was studied at 923K, 973K and 1023K up tp 1000 h of ageing. Coarsening was found to follow LSW kinetics (Figure 6.12). Thermal ageing will also be of significance in non-standard ageing conditions like welding where estimations of strength of the weld HAZ zone is important.

An activation energy analysis was also carried based on JMA kinetics and is a part of a preceding work in the same material [V Chaswal, MS Thesis 2011].
Figure 6.11750C /500h aged IN718Plus, non-standard age forms brittle η-Ni3Ti precipitates (red arrows) and weak precipitate free zone (white arrows)
Figure 6.12: Coarsening behavior of $\gamma'$ following LSW kinetics [V Chaswal, 2011]

Activation Energy for $\gamma'$ Coarsening

$E_a = 327.595 \text{ kJ/mole}$
6.1.5 Tensile and 3PB compression

Monotonic uniaxial loading tests were carried out on IN718Plus samples to obtain baseline strength data, which is required to plan fatigue tests.

a) 3PB compression: Tests were conducted in 3-point bend samples corresponding to subsequent fatigue geometries. Yield load is found using the 0.2% offset technique (Figure 6.13). Corresponding stress is calculated using:

\[
\sigma = \frac{3PS}{2bh^2}
\]  

Where \( S = \) span = 28 mm, \( P = \) load, N

\( b = \) thickness = 2.75 mm

\( h = \) width = 6.67 mm

gives a surface stress value of 1956 MPa at gross yielding.

b) Uniaxial tension: Tensile tests are conducted on an MTS 810.21 machine on flat tensile samples used for SN-curve fatigue with a gauge length of 60mm and a cross sectional area of 1.69mm x 1.0 mm. Yield strength of IN718Plus alloy found using 0.2 % offset technique is 720 MPa (Figure 6.14).

Stress values under a bend test always are higher and cannot be directly equated to monotonic yield values [Lipetzky 1996] due the different nature of yielding in the two tests. More details on the bilinear bend test behavior are discussed in the following sections 6.5.3 on 3PB fatigue.
Figure 6.13: 3PB compression stress strain behavior

Monotonic 3PB compression
IN 718P, Aged 1023K/50h
Span 28, thickness 2.75,
Width 6.67

Stress, MPa

-3000 -2500 -2000 -1500 -1000 -500 0
-0.16 -0.14 -0.12 -0.10 -0.08 -0.06 -0.04 -0.02 0 0.02

Strain

-1956 MPa
Figure 6.14: Tensile stress strain behavior
6.2 Laser Shock Peening

The efficiency of LSP in generation of residual stresses is dependent on the type of plasma generated on the impact of laser with target surface. This plasma depends upon several parameters e.g. pulse power density, duration, rise time, overlay absorption, water transmittance, material properties. Since the area of the laser spot can be varied while keeping the energy constant, it becomes important to characterize the incident energy in terms of pulse power density by normalizing with the area and the pulse width. This is calculated for a circular laser beam of spot diameter ‘d’ as:

\[
\text{Power Density, } P = \frac{4E}{\pi D^2 t} \text{ GW/cm}^2
\]  

(8)

Where \( E = \text{incident energy, Joules,} \)

\( D = \text{laser spot size, mm; } t = \text{laser pulse width, ns} \)

Pulse width, t, is measured from the oscilloscope readout, laser energy from molelectron readouts calibrated with Scientech calorimeter, and the laser spot size is measured before high energy laser peening by taking low energy shots on the photosensitive laser paper. Details are described in experimental procedures, Chapter 5. As we acquire LSP waveforms (Figure 6.15) using a storage oscilloscope and calibrate the energy with a laser calorimeter, we have a good estimate of the incident energy per laser pulse. What remains to be understood is how much of the incident energy gets converted into useful energy absorbed by the material in the form of mechanical shock wave.
Recent analysis [Wu 2011] finds a reasonable coupling coefficient match with the experimentally observed pressure profile using around 30% energy conversion of the process similar to literature [Berthe 1997]. Berthe et al also report no increase in pressure with power density higher than 10 GW, due to parasitic losses and breakdown in water confinement. While initially, we spent around 2 years to increase the energy density to get the highest levels possible for greater compression depth through thickness, later comparison with simulations help us decide that lower energy values of 2-3J are where the LSP behavior was best optimized for fatigue.
6.2.1 Laser material interaction: laser material interaction is a complex phenomena with the plasma formation as well and the shock transients. We try to summarize observation of our interest with a limited treatment of this field. As will be discussed later, the degradation of surface due to LSP is hypothesised to not have a significant effect on fatigue crack nucleation and growth due to the available compressive stress field as well as the nature of stress concentration at small porosities [Timoshenko and Goodier 1970]. Though this does not rule out the importance of surface condition for other areas of research like corrosion, more important at this stage of our discussion, is the interaction of the surface layer in generating the plasma and compressive residual stresses.

6.2.1.1 Effect of ablative media

3M type 471 black tape is used as the ablative media. It absorbs the LSP energy and gives rise to a plasma that results in the creation of a shock wave. Loomis et al [Loomis 2005] use Lindl equation to estimate the pressure (P) generated on sample surface as:

\[ P = 40 \left( \frac{I}{\lambda} \right)^{2/3} \]  

(9)

Where \( I \) is the incident radiation flux \( 1 \times 10^{15} \) W/cm\(^2\), \( \lambda \) is the wavelength of laser = 1100 nm. They suggest around 15% of the laser energy is lost during the impact due to scattering. Since they used direct peening without an overlay of NiAl bicrystals, the losses may be higher than those in our testing. In our tests the sample was covered with a black ablative tape. Using a coupling constant for LSP to be 90%, we get:
For 3 mm spot size and 2J laser energy and a pulse width of 30 ns,

\[ P = \frac{2J}{(30 \times 10^{-9} s) \pi (3.0 \times 10^{-1} cm)^2} = 2.359 GW / cm^2 \]

\[ P_{\text{effective}} = P \times 0.9 = 2.15 GW/cm^2 \]

### 6.2.1.2 LSP surface: morphology

Surface morphology is important for understanding fatigue behavior, as the crack initiation takes normally place at a free surface. The microstructure and mechanical properties of LSP are reported to be altered up to ≈100 μm in depth [Chu 1999]. Typical optical, SEM and interferometric surface profile of LSP surface are presented (Figures 6.16, 6.17 and 6.18 respectively). While the optical and interferometric profiles show uniform LSP patch, the SEM surface shows a local recast layer when peened without a tape overlay. A recast layer forms due to local melting and re-solidification of the metal surface in the absence of tape overlay. The recast layer regions often have micro-cracks as shown (Figure 6.17) and can grow into a macro-crack, though normally compressive stresses associated with the outer surface prevent the same. The depth of LSP dimples is around 20 -30 microns as seen from the interferometric scan and is a direct measure of the plastic deformation of the surface. Depth increases with increasing LSP energy density as will be seen in section 6.2.2 indicating higher cold work associated with the increasing energies.
Figure 6.16: Optical profile of LSP surface
Figure 6.17: SEM profile of LSP surface region peened without tape overlay
Figure 6.18: VEECO profile of LSP surface
6.2.2 **Effect of laser shot energy:** Effect of laser energy was studied on single-spot laser patterns with energy ranging from 2J to 8J. The surface deformation, which is a direct measure of the damage, was characterized using interferometric profilometry, and is presented (Figure 6.19). At higher energy, laser impact produced greater surface deformation and cold work. Our simulations suggested that higher energies produced greater tensile stresses in the center of sample. Hence, we found the energies of 2J – 3J to be optimum for producing good compressive residual stresses with tensile stresses in control. It was reported that for two laser generated shock wave pressures of 3.5 and 6 GPa, shock pressure increased the hardness with both one and five repetitions. 3.5 GPa shock wave only increased the hardness with one repetition and not with five wave pressure [Montross 2001], this allowed us to conclude that additional repetition will not create additional tensile stresses in the material but can be considered more as a homogenization step. Hence, we employed double layer of laser shots to minimize the local inhomogeneties that may arise due to instrumental shot-to-shot variation and limit fatigue life by creating local hot spots.
Figure 6.19: VEECO profilometry of LSP impact at different energies
6.2.2.1 Nano indentation: Nano-indentor microhardness measurements were carried on the laser impacted surface after laser shock peening at different energies. Results are presented (Figure 6.20) which show a direct proportionality between laser energy and increase in hardness, in agreement with the reports on LSP energy vs. hardness measurement carried on Hadfield Mn steel [Chu 1995] where up to 130% increase in hardness were reported. The reported high hardness values were due to the formation of ε-hexagonal close-packed (hcp) martensite (35 vol %). Since IN718Plus does not undergo a strain induced martensitic transition, the increase in hardness, which is up to 50% can be attributed to defect production and work hardening. As can be seen the hardness curves overlap at higher energies indicating saturation takes place in hardening with higher energies. This suggests a cold work based mechanism for the hardening.

6.3 Simulations

The sample geometry was modeled with Hypermesh software using finite element method (FEM) for single and multiple laser impacts to predict the residual stress fields and deformation behavior under different geometries and energy, spot patterns, sequencing were carried out. We use an explicit non-linear FEA code LSDYNA on Unix based Dell precision quad-core servers. The LSDYNA and Matlab codes for LSP were originally developed by the Co-PI in our AFRL-funded project, Professor Dong Qian’s research group in Mechanical engineering department at UC [Zhou 2012]. Thereafter modified by us to meet the design specifications for our IN718plus test coupons and fatigue samples. Since at room temperature properties of IN718 and IN718Plus have close match in chemistry, and the dynamic response of IN718Plus is not yet available in literature, the material model for IN718 was employed in the constitutive analysis for generating
the pressure pulse using MATLAB software. The use of LSDYNA for LSP simulations is in line with research elsewhere who model laser processing in friction stir welded [Carney 2011] and non welded materials [Hu 2006; Arif 2009].

![Figure 6.20: Nanoindentation microhardness of LSP impact at different energies](image)
The results are very helpful in guiding the correct scheme of LSP. Firstly, we found higher energy is not always good. Despite the common belief that high energy produces greatest benefits for researchers trying to achieve through thickness compression, the FEM results indicate large tensile stresses inside the material. This was reflected in our initial fatigue tests on high energy LSP samples which without numerical optimization gave poor fatigue life. Energies of around 2J - 3J were best for the double-side laser peening to give improvements in fatigue life. Simulations indicate that small changes in sequencing, sample geometry or alignment can lead to a dramatic change in the residual stress profile. Hence, laser shock peening process parameters have to be chosen in order to realize the beneficial effects of the compressive residual stress field [Altenberger 2002]. Different steps and applications of simulations are shown below. First image shows an indentation profile (Figure 6.21) that was observed from our microhardness measurements across single-spot LSP impact. Modeling this single-spot LSP impact was the first step to LSDYNA simulations. Figure 6.22 shows the single-spot simulation. Single shot simulation was followed by simulating a multiple-shot LSP sequence on a non-optimized bar geometry, which is shown in Figure 6.23. The non-optimized geometry has high tensile stresses (shown in red).

Fig 6.21: Nanoindentation impression
Fig 6.22: Simulation of single spot Laser shot
Figure 6.23: Non-optimized bar geometry
Figure 6.24: Optimized test sample geometry
Finally the geometry was optimized to a standard flat tensile sample, based on our raw materials, testing facility and standard testing codes. The optimized geometry exhibits a uniform compressive region (shown in blue) in Figure 6.24. Once this reliable geometry had been established, the effects of LSP on generation of compressive residual stresses, including variation in shot sequence, number of layers, instrumental misalignment, etc. were examined.

6.3.1 X-Y Strain anisotropy

It has been reported that the strain components longitudinal and transverse to the peen line are not identical, with the transverse component being much less compressive [Toparli 2011]. We confirm the strain anisotropy, the strain in X direction (Figure 6.25), is smaller than those in Y direction given in Figure 6.26.

6.3.2 Effect of laser shot sequence

Moving from a poor laser shot sequencing scheme (Figure 6.27) to a better scheme (Figure 6.28), keeping all other parameters constant, improves the distributions of tensile stresses, shown in red, across the section. The optimized scheme (b) has less tensile stresses in the central z-section compared to the poor scheme. Thus, scheme (b) has better probability for fatigue life enhancement. X-direction compressive residual stresses are of the same order in both the cases.

6.3.3 Effect of overlap

The spacing between peening zones is critical for the uniformity of mechanical properties across the surface. The greatest uniformity and largest stress magnitudes are achieved by overlapping of the laser spots [Warren 2008]. While the optimized geometry shown (Figure 6.28) has 50% overlap, simulations were conducted to see if increasing the shot-to-shot overlap improved the
stress distribution. A 90% overlap was attempted keeping all other parameters constant. There is high accumulation of tensile stresses in the sample (Figure 6.29). This very high overlap percentage can deteriorate the stress distribution. Overlap in dynamic multiple shot laser impacts entails complex relaxation and deformation dynamics that is dependent on the spot, energy, geometry and sequence.
Figure 6.26: Y strain Max = 0.056 Min = -0.0018
Figure 6.27: Improper 50% overlap sequence
Figure 6.28: Proper 50% overlap sequence
Figure 6.29: 90% overlap
Figure 6.30: 0.8mm offset two layer LSP
A general trend cannot be predicted based on any specific geometry, but the results highlight its importance for ensuring reliable LSP performance, in agreement with the literature cited above.

### 6.3.4 Effect of second layer offset

While simulations can be conducted at very high precision, practical laser peening involves physical alignment of the sample and the beam. Often small deviations in sample to sample alignment occur. Hence, a 0.8 mm offset misalignment was simulated between two successive layers of LSP impacts one above the other on the same sample. Simulation (Figure 6.30) shows that the second layer offset creates a region of compression on one side and another adjacent region of tension (shown in red arrows) along the sample. Thus, subjecting the sample to greater probability of failure from the tensile side, limiting its useful life to the tensile region. Thus, the role of small misalignments in generating tensile stresses emphasizes the importance of good alignment especially of small samples and curved edges.

### 6.3.5 Effect of center terminating sequence

It has been suggested that a centrally terminating sequence would be good for tensile fatigue since the last shot has the highest compression. This sequence was simulated. The sequence does lead to highest compression in the center, but there are periodic regions of high tensile stresses distributed around the central thin section (shown in red arrows), decreasing in intensity with distance from sample center (Figure 6.31). Thus the sample will have a high probability of failure at these tensile regions.
6.3.6 Effect of number of layers

Since the number of layers of LSP needs to be optimized to get efficient use of laser time, simulation results (Figure 6.32) of increasing the number of layers in a 50%-overlap optimized sequence are presented. An increase from 2 layers to 6 layers had no effect on residual compression, indicating that successive layers may have a homogenizing role, but do not contribute toward enhancing the residual compression in any significant way.

The simulation results presented above are used as a guideline for optimizing the LSP procedure. Trends are more significant here than absolute values. Absolute values of residual stresses were not attempted to fit to the actual measurements on the samples with X-ray diffraction, since the complexity of microscopic origin of residual stress is difficult to be captured using FEM alone. Accordingly, absolute values from FEM simulations were not used for predictions. Hence, for simplicity energy coupling parameter of 100% was used, though values around 30% are expected based on literature. However, as tensile stresses cannot be eliminated and always accompany compressive stresses in LSP, trends of residual stress distribution were used in saving important laser and test time and achieving better LSP success rate by controlling the tensile stresses to benign configurations.

6.4. Residual stress

Residual stresses were determined using XRD. Initial measurements were conducted using synchrotron XRD at APS, Argonne. Some of the results were presented in an earlier work and are compared here. Local measurements were later attempted with conventional PANalytical
X-Ray Diffractometer measurements at University of Cincinnati, and gave results in agreement with the Advanced Photon Source, Argonne, Synchrotron XRay data. The main workhorse for XRD measurement was the Proto line focus dedicated XRD measurement equipment (LXRD). LXRD saves measurement time and allowed detailed characterization.

Figure 6.33 shows a typical LXRD residual stress map for IN718Plus. The coupon is the same thickness as our room-temperature fatigue test sample with 50% overlap. The surface residual stress generated on the LSP patch was mapped across an 18mm x 18mm square including the patch (Figure 6.33 a,b). The resulting stress map shows highest compression in the central part and characteristic inhomogeneity associated with LSP due to shot-to-shot variation in laser beam associated with the instrument. It also captures partly the tensile stresses associated with LSP at the boundaries of the patch. The highest compressive stress is around 719 MPa which is in close agreement with our subsequent measurements.
Figure 6.31: Central terminating sequence
Figure 6.32: 6-layers LSP with similar stresses
Figure 6.33a: LSP patch LXRD: 3mm outside patch edge, 3 mm increment, 18 x 18 mm map
Figure 6.33b: LSP patch LXRD: 3mm outside patch edge, 3 mm increment, 18 x 18 mm map

Residual stress measurement points
6.4.1 Effect of overlay material

In our measurements we observed that samples peened with and without tape overlay had significantly different residual stress. Almost all the samples without tape show tensile stresses on the surface compared to the samples with tape overlay, which show the expected compressive stresses on the surface. This occurrence of tensile stresses when LSP treated without a tape overlay is in agreement with literature, where it has been reported that overlay makes compressive residual stress profile move closer to the surface [Rubio-González 2006]. The removal of overlay moves the compressive stresses away from the surface. Tensile stresses start dominating the residual stress measured with conventional X-Rays, which only sample the top few microns of the material.

6.4.2 Through-thickness compression

To ensure crack retardation throughout the sample cross thickness compression is important. Since LSP gives deeper compressive stresses, through thickness compression can be ensured at least in thin sections [Ivetic 2011]. As reported in literature, residual stresses generated are compressive for a depth of up to 1 mm [Masse 1995] with maximum residual stress at 20-30 μm from surface. We attempt to confirm the same for IN718Plus. Our LXRD depth profile results are presented (Figure 6.34). The depth profile was generated by electrochemically polishing in a 10% H₂SO₄ - 90% Methanol solution and removing 30 -100 microns of surface layer material and making the stress measurements using the Proto LXRD system. This process was incrementally repeated to obtain the depth profile. The results show residual stresses of the order of 800 MPa on surface that gradually decrease to zero around 0.7- 0.8 mm. The depth of penetration depends on the number impacts and has been reported to increase to 1 mm with
increasing number of impacts [Ding 2006]. Our simulations on the effect of number layers have also shown an increase in the compressive residual stress, but beyond the second layer there was not much increase in residual stresses as discussed in section 6.3.6. Hence, our samples were peened with double layer LSP to get the maximum possible compression without spending excessive machine time in putting repeat layers. More importantly, based on the above, the fatigue samples were designed to be 1.7 mm thick to ensure complete through thickness compression in all our tests.

![Residual Stress Depth Profile](image)

**Figure 6.34: Residual stress depth in LSP treatment**
6.4.3 XRD, LXRD, SXRD comparison

Residual stress relaxation is a vital issue for laser shocked alloys. Residual stresses can relax due to thermal exposure [Buchanan 2011] or mechanical processing [Golden 2008]. Several authors have explored both types of relaxation. There are classic texts [Noyen & Cohen 1987] estimating the residual stress. We used synchrotron XRD, conventional XRD as well as LXRD for investigating these relaxation effects. While Synchrotron XRD was used in transmission geometry on 5 mm thick LSP treated IN718Plus coupons using a highly penetrating 114 KeV X-Ray beam ($\lambda = 0.107877$ Å), later measurements with conventional XRD and Proto LXRD are done on the surface of the LSP-treated samples.

6.4.4 Thermal relaxation effect

For conventional shot peened alloy, surface residual stress has been reported to relax to less than 40% of as treated value after 1- hr heat treatment at 943K. In our results (1 hr at 1023K heat treatment), the residual compressive strains remain about 45% in IN718Plus alloy SXRD measurements. SXRD requires analysis of viz. peak positions, width and shape to give depth-resolved 3-D structural, strain, stress and cold work information from individual phases at high spatial resolution. SXRD experiments were conducted at APS Argonne in Dr. Yang Ran’s beamline 11-1D-C (Jan 29, 2008 to Feb 4, 2008) and beamline 01-1D-C under Dr. Uhlrich Leinert and Dr. John Almer (January 2010). Incident beam energy was 114 KeV and corresponding wavelength 0.107877 Angstroms. Incident photon beam was 0.1 mm x 0.4 mm. It was incident on the LSP treated area of a rectangular sample in the transverse direction. The diffraction pattern was recorded on an image plate sequentially from the top to the bottom specimen surfaces at 50-100 μm steps. Calibration (CeO₂ standard) and sample data were
resolved with data conversion (Fit2D) and fitting routines (MatLab) of Dr. John Almer and Dr. Uhlrich Leinert at APS, Argonne. LSP introduces compressive in-plane strains/stresses ($X_1$, $X_3$ direction) and out-of-plane tensile strains/stresses (in the peening direction $X_2$) to some depth in the material. Strain anisotropy results in elliptical diffraction rings: the radii of circular rings shrink (i.e. increase in $d$) in the out-of-plane peening direction $X_2$ (azimuthal angles $h=90^\circ$ and $270^\circ$), but expand (i.e. decrease in $d$) in the in-plane direction $X_1$ normal to peening (azimuthal angles $h=0$ and $180^\circ$).

Figure 6.35: SXRD diffraction pattern from IN718Plus
Typical diffraction rings obtained SXRD transmission mode are plotted in fit2D software and analyzed for distortion (Figure 6.35). Ring distortion is the basis for diffraction strain measurement and corresponding stress by calculation [J. Almer, U. Lienert et al 2003]. Examples from an LSP-treated IN718Plus sample are shown in (Figure 6.36). From the measurements and plots of the radius (and hence the Bragg angle and d-spacing) the in-plane (0/180° ε11) and out-of-plane (90, 270° ε22) strains are obtained from a pseudo-Voigt fit to the data, and the following equations:

$$e_\eta = \frac{R_\eta - R_o}{R_o} = \frac{d_\eta - d_o}{d_o}; \quad e_{11} = \frac{R_{0180^\circ} - R_o}{R_o} = \frac{d_{0180^\circ} - d_o}{d_o}; \quad e_{22} = \frac{R_{90270^\circ} - R_o}{R_o} = \frac{d_{90270^\circ} - d_o}{d_o} \quad (10)$$

The residual stress is then determined by employing the 3-D Hooke’s Law assuming equal bi-axial in-plane strains as follows [Hauk 1997]:

$$\sigma_{ij} = \frac{1}{2} s_2 \left[ e_{ij} - \delta_{ij} \frac{s_1}{s_2 + 3s_1} (e_{11} + e_{22} + e_{33}) \right] \quad (11)$$

where

$$\delta_{ij} = \begin{cases} 1 & \text{if } i = j \\ 0 & \text{if } i \neq j \end{cases}$$

and

$$s_1^{bkl} = \left( \frac{\nu}{E} \right)^{bkl}; \quad \frac{1}{2} s_2^{bkl} = \left( \frac{1 + \nu}{E} \right)^{bkl} \quad (12)$$

The in-plane residual strains as function of depth from the top to the bottom surface are shown (Figure 3.7). The in-plane strains are compressive in the near-surface regions to a depth of ~1 mm, then become tensile further in the depth, while diminishing in magnitude [Chaswal, Vasudevan 2009]. Synchrotron LSP results were used to estimate volumetric strain profile, there
is a reduction in residual diffraction strain to around 50% of the as peened value in the sample aged after peening (Figure 6.36, 6.37). This reduction in residual strains implying a corresponding reduction in residual stresses is in agreement with data reported in literature where similar reduction in residual stress is reported within first hour of higher temperature exposure [Clauer 2001]. Under peened conditions the region outside the patch shows strong gradients from compressive to tensile as expected from overall force balance. As the material is heat treated its compressive residual stresses relax below the shock treated region. The compressive-to-tensile gradient also decreases. Hence, while thermal treatment relaxes residual compressive stresses, it also decreases the compensating tensile stresses in the region adjacent to shock boundary. Our work on 718Plus studying the effect of LSP stress relaxation on microhardness (Figure 6.38) shows corresponding decrease in microhardness upon thermal ageing. The results are in agreement with literature confirming that a high percentage of initial residual stresses are retained after thermal exposure, which can be beneficial in delaying fatigue failure [Buchanan 2011].
Figure 6.36: LSP treated SXRD residual strain [V Chaswal, MS Thesis, 2011]
Figure 6.37: LSP + Heat Treated (700C/1h, 750C/1h) SXRD residual strain
6.4.4.1 Eigenstrain analysis:

In a multiple shot peening sequence, an important concern is the relative contribution of each shot to the overall compressive residual stresses in the component. While numerical simulations by FEM give us an idea, we try to arrive at the result analytically using eigenstrain analysis.

One of the ways of measuring the residual stress distributions in shot peened coupons has been the eigenstrain analysis [Korsunsky 2005]. This procedure allows calculation of the internal residual stresses in a shot peened plate using the observed surface strains. To be able to corroborate our strain measurements with this technique, we conducted interferometric
measurements of the edge deflections along with the LSP patch profile in our LSP coupons (Figure 6.39). This edge profile is useful in getting the maximum deflection of the edge (Figure 6.40, 6.41). This maximum deflection was found to be the difference between the lowest (Blue) and the highest (Red) points of edge. We this related this directly to maximum Eigenstrain developing in the material. For the analysis of our 5 mm thick coupon, we can use the infinite plate type geometry as used by [Korsunsky, 2005] (Figure 6.42) within LSP impact plane in x and y direction, and thickness h in z direction along the direction of laser. In the uniform eigenstrain problem the eigenstrain domain is infinite in x and y, but varies in z direction. Assuming normal impact, the eigenstrains \( \varepsilon^*_x = \varepsilon^*_y = \gamma(z) \). For the depth function \( \gamma(z) \), the biaxial stresses in the plate are \( \sigma_x \) and \( \sigma_y \). Depth of the peening affected zone is given by region \( d \), where \( \gamma(z) > 0 \). For a sample of thickness h, the depth average eigenstrain \( (\Gamma) \) and its first moment \( (\Gamma_1) \) are defined as:

\[
\Gamma = \frac{1}{h} \int_0^d \gamma(z)dz \quad \text{and} \quad \Gamma_1 = \frac{1}{h^2} \int_0^d (\gamma(z))zdz
\]  

(13)

that they solve to get stress immediately below the peened surface:

\[
\sigma(0) = \frac{E}{1 - v} \left[ \gamma(0) + 6\Gamma_1 - 4\Gamma \right]
\]

(14)

For a thick plate as used by us, we can obtain an empirical form of the function, \( \gamma(z) \) from our depth profile measurements.
Since the plate is not infinite and the exact profile will vary with geometry, we fit an exponential decay to our residual stress depth profile data as shown in Figure (6.34). We get the following depth dependence for stress $\sigma(z)$ with depth ($z$)

$$\sigma_1(z) = 18.676 - 874e^{-z/123.06}$$

(15)

Where $z$ is in $\mu$m and $\sigma(z)$ in MPa. For isotropic deformation we can use the same profile to obtain the moments $\Gamma$ and $\Gamma_1$ as:

$$\sigma = \frac{Ee}{(1-\nu)}$$

(16)

Assuming the residual stress dependence is directly related to the plastic strain, and the material deforms isotropically under circular laser, we can propose that measured residual stress at any depth, arising from residual elastic straining of lattice corresponds to the biaxial plastic strains at that depth:

$$\sigma_1(z) = \frac{E\varepsilon_y * (z)}{(1-\nu)}$$

(17)

$$\varepsilon_y * (z) = \frac{(1-\nu)}{E} \sigma_1(z) = \frac{(1-\nu)}{E} (18.676 - 874e^{-z/123.06})$$

(18)

$$\text{as } \varepsilon_y * (z) = \gamma(z),$$

(19)

$$\Gamma = \frac{1}{h} \int_0^d \gamma(z)dz \quad \text{and} \quad \Gamma_1 = \frac{1}{h^2} \int_0^d (\gamma(z))dz$$

Substituting,

$h = 5\ mm = 5000\ \mu\m = \text{coupon Thickness}$

$d = 0.6\ mm = 600\ \mu\m = \text{depth of residual stress}$

$\nu = 0.31, E = 214\ GPa \quad \text{From} [ATI - Allvac, 2008]$

$$\Gamma = \frac{(1-0.31)\times 600\times (18.676 - 874e^{-z/123.06})}{5000}$$

$$\Gamma = \frac{0.69\times 600}{5000} \times [18.676 - 874(-123.06)e^{-600/123.06} - 874]$$

(20)
\[ \Gamma_1 = \frac{1}{h^2} \int_0^d (\gamma(z)) dz \]

\[ \Gamma_1 = \frac{(1 - 0.31)}{5000^2} \int_0^{600} (18.676 - 874e^{-z/123.06}) dz \]

\[ \Gamma_1 = \frac{0.69}{5000^2} [18.676 \times (600) - 874[(-123.06)^2 (e^{-600/123.06} (-600/123.06 - 1) - 1)] \]

\[ \Gamma_1 = (0.69 / 25000000)(11205.6 - 874[(15143.7636)(0.007623(-5.87567) - 1)] = 0.38 \mu \] (21)

Corresponding values are calculated assuming main plastic deformation is restricted to the strongly deformed surface layer of 30 microns from SEM fractography and gives \( \Gamma = 11.59 \mu \) and \( \Gamma_1 = 0.72 \mu \). This depth also corresponds to the plastically deformed surface depth obtained from VEECO interferometry (Figure 6.35). For a depth average deformation strain \( \Gamma = 11.59 \mu \) and the first moment \( \Gamma = 0.72 \mu \) about the mean we can now estimate the X and Y direction cumulative strains.

Assuming the strains are equally distributed in \( \pm X \), \( Y \) directions, a multiple shot sequence of 13 shots in one dimension along the center of the patch gives a value close to the total cumulative strain as:

\[ \varepsilon_{x}^{*} = n\Gamma = \sum_{1}^{13} \varepsilon_{i} \] (22)

Due to alternate sequence of shots in a LSP sequence, if mainly the first and last shots contribute the most to the plastic strain, permanent deformation occurs in both directions and manifests as the bulging of the outer edge along x-direction of the sample. This bulge is measured using VEECO interferometric profilometry, the maxima of which occurs around the center of an edge and can be taken as being closest to the cumulative total displacement in one dimension.
It is possible that subsequent shots occurring within the overlapping LSP sequence tend to cancel each other’s displacements due to overlap and non-sequential peening. Hence the effective total displacement \( s \) of the outer edge must at least equal the displacement arising from first and last shot, and at most be equal to the cumulative sum of all the shots i.e.

\[
\sum_{i=1}^{2} \varepsilon_i \leq \varepsilon \leq \sum_{i=1}^{13} \varepsilon_i \tag{23}
\]

\[
d \times (\varepsilon_1 + \varepsilon_{13}) \leq s \leq d \times 13\bar{\varepsilon} \tag{24}
\]

where \( d = \text{spot diameter} = 3 \text{mm} \)

substituting :

\[
\frac{3 \times 1.539 \times 2}{100} \text{mm} \leq s \leq \frac{3 \times 1.539 \times 13}{100}
\]

\[
0.09324 \text{mm} \leq s \leq 0.6 \text{mm}
\]

since this strain is divided equally among \( \pm x \) direction to the two sides of the coupon, individual displacement on one side is :

\[
0.047 \text{mm} \leq s / 2 \leq 0.3 \text{mm}
\]  

Comparing with the observation on sample sides, we can see it matches closely with the lower limit as the total deformation observed on the sides is around 40 \( \mu \text{m} \) (Figure 6.41 and 6.42).

This exercise also confirms an important hypothesis we made above, that the first and last spots are more important in imparting LSP strains, and those in between serve more of a function of homogenization and redistribution of stresses and strains arising from earlier shots for the plate geometry and LSP treatment considered.
Figure 6.39: LSP coupon used for eigenstrain analysis

Figure 6.40: Plate geometry for eigenstrain analysis, following [Korsunsky 2005]
Figure 6.41: Edge of LSP treated coupon used for eigenstrain analysis

Figure 6.42: Edge of unpeened coupon used for eigenstrain analysis
6.4.5 Fatigue relaxation effect: Cyclic relaxation of compressive stress is important for service stability and life prediction [McClung 2007], the salient features are the fact that while LSP has been reported to have less relaxation of fatigue induced stresses [Nalla 2003], there is always a concern since fatigue and thermal effects combined can lead to severe relaxation of stresses [Leverant 1979]. Complete relaxation was observed only under severe cycling [McClimont and Cohen 1982]. Our results on fatigue crack induced relaxation of LSP samples are presented in Figure 6.43 and 6.44. There is reduction in compressive residual stresses to around 50% upon fatigue cycling. Since the reductions are of the order observed in thermal relaxation, they are important for estimating total fatigue life at service temperature.

![Figure 6.43: LSP patch LXRD map before FCG](image1)

![Figure 6.44: LSP patch LXRD map after FCG](image2)
6.5 Fatigue

Fatigue resistance has traditionally been characterized by the total life approach involving stress (S) vs number of cycles to failure (N) curves. Fatigue testing was conducted on 2.5 and 5mm thick samples after LSP with a double side patch overlay of 2.5 mm spots at 5 to 8 GW/cm² using pulsed Nd:glass laser.

**Choice of specimen geometry:** two commonly used specimen geometries were selected based on viability of application of LSP on a flat surface as well as the industrial appeal and application.

**a. Flat tensile geometry:** Tensile test is one of the most commonly used tests. It is easy to interpret and relate with the monotonic tensile data and there exists large S-N curve data on tensile samples in literature. This simple geometry allows direct calculation of stresses from applied load by dividing the load by cross sectional area.

**b. 3-Point bend geometry:** Often it is not possible to be able to test specimens in tension due to their semi-brittle or brittle nature. Almost all brittle materials are tested in compression. 3PB tests are again one of the most common tests conducted in compression. Though it is difficult to measure a unique cross sectional stress as the stress is tensile at the top surface and compressive at the bottom surface. A typical stress-strain behavior of the bend test is shown in (Figure 6.45), and exhibits a bilinear behavior. This deviation from elastic stress maxima indicates the onset of plastic deviation [Lipetzky 1996]. This onset of plastic deviation was taken as yield stress in our fatigue tests. In order to be able to relate the effects of LSP under this important type of loading, 3PB was the other geometry chosen.
**Choice of load cycle:** Since, cracks tend to grow under tensile loading, tension-tension fatigue tests were conducted. Further as we also wanted to investigate the role of crack closure, the stress ratio R was kept to a low value (R=0.1) so that the differences in crack closure could be estimated before and after application of LSP treatment.

![Diagram of typical bilinear elastic-plastic behavior](image)

*Figure 6.45: Typical bilinear elastic-plastic behavior of 3PB geometry, reprinted with permission [Lipetzky 1996]*

### 6.5.1 Fatigue: 298K

Initially room temperature (RT) fatigue tests are conducted on 3PB compression samples at stress ratio (R) = 0.1 on 750°C/50h aged to simulate high hardening based on earlier work on
ageing kinetics [V Chaswal, MS Thesis, UC 2011], and monitored with an MTS clip-on strain
gauge to establish a baseline S-N curve. The load values are determined on the monotonic 3PB
compression response found out earlier (Figure 6.13). The results are shown in Figure 6.46. The
3PB tests were not carried out in high temperature as the MTS high temperature furnace was
designed for tensile fatigue tests only.

A tensile sample geometry optimized using simulations (section 6.3) was subsequently used for
both room and high temperature tests. All samples were heat treated as per manufacturer
recommendation (Cao 2006) by solutionizing at 788 °C for 8 h in a vacuum furnace followed by
fast cooling (>50 °C/h) to 704 °C and held for another 8 h before cooling in flowing argon.
Fatigue tests were conducted based on the best LSP treatment identified from simulations
(section 5.3) at 30 Hz and R=0.1 at room temperature for both LSP treated and untreated
samples. The load values for fatigue were based on the monotonic tensile test data generated
earlier (Figure 6.14). The results (Figure 6.47) show improvement in fatigue life in high cycle
fatigue (HCF) conditions though at high stresses under low cycle fatigue (LCF) conditions, the
improvements are not as significant. Our results are in agreement with literature where an
improvement of the fatigue life was found for an R ratio of 0.1 of up to 25-35%, depending on
the load level [Heckenberger 2011].
Figure 6.46: Peak stress vs failure cycles in at room temperature
6.5.2 LSP Fatigue: 298K and 923K

High temperature (HT) fatigue tests are subsequently conducted at R=0.1 on identical geometry and LSP treatment for both LSP treated and untreated samples. Sample displacement was monitored with a HT clip-on strain gauge. Simultaneous AC potential drop (ACPD) measurements were conducted on select samples to ascertain crack initiation and as a safety interlock to stop the machine at sample breakpoint since resistance changes much faster compared to the displacement near the sample breakpoint. The load values for fatigue were determined based on the monotonic tensile test data generated at 923K. The results (Figure 6.47) show improvement in fatigue life under HCF conditions. Interestingly, the high temperature fatigue strength is significantly greater than room temperature. This is related yield anomaly of superalloys due to their inherent microstructure that gives rise to higher yield stress at high temperatures compared to lower temperatures [Nembach 2003]. Dynamic precipitation in the alloy occurring at high temperature due to interaction between dislocations and precipitates[Sundararaman 1990] could enhance the contribution to strength of the material. Though, we did not observe precipitate fragmentation, any such occurrence would effect residual stress relaxation under cyclic loading conditions [Altenberger 2006]. Based on our TEM observations, there are slip band similar to those seen in most superalloy fatigue though precipitate fragmentation was not seen along the bands. It could be attributed to the asymmetrical loading in our stress controlled tests compared to the symmetrical loading in almost all strain controlled fatigue with the TEM observations of LSP treated alloy. As reported by [Lukas 2001], PSBs do not form under asymmetrical loading.

\[
D = \frac{4(1-v)\sigma_y a}{\pi \mu} \ln(\frac{b_0}{a}) + \frac{16n(1-v)\sigma_y a}{\pi \mu} \ln(\frac{b_1}{a})
\]

(26)

where

\[
b_0 \quad \text{and} \quad b_1 = \sec\left(\frac{\pi}{2} \frac{\sigma_{\text{max}}}{\sigma_y}\right) \quad \text{and} \quad b_1 = \sec\left(\frac{\pi}{4} \left(\frac{\sigma_{\text{max}} - \sigma_{\text{min}}}{\sigma_y}\right)\right)
\]

They match this theoretical measurement with their SEM observations of fatigue striation. Each fatigue striation corresponds to a single loading cycle and conclude it corresponds to stage I fatigue.

We find similar striations in our samples (Figure 6.48). Though our striations spacing (around 2-3 microns) is greater than those observed by Gell et al (0.9 microns), they fall within the same fatigue regime. Indicating the mode of fracture remains same. But our FCG results on LSP treated samples (Section 6.6.1), show that crack growth rates of 3-4 microns fall in the Stage II steady state fatigue. This stage follows the Paris equation and is known to be less crystallographic in nature. Hence, we can conclude that similar mechanism as that reported by Gell et al operates in fatigue of IN718Plus though striations are also operative in the Paris regime.
Figure 6.47: Effect of LSP on room and High temperature Fatigue in IN718Plus
Figure 6.48: Fatigue striations in IN718Plus
6.5.3 Effect of heat treatment on LSP fatigue

Fatigue tests were carried out in as-received IN718Plus aged at 650 °C/500h to generate the plate shaped feature of IN718Plus precipitation that forms upon thermal ageing at longer durations. These plate shaped precipitates are associated with a precipitate free zone adjacent to them [Chaswal, 2010]. Lack of precipitates makes the region adjacent to the plate shaped precipitates weaker compared to the matrix. Since these plate shaped precipitates are brittle, they can fracture under laser shock impact, creating potential stress concentration regions and reducing the fracture and fatigue resistance of the alloy (Figure 6.11). Precipitate free zones are common in many alloys and often associated with a decrease in mechanical properties as discussed literature review. Since these precipitates are not seen under manufacturer recommended heat treatments [Cao and Kennedy 2006], it is important to follow suitable heat treatment for this alloy. Hence it is vital to check microstructure in service-aged samples before utilizing LSP as an in service repair and/or life extension technique. These tests showed reduced fatigue life under heat treatment conditions from run-out to 275K cycles. The SEM surface of aged + LSP treated and fatigued sample (Figure 6.49) show sharp regions of the order of dimensions of these precipitates that may have fractured due to deformation by multiple slip.

**Mechanism for formation of precipitate free zones:** Similar precipitate free zones are observed in literature for other γ’ strengthened Ni-base superalloy [Kroll 2004 Nembach 2005] close to grain boundaries. Mechanism of formation of precipitate free zones depends upon several factors. Normally in γ’ strengthened Ni-base superalloys, the mismatch between Ni₃(Al,Ti) and the Ni matrix is quite small, hence strain induced partitioning is not significant.
Instead, the difference in chemical Ti for grain boundary carbides was reasoned to be the main cause for the dissolution of $\gamma'$. A double heat treatment attempted to reduce the precipitate free zones was not found to be successful. These zones have been attributed to greater affinity for grain boundary.

Based on above reports, difference in Ti-partitioning can be attributed to be the reason for the precipitate free zone seen in our samples as well. Though, in our case the $\gamma'$ precipitates dissolve to form more stable hcp $\eta$- Ni$_3$(Ti, Al, Nb) precipitates which are rich in Ti and are stoichiometrically close to Ni$_3$Ti. Thus formation of $\eta$- Ni$_3$Ti requires greater Ti content viz. 25%, than is available in the matrix which has an average concentration of 0.71 wt% (Table 5.1). This it tends to draw Ti from a large region adjacent to it which depletes the matrix of Ti. The depletion would results in the dissolution of $\gamma'$ - Ni$_3$(Al,Ti) in the Ti-lean zone adjacent to the $\eta$-Ni$_3$Ti precipitates.

**Effect of Ageing:** A detailed study on effect of ageing was carried out in an earlier work [V. Chaswal, MS Thesis 2011]. A conventional X-Ray plot of aged IN718Plus vs unaged IN718Plus is shown (Figure 6.50). In the unaged and solution annealed condition all, the matrix does not have much precipitation. As the the sample is aged $\gamma'$ - Ni$_3$(Al,Ti) starts precipitating and coarsening. This is reflected in the XRD plots where the peak intensities for some of the fcc matrix start decreasing as the scattering is shared by the ordered precipitates forming in the matrix.

**6.5.4 LSP fatigue fracture:** Stage 1 fatigue fracture is crystallographic in most alloys. In fcc alloys it takes place on (111) planes preferably in $<$110$>$ direction. Crystallographic slip traces as
reported [Gell and Leverant, 1968] in a high strength Nickel alloy are comparable with crack tip slip facets observed for our LSP treated and fatigued sample (Figure 6.49). The slip traces are of the same order as those reported. This indicates that fatigue fracture under LSP retains its crystallographic slip dependence. Though, the dominant slip traces are unidirectional in the Gell and Leverant’s work but under LSP we see more predominance of bending of slip lines and slip lines occurring in multiple orientations indicating the propensity of multiple slip may have increased due to LSP treatment. Fracture mode is also seen to be crystallographic faceted mode in IN718Plus.

**298K LSP fatigue** (Figure 6.51, 6.52) depicting fatigue and fast fracture region shows the fine striations and a micro-void nucleation and growth type fracture respectively.

**923K LSP fatigue** (Figure 6.53, 6.54) depicting fatigued and fast fracture region shows pronounced striations that rise due to oxidation of the fracture surface and a weaker micro-void nucleation fracture respectively.
Figure 6.49: SEM of LSP fatigued surface and slip band spacing (inset)
Figure 6.50: Effect of Ageing on IN718Plus diffractometer scan
Figure 6.51: SEM of 298K LSP fatigued surface showing striations

Figure 6.52: SEM of 298K LSP fatigued surface in the fast fracture region

Figure 6.53: SEM of 923K LSP fatigued surface showing striations and secondary cracking

Figure 6.54: SEM of 923K fatigued surface in the fast fracture region near LSP edge
Figure 6.55: SEM of unpeened HT fatigued surface in showing striations

Figure 6.56: SEM of HT LSP treated fatigued fracture surface showing slip lines
923K unpeened fatigue (Figure 6.55) tested sample shows regular striations with greater irregularity in surface topography indicating increased roughness, with crystallographic failure at the edge. Comparing this with the edge failure in peened HT (Figure 6.54) fatigue we see less incidence of crystallographic failure which can be due to high damage in the LSP treated zone in the vicinity of the surface. On the other hand LSP treated peened fracture surface shows crystallographic failure. While the central region of crack shows slip line based failure (Figure 6.56) the near edge regions show faceted appearance (Figure 6.57).

![Image](image.png)

**Figure 6.57: SEM of unpeened 923K fatigued edge in showing faceted failure**
6.5.5: TEM substructure and mechanistic description:

**Age Hardened IN718Plus:** Transmission electron micrograph of age hardened IN718Plus following manufacturer recommended heat treatment shows extensive precipitation of uniformly sized γ’ precipitates (Figure 6.58).

**Fatigue microstructure:** Transmission electron microscopic observations of fatigued samples (Figures 6.58 and 6.59, 6.61) show slip band formation in an alternating network at low temperature. At lower temperature the TEM foil also shows occurrence of sharp failure points (Figure 6.60). At higher temperature of (923K) such a profuse network is not seen (Figure 6.62 - 6.64). This indicates a change in slip mechanism at higher temperatures, which is in agreement with the change in deformation mode from glide to climb proposed by [Kear, 1974].

**LSP microstructure:** Transmission electron microscopic observations of LSP treated (Figure 6.65 and 6.66) material show for the first time a clear separation of LSP and non-LSP regions in the same picture. A sudden increase in dislocation density within a single grain is observed, that separates the grain into a region of high dislocation density vs. a region of low dislocation density. This confirms that high dislocation density is generated in LSP treatment. High magnification of the deformed zone (Figure 6.68) indicates the dislocation density consists of pairs of dislocations. LSP treated region also shows a deformed zone adjacent to precipitates (Figure 6.68) and incidence of precipitate fracture under LSP (Figure 6.69) was also seen.

**Effect of increased dislocation density:** Increase of dislocation density affects the degree of Bauschinger effect operative by changing the number density of dislocations completely
reversing per cycle. This can be related to plastic back strain in a cyclic test of the material as [Kocks and Mecking, 2003]:

\[ \gamma_B = n \frac{b}{d} = \frac{\tau_p}{\alpha \mu} n\left(\frac{\tau}{\tau_p}\right) \]  

(27)

Where

\(D = \text{slip line spacing}\)

\(n(\tau/\tau_p) = \text{the number of dislocations per potential slip plane that have moved back completely.}\)

Plot of normalized plastic back strain as a function of normalized applied stress (Figure 6.70) is from [Kocks and Mecking 2003]. As the number of dislocations per potential slip plane that are reversing increases, the plastic back strain increases. Our observations of dislocations density increase in the LSP affected region show an increase of 1 to 2 orders higher than the normal fatigued microstructure. Thus, incidence of slip reversibility is expected to be higher dislocation under LSP. But the reversible dislocations are only the ones lying on the slip bands already existing in fatigue. Since the overall area covered by slip bands is considerably low, we can assume an area dependence of the number of dislocations that are reversed by the slip bands. Area covered by slip bands the order of 50% in a highly fatigued region. Accordingly, slip reversibility is higher for the same value of applied strain for LSP treated material, but it does not rise linearly as shown in the figure 6.70 [Kocks & Mecking 2003]. The initial decrease in LSP residual stresses upon fatigue cycling to around 50% of their value corresponds to the adjustment of some of the new dislocations into the slip band structure.
At the same time, the increase in overall dislocation density by LSP increases the applied stress. This increases the lattice resistance to dislocation motion and increases the endurance limit.

Assuming that the complete reversibility of slip takes place in the slip bands (PSBs), the rate of increase of stress reversibility could be related to the rate dependence of nucleation of slip bands with increasing plastic strain. This has been given by Mughrabi [Mughrabi 1978]. As the plastic strain increases the rate of nucleation also increases and the stress required to nucleate a PSB reduces. The reduction in stress is due to the contribution from the long range internal stresses developing inside the material with increasing plastic strain and cold work. Accordingly, in a highly cold worked material like that produced by LSP, higher long range internal stresses are already present.

Mughrabi’s results are for PSB’s, but PSB’s are seen only under symmetrical tension-compression loading. Under such loading, the long range stress that are developed in the material are isotropic in nature. Cracks grow under a tensile state of stress. Hence, even though the tension-compression loading initiates a crack by forming intrusions and extrusions, the useful part of a tension-compression loading in growing the crack subsequently is primarily the tensile part. Accordingly, application of a compressive residual stress decreases the crack growth as well as crack nucleation tendency. This is particularly highlighted if the loading is asymmetrical tension-tension type (as in our case with $R = 0.1$). In such cases, a compressive residual stress will results in crack retardation for the complete the load cycle.

This implies the nucleation of slip bands takes place with greater difficulty in LSP treated materials.
Relation to increase of endurance limit:

For smooth uncracked specimens, fatigue damage initiates with the creation and growth of intrusions and extrusions. Intrusions and extrusions occur by slip based mechanism in the surface grains of the material [Greenfield 1971]. These intrusions and extrusions correspond slip localization, often in persistent slip bands [Liard 1976]. Nucleation of PSBs depends upon the long range internal stresses in the matrix (Figure 6.71) [Mughrabi 1978]. Existence of a threshold stress required for creation of PSBs has been linked to the existence of an endurance limit in materials [Mughrabi 1978].

Due to the introduction of compressive residual stresses from LSP, the long range internal stresses are modified. As the LSP induced residual stress opposes the cyclic tensile stresses, effective stress available for crack growth is reduced, thereby reducing the tendency for creating of a PSB structure. This increases the endurance limit. Thus, endurance limit rise is directly due to the reduced nucleation of slip-bands because of the compressive residual stresses introduced in the material. Though PSB formation may not always occur (e.g. PSBs are reported not to form under asymmetrical loading), the basic process of microcrack initiation still requires slip localization. In the absence of PSBs, a cellular structure has been found to be operative in Ni [Lukas 2001]. Since the cellular structure also represents slip localization, the effect of long range matrix stresses in contributing to the endurance limit [Mughrabi 1978] still holds good.

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Figure 6.58: TEM of unpeened and Age hardened IN718Plus showing γ’

Figure 6.59: TEM of 923K non-LSP fatigued sample near the fracture region occurring outside furnace at lower temperature
Figure 6.60: TEM of 923K non-LSP fatigued sample near the fracture region occurring outside furnace at lower temperature

Figure 6.61: TEM of 923K non-LSP fatigued sample near the fracture region occurring outside furnace at lower temperature
Figure 6.62: TEM of 923K non-LSP fatigued sample from gauge section lying inside the furnace at 923K
Figure 6.63: TEM of 923K non-LSP fatigued sample from gauge section lying inside the furnace at 923K showing dislocations bowing and lower slip bands

Figure 6.64: TEM of 923K non-LSP fatigued sample from gauge section lying inside the furnace at 923K
Figure 6.65: TEM of LSP treated sample showing a clear zone of sudden increase in dislocation density (red arrow)

Figure 6.66: TEM of LSP treated sample showing a zone of increase in dislocation density
Figure 6.67: TEM of LSP induced dislocations occurring in pairs
Figure 6.68: TEM of LSP treated sample showing deformation zone adjacent to γ' precipitate

Figure 6.69: TEM of LSP treated sample showing precipitate fracture
Figure 6.70: Variation of plastic back-strain with stress after prior straining, reprinted with permission [Kocks & Mecking 2003]

Plastic back-strain, $\varepsilon_B$, in a Bauschinger test as a function of the applied stress, $\sigma$, during continuous stress reduction followed by a stress-reversal after prestraining to different levels of the flow stress $\sigma_p$. Normalized by the shear modulus and the flow stress according to Eq. (41) leads to a unique curve [41].

Courtesy ref: U.F. Kocks, H. Mecking 2003]
Dependence of the nucleation stresses, $\tau_n$, for persistent slip band formation and of the parameter, $R$, related to the rate of propagation of persistent slip bands, on $\gamma_{pl}$. The plot demonstrates that the decrease of $\tau_n$ and the increase of $R$ with increasing $\gamma_{pl}$ are related to the transition from cyclic hardening mechanism I to II'.

Courtesy: ref[Mughrabi 1978]

Figure 6.71: Variation of nucleation stress for PSB formation and propagation, on plastic strain, reprinted with permission [Mughrabi 1978]
6.6 Fatigue Crack Growth

As per the second law of thermodynamics, any irreversible process increases the entropy of the universe. Accordingly, human tendencies of use and throw have been instrumental in contributing to increasing the entropy of our environment that reflects in global warming and irreversible loss of natural habitats. These trends have lead to extinction of multiple species till now and are a serious threat to all life eventually. To be able to combat these concerns the demand on increasing the lifetime of components has been ever increasing. Improving fatigue life by application of several surface modification techniques like shot peening, cold working, LSP, low plasticity burnishing, water jet peening etc by introducing compressive stresses has been helpful in uncracked samples and improving total life fatigue, and has also shown promising results for LSP discussed in section 6.5. But application of these surface modification techniques in improving fatigue life of components containing a pre-existing macroscopic crack have seen limited success.

Basic concept towards improving fatigue crack growth life lies is being able to bridge or heal the crack. While several researchers have attempted to find crack healing mechanisms [Hager 2010] e.g dynamic precipitation oxidation induced microencapsulation [White 2001], and thermal reversibility in polymeric materials, we investigate the role of LSP induced stresses in reducing fatigue crack growth rates. Based on the geometry of most commonly tested materials, we have
chosen 3-Point bend (3PB) (Figure 6.73, 6.74) and middle tension (M(T)) geometries. Both the geometries are amenable to LSP treatment and are directly related to our tests carried out on uncracked specimens (section 6.5). While M(T) specimen was used for constant amplitude FCG with and without LSP, the 3PB geometry was used to study the effect of LSP on crack tip overload reflecting loading variability.

**6.6.1a Test method:**

The middle tension, M(T) sample Geometry was chosen for FCG testing of LSP treated materials. Tests were conducted at room temperature. Standard M(T) samples were prepared and tested as per ASTM e647-00. Constant-amplitude tension-tension FCG was conducted at R = 0.1 in the Paris regime on unpeened material as follows:

**i) Pre-crack:** a small hole was drilled in the center of the specimen which was subsequently notched on opposite sides using an ACCUTEX wire EDM machine. A sharp fatigue crack was grown to same dimensions for high temperature as well as room temperature samples following ASTM [ASTM e647] recommendations.

**ii) FCG testing:** constant amplitude FCG testing was conducted at R=0.1 on both the room and high temperature on as received IN718Plus samples age hardened as per manufacturer recommendation. Crack was allowed to grow for a ΔK increment of around 10-15 MPa√m in the Paris regime, to determine the exponent ‘m’ using equation (3):
\[
d\frac{a}{dn} = C\Delta K^m
\]

**Crack length measurement:** Crack growth rate was estimated based on ACPD measurement using a two-point calibration with an optical microscope at the beginning and end of test and dividing it by number of cycles. Though there have been reports of crack bridging due to local crack welding arising due to applied currents which can lead to incorrect measurement of fatigue crack growth life [Hosoi 2012]

Since the crack increment was not large between two successive measurements, ACPD measurements can be linearly related to crack increment. Crack closure monitored using MTS COD gauge corroborated ACPD measurement. Crack length was optically measured every time the sample was removed from the test machine using a metallurgical microscope.

**iii) LSP treatment:** Test is unloaded and sample is double side laser peened with 6J, 25 ns, 2 mm spot size Nd-glass laser multiple overlay patch with 50% overlap using adhesive tape overlay and water confinement.

**iv) Post LSP FCG:** constant amplitude FCG is resumed after LSP to see the effect of LSP on FCG rates. Crack tip profilometry was done on the crack tip XRD patch to measure the residual stress relaxation. Since the resistance of the material changes after LSP treatment, ACPD was recalibrated after peening sample and also during every unloading. Subsequent fatigue-induced residual stress relaxation is determined using LXRD system. Conditions for crack initiation and branching are investigated using scanning electron microscopy.
6.6.1b Fracture mode in IN718Plus

Fatigue crack path was studied using optical microscopy in 3PB samples. A straight wire EDM cut notch was used as crack starter (Figure 6.74). The 3PB samples were polished and etched on one side prior to fatigue. Etched microstructure shows the crack initiation from the notch tip as well as propagation (Figure 6.75) across IN718+ material to be transgranular with reasonable roughness. While the effect of LSP on crack path, causes a deflection as it enters the LSP patch (Figure 6.76). As the samples are not very thick it is important to check if they follow plain strain conditions and ensure that application of surface treatment does not create a plane stress situation. Crack deflection is measured on both sides of the sample and its sense is found to be parallel to the crack plane indicating the sample is still under plane stress (Quantitative validation presented later in section 6.6.4 for FCG after overload + LSP case where greater thickness reduction is observed also confirms that sample is in plane strain).

Reports in literature on effect of LSP on fatigue crack growth are varied. In 6061-T6 aluminum alloy [Rubio-González 2004], steel [Rubio-González 2011] and friction stir welded and unwelded Aluminum Alloy (AA) 7075-T7351 [Hatamleh 2008] LSP reduces fatigue crack growth and increases fracture toughness. It has been suggested, however that fatigue lives of LSP-treated specimens may be reduced due to the occurrence of internal cracking [Liu 2007]. Measurements of residual stress fields generated by LSP using neutron diffraction show compressive stress near the surface and tensile stresses in the mid-thickness regions. Growth rates of cracks were observed to be more affected by the tensile core than by the compressive surface stresses [Chahardehi 2010]
Figure 6.73: 3PB testing on LSP treated FCG sample on MTS 810

Figure 6.74: Pre-crack initiation from a notch root, sample etched side prior to fatigue
Figure 6.75: Transgranular Fatigue

Figure 6.76: Crack deflection upon Laser peening
6.6.2 Fatigue Crack growth

Fatigue crack growth tests were conducted on LSP treated and untreated M(T) specimens of IN718Plus as per ASTM e647-00. The results are presented in Figure 6.77- 6.80. Cracklength progressively increases with number of cycles for unpeened and peened samples (Figure 6.77 and Figure 6.79 respectively). Fatigue crack growth plots of plane strain stress intensity factor range (ΔK) with crack growth rates in (da/dn) corresponding to the Paris regime are shown (Figure 6.78 and Figure 6.80 respectively). After LSP treatment, the FCG rate has increased compared to unpeened condition indicating that beneficial effects of LSP are not seen in FCG, though the slope of the curve remains almost the same indicating that Paris law (Eqn 3) continues to be followed. The literature suggests that primary improvement of LSP occurs during the crack initiation stage [Peyre 1996], as the compressive layer delays fatigue crack initiation and retards small crack propagation [Prevéy 2000]. This idea explains our result. While fatigue crack initiation controls the HCF fatigue behavior where improvements were seen in our fatigue tests (section 6.5), fatigue crack growth test, being a long crack test, does not show such improvements. In fact, since our MT sample geometry was chosen to represent an internal preexisting flaw growing from a hole drilled in the sample, and is similar to a fastener hole fatigue in some sense. Studies on LSP of fastener hole cracks have reported negative effects on fatigue lives on the specimens with the hole already present [Ivetic 2012]. This is in agreement with our results, where a crack grows from a pre-existing hole.

Estimating the contribution of LSP on fatigue crack growth ref [Richards 1971]:

\[ R = \text{plain strain plastic zone size}, \ COD = \text{crack opening displacement} \]
\[ \sigma_y = \text{yield stress} \]
E = Young’s modulus

Upon the application of the LSP, let the plastic zone size change to $R_{lsp}$ and COD to $COD_{lsp}$:

*From* [Richards, 1971]*

$$COD_{lsp}^2 \propto \frac{da_{lsp}}{dn} \quad \text{and} \quad COD_{lsp} \propto R_{lsp} \frac{2\sigma_y}{E} \tag{28}$$

$$\sqrt{\frac{da_{lsp}}{dn}} COD = COD_{lsp} \tag{29}$$

substituting observed values

300$*COD = COD_{lsp}$

such an increase of 2 orders is not observed.

*Applying* Irving's [plastic zone size correction]*

$$COD \propto \left( \frac{(\Delta K)^2}{5.6\pi(2\sigma_y)E} \right) \tag{30}$$

$$\frac{da}{dn} \propto \left( \frac{(\Delta K)^2}{2\pi(2\sigma_y)E} \right)^2 \tag{31}$$

$$\frac{da_{lsp}}{dn} = \left( \frac{(\Delta K_{lsp})^2}{(\Delta K)^2 \sigma_{y_{lsp}}} \right)^2 \tag{32}$$

$$\sigma_{y_{lsp}} = \left( \frac{\Delta K_{lsp}}{\Delta K} \right)^2 \sqrt{\frac{da}{dn}} \frac{\sigma_y}{da_{lsp}} \tag{33}$$

substituting values from our results:

$$\sigma_{y_{lsp}} = \left( \frac{55}{25} \right)^2 \sqrt{\frac{9 \times 10^{-7}}{9 \times 10^{-3}}} \sigma_y \leq \sigma_y \tag{34}$$
Which indicates an LSP induced softening or reduction in effective yield stress ahead of the crack tip. Since the absolute values of COD can change due to sample loading/unloading, but the relative change in COD with K remains independent, we can arrive at a corresponding analysis of effective contribution rate of change of COD in the Paris regime where:

\[
\frac{da}{dn} = C\Delta K^m \quad \text{From (3)}
\]

Let

\[
\zeta = COD
\]

\[
\zeta^2 \propto \frac{da}{dn} \quad \text{From (28)}
\]

\[
\zeta^2 = C'\frac{da}{dn}
\]

(35)

where \( C' \) depends upon the material and sample geometry

\[
\zeta = C\sqrt{C'}\Delta K^{m/2}
\]

(36)

\[
\frac{\partial \zeta}{\partial \Delta K} = \frac{C\sqrt{C'}m\Delta K^{-1+m/2}}{2}
\]

(37)

\[
\frac{\partial \zeta_{lsp}}{\partial \Delta K_{lsp}} = \frac{C_{lsp}\sqrt{C'm_{lsp}\Delta K_{lsp}^{-1+m/2}}}{2}
\]

\[
\frac{\partial \zeta}{\partial \Delta K} = \frac{C\sqrt{C'm}\Delta K^{1+m/2}}{2}
\]

(38)

Since the material and sample geometry remain unchanged, \( C' \) is assumed to be independent of LSP.

\[
\frac{\partial \zeta_{lsp}}{\partial \Delta K_{lsp}} = \frac{C_{lsp}m_{lsp}\Delta K_{lsp}^{-1+m_{lsp}/2}}{Cm\Delta K^{-1+m/2}}
\]

(39)

assuming \( C \) and \( m \) do not change significantly

\[
\Delta K_{lsp} = \left(\frac{\partial \zeta_{lsp}}{\partial \Delta K_{lsp}}\right)^{m-1}\Delta K
\]

(40)
Substituting for $K$:

\[
K = f(a/w)P\sqrt{a}
\]

\[
\Delta P_{lsp} \nabla a_{lsp} = \left(\frac{\partial \Delta P_{lsp}}{\partial \zeta_{lsp}}\right)^2 \Delta P \sqrt{a}
\]

in a constant amplitude test $\Delta P_{lsp} = \Delta P$

\[
a_{lsp} = \left(\frac{\partial \zeta_{lsp}}{\partial \Delta P}\right)^4 a
\]

but $\frac{\partial \zeta}{\partial \Delta P} =$ compliance, $S$

\[
a_{lsp} = \left(\frac{S_{lsp}}{S}\right)^4 a
\]

for $m = 3$,

substituting values from COD first cycle

\[
a_{lsp} = \left(\frac{0.041}{0.047}\right)^2 a = 0.76a
\]

for $m = 3$,

substituting values from COD average of 6 cycles before and after LSP

\[
a'_{lsp} = \left(\frac{0.040}{0.0245}\right)^2 a' = 2.6655a'
\]
The above analysis shows that immediately after LSP, the material is having some residual crack closure due to LSP effect in the first cycle, but within the first 5 cycles the average crack growth rate increases to twice than that of the 5 cycles before LSP indicating the rapidly accelerating overall crack growth. This analysis based on COD compliance corroborates the fatigue crack growth data based on ACPD measurements (Figure 6.77 - 6.80: crack growth rate is plotted with $\Delta K$ values derived from ACPD measurements), where also increased crack growth rates were observed after LSP.
Figure 6.78: Paris Regime FCG unpeened

Fatigue Crack Growth non LSP - IN718Plus

\[ \frac{da}{dn} \text{ mm/cycle} \]

\[ \Delta K \text{ MPa } (m)^{0.5} \]

\[ B \quad \text{Linear Fit of Data2_B} \]
Figure 6.79: Crack growth laser peened
Fatigue of LSP treated IN718Plus

Paris slope LSP/ Paris slope non LSP = 0.8

Figure 6.80: Paris Regime Laser Peened
6.6.3 Fatigue Crack growth: crack closure

Since fatigue crack growth is dependent on crack closure [Suresh and Ritchie], we also made crack closure measurements to identify if there is any correlation between the crack growth response to LSP and crack closure. The results are presented in (Figure 6.81- 6.86)

Pre LSP compliance is seen to be smaller compared to the Post LSP values as the slopes of the crack opening displacement curves are higher post LSP (since absolute values are reported y-axis scales are different), in accordance with the LSP response discussed above, although there is no outward indication of closure in these curves. It can be seen that the ACPD hysteresis loop changes from pre-LSP to post LSP. From this curve the crack opening loads seem to have undergone a 10% - 20% increase. The most pronounced feature is the reduction in signal noise, which is a measure of dissipative loss of energy through crack closure. Lower noise after LSP also indicates that less energy is getting lost through crack closure. This indicates greater fatigue induced damage accumulation in the material, that offsets benefits of LSP induced compressive residual stress and also contributes to higher crack growth rates.

Deviations observed from ideal LEFM behavior reflect the high dissipative loss in unpeened material compared to lower dissipation in laser peened materials (Figure 6.83 and 6.84). The curves also are indicative of the corresponding closure displacements occurring at higher values in LSP-treated alloy in accord with the increased compliance observed earlier.
Figure 6.81: Pre LSP compliance

Figure 6.82: Post LSP compliance
Figure 6.83: Pre LSP Load vs COD

Figure 6.84: Post LSP Load vs COD
**Figure 6.85: Pre-LSP closure**

**Figure 6.86: Post LSP closure**
6.6.4 FCG: overload effect

Overload measurements involve application of LSP to fatigue tests simulating real life loading scenarios. Most standardized testing [ASTM e647] for FCG measurements are conducted under controlled set of conditions with fixed load amplitudes being repeated over millions of cycles. But real life scenarios involve loads which are not identical in successive cycles, and on complex components that do not have simple lab geometries. Though the variation in component geometry can be addressed using continuum numerical modeling tools, variation in cycle-to-cycle load response is difficult to model. This variation becomes especially important if during operation the component can undergo a sudden cyclic overload of up to 50% more than the normal design [Nicholas 1991]. Here, it is essential to characterize the acceleration due to overload or retardation due to crack blunting on crack growth rates to be able to continue using the component reliably for intended design life after the occurrence of such an overload. Overload with LSP is an unexplored area and overload studies are significant due to the presence of residual stresses induced in the material during LSP. In similar systems of conventional-peened materials, effect of overloads diminish the advantages of peening [De Los Rios 1996]. Crack growth tends to get arrested in metals after a sudden overload due to crack tip blunting and enhanced plastic zone size. McEvily et al [McEvily 2004] reported that the number of overload delay cycles to return to fast crack growth rate depends upon sample thickness, since the overload deformation zone ahead of the crack tip has a larger plane stress component compared to the sharp crack. They assume that the sample follows small scale yielding (SSY). SSY is valid when the sample size is small compared to the crack tip deformations, a requirement of linear elastic fracture mechanics (LEFM) [Griffith 1921] [ASTM e647]. But SSY may not be valid in case of large overloads coupled with LSP treatment. For large overloads experimental validation
is necessary wherever the J-integral approach [Rice 1974] cannot be applied due to testing limitations. As LSP modifies the surface state of stress, our model for overload delay cycles was validated with a single spot LSP pattern at the crack tip (Figure 6.87). 5 mm thick fatigue crack growth samples to match with the earlier results were pre-cracked by the ASTM load shedding technique. Initial crack was selected to be equivalent to typical NDT detection limit of 2 mm as per AFGROW recommendations.
Figure 6.88: LSP patch LXRD just after Overloading

Figure 6.89: LSP patch LXRD Fatigue cycled after overloading
Figure 6.90: Crack tip LSP after an overload, surface depth profile map

<table>
<thead>
<tr>
<th></th>
<th>Before LSP</th>
<th>After LSP</th>
</tr>
</thead>
<tbody>
<tr>
<td>da/dn mm/cycle</td>
<td>0.004</td>
<td>0.0042</td>
</tr>
<tr>
<td>$\Delta K$ MPa√m</td>
<td>14.04</td>
<td>19.696</td>
</tr>
</tbody>
</table>

Table 6.1: Effect of crack tip LSP on FCG rates
A single overload cycle is applied to the sample followed by FCG at constant amplitude. LSP is applied in the overload plastic zone to estimate the effect of LSP on crack overloads. Our results show that a higher $K$ is needed to get same crack growth after LSP treatment. The minimum crack growth rate after overload is [McEvily 2004]:

$$\frac{da}{dn} = A(K_{max} b - K_{op2 max} - \Delta K_{effth})^2$$

(47)

For the same crack growth rate corresponding $K$ values (Table 6.1) can be used to calculate the contribution of LSP residual stress on fatigue crack growth after an overload. In this case LSP Avg. residual stress = 394.24 - 513.82 = -119.58 MPa, which is smaller than our residual stress measurements, but considering that the samples are thicker than through thickness compression and the centre has quite low compressive stresses, it seems reasonable. But since overload effect is also involved in above estimates, it is important to separate the contribution of two effects. As per Hill, measurements of residual stress on the crack plane, and measurements of the residual stress intensity factor, enable correction of the toughness data through superposition and linear elastic fracture mechanics [Hill 2008]. We map the residual stresses along the crack just after the overload (Figure 6.88) and then later after cycling the material to advance the crack through the overload (Figure 6.89). Overload introduces an increased compressive stress ahead of a growing crack apart from the blunting, which tends to reduce to equilibrium values after cycling. This additional compressive stress at the crack tip explains the crack retardation effect.

**Plane strain condition:** An excessively large plastic zone can lead to violation of small scale yield criteria as discussed earlier. Further, since LSP reduced sample thickness, it becomes important to re-establish if the sample size decreased substantially to consider it a plane stress
failure. Crack tip profilometry on the overload was done to check the effect on crack profile, the double shot LSP produces around 45 µm deep dimple ahead of the crack tip overlapping the crack plastic zone (Figure 6.90). Since the total thickness reduction is not substantial (0.1 mm) even at the maximum depth, crack is in plane strain condition. In order for the tests to be valid as per [ASTM E399-90, ASTM 1820]:

Both thickness as width of the sample must exceed:

\[ B, W \geq 2.5 \left( \frac{K_{\text{max}}}{\sigma_{YS}} \right)^2 \]  
substituting for \( K \) values

\[ B, W \geq 2.5 \left( \frac{0.9}{958} \right)^2 = 1.214 \text{ mm} \]  

\( K_{\text{max}} \) should be corrected for reduction in 0.1 mm

\[ K = \left[ \frac{PS}{(BB_N)^{1/2}W^{3/2}} \right] f(a/W) \quad \text{From ref [ASTM e399]} \]

where

\[ f(a/W) = \frac{3(a/W)^{1/2}[1.99 - (a/W)(1 - a/W)(2.15 - 3.93(a/W) + 2.7(a/W)^2)]}{2(1 + 2a/W)(1 - a/W)^{1/2}} \]

\[ K' = K(5/\sqrt{5(4.9)}) = (1.01)K \]  

\[ K'_{\text{max}} = 1.01 \times \frac{19}{0.9} = 21.325 \text{MPa}\sqrt{m} \]

\[ B, W \geq 2.5 \left( \frac{K'_{\text{max}}}{\sigma_{YS}} \right)^2 = 1.2388 \text{ mm} \]
Since both B and W exceed 1.24 mm, the sample is under plane strain condition under overload and LSP.

6.6.5 Fractography

Peening processes alter the failure mechanism in materials [Lavender 2008]. SEM fractography of our LSP treated and fatigued samples post failure reveals crack branching and presence of secondary crack in the center of samples (Figure 6.91), indicating the centre of the sample did not show much resistance to cracking and the benefits are limited to the surface layer. A transition in fracture mode visible macroscopically at the onset of LSP zone (Figure 6.92). This could be a precursor to crack tunneling and fast crack growth, reaffirming the importance of validating the stress state ahead of crack tip. Figure 6.93 shows the surface of the LSP fracture sample, a highly deformed zone due to laser impact extends from the surface to around 45 - 30 µm. Though this LSP surface layer is highly deformed, crack initiation did not take place here. We address this further in the concluding part of this section.

Effect on crack tunneling:

LSP induces in-plane compression and out of plane tension in the sample. As the region ahead of the crack tip is in a triaxial state of stress, enhanced tensile stress out of plane dimension can have two effects:

1. **Change of fracture mode:** It can promote a transition to plain stress fracture condition ahead of crack tip. This behavior can be detected from the fracture surface morphology and leads to non-conservative estimates of FCG behavior. Plane stress samples tend to have an inclined fracture normal to tensile axis. This would result in a unique deflected appearance of crack path.
of two sides of the sample. Crack path deflects upwards on one side and downwards on the opposite side of the sample.

Our optical micrographs of crack growth after LSP show similar a crack deflection, but the deviation of crack front is in same direction on both sides of the sample indicating the sample did not fail under plane stress.

2. Crack tunneling: Out of plane tensile stress can enhance the tri-axial stresses ahead of a crack tip leading to tunneling and enhanced crack growth rates. We see a transition zone of around 0.5 mm in our FCG tests (Figure 6.92) adjacent to the boundary of LSP region. Fractographic evidence support the observed FCG behavior. This is in agreement with the change in crack length and corresponding increase in compliance seen after LSP treatment.

6.6.6 General Discussion on fatigue life improvement under LSP

While the basic understanding of improvement in fatigue life by introduction of compressive residual stresses has been available for more than a century, advances in our understanding of material behavior demand a more in-depth mechanistic description of this phenomena. Tensile stresses grow cracks and compressive stresses close them, hence application of residual compressive stresses should increase fatigue life. Here we attempt to address this issue further, light of our observations.

A. High temperature anomaly: It is observed that IN718Plus has endurance limit and greater fatigue strength at high temperatures compared to lower strength. This appears unusual as most metals and alloys tend to lose their strength at high temperatures. But this contradiction is not unique to IN718Plus, and is rather common to most superalloys [Nembach 2003]. Most ordered
superalloys exhibit higher strength at high temperatures compared to lower temperature and this phenomena has come to be known as the yield strength anomaly. While there are many competing theories to explain this behavior the most common reasons are listed below:

a. **Kear-Wilsdorf Locking of partial dislocations**: dislocation locking takes place by motion from octahedral to cube planes [Sada 1992, Devincre 1996] Dislocations in ordered superalloys move in pairs since the passage of one dislocation tends to create an antiphase boundary which is a region of high energy. To reduce this extra surface energy a second dislocation closely follows the first one and restores the order. In γ’ strengthened Ni-base superalloys, these dislocation pairs are often 1/6(110) type Shockley partials which slip on (111) close packed planes at low temperatures. As the temperature increases, they acquire enough energy to cross slip to alternate slip systems. It is observed that the (100) plane in these ordered alloys has as low anti-phase energy. Hence, one of the leading partials tends to cross slip to this plane of lower energy. But (100) being a non-close packed plane is not a slip plane for these partials. This makes any further glide of the partials on the slip plane impossible and they can only move by non-conservative means like climb which are relatively slow and often do not become active unless temperature is raised higher. Hence, in this intermediate temperature range, the material shows anomalous hardening due to increased sessile dislocation density. For Ni-base superalloys this corresponds a temperature from 873K -1123K C.

b. **Diffusion controlled Frictional effects**: Dislocation motion is dependent on diffusion and solute interaction for these mechanisms. It is also active in ordered alloys like Al3Ti. The equilibrium dislocation configurations is dependent upon a temperature dependent relaxation time that may arise from temperature assisted climb, softening of APBs, and pinning
atmospheres Three types of domains are possible [Cilliard 1996, Morris 1993, Potez 1992, Strudel 1979] based on the flow stress ($\sigma$), temperature, ($T$), and dislocation velocity ($v$):

i) -ve temperature dependence of $\sigma$: High $v$, low $T$

ii) -ve temperature dependence of $\sigma$: high $T$, low $v$

iii) PLC effect, jerky flow: intermediate $T$, intermediate $v$

c. Lattice resistance controlled frictional effects: Dislocation cores have a non-planar structure in case of superpartials seen in ordered alloys like IN718Plus, hence the Peierl’s stresses can give rise to additional frictional component and cause a yield anomaly. Commonly seen in Be [Cilliard 1996]

d. Multiple slip activation: thermal activation of multiple slip systems in precipitates can also results in yield anomaly and has been reported in Co based intermetallics [Suzuki 2007]

B. Endurance limit increase: It is observed that endurance limit increase in case of LSP is similar or higher than those observed for conventional techniques like shot peening. The basic reason behind this lies in the difference in stress profiles and their interaction with crack nucleation and growth. While it is well established by now based on literature, and our results also agree with it, that the compressive residual stresses introduced by LSP are much deeper (up to $0.6 - 1.0$ mm) compared to shot peening ($0.1 - 0.2$ mm). This implies that crack nucleation takes place much deeper in LSP treated sample compared to the shot peened sample. Fractographic analysis of our fatigue tested samples (Figure 6.94) confirms this hypothesis. White arrows mark the location of crack nucleation and black arrows show the direction of application of LSP. Nucleation of fatigue crack is seen to take place sub-surface and not at the
surface. The distance of this crack nucleation depends upon the microstructure, but is found to be around 150-200 μm. This is close to the maximum limit of compressive stresses in shot peening. Accordingly, shot peening would have little or no effect at these depths, since the stresses would have reduced to zero and beyond this depth may be even tensile from force balance considerations. On the other hand, LSP provides compressive stresses to a greater depth within the sample. This increase in depth has important implications on crack nucleation and growth.

**LSP surface layer condition:** An important aspect of LSP is that it creates a surface layer which has high damage. As can be seen from SEM fractographs of LSP edge, the region of up to 30 - 45 microns near the surface has its structure highly deformed and modified by LSP (Figure 6.93). Why is it that this surface that offers sufficiently large crack initiation sites does not become the source of sub-surface nucleation. There are 2 aspects we could attribute to this:

1) the near surface stress is highly compressive in LSP treated materials as can be seen from out depth profile of LSP coupon, the peak LSP stress is maintained near the 50 micron depth, and even after fatigue and thermal relaxation of up to 50%, the stress profile remains unchanged. Hence the surface remains in bulk compressive stress. But this still does not rule out the possibility of local stress concentration from the wide area of deformed zone available all over the surface. This brings us to the second reason.

2) Each inhomogeneity can be considered as a small porosity from which crack can nucleate. Only difference between the inclusions inside the material from which the cracks nucleate at sub-surface, and those on the LSP damage zone is their size; those in the damage zone being bigger. The maximum stress concentration factor Kt for a spherical cavity has been shown to be independent of the cavity size as [Timoshenko & Goodier 1970]:

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Where ‘ν’ is the Poisson’s ratio. Hence, as long as the pore diameter is small compared to the sample, most of the crack initiation at low loads has equal stress concentration for small cavities, for sub-surface as well as those in the LSP damage zone. This leaves only the residual stress distribution profile as the determining factor, and the crack nucleates at the sub-surface depth where the residual stress has decreased to a value lower than the nominal tensile stress of the material. Hence, our endurance limit should give us an idea of the depth at which the crack will initiate. For our room temperature fatigue tests, the mean cyclic stress based endurance limit for LSP treated IN718Plus is around 300 MPa from our observations (Figure 6.47). Comparing this with our depth profile measurements on LSP treated IN718Plus coupon (Figure 6.34), we can see that the depth at which the residual stress is reduced to 300 MPa is around 100µm. Hence we can safely expect the subsurface depth of around 100 microns to be where the crack nucleation can be expected. This is in agreement with fractographic observations (Figure 6.96) where the crack nucleation site occurs around 100 microns depth.

Though the results are in agreement, we cannot directly generalize this mechanism to all materials and conditions. $K_t$ has geometrical dependence, while sub-surface nucleation has strong microstructural dependence. Hence, we qualify these observations to our material, LSP conditions and geometry.

**LCF:** While most of the life of a sample at low stresses under high cycle fatigue consists of nucleation of cracks. Since LSP creates a shell of compressive layer outside the sample, these
cracks have to nucleate from subsurface regions. This makes it more difficult to nucleate the fatigue crack and increases the endurance. On the other hand as we increase the stresses and reach closer to the $\sigma_{ys}$ of the material, most of the life of material is spent in crack propagation (low cycle fatigue). Accordingly, nucleation is no longer as important in determining fatigue life. This can be seen from the general dependence of fatigue failure mechanism with temperature and stress level that shows three stages (Anton 1985). LSP has greatest effect on the porosity nucleation initiated fatigue and at higher stresses in LCF region, LSP effects will be reduced. This is reflected in our S-N curve results of LSP treated and non-LSP samples (Figure 6.47).

Effects of LSP tend to diminish as number of cycles to failure is reduced by increasing the mean cyclic stress. Both peened and unpeened fatigue lives tend to converge near the $\sigma_{ys}$. Along with reduced contribution of crack initiation, additionally residual stress relaxation by yielding tends to diminish the effects of LSP at higher mean stresses and the material starts behaving more like unpeened material.
Figure 6.91: Crack branching in sample centre (red arrow)
Figure 6.92: Transition in fracture mode under LSP region
Figure 6.93: Deformed LSP surface layer
Figure 6.94: SEM of 923K LSP fatigued surface showing location of LSP edge and nucleation site
Chapter 7: Conclusions

1) **Total life fatigue:** Room and high temperature S-N fatigue curves have been generated with and without LSP for IN718Plus. Simulations, XRD residual stress measurements and nano-indentation are used to find conditions for fatigue life improvement. High cycle fatigue life improves with LSP at both room and high temperatures, for simulation optimized flat tensile geometry and LSP procedure. High temperature fatigue strength at 923K is significantly higher than room temperature attributed to dynamic precipitate defect interactions. Fatigue limiting factors have been identified in materials treatment as well as LSP processing conditions.

2) **Fatigue crack growth:** Based on a conservative approach considering critical applications, fatigue crack growth is reported to be insensitive to LSP based improvements. Room temperature results on MT geometry are presented, and found to match lack of improvement reported in literature. Crack tip plasticity induced closure activity increased though crack opened after peening leading to faster crack growth rates. Crack tip overloads improve in fatigue resistance not just due to crack tip blunting, but also by inducing compressive stress ahead of crack tip.

3) **Residual stresses:** An XRD based residual stress measurement procedure was used. Thermal exposure reduces residual stresses to about half their original value based on LXRD results, and in agreement with our earlier SXRD results that show strain relaxation up to around 50-60% value with heat treatment. The distribution of stresses across the sample tends to remain unaffected. Fatigue cycling induced stress relaxation is observed in overload samples. Samples with nominal compressive stress show better fatigue behavior that varies with peening conditions.
and geometry. Hence, simulations are vital to controlling the tensile distribution and in conjunction with residual stress measurements. These are an important quality control parameter to minimize residual stress variability associated with high energy LSP.

4) **Laser shock peening:** Effects of LSP were observed and residual stress measurements used with simulations to optimize the LSP process. Single spot nano-indentation hardness increases with LSP energy indicating improved cold work in the material. Multiple shot LSP sequence overlap and number of layers are optimized, using FEM simulations and experiments to give improved fatigue results. Increasing number of layers does not generate a proportionally increasing compression. Very high energy density (8 - 10 GW) LSP can generate high tensile stresses in the centre of the thin specimens. An energy of 2J to 3J was found to be most beneficial for fatigue. Highest compression is seen in the final shots in a multiple shot sequence. Each subsequent shot also partly relaxes compressive stresses induced by earlier shots unless it is on the same location. Maximum specimen thickness that gives through thickness compression was found to be around 2 mm.

5) **IN718Plus:** Brittle plate shaped phases are observed to be potential detriment in fatigue crack growth resistance and fracture strength over long term aging. Being stress concentrators they lead to higher probability of crack initiation, if allowed to form as in case of untested heat treatments, and reduce HCF fatigue life. Dislocation density seen in as received microstructure reduces to low values upon static ageing at service temperatures. LSP induced dislocations coupled with fast coarsening kinetics of \( \gamma' \) may effect dynamic precipitation, which could account for improved fatigue resistance at higher temperatures, compared to room temperature.
A topical summary of conclusions from the perspective of the main objectives of this study that were intended are:

A. Effect of laser shock peening was studied on fatigue and fatigue crack growth behavior of IN718Plus superalloy at room and service temperatures. We also investigated relaxation of LSP residual stresses by fatigue and thermal effects. The underlying microstructure was studied using transmission electron microscopy.

B. Simulations of LSP-induced stresses are done using LSDYNA software. Experimental validation is done from residual stress measurements using XRD, nano-indentation and electron microscopy.

C. This work adds confidence in long-term fatigue life prediction and remnant life assessment of LSP treated alloys while attempting a general treatment of damage for similar systems in an experimental and simulation based synergistic framework, the role of precipitates in IN718Plus is investigated at high temperatures under monotonic and cyclic loading. Finally, we investigated residual stress retention after laser shock treatment upon thermal ageing in the alloy, which is vital towards estimating design life of the components subjected to high-energy laser shocks for inducing residual compressive stresses to enhance the fatigue life of components.

A) Fatigue

1) Total life fatigue: Room and high temperature S-N curves have been generated with and without LSP for IN718Plus under tension-tension loading on a flat tensile geometry. LSP shows
improvement in fatigue life at both room and high temperature. This is in agreement with similar improvements reported in literature by application of LSP.

A methodology has been arrived at using FEM Simulations and residual stress measurements using XRD and nano-indentation technique that is found to give repeatable fatigue life improvements. This methodology can be used by subsequent researchers. Limitations of the process have been identified in materials (heat treatment, brittle precipitates) as well as LSP processing (effect of overlap, effect of poor sequencing, effect of shot energy, ablative media, misalignment etc).

2) Fatigue crack growth: Fatigue crack growth results are presented in a conservative manner considering the critical applications of this superalloy in aircraft components. Lack of improvement is reported for fatigue crack growth rates upon application of LSP in a standard M(T) geometry used in out tests, in agreement with the most conservative reports in literature. Overload and crack closure effects are identified and reported in LSP fatigue. Application of overload followed by crack tip LSP are found to slow the fatigue crack growth rate.

B) LSP

LSP parameters were optimized for fatigue life improvement in IN718Plus using FEM simulations, experiments, literature as:

B1) LSP procedure

1) Laser material interaction: Physical (SEM, interferometric) and mechanical (nanoindentation, XRD) effects were studied and observations are reported. LSP induced
severe plastic zone just below the surface is found to be of the order of 30-50 microns. Brittle precipitates are found to reduce the fatigue strength of LSP treated alloy. Hardness profile across the LSP region is plotted and shows a sharp increase of hardness at the boundary, TEM investigations show a corresponding sudden increase in dislocation density in the LSP treated region compared to non-LSP treated region accounting for the increased hardness.

1) Effect of shot energy on LSP residual stresses: Higher energies are found to be not necessarily beneficial, lower to intermediate energies found to be better in improving fatigue life.

2) Effect of shot sequence on LSP residual stresses: Good sequencing and sequence optimization is found to be critical to controlling residual tensile stresses associated with outer edge of LSP shot that can limit fatigue life.

3) Effect of shot overlap on LSP residual stresses: Partial overlap is found to be helpful but excessive Shot overlap does not always improve residual compressive stresses.

4) Effect of number of layers on LSP residual stresses: Successive layers are found not to generate proportionally higher residual compressive stresses, and tend to show saturation in behavior beyond the second layer.

B2) LSP Specimen

1) Optimizing specimen geometry for LSP fatigue life: Specimen was geometry optimized based on project goals, simulations, materials available, the testing facilities and test standards.
2) Effect of specimen thickness: A thickness of around 2mm was found to be the transition beyond which through thickness compression is not achieved. Samples smaller than 2 mm show exhibit through thickness compression, but may not be representative of real life situations, hence 2 mm thickness was chosen for most testing.

3) Effect of specimen misalignment on LSP residual stresses: Specimen misalignment was found to result in redistribution of stresses and often harmful by generating uncompensated tensile stresses.

4) Effect of heat treatment on LSP fatigue life: Suitable heat treatment was found to be vital to improving fatigue life. Non-standard heat treatment like those encountered during repair welding could lead to precipitation of brittle plate shaped $\eta$-$\text{Ni}_3(\text{Al},\text{Ti})$ that can reduce fatigue strength.

**B3) Residual stresses**

1) A residual stress measurement procedure was established using LXRD using FEM simulations as a guiding tool. Absolute, non-destructive measurement of residual strains was demonstrated using SXRD. An inexpensive alternate Eigenstrain analysis was demonstrated for plate geometry for the given LSP conditions to be able to corroborate the XRD residual stress observations of residual stress profile based from surface distortions measurements from VEECO profilometry, and can be useful for quick estimation of residual stress with simple geometries.
2) Thermal relaxation of residual stress was characterized using LXRD mapping and SXRD measurements. Stresses are found to reduce to around 50% of their original value upon service temperature exposure.

3) Fatigue induced relaxation of residual stress was characterized using LXRD mapping. Stresses are found to reduce to around 50% of their original value upon fatigue cycling and in an overload crack tip plastic zone.

C) IN718Plus Superalloy

1) Fracture of Brittle plate shaped precipitates was identified as a microstructural detriment under application of LSP

2) High dislocation density was retained under LSP, indicating strong strengthening effect. Dislocation interaction with precipitates in IN718Plus takes place in pairs by precipitate shearing as in other γ’ strengthened superalloys. Dislocation density for LSP treated specimens was found to be higher than both fatigued and unfatigued specimens. Dislocation density for fatigued specimens is found to be higher than unfatigued specimens.
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Peer reviewed publications and presentations on this work

Journal:


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