Swept frequency eddy current (SFEC) measurements of Inconel 718 as a function of microstructure and residual stress

Ramya Chandrasekar
Iowa State University

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Swept frequency eddy current (SFEC) measurements of Inconel 718 as a function of microstructure and residual stress

by

Ramya Chandrasekar

A dissertation submitted to the graduate faculty

in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

Major: Materials Science and Engineering

Program of Study Committee:
L. Scott Chumbley, Major Professor
    Alan Russell
    Iver E. Anderson
    Nicola Bowler
    Stephen Holland

Iowa State University
Ames, Iowa
2013

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DEDICATION

To my

Dad Chandrasekar

Mom Manonmani

Professor R. Bruce Thompson
# TABLE OF CONTENTS

DEDICATION .................................................................................................................. ii

TABLE OF CONTENTS .................................................................................................. iii

ABSTRACT ...................................................................................................................... vi

CHAPTER 1: INTRODUCTION ....................................................................................... 1
  1.1 Background ........................................................................................................... 1
  1.2 Nickel based super alloys .................................................................................. 1
    1.2.1 Solid Solution Hardened alloys ................................................................. 2
    1.2.2 Dispersion Strengthened alloys ................................................................. 3
    1.2.3 Precipitation Hardened alloys ................................................................. 3
    1.2.4 Microstructure of Precipitation Hardened Ni-based alloys ..................... 4
    1.2.5 Mechanical Properties of Precipitation Hardened Ni-based Super Alloys ...... 7
    1.2.6 Failure of Ni-based superalloy parts ....................................................... 10
  1.3 Residual Stresses ............................................................................................... 11
    1.3.1 Compressive Stresses ............................................................................... 11
    1.3.2 NDE Measurement of Residual Stress .................................................... 12
  1.4 Nondestructive Evaluation ................................................................................ 13
    1.4.1 Eddy Current Measurements ................................................................... 14
    1.4.2 Limitations of Eddy Current Signals ....................................................... 15
    1.4.3 Factors Contributing to Eddy Current Signals ......................................... 16
  1.5 Swept Frequency Eddy Current (SFEC) Measurements .................................. 23
    1.5.1 Theory ....................................................................................................... 23
    1.5.2 Instrument ................................................................................................. 23
    1.5.3 Instrumental Factors Affecting SFEC Measurements .............................. 26
  1.6 Measurement of Stress Induced by Shot Peening ........................................... 27
  1.7 Problem Statement ........................................................................................... 30
  1.8 References ......................................................................................................... 31

CHAPTER 2: EXPERIMENTAL PROCEDURE ............................................................ 39
  2.1 Sample Preparation .......................................................................................... 39
CHAPTER 5: ANALYSIS OF EDDY CURRENT SIGNALS ................................................................. 98
  5.1 Conductivity Deviation Profiles .................................................................................. 98
  5.2 Determination of Near Surface Conductivity Variation ........................................... 107
  5.3 References .............................................................................................................. 120

CHAPTER 6: DISCUSSION .................................................................................................. 121
  6.1 Effect of heat treatment on Eddy Current ............................................................... 121
  6.2 Effect of Shot peening on Eddy Current ................................................................. 123
  6.3 Combined effect of heat treatment and shot peening on eddy current signals .. 124
  6.4 References .............................................................................................................. 125

CHAPTER 7: CONCLUSION ............................................................................................... 126

CHAPTER 8. RECOMMENDATIONS FOR FUTURE WORK ............................................ 128

ACKNOWLEDGEMENTS ................................................................................................. 129
ABSTRACT

The goal of this thesis was to determine the dependency of swept frequency eddy current (SFEC) measurements on the microstructure of the Ni-based alloy, Inconel 718 as a function of heat treatment and shot peening. This involved extensive characterization of the sample using SEM and TEM coupled with measurements and analysis of the eddy current response of the various sample conditions using SFEC data. Specific objectives included determining the eddy current response at varying depths within the sample, and this was accomplished by taking SFEC measurements in frequencies ranging from 100 kHz to 50 MHz. Conductivity profile fitting of the resulting SFEC signals was obtained by considering influencing factors (such as surface damage). The problems associated with surface roughness and near surface damage produced by shot peening were overcome by using an inversion model. Differences in signal were seen as a result of precipitation produced by heat treatment and by residual stresses induced due to the shot peening. Hardness of the material, which is related both to precipitation and shot peening, was seen to correlate with the measured SFEC signal. Surface stress measurement was carried out using XRD giving stress in the near surface regions, but not included in the calculations due to shallow depth information provided by the technique compared to SFEC. By comparing theoretical SFEC signal computed using the microstructural values (precipitate fraction) and experimental SFEC data, dependency of the SFEC signals on microstructure and residual stress was obtained.
CHAPTER 1: INTRODUCTION

1.1 Background
Superalloys are among the most compositionally complex alloys ever developed. They contain many alloying elements, producing different phases which are responsible for the mechanical properties possessed by the superalloys. Superalloys are used in extreme conditions, such as corrosive environments or high temperature structural applications where oxidation is problematic. Ni superalloys in particular possess the mechanical properties necessary for use at high temperature operating conditions. Due to the number of alloying elements, a wide range of alloys can be developed by slightly varying the composition. This allows enhancement of the properties of the alloys depending on the intended applications.\textsuperscript{1,2}

The ability to operate gas turbine engines at high temperatures and stress was the major focus of this industry as companies sought to develop an alloy that can withstand these needs. Development of gas turbines began in 1872 when Dr. Frank Stolze described a device similar to a gas turbine engine, while in the early 1900s Charles Curtis produced the first working models of gas turbine engines.\textsuperscript{3} In the 1910s austenitic stainless steels became the primary choice for an alloy that could handle the high-temperature applications required.\textsuperscript{3} The next major step occurred in the 1950s, when Eiselstein introduced Inconel 718, a Nickel based alloy with an operating temperature of 1300°F, and is arguably the first superalloy that was introduced. Continuous improvement has occurred since that time.

1.2 Nickel based super alloys
The classification of superalloys is based on the alloying elements present in them. The main alloying elements are iron, cobalt and nickel. The general operating temperature range of superalloys is above 1000°F. The characteristics of Nickel based superalloys
apart from high operating temperature are their improved mechanical properties such as high toughness, ductility, and low cost. The low cost of nickel–iron–based superalloys is obtained by adding substantial amount of iron to the alloy without compromising the properties necessary for the desired application. They are widely used in applications such as turbine discs or forged rotors.

Ni-based superalloys are developed by adding various alloying elements to the base metal to achieve the desired properties. The chemical composition of Ni-based superalloy consists of 10-20% Cr, 5-10% Co, 25% Mo max, with 8% Ti and Al max. In the case of nickel-based superalloys, the base Ni alloy is commonly alloyed with Al, Ti, and Nb. These additions help the superalloy obtain high strength. They also form intermetallics which can melt at high operating temperatures. For aircraft engine applications the strength-to-weight ratio needs to be considered when considering the mechanical property of any alloy. In the case of Ni-base superalloys, additions such as Al and Ti, which are low density elements, reduce the weight of the superalloys while still providing good mechanical properties. Based on the elemental addition used and the strengthening mechanisms provided, superalloys can be developed to provide a) solid-solution hardening, b) precipitation hardening and/or c) dispersion strengthening. These basic mechanisms of strengthening are briefly described below.

1.2.1 Solid Solution Hardened alloys
Solid solution hardening is achieved by the addition of a different soluble element to the matrix to increase its strength. Misfit of atomic radius between the solute and the matrix results in distortion of the atomic lattice. The subsequent strain caused by this misfit may be either tensile or compressive in nature, depending on the size of the atom introduced. In either case the strain inhibits dislocation movement. The typical elements used in solid-solution hardening of Ni-based superalloys are aluminum, iron, titanium, chromium, tungsten, and molybdenum. This type of hardening also decreases the stacking fault energy present in the crystal lattice of the alloy. Due to low stacking
fault energy the movements of dislocation cross slip are arrested. This prevents the deformation occurring at high temperatures$^8$.

1.2.2 Dispersion Strengthened alloys
Dispersion strengthening is a mechanism where a strengthening agent is added to the alloy. The strengthening agent may be particles of an entirely different phase or material with properties vastly different from the matrix. For example, oxide particles added to a melt can strengthen the matrix by blocking dislocation motion present in the matrix alloy. The advantage of dispersion strengthening is that usually a suitable particle, which is inert with respect to the matrix, can be added to almost any melt. The drawback of such adding strengthening agents is that their crystal structure is incoherent with the matrix phase. Therefore, this mechanism provides strengthening through Orowan bypassing mechanism, where dislocations move either by cutting through or bypassing the precipitates$^9$. This will be discussed in section 1.2.5.

1.2.3 Precipitation Hardened alloys
Precipitation hardening is achieved by generating finely distributed precipitates in the matrix from a supersaturated solid solution during heat treatment. In the case of nickel-based alloys, finely distributed precipitates are produced by adding elements such as titanium, aluminum, and niobium. As these elements have limited solubility in the alloy matrix, low temperature anneals of solid solutions enables the production of finely distributed precipitates. Secondary phase precipitates such as γ’- Ni$_3$ (Ti, Al) or γ”-Ni$_3$Nb phase reduce the movement of dislocations and hence increase the strength. As the precipitates are produced within the matrix they are coherent with the matrix. In this case as stated by Stoloff et. al, "Dislocation movement is inhibited both by the strain field surrounding coherent precipitates and the particle itself. Thus, precipitation hardening is inherently more effective than simple dispersion strengthening since two different strengthening mechanisms are operative. Movement of a dislocation in the matrix containing precipitates can only take place either by cutting through or by
bypassing the particles. The drawback of precipitation hardening is that since the precipitates develop from the matrix, heat treatment becomes critical in creating the right size and/or dispersion of precipitates for maximum strengthening. Since the alloys examined in this study are strengthened by precipitation hardening a brief discussion of microstructure is now in order.

1.2.4 Microstructure of Precipitation Hardened Ni-based alloys

Nickel has a face centered cubic (FCC) crystal structure and the major phase is designated as \( \gamma \) after the high temperature FCC Fe phase. In precipitation strengthened Nickel-based super alloys containing titanium and/or aluminum, the strengthening secondary phase precipitates are \( \text{Ni}_3\text{Al} \) or \( \text{Ni}_3\text{Ti} \). Since these precipitates are also FCC and ordered, both Al and Ti atoms occupy specific sites within the FCC framework and are designated as \( \gamma' \). The crystal structure of \( \text{Ni}_3\text{Nb} \) precipitates as body-centered-tetragonal (BCT) and are designated \( \gamma'' \). In the early stages of heat treatment when the precipitates are still small, the precipitates are generally coherent with the matrix, thus restricting the dislocation motions and providing maximum strengthening effect. In such a case, movement of dislocation can only take place by cutting through or by bypassing the particles.

The alloys A-286, V-57, Nimonic 901 and Inconel 718 are the major precipitation strengthened alloys. In these alloys \( \gamma' \) is the major strengthening precipitate with \( \gamma'' \), also present in Inconel 718. Inconel 718 is a highly weldable superalloy and the presence of \( \gamma'' \) is due to the addition of niobium. Thus, dual strengthening precipitates occur when combining the elements such as aluminum, titanium and niobium, resulting in strengthening of the superalloy by the presence of both the secondary phases (\( \gamma' \) and \( \gamma'' \)). The chemical composition of Inconel 718 is given by Table 1.1. Secondary alloying elements are added to change the corrosion resistance, creep properties, strength, and grain refinement behavior of Ni-alloys.
Inconel 718 is frequently used for cryogenic storage tanks, turbine engine components, and in corrosive environments such as oil well drill-shafting and well-head parts. Other uses include jet rocket, nuclear fuel, and pump body components. Inconel 718 is also ideal for hot extrusion tooling and is used for any number of parts including nuclear fuel element spacers and high-strength bolts due to its attractive combination of durability and weldability. Inconel 718 also can withstand a wide range of temperature extremes, which makes it a useful alloy for both cryogenic and high temperature applications.

**Table 1.1** Chemical composition of Inconel 718.

<table>
<thead>
<tr>
<th>Alloying Element</th>
<th>Ni</th>
<th>Cr</th>
<th>Nb</th>
<th>Mo</th>
<th>Ti</th>
<th>Al</th>
<th>Co</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max (wt. %)</td>
<td>-</td>
<td>21.00</td>
<td>5.50</td>
<td>3.30</td>
<td>1.15</td>
<td>0.80</td>
<td>1.00</td>
<td>0.08</td>
<td>0.35</td>
<td>0.35</td>
<td>0.30</td>
</tr>
<tr>
<td>Min (wt. %)</td>
<td>50.00</td>
<td>17.00</td>
<td>4.75</td>
<td>2.80</td>
<td>0.65</td>
<td>0.20</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Figure 1.1 -1.4 shows several typical microstructures of Inconel 718 at various heat treatment conditions. The γ’ (Ni₃Al and Ni₃Ti) precipitates that form upon heat treatment to strengthen the matrix generally occur as spherically shaped precipitates while γ’’ (Ni₃Nb) tends to be disc shaped.

![Figure 1.1 SEM micrograph of ring-rolled IN 718 after solution treatment at 1025°C/1h]
Figure 1.2 TEM micrograph (bright field) of spray-formed IN 718, heat treated at 875 °C/6 h showing early precipitation of delta plates. The larger ellipsoidal particles present are $\gamma^\prime$.

Figure 1.3 SEM micrograph of spray-formed IN 718, heat treated at 850°C/24 h.
As for all precipitation hardened alloys, the actual strength reached depends on a combination of heat treatment temperature and time-at-temperature\textsuperscript{12}. The mechanical properties of the precipitation hardened alloys changes rapidly in the vicinity of the solvus temperature of the strengthening precipitates\textsuperscript{1}, and so heat treatments must be carried out very carefully. A brief review of mechanical properties associated with precipitation hardening in Ni-based alloys is provided in the next section.

### 1.2.5 Mechanical Properties of Precipitation Hardened Ni-based Super Alloys

High creep and creep-rupture strengths are required for applications such as turbine airfoils and disk engines\textsuperscript{2} due to the high operation temperatures and high stresses encountered in service. Good ductility is also required to avoid fracture, which will lead to engine failure.\textsuperscript{2} Such mechanical properties and creep strength for high temperature applications can be obtained by precipitation hardening, where the precipitates act to pin dislocations and grain boundary motion. Pinning of dislocations helps improve the tensile strength while pinning of grain boundaries improves creep resistance\textsuperscript{1,2,9}. 

![SEM micrograph of spray-formed IN 718 after heat treatment at 950 °C/50 h\textsuperscript{11}.](image)
The effect of precipitate hardening depends on several factors such as:

1. Coherency strains\textsuperscript{1,2} that exist between the matrix (\(\gamma\)) and the precipitate (\(\gamma', \gamma''\)).

2. Antiphase – boundary (APB) energy\textsuperscript{1,2}. The APB represents the energy needed for the dislocation to cut through the ordered precipitate. Cutting of dislocations creates disorder between the matrix and the precipitate.

3. Volume fraction of the secondary phase precipitates\textsuperscript{1,2} (\(\gamma', \gamma''\)) present in the matrix.

4. The average particle size of the precipitates\textsuperscript{1,2}.

**Coherency Strains:** Dislocations can either cut through precipitates (smaller precipitates) or bypass the precipitates (larger precipitates) depending on their size. As the precipitate size changes, the strengthening obtained by precipitate hardening will also change. Therefore, an increase in precipitates size increases the hardening effect as long as the precipitate remains coherent, due to coherent strains and ordering. However, once the precipitates become incoherent then increase in strength with increasing particle size is given by the Orowan equation (Equation 1.1)\textsuperscript{13}.

\[
\frac{dy}{dt} = \rho b v \tag{1.1}
\]

where \(\frac{dy}{dt}\) is the shearing rate, \(\rho\) is the dislocation density, \(b\) is the magnitude of the burgers vector (denotes the magnitude of lattice distortion by a dislocation), and \(v\) is dislocation velocity. Strength can be seen to increase but is limited by Orowan bowing, where the dislocation will bypass the particle.\textsuperscript{13}.

The shape of coherent particles depends on a balance between the elastic strain energy associated with the lattice mismatch between precipitate and matrix and by the interfacial energy of the particle-to-matrix boundary\textsuperscript{14}. The elastic strain energy depends
on the shape, habit, and volume of the precipitates, while interfacial energy merely depends on the surface area of the precipitates. For a matrix with similar lattice parameters, a spherical precipitate is formed, while for large differences in lattice parameters a cuboidal precipitate is usually formed\(^1\).

**Antiphase Boundaries:** Similar to coherency strains, APBs are effective in preventing dislocation motion and act as a hardening mechanism. The general strengthening equation for solid solution strengthening was estimated by the Fleischer model\(^{15}\) and is given by equation 1.2.

\[
\tau_y = \frac{G \varepsilon_s^{3/2}}{700} \sqrt{c}
\]

where, \(\tau_y\) - shear stress, \(G\) - shear modulus, \(\varepsilon\) - total strain and \(c\) - concentration of solutes. This equation can be used to estimate the approximate stress that can be withstood by Inconel 718 material with different solute concentrations.

**Volume Fraction and Particle Size:** The effect of volume fraction and particle size of precipitates (which determines mean particle spacing) on mechanical properties is given by the well-known Hall-Petch Relationship (Eq 1.3)\(^{16}\):

\[
\sigma_y = \sigma_i + \frac{\kappa_y}{\sqrt{D}}
\]

where \(\sigma_y\) - yield stress, \(\sigma_i\) - materials constant for the starting stress for dislocation movement, \(\kappa_y\) - Petch Parameter (unpinning constant), \(D\) - average grain diameter and/or particle spacing. The degree to which hardening of material occurs depends on the material’s Hall-Petch coefficient and the degree of grain-size / particle spacing refinement possible in the material\(^{17}\). This equation helps to correlate the effect of precipitate spacing and concentration, present in different microstructures, to the strength of the materials.
1.2.6 Failure of Ni-based superalloy parts

During service, failure of Ni-based superalloy parts due to insufficient strength of the material to withstand the applied load is rare. Rather, overload failures occur when failure of another component causes a stress to be applied at a level that far exceeds the initial design criteria. At high operating temperatures, creep can play an important role in the rupture life of a Ni-based turbine blade part\textsuperscript{2}, and for this reason directionally solidified and single crystal blades were developed. Currently the most common mode of failure is cracking / rupture due to fatigue. Fatigue is defined as a progressive structural damage that occurs when the material is subjected to a cyclic loading that is below the tensile yield stress limit. Failure due to fatigue is common as virtually all alloys and parts under cyclic loading show fatigue in service applications. Low-cycle fatigue occurs in gas turbine disks, cases, and other structures highly loaded in tension but not in creep-rupture conditions. Fatigue life is related to many factors such as defects, surface finish, applied load, and the inherent non-cyclic strength properties of an alloy, etc. Low-cycle fatigue is related to the yield strength of the alloy, while high-cycle fatigue is related more to the ultimate strength of the material. Both are related to the amount of residual stress present in the material since the presence of a tensile stress is one of the prime contributors for fatigue to occur. Successful incorporation of residual stress measurements in life management models will require a good understanding of the surface and sub-surface residual stress changes\textsuperscript{18,19} since these values play a critical role in accurate determination of predicted life as a function of applied stress. In stress and strain-life calculations residual stress is usually regarded as a static stress that is added to the mean stress during each load cycle\textsuperscript{19,20}. The introduction of residual compressive stresses to offset either residual or applied tensile stresses is thus of major interest and concern when evaluating the life of any part, especially life-critical parts such as used in jet turbine engines. A summary of how compressive stresses can be introduced and measured is given in the next section.
1.3 Residual Stresses
Residual stresses can be defined as stresses that exist in the absence of any external loading or thermal gradients within the material. Manufacturing processes are the most common causes of residual stress. Virtually all manufacturing and fabrication processes such as casting, welding, machining, molding, heat treatment, plastic deformation during bending, rolling or forging introduce residual stresses into the manufactured object.

While residual stresses are introduced as a result of plastic deformation they penetrate further into the sample than the deformed region. Consider the case of shot peening. As the surface of the material is deformed it first yields elastically, up to the yield point, then plastic deformation begins. This means the elastic region is distributed into the material and extends well into the sample, often hundreds of microns, while the plastic deformation may be limited to a few tens of microns on the surface. After the ball rebounds from the surface the material tries to regain the elastic deformation that has occurred. However, since the deformation on the surface has essentially produced a longer distance (that must somehow be accommodated as the material tries to recover elastically) the elastic stress distributed throughout the depth of the part near the surface is unable to fully relax. The material is thus left with a residual compressive stress layer, which can be very beneficial in certain applications.

1.3.1 Compressive Stresses
Surface enhancement treatments are widely applied to fracture-critical metallic components such as turbine disks of aircraft engines. These treatments significantly improve fatigue resistance of the components by introducing protective compressive residual stresses in the surface layer. Surface compressive stresses are known to be beneficial to slowing crack initiation and propagation associated with the phenomenon of fatigue$^{9,21}$.
Improvement of cyclic fatigue performance of engineering components has been achieved by the application of various surface treatments such as shot peening, laser peening, ion implantation or processing methods after a component has been manufactured by such processes as casting, welding, forming, machining, etc. Among the surface treatment methods shot peening, i.e., the rapid bombardment and subsequent shallow deformation of the work piece surface by hard projectiles (e.g., shot) has been widely used in industry. The shot peening process induces compressive residual stresses on the surface, thus improving the cyclic fatigue life and decreasing the susceptibility of the component material to corrosion cracking\textsuperscript{22-30}.

1.3.2 NDE Measurement of Residual Stress
As stated above, residual stress needs to be included in life prediction of many components. It has been over 100 years since the first reporting of residual stress measurement\textsuperscript{31} occurred. All early measurement techniques relied on destructive means where the part is cut to observe any dimension changes occurred in the component\textsuperscript{32}. In recent decades non-destructive evaluation (NDE) diffraction techniques, such as high energy neutron and synchrotron diffraction, have become powerful tools for residual stress studies. Unfortunately, an accurate nondestructive means of determining residual stress that can easily be applied in a manufacturing environment or routinely conducted as a part of regular maintenance of a component remains elusive\textsuperscript{18}. Because of the size, availability and lack of portability of diffraction techniques these measurements are primarily limited in application to parts in the development stage.

While X-ray diffraction (XRD) is the standard method for residual stress measurements, penetration to \(\sim 200 \, \mu\text{m}\) in depth without surface material removal requires higher energy X-ray sources, such as synchrotron sources or a dedicated laboratory system\textsuperscript{33}. Such instruments are expensive and, therefore, not routinely employed for manufacturing. What is desired is a process that enables accurate monitoring of
residual stress at life limiting critical locations using nondestructive evaluation (NDE) techniques. NDE of the surface and subsurface residual stresses is the key to fully exploiting the benefits of the surface treatments, thereby extending the service lives of the components and ensuring system reliability.  

Potential NDE techniques available for measuring residual stresses are 1) Ultrasonic testing 2) X-ray diffraction and 3) Electromagnetic methods. All these NDE techniques have been studied for residual stress profiling. Ultrasonic testing can be used to measure bulk residual stresses; X-ray diffraction can be used to measure surface residual stresses approximately 10-20 microns deep. Among electromagnetic methods, the eddy current (EC) technique was identified as a leading candidate for nondestructive characterization of near-surface residual stress profiles based on the piezoresistivity effect, which refers to the stress-induced changes in electrical conductivity. The basics of eddy-current measurement are discussed in the next section.

1.4 Nondestructive Evaluation

Nondestructive evaluation/ testing (NDE/ NDT) is used to examine an object, material or system without causing damage. NDE techniques can be divided into different techniques, each based on a scientific principle. The most commonly used NDE techniques are electromagnetic testing, ultrasonic testing, magnetic particle testing, acoustic emission testing, acoustic resonance testing, infrared testing, dye penetrant testing, radiographic testing, and visual testing. Electromagnetic testing is the process of inducing electric currents and/or magnetic fields inside a test object and observing the response. A defect in the test object may be detected where electromagnetic interference creates a measurable response, e.g. an eddy current measurement.
1.4.1 Eddy Current Measurements

The eddy current method consists of passing an alternating current through a coil so as to induce circulating currents, i.e. eddy currents, in an electrically conducting object in its vicinity. A basic eddy current device is shown schematically in Figure 1.5. An impedance analyzer measures the impedance produced in the applied current that results due to the eddy currents within the object. Depending on the sample conditions (homogenous sample, cracks, etc.), the impedance signal will change. As the penetration of the eddy current is determined by the frequency used, one can accurately measure at which depth the sample loses its homogeneity. Eddy current tests can be made on all materials that are electrically conducting. Applications include the sizing of surface and sub-surface cracks, measurement of the thickness of metallic plates and of non-metallic coatings on metal substrates, assessment of corrosion and measurements of electrical conductivities and permeabilities.

Figure 1.5 Basic eddy current test setup.
Eddy currents are confined to the near surface region by the skin effect (Figure 1.6)\textsuperscript{42}. The depth that eddy currents penetrate into a material is affected by the frequency of the excitation current and the electrical conductivity and magnetic permeability of the specimen. The depth of penetration decreases with increasing frequency and increasing conductivity and magnetic permeability. Due to this skin effect, the eddy current method can be expected to accurately characterize surface modification at different depths in the range of a few mm to hundreds of mm, have good spatial resolution, and provide the ability to scan large areas rapidly\textsuperscript{43}.

1.4.2 Limitations of Eddy Current Signals
While eddy current NDE has its own advantages, it also has its own limitations. Eddy current testing is extremely sensitive to surface variations and, therefore, requires a smooth flat surface when observations of small variations are required. A second drawback is that it is applicable to electrically conductive materials only. While eddy current testing can be used on both magnetic and non-magnetic material it is not reliable on some carbon steels for the detection of subsurface flaws as its depth of
penetration is limited and the measured signal depends on the frequency, crack tightness, material property and orientation of eddy current flow to the crack or linearity\textsuperscript{42} of interest.

### 1.4.3 Factors Contributing to Eddy Current Signals

From the discussion above it should be clear that the impedance of an eddy current probe may be affected by number of factors including 1) frequency at which the eddy current signals are measured; 2) electrical conductivity and the magnetic permeability of the test objects of a material, which in turn depends on microstructure, e.g. grain structure, presence of a second phase, work hardening, heat treatment etc.; 3) changes in contact of the eddy current probe with the surface (i.e. lift-off) or fill-factor from probe “wobble” (electromagnetic coupling in the case of rods, tubes, uneven surfaces); 4) the presence of surface defects, such as cracks, and sub-surface defects such as voids and non-metallic inclusions; 5) dimensional changes, e.g. thinning of the object that occurs during service due to wear or corrosion; 6) the presence of supports, e.g. brackets, that may be below the surface being examined; and 7) the presence of discontinuities such as edges\textsuperscript{31}. These effects can be divided into two broad categories, namely, those inherent to the material itself (i.e. material conductivity, microstructure of the material, etc.) and those inherent with the quality of the measurement (e.g. lift-off noise, temperature of the sample when measured, etc.). These will be discussed in turn.

#### Material Conductivity

The conductivity of a material has a direct effect on the eddy current density: the greater the conductivity of a material, the greater the density of eddy currents near the surface. Conductivity in turn is affected by microstructural factors such as 1) “conductivity of the pure (base) metal; (2) the conductivity and volume fractions of the phases within the matrix; (3) the scattering of electrons by small (nanometer-sized) precipitates/zones; and (4) alloying atoms dissolved in the matrix phase" as stated by Blitz et al\textsuperscript{43}. All of these factors come into play when one considers precipitation hardening of an Al-Cu
alloy. This is shown in Figure 1.7. Upon heating a solutionized sample, while the material is still in the pre-precipitation period, one observes a decrease of conductivity followed by an increase as the precipitates starts growing in their size. This might be due to the coherency strains produced by the precipitates; another possibility is that abnormal scattering zones result when the precipitates size is of the same order as the wavelength of the electrons ($\sim 10\text{Å}$) $^{44-50}$. As solute atoms then leave solution and precipitate to harden the alloy a corresponding change in conductivity is seen again. As solute atoms leave the matrix, strain is released, resulting in increased conductivity. However, this must be balanced by the fact that a new, second phase is being created. Thus phase creation has two effects on conductivity a) the phase itself can be expected to have a different conductivity than the matrix, either greater, or less than; b) new interfaces are created as the phase precipitates. The actual conductivity change is a balance of these competing factors.

![Figure 1.7](image)

**Figure 1.7.** Variation of aluminum conductivity with heat treatment$^{51}$.

The entire progression from solution $>$ hardened $>$ overaged $>$ re-solutionizing heat treatment can be monitored using conductivity / eddy current measurements and is
often monitored using simple hardness tests. The above example shows that while microstructural changes can be monitored using eddy current measurements, the exact nature of the change occurring is unknown. A summary of studies that have employed eddy currents to study / monitor various microstructural effects is given in the next section.

**Microstructure**

There have been numerous studies to evaluate microstructure changes using the eddy current method. Eddy current testing has been used to characterize microstructural changes such as grain size and hardness changes after thermal treatments based on measurement of conductivity and magnetic permeability changes. Other examples include measurement of pearlite percentage in plain carbon steels and ductile cast irons, surface carbon content of carburized steels, measurement of case depth of case hardened steel rods and the effect of mechanical micro-hardness on impedance variations.

Evaluation of decarburizing depth of steels with a martensitic base microstructure has been investigated using harmonic analysis and magnetic Barkhausen noise (MBN) emission. An investigation was made on magnetic properties as well as complete eddy current responses of decarburized steel parts. The difference in magnetic properties of the decarburized zone (with a higher percentage of ferrite microstructure) and the core of the specimen (martensite) is the basis of the eddy current method’s capability to determine the decarburized depth of steel rods.

Note that the studies above do not seek to separate the various factors that could be contributing to the measured signal. For example, in precipitation hardening, the overall signal is a sum of the conductivity increase that occurs as solute atoms leave the matrix, and the conductivity decrease that results as precipitation of a second phase
occurs, bringing with it a different conductivity for that phase and introducing strain that affects the conductivity of the parent matrix. Similarly, decarburization of a surface results not only in a change of microstructure from martensite to ferrite but also a release of the residual stress inherent in the formation of the martensite. Thus, the operating assumption is that the microstructure being studied is relatively uniform and that the conductivity differences that exist between the various phases present in the structure are either irrelevant or minor. When measuring the amount of decarburization and its effect on pearlite content of a steel, for example, similar normalization treatments on the samples is assumed to place residual stress at its minimum value and, more importantly, similar for all. In this study it is reported that eddy current outputs are not affected by grain size as the average grain size for the core of steel samples is similar. Thus, the ferrite-pearlite content change is the main factor that affects the eddy current outputs (impedance signals)\textsuperscript{52}.

If one wishes to know the exact contribution of any particular microstructure on the eddy current response, the problem becomes significantly more difficult. Such studies require not only a measurement of the eddy current response, but also a careful monitoring of the microstructural changes that can occur during any surface treatments. Such detailed studies require expertise in both eddy current testing methods and knowledge of material microstructure along with material characterization techniques, such as scanning and transmission electron microscopy. Even in the heavily studied Fe-C system no studies of this type exist since multi-phase systems where the microstructure can vary greatly as a function of heat treatment are especially difficult to analyze.

The same lack of microstructural data as related to eddy-current response exists in Ni-based superalloy systems, where numerous precipitates can occur. Currently no studies of eddy current response as a function of microstructure exist in these complex
systems. The study of eddy current response as a function of microstructure on Ni-based superalloy systems forms the main focus of this thesis.

**Permeability**

Permeability of the material has a significant influence on the eddy current response. It is not uncommon for the permeability to vary greatly within a metal part due to localized stresses, heating effects etc. The disadvantage of inspecting ferro-magnetic materials is that permeability changes generally have a greater effect on the eddy current response than conductivity variations. Crack detection becomes difficult when permeability changes randomly. To account for heterogeneity (such as cracks, non-uniformity in microstructure) existing in the material due to the manufacturing process, a reference sample is commonly used, which is obtained from the same manufacturing process along with the testing sample\(^6\).

Several studies have been conducted to take magnetism into account in eddy current analyses. For example, Uzal et al. calculated the impedance of a cylindrical air-cored probe over layered metallic materials whose conductivity and permeability varied continuously as a function of the depth\(^6\). Also, Kasai et al. have used magnetization to cancel external magnetism\(^6\). In this study, an external magnetic field is applied to the magnetic sample which is being tested, by using a C-core probe. The external magnetic field cancels the magnetic effect of the testing sample. By cancelling out the magnetism effect, the eddy current responses corresponding to the conductivity of testing samples were studied.

While permeability can have a large effect, it is important to note that it causes problems only in materials that are ferro-magnetic. Thus, all irons and many steels are susceptible to permeability problems as are certain Ni-based alloys. For example, Inconel 600 displays this problem. The alloy studied in this thesis, Inconel 718, is a non-
ferromagnetic Ni-based alloy. Thus permeability is not expected to play a role in the analyses conducted below.

**Residual Stress**

The presence of residual stress in a material can also affect the conductivity of the material and thus, affects the eddy current response obtained from the material. Residual stress and the conductivity of the material are related by the positive piezoresistivity effect. The piezoresistivity effect is defined as the change in electrical conductivity of the material when mechanical stress is applied. A review of research by Javier et al showed that many authors have measured residual stress using eddy current techniques. Coils can detect small stress variations in ferromagnetic steels due to the magnetic-elastic effect. Stress can be measured based on the changes in the impedance of an electromagnetic coil. The impedance change occurs due to variations in the electrical conductivity and the magnetic permeability of the test piece in comparison to a calibrated sample.

Recently, Blodgett, Nagy and Yu showed that the apparent conductivity of a nickel-based alloy increased after shot peening. The observations by Blodgett, Nagy and Yu have cast new light on the feasibility of using eddy currents for quantitatively measuring residual stress of nickel-based alloys, which are extensively used in the aviation industry. Yu and Nagy have developed empirical models to relate the measured apparent electric conductivity change (AECC) to the residual stress.

Residual stress may be developed by various surface treatment processes such as shotpeening, laser peening etc. Shot peening a surface causes plastic deformation, which induces a residual compressive stress on the surface, balanced by a residual tensile stress in the interior. The plastic deformation causes the increase in resistivity of
the material. The surface compressive stresses confer resistance to metal fatigue and to some forms of stress corrosion, which is the reason for peening in the first place.

Surface enhancement treatment by shot peening has been widely used in industrial applications, especially for aircraft engine components, to produce compressive residual stresses. Typical peening processes use small shots of a few hundred micrometers in diameter forcefully impinging on component surfaces, resulting in compressive residual stress from the surface to a depth range of a few hundred micrometers nominally. Compressive surface residual stress is useful for improving crack initiation resistance that prolongs service life of the part. To implement this highly desirable maintenance strategy, an in-service nondestructive method is needed to monitor the residual stress state of parts periodically, so that appropriate maintenance actions can be taken when residual-stress protection is lost (relaxing of residual stress occurs), by either replacing or re-treating the part.

Based on the results of, the effects of piezo-resistivity on the eddy-current response can be accounted for, however, the effect of microstructure is still not understood. This research work focuses on examining and understanding the effects of microstructure on eddy current response of shot peened nickel based superalloys. By understanding the effects of microstructure, in relation to the eddy-current response obtained from the material as the microstructure varies, it should be possible to differentiate between microstructural effects and the effects of stress alone. Thus, microstructure observations coupled with eddy current measurements are used in this study. The particular type of eddy-current measurements employed, namely, swept-field measurements, are discussed in the next section.
1.5 Swept Frequency Eddy Current (SFEC) Measurements

1.5.1 Theory
Conventional eddy current measurements are performed under 10 MHz with the smallest penetration depth of around 200 μm for typical aircraft engine materials. However, there is a strong desire to determine residual stress profiles in shot-peened engine components within 200 μm from the surface. Thus higher frequency operation with smaller penetration depths is needed. A swept frequency eddy current (SFEC) system which can operate up to 50 MHz has been developed for electromagnetic nondestructive characterization of residual stresses in shot peened aerospace materials such as nickel-based superalloys with typical conductivities of one to several percent International Annealed Copper Standard (IACS). In this approach, shot-peened surfaces are regarded as modified surface layers of varying conductivity, and the conductivity deviation profile is determined by inversion of the SFEC data. The instrument used is described below.

1.5.2 Instrument
The SFEC measurement system employed (shown in Figure 2.11) consists of a pair of closely matched printed-circuit-board coils driven by laboratory instrument under software control. This provides improved sensitivity and high frequency performance compared to conventional coils, so that swept frequency EC measurements up to 50 MHz can be made to achieve a skin depth of 80 μm for nickel-based superalloys. SFEC measurements up to 50 MHz or higher are prone to liftoff noise and spurious instrumentation effects. This problem was circumvented here by the use of the liftoff-normalized vertical component EC signal $V_{EXP}$ ($V$-component signal, j denotes the imaginary part of the signal) defined in Equation 1.3.
where $H$ is the horizontal component of the signal, $S_T$, $S_R$, and $S_L$ are experimental, complex-valued EC signals, and correspond to, respectively, the test signal, reference signal, and the extra lift off signal. The detailed experimental procedure for this device is discussed in section 2.9.

Figure 1.8 shows an example of horizontal and vertical component signals obtained during 5 rounds of measurement from a solutionized Inconel 718 sample. Examination of Figure 1.8a shows the horizontal component of the signal while Figure 1.8b shows the vertical component. The horizontal component is dependent on lift-off as it is measured in the direction parallel to lift-off. However, the vertical component does not depend on the lift-off variation. This is easily seen in Fig 1.8b by conducting repetitive measurements.

**Figure 1.8** Eddy current signals obtained during repeated measurements from a solutionized Inconel 718 sample. a) horizontal component b) vertical component. Observed variation is due to lift-off variation.
Hence, the use of $V_{\text{EXP}}$ alone offers the advantages that it helps suppress the liftoff noise, while avoiding mismatch due to variation across different instruments used in the frequency range studied. It thereby provides the basis of model-based inversion of conductivity profiles by allowing direct comparison of $V_{\text{EXP}}$ with the theoretical vertical component signal $V_{\text{TH}}$ defined in Equation 1.5.

$$V_{TH} \equiv \text{Im} \left\{ \frac{(Z_T - Z_R)_{TH}}{(Z_L - Z_R)_{TH}} \right\}$$

where $Z_R$, $Z_L$, and $Z_T$ are the corresponding theoretical coil impedances calculated for the reference, liftoff, and test configurations, respectively\textsuperscript{39,72,73}. An additional advantage is that $V_{\text{TH}}$ thus defined takes a particularly simple form under the approximation that holds valid under our measurement conditions used. Specifically, when the relative conductivity changes are sufficiently smaller than unity, and when the outer diameter of the detection coil (assumed a cylindrical air-cored coil) is much larger than any other length parameters, such as the skin depth $\delta$ or coil liftoff $l$, $V_{\text{TH}}$ can take an approximate form shown in Equation 1.6 where $\Delta \sigma(z)/\sigma_{\text{Ref}}$ denotes the small conductivity deviation $\Delta \sigma(z) = \sigma(z) - \sigma_{\text{Ref}}$ relative to the reference conductivity $\sigma_{\text{Ref}}$ as a function of depth $z$.

$$V_{TH}(f) = -\frac{1}{l} \text{Im} \int_0^\infty \exp \left[-(1 + j) \left(\frac{2z}{\delta}\right)\right] \frac{\Delta \sigma(z)}{\sigma_{\text{Ref}}} dz$$

Somewhat remarkably, Equation 1.5 indicates that, under the aforementioned conditions, the $V$-component signal is entirely independent of the coil parameters such as its dimensions and number of windings\textsuperscript{73}. This allows us to use multiple coils optimized for EC measurements in different frequency bands, while yielding continuous broad-band EC spectra so that both the near-surface conductivity profile and the bulk
conductivity can be determined by model-based inversion \(^{57}\). The profiling capability offers the opportunity to detect any pre-existing spatial variations of bulk conductivity that are commonly found in forged components, so that their effects on the detected EC signals can be separated from those induced by surface treatments.

By measuring SFEC signals on samples with different microstructures, and different shot peening intensities with the same microstructure, one can quantify the effect of microstructure and residual stress on the eddy current signals obtained. However, care must be taken to ensure that signal variation due to instrumental factors does not overwhelm the signal due to the effect being studied. This requires careful consideration of the test methodology, in particular addressing concerns due to surface roughness that can produce lift-off. This is discussed in the next section.

### 1.5.3 Instrumental Factors Affecting Swept Frequency Eddy Current Measurements

SFEC measurements will be affected by the same material factors that affect all eddy current measurements, as discussed in section 1.4.2. In addition, there are instrumental factors that must be considered when evaluating any eddy current response, such as lift-off and frequency.

**Lift-Off:** The lift-off is the impedance change that occurs when there is variation in the distance between the inspection coil probe and the test piece. Lift-off variations can be caused by varying coating thicknesses and irregular surfaces encountered during movement of the probe across a surface\(^{74}\). In many applications, eddy current measurements are adversely affected by lift-off\(^{75}\). Lift-off is often considered a noise source and it is especially undesirable in defect detection. For example, the measured signal in the impedance plane due to lift-off could occur in the same direction as that
due to a crack, thereby concealing the crack response. Therefore, the distance between the probe and metal must be as constant as possible in order to avoid lift-off noise $^{51}$.

There are methods for lift-off compensation when eddy currents are used in order to detect cracks and lift-off becomes an undesired variable. Yin et al. researched dual excitation frequencies and coil design to minimize the lift-off effect $^{75}$. Tian et al. have researched the reduction of lift-off effects via normalization techniques $^{74}$. In this work, lift-off is overcome by using the normalization technique as explained in section 1.5.1.

**Frequency:** Eddy current response is strongly affected by the frequency chosen for the investigation. This is the factor controlled by the operator which is chosen based on the application needed. Higher frequencies are chosen for surface characterization while lower frequencies are chosen for bulk characterization. By choosing multiple frequencies covering low to high frequencies ($100$ kHz to $50$ MHz), different depths (~ $100$ μm to $500$ μm) for a Ni-based superalloy sample can be examined $^{76}$.

### 1.6 Measurement of Stress Induced by Shot Peening

X-ray and neutron diffraction methods currently are the only two standard methods considered as being reliable for residual stress measurements associated with shot peening. However, conventional XRD methods achieve relatively low penetration depth (<$10$ μm for most metals), and destructive layer removals are needed for measuring residual stress profiles which typically range from $200$ μm to $2000$ μm in depth for shot-peened materials of practical interest. The neutron diffraction method has a practical limitation in terms of its cost and resulting radioactivity $^{51,65}$.

Several factors need to be considered and evaluated if eddy current NDE is to be used successfully to measure depth of surface residual stress. Nagy and his group developed
a new measurement technique to measure residual stress using eddy current NDE. In this technique, the measured probe coil impedance is evaluated for an “apparent” eddy current conductivity (AECC). Abu- Nabah et. al stated that “At a given frequency, AECC is defined as the electrical conductivity of an equivalent homogeneous, non-magnetic, smooth and flat specimen placed at a properly chosen distance from the coil that would produce the same complex electrical coil impedance as the inhomogeneous specimen under study.” The surface of the specimen is aligned with the scanning plane of the probe and the probe is adjusted to a constant normal lift-off distance ($l = 0.1$ mm). The complex impedance plane is then rotated by changing the phase angle so that the lift-off direction appears horizontal, and the vertical component of the impedance variation is used to assess the apparent eddy current conductivity. The adverse effects of inevitable lift-off variations during the scanning are effectively reduced by the choice of phase angle.

Nagy’s group performed eddy current measurement on three different materials: Waspaloy, IN 100, and Ti-6Al-4V. These samples were shot peened at different Almen intensities such as 4A, 8A, 12A and 16A (Almen strips are SAE 1070 steel used to measure the intensity of shotpeening by using an arc measurement gauge and the intensity is measured by using a calibration graph of Almen strip arc curves vs the exposure time of shot peening). The measured AECC conductivity was found to be proportional to peening intensity. The AECC measured was inverted to obtain the corresponding conductivity profile using the Cheng-Dodd-Deeds approximation. In this study, Nagy’s group used a single coil to measure eddy current signals for the complete frequency range (1 MHz -100 MHz). Eddy current signals obtained were found to depend on the inductive effect and capacitive effect of the coil used. Lift-off effects were observed at high frequencies like 50 MHz. The obtained apparent eddy current conductivity is not reduced significantly by increasing dislocation density and other microstructural defects due to cold work.
Hillmann et. al \textsuperscript{78} observed that microstructure of the material may affect the eddy current signal obtained. Difference in eddy current signals obtained from shotpeened solutionized samples and shotpeened precipitate hardened samples of Inconel 718 were observed. Both the samples were shot peened at the same shotpeening intensity. This led to the conclusion that microstructure of the sample may affect the signal apart from the residual stress induced in the samples.

Based on both these observations, two important factors need to be taken into account while measuring residual stress of the samples using the eddy current technique, namely, 1) lift-off (instrument factor) and 2) microstructure (material factor). In this thesis work, the main focus is on microstructure of the material. Lift-off studies were performed by another member of the same research group, and their results are used extensively in this thesis. Details of this earlier work are given in the bibliography\textsuperscript{39}. Briefly, the lift-off factor, which was a major drawback in the studies performed by Nagy’s group, was overcome by using the normalized V-component and approximation theory. This acts to eliminate any instrument dependent factors such as specific coil design, and measurement dependent factors such as lift-off.

By using the swept frequency eddy current technique developed here\textsuperscript{39}, combined with using normalized V-component and approximation theory, the ability to measure the eddy current signals from the frequency of 100 kHz to 50 MHz is possible. The obtained SFEC signals can be inverted into corresponding conductivity profiles using Cheng-Dodd-Deeds theory and approximation theory. By using Inconel 718 (a Ni- based superalloy) samples of various heat treatments which are solutionized, under-aged, peak aged and over-aged combined with different shotpeening intensities like 4A, 8A and 12A will enable study of the microstructure dependency of the material and residual stress induced in the material.
1.7 Problem Statement

This thesis is the first attempt made to separate the effects the microstructure and residual stress (induced by shot peening) using eddy current measurement technique in the Nickel based superalloy Inconel 718. This problem is approached by understanding the microstructure of the material at different heat treatments and the effects of shot peening at different shot peening intensities.

This dissertation discusses how the eddy current signals are affected by microstructure of the samples, and a systematic investigation has been carried out to investigate these effects. Specifically, the effects of precipitation as a function of heat treatment and damage due to shot-peening on the eddy current signal were studied. A set of Inconel 718 samples, heat treated to possess different amounts of precipitation, were chosen for the study. This was done for several reasons. Firstly, Inconel 718 is a major alloy using in turbine engines, and thus is of great importance. Secondly, Inconel 718 is non-ferromagnetic, which means the permeability factor may be excluded from the eddy current responses measured in this investigation. Thirdly, residual stresses in Inconel 718 have been measured using commercial XRD techniques and residual stress profiles have been measured using surface layer removal \(^{79-82}\), giving some idea of the level of stresses that may be encountered.

Microstructure of the samples was studied using various microscopy techniques like Scanning electron microscopy and Transmission electron microscopy. Microstructure of the samples was quantified in terms of their secondary phase precipitates. This was approached by using the micrographs obtained from the above mentioned materials characterization techniques. At various levels of heat treatment the size and volume percentage of strengthening precipitates can be expected to change. Therefore, the volume fraction of the secondary phase precipitates present in the Inconel 718 samples chosen for the study needs to be determined with some degree of reliability in order to relate microstructure to eddy current response. The correlation of eddy current signals
with the corresponding samples precipitates (quantified), helped to determine the impact of microstructure on the eddy current signals apart from residual stress.

Samples were shot peened at three different intensities and swept frequency eddy current signals were measured before and after shot peening. For the eddy current measurements different frequencies were used so that different depths of the sample could be explored, encompassing both the shot peened layer, the center bulk of the sample, and the bottom region where an edge is present due to the nearby free surface.

The lift-off effect was observed in the eddy current measurement technique used by Nagy’s group and it was more pronounced at higher frequencies above 10 MHz. This lift-off effect was cancelled out by using the normalized V-component signal, which is not affected by the lift-off. The other problem that might be faced is edge effects while using eddy current technique. In this study, comparison of the free edge results to the bulk and shot peened results allowed edge effects to be accounted for by making sure the measurements are always performed in the center of the sample to avoid edge effects. By comparing the swept frequency eddy current signals to the extensive microstructure characterization allowed a calibration curve to be obtained relating conductivity profiles to microstructure.

1.8 References


43 Blitz, J. *Electrical and magnetic methods of non-destructive testing*. Vol. 3 (Springer, 1997).


CHAPTER 2: EXPERIMENTAL PROCEDURE

2.1 Sample Preparation
A series of Inconel 718 samples were chosen for the study. Six samples of sizes 0.250" x 3.00" x 5.00" solution heat treated (1040°C) for 1 hour and air cooled and one sample of size 0.250" x 3.00" x 5.00" stock Inconel 718 per ASM 5596 specification was purchased from Advanced Alloys, Deer Park, New York. The samples were cut into 0.250" X 1.5" X 2.5" coupons using electric discharge machining (EDM). Different heat treatment conditions were selected using the time–temperature-transformation (TTT) diagram\(^1\) shown in Figure 2.1 to produce various microstructures with different amounts and sizes of secondary precipitates.

![TTT Diagram](image)

**Figure 2.1** Time-Temperature-Transformation for Inconel 718\(^1\). Red color dots indicate the sample conditions chosen for the investigation. Red line for condition V indicates that condition V is subjected to double aging at two temperatures indicated by red dots.
Sample I (AR) was kept in the as-received condition. To ensure a homogeneous starting microstructure all the other samples were again solutionized at 1024°C for 0.5 hour using a controlled atmosphere GCA box furnace and then furnace cooled to ensure homogeneity. Thus, Sample II (SHT) is designated as the solution heat treated sample although it in actually was solutionized twice. The remaining samples were aged under different conditions as given by Table 2.1. In accordance with the TTT diagram of Figure 2.1, Samples III and IV are expected to be underaged, and are designated UA1 and UA2, respectively. Sample V is expected to be in the peak aged condition and is designated PA, while Sample VI and VII should be overaged and are designated OA1 and OA2, respectively. It should be noted that Sample V underwent a two-step aging process, being held at initially at 718°C then furnace cooled to 621°C. This means that Sample V actually was held in two different regions of the TTT curve shown in Figure 2.1. This is reflected on the figure by showing two points for Sample V joined by a line.

Table 2.1 Heat-treatment conditions for the set of Inconel 718 samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Heat Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>I (AR)</td>
<td>As-received (solutionized)</td>
</tr>
<tr>
<td>II (SHT)</td>
<td>Solutionized at 1024°C/0.5hr</td>
</tr>
<tr>
<td>III (UA1)</td>
<td>Solutionized, aged at 620°C/10hrs</td>
</tr>
<tr>
<td>IV (UA2)</td>
<td>Solutionized, aged at 680°C/50hrs</td>
</tr>
<tr>
<td>V (PA)</td>
<td>Solutionized, aged at 718°C/8hrs,furnace-cooled to 621°C, aged at 621°C/8hrs</td>
</tr>
<tr>
<td>VI (OA1)</td>
<td>Solutionized, aged at 850°C/10hrs</td>
</tr>
<tr>
<td>VII (OA2)</td>
<td>Solutionized, aged at 900°C/20hrs</td>
</tr>
</tbody>
</table>
2.2 Shot peening
Each heat-treated sample was divided into four quarters (Figure 2.2) for the shot peening study. One quarter was kept in the pristine condition and was used as the reference for swept frequency eddy current (SFEC) measurements, while the other three were shot peened at Almen intensities of 4A, 8A and 12A with 100% coverage using ceramic shot (Zirshot Z850®, Saint-Gobain ZirPro) with average diameter of 0.8 mm. Shot peening was conducted using a Trinc Dry blast instrument, standard model 24/BP. The shot peen intensities were measured using standard shot peening control “A” strips bought from Electronics, Inc.

![Figure 2.2 Shot peened Inconel 718 sample with different Almen intensities (4A, 8A and 12A).](image)

2.3 Electrical Conductivity Measurements
The bulk electrical conductivities of the samples before and after shot peening were measured using a commercial conductivity gauge (Sigma scope by Fischer) operated at
60 kHz with measurement accuracy ≤ 1 %. The accuracy of the gauge was calibrated using seven different standards for conductivities ranging from 1 % international annealed copper standard (IACS) to 99.99% IACS.

2.4 Mechanical Property Measurements
Sample hardness was also measured before and after shot peening using a Leco indenter employing the Rockwell B and C scales. Calibration of the hardness testers was conducted using known standards before measurements were taken. This step controls uncertainty to ±1.0 Rockwell number. Figures 2.3 and 2.4 show the example of a hardness indentation of the samples, which measured approximately 430 – 595 microns diameter, covering more than one grain. The average hardness value was obtained by averaging over 10 measurements.

Figure 2.3 Example of hardness indent using Rockwell B indenter on solutionized Inconel 718 sample.
2.5 Residual Stress Measurements

The samples examined in this study and the shot peening values selected were chosen based on previous work\(^2\) which determined what residual stress levels should be expected in these alloys. Average residual stress magnitudes were estimated from the literature to be nominally equal for each different conditions, falling in the range 1 GPa to 1.5 GPa\(^2\) as condition varied. An attempt was made to verify these values independently using X-ray diffraction. Since X-ray penetration is limited to the top 40 microns of a material, with the actual depth depending on the specific absorption coefficient of the material in question, measuring bulk residual stress using X-ray diffraction involves a series of measurements, with material being removed sequentially by chemical means between successive x-ray scans to produce an overall measurement of stress vs. depth. The residual stress profile of sample OA2 was measured in this way by Proto Manufacturing, Ltd., Taylor, Michigan in accordance with SAE HS-784.

**Figure 2.4** Example of hardness indent using Rockwell C indenter on peak aged Inconel 718 sample.
Surface residual stresses were measured on all samples using a Bruker D-8-Diffractometer (Figure 2.5) equipped with a two-dimensional general area detector diffraction system (GADDS) using a Cr tube to acquire the XRD data.

![Bruker X-ray Diffractometer with 0.8 mm collimator and Cr-Source.](image)

The size of the collimator used was 0.8 mm. All the shot peened conditions and pristine conditions were measured for all the samples. The (220) peak ($2\theta = 128^\circ$) was examined, and each acquisition was obtained using 30kV, 50mA, by the $\psi$ method. Stress analysis was conducted using proprietary Bruker software.

In the method used, the principle stresses in the $\sigma_1$, and $\sigma_2$ directions were determined, Figure 2.6. Because the penetration of the X-ray beam is extremely shallow, the
diffracting volume can be considered to represent a free surface under planar stress. In this image, $\sigma_1$ corresponds to the stress in the direction in which the sheet was rolled, while $\sigma_2$ represents the stress in the transverse direction. Stress $\sigma_3$, out of the plane of the sheet, is generally not measured for two reasons, the principal one being that this stress is generally extremely small or no longer present$^{3,4}$. The values obtained by XRD were then compared to the expected literature values and to swept frequency eddy current measurements.

**Figure 2.6** Schematic diagram of the stresses present in the shot peened sample.

### 2.6 Microstructure Characterization

The secondary precipitates in Inconel 718 are $\gamma'$, $\gamma''$ and $\delta$ precipitates, of which $\gamma''$ is considered to be the primary strengthening precipitate. The expected microstructures from each heat treatment condition$^1$ according to the referenced TTT diagram (Figure 2.1)$^1$ are given in Table 2.2. In Inconel 718, $\gamma'$ is spherical in shape having an ordered FCC structure, $\gamma''$ is disc-like having a BCT structure, and $\delta$ has an orthorhombic structure$^1$. 

![Schematic diagram of the stresses present in the shot peened sample.](image)
Table 2.2 Expected microstructure from each heat treatment condition.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Expected Microstructure</th>
</tr>
</thead>
<tbody>
<tr>
<td>AR</td>
<td>Homogeneous microstructure free of γ', γ'' &amp; δ precipitates</td>
</tr>
<tr>
<td>SHT</td>
<td>Homogeneous microstructure free of γ', γ'' &amp; δ precipitates</td>
</tr>
<tr>
<td>UA1</td>
<td>Small amount of γ' and γ'' precipitates</td>
</tr>
<tr>
<td>UA2</td>
<td>Large amount of γ' and γ'' precipitates</td>
</tr>
<tr>
<td>PA</td>
<td>Homogeneous mixture of γ' &amp; γ'' throughout the matrix</td>
</tr>
<tr>
<td>OA1</td>
<td>γ'' precipitates will coarsen and get converted into δ precipitates</td>
</tr>
<tr>
<td>OA2</td>
<td>Predominantly δ precipitates</td>
</tr>
</tbody>
</table>

Strips were cut from each heat-treated sample to make coupons, which were then polished and etched for microstructure characterization using an FEI Quanta-250 field-emission scanning electron microscope (SEM). Examination involved sectioning the samples in cross section and polishing to reveal the microstructure changes that result due to shot peening. Thus, one edge of the sample exhibited the shot peened side, while the opposite edge represents the base material in the as-received condition. This sectioning is illustrated in Figure 2.7. A 6-mm-wide sample was sectioned from the initial 2.5 cm x 1.5 cm x 6 mm piece (first cut in Figure 2.5) then sectioned again (second cut) to produce a smaller sample for SEM observation.
Figure 2.7 A schematic of sample cross-section obtained for SEM characterization. Note that the thickness of the piece is greatly exaggerated for clarity.

Coupons of the samples were mounted and polished using sand paper grits from 120 to 1200 followed by fine polishing using a diamond solution of 1 micron, followed by etching using 50 ml HCl, 30 ml H$_2$O, 20 ml HF and 10 ml HNO$_3$ solution. From the coupons, 3 mm discs were punched, and thin foils were prepared by mechanical dimpling followed by jet electropolishing using 10% perchloric acid and 90% methanol to perforation at a temperature of -30°C at 15 KV. The foils were examined using a Tecnai G2 F20 and a Phillips CM30 transmission electron microscope (TEM). 3 mm discs were punched from different locations to ensure that samples examined were representative of the actual condition of entire coupon.

Additional SEM images were obtained in three different regions of sample OA2, namely, the shot peened region, the bulk region and the free (unpeened) region, representing different depths in the sample. A schematic of the sampled regions is provided in Figure 2.8. For this sample images were taken at five regularly spaced locations across the sample. At each location six images were analyzed. For images taken in the shot-peened and free surface regions, measurement features available in the SEM software were utilized in an effort keep the distances from the edges constant for the images taken. This involved using the corresponding edge of the sample as a reference point for subsequent depth measurements. Distances from these edges were in the range ≈
20-130 microns. Measurements taken in the center of the sample representing the bulk region were not as carefully controlled since the microstructure did not change over the extent of this region, making careful measurement unnecessary.

Figure 2.8 Schematic showing the location of images taken and analyzed from Sample VII as a function of distance from the opposite surfaces.

2.7 Quantification of Precipitates
The micrographs taken using SEM and TEM were processed using the commercial software package ImagePro. The contrast and brightness of the images were enhanced, and the secondary precipitates were highlighted. The apparent sizes (i.e. diameter for γ', diameter and disc thickness for γ'') and volume fraction of the secondary precipitates were calculated for each of the images, and average apparent sizes and average volume fraction of the secondary precipitates were calculated based on analysis of 5 SEM micrographs and 10 TEM micrographs for each data point plotted.
An example of this process is shown in Figure 2.9. The apparent sizes (i.e. diameter for γ', diameter and disc thickness for γ") and volume fraction of the secondary precipitates were calculated for each image and average apparent size and average volume fraction of the secondary precipitates were calculated. Figure 2.9 shows an unprocessed SEM micrograph of Sample OA2 on the left and the processed image using ImagePro software in the right.

Micrographs such as these were taken and analyzed for all the samples to quantify the precipitates’ volume fraction, and the values tabulated, the lone exception being. Sample UA1. This could not be calculated since the extremely small size of the precipitates (~2-4 nm as shown in the results section) present in UA1, prevented clear distinction between the separate phases.

![Figure 2.9 SEM micrograph of sample OA2 with high contrast and brightness in the left and Image processed using ImagePro software in the right](image.png)
2.8 Surface Roughness and Grain size Measurements

Sample surface roughness measurements were done using a laser profilometer manufactured by Solarius instruments. For all the samples, roughness measurements were done on a scan area of 30 mm x 50 mm with a scan speed of 25 mm/s and a back speed of 10 mm/s with step size of 0.025 mm.

Grain size of the samples was measured on optical micrographs using the linear intercept procedure also known as the Heyn method as per section 11, ASTM E112 standard. For each sample condition, five repeated measurements were made at 100X magnification. Figure 2.10 shows an example of the grain size measurement of one of the samples using the linear intercept method. In this method lines of known length L are drawn on the micrograph. The number of intercepts between these lines and the grain boundaries is counted. By dividing the number of intercepts by the total length and using the equation described in ASTM E112 standard, the grain size number is calculated.

![Optical Microscopy image of a PA sample showing the grain size measurement using linear intercept method.](image-url)
2.9 Swept Frequency Eddy Current (SFEC) measurements

SFEC measurements were performed in two different but partially overlapping frequency bands using different sets of instrumentation and detection coils\(^6\), as shown in Figure 2.9. The instruments are (i) a network analyzer (Agilent E5061A) and a pair of differential detection coils (14 turns, 12 mm diameter) fabricated on a printed circuit board for measurements from 1 MHz to 50 MHz\(^7\) and (ii) an impedance analyzer (Agilent 4292A) and an air-cored pancake coil (244 turns) for measurements from 100 kHz to 4 MHz. The coil parameters employed are listed in Table 2.3.

**100 kHz to 4 MHz**

- Impedance analyzer
- Sample
- Pancake Coil

**1 MHz to 50 MHz**

- Network analyzer
- Differential amplifier
- PCB Coil

**Figure 2.11** Instruments used for SFEC measurements, namely impedance analyzer (for measuring 100 kHz to 4 MHz range) and network analyzer (for measuring 1 MHz to 50 MHz range).
Figure 2.12 PCB coil used for SHEC measurements for measuring 1 MHz to 50 MHz range.

Table 2.3 Coil parameters of two coils used in SFEC measurements.

<table>
<thead>
<tr>
<th>PCB Coil</th>
<th>Coil parameter</th>
<th>Pancake coil</th>
</tr>
</thead>
<tbody>
<tr>
<td>14</td>
<td>No. of turns</td>
<td>50</td>
</tr>
<tr>
<td>12</td>
<td>Outer diameter (mm)</td>
<td>12.7</td>
</tr>
<tr>
<td>1.2</td>
<td>Inner diameter (mm)</td>
<td>3.8</td>
</tr>
<tr>
<td>&gt; 100</td>
<td>$f_{\text{Resonant in air (MHz)}}$</td>
<td>10.3</td>
</tr>
</tbody>
</table>

Liftoff signals $S_L$ were measured at an additional coil liftoff of 25 microns from the sample surface.
Figure 2.13 shows a schematic representation of the SFEC measurement procedure. SFEC measurements required 3 steps.

Step 1: SFEC Measurement on the pristine surface of the sample.

Step 2: SFEC measurement on the pristine surface after placing a spacer of known thickness on the pristine surface.

Step 3: SFEC measurement on the shot peened surface.

The vertical component of the eddy current (EC) signal $V_{\text{EXP}}$ (V-component signal) is defined in Equation 2.1,

$$V_{\text{EXP}} \equiv \text{Im} \left\{ \frac{(S_T - S_R)_{\text{EXP}}}{(S_L - S_R)_{\text{EXP}}} \right\}$$  \hspace{1cm} 2.1

where $S_T$, $S_R$, and $S_L$ are experimental, complex-valued EC signals that correspond to, respectively, the test signal, reference signal, and the extra lift-off signal.

The use of $V_{\text{EXP}}$ offers the advantages that it helps suppress the liftoff noise, while canceling out the instrument transfer function in the ratio. It thereby provides the basis
of model-based inversion of conductivity profiles by allowing direct comparison of \( V_{\text{EXP}} \) with the theoretical vertical component signal \( V_{\text{TH}} \) defined in Equation 1.5. As mentioned previously, \( V_{\text{TH}} \) thus defined takes a particularly simple form under the approximation that holds valid under our measurement conditions. Specifically, when the relative conductivity changes are sufficiently smaller than unity, and when the outer diameter of the detection coil (assumed a cylindrical air-cored coil) is much larger than any other length parameters, such as the skin depth \( \delta \) or coil lift-off \( l \), then, \( V_{\text{TH}} \) can take the approximate form shown previously in Equation 1.6.

SFEC measurements were repeated five times at each single location on each sample and an average SFEC value computed. An example of a typical measurement is shown in Figure 2.14, which shows the eddy current signals obtained from Inconel 718 sample shotpeened at 4A (Almen intensity) using two different coils. 14-turn PCB coil was used to measure from 100 kHz to 3 MHz and 244-turn coil was used to measure from 1 MHz to 50 MHz with step size of 100 kHz.

![Figure 2.14](image)

**Figure 2.14** Vertical component signals obtained for shot peened (4A) sample using two different coils, namely 14-turn PCB coil and 244-turn coil averaged over five repeated measurements.
Note that although two different coils were used to measure eddy current signals from 100 kHz to 50 MHz, we observe from Figure 2.14 that the signals obtained using two coils are continuous, and the signals from 1 MHz to 3 MHz are overlapping. The error bar corresponds to the average of five repeated measurements.

2.10 References

5 Dumont, M., Lefebvre, W., Doisneau-Cottignies, B. & Deschamps, A. Characterisation of the composition and volume fraction of $\langle i \rangle$ $\eta$/$\langle i \rangle$' and $\langle i \rangle$ $\eta$/$\langle i \rangle$ precipitates in an Al–Zn–Mg alloy by a combination of atom probe, small-angle X-ray scattering and transmission electron microscopy. Acta materialia 53, 2881-2892 (2005).
CHAPTER 3: EXPERIMENTAL RESULTS

3.1 Bulk Conductivity and Hardness

Results for the bulk electrical conductivities for the seven samples measured are given in Table 3.1\(^1\). The measured bulk electrical conductivities reported are an average of 5 measurements taken from each sample. The conductivity gauge was calibrated using the calibration standards before each measurement.

Table 3.1 Heat-treatment conditions and measured bulk conductivity of the Inconel 718 samples\(^1\).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Heat treatment</th>
<th>Measured Bulk electrical conductivity (%IACS)(^*)</th>
<th>Standard deviation in 5 measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td>AR</td>
<td>As-received</td>
<td>1.38 ± 0.01</td>
<td>0.001</td>
</tr>
<tr>
<td>SHT</td>
<td>Solutionized at 1024°C/0.5 hr</td>
<td>1.38 ± 0.01</td>
<td>0.001</td>
</tr>
<tr>
<td>UA1</td>
<td>Solutionized, aged at 620°C/10 hrs</td>
<td>1.35 ± 0.01</td>
<td>0.002</td>
</tr>
<tr>
<td>UA2</td>
<td>Solutionized, aged at 680°C/50 hrs</td>
<td>1.47 ± 0.01</td>
<td>0.001</td>
</tr>
<tr>
<td>PA</td>
<td>Solutionized, aged at 718°C/8 hrs, furnace-cooled to 621°C, aged at 621°C/8 hrs</td>
<td>1.48 ± 0.01</td>
<td>0.001</td>
</tr>
<tr>
<td>OA1</td>
<td>Solutionized, aged at 850°C/10 hrs</td>
<td>1.46 ± 0.01</td>
<td>0.002</td>
</tr>
<tr>
<td>OA2</td>
<td>Solutionized, aged at 900°C/20 hrs</td>
<td>1.43 ± 0.01</td>
<td>0.005</td>
</tr>
</tbody>
</table>

\(^*\)International Annealed Copper Standard (100\% IACS = 5.8108 X 10\(^7\) S/m). Measurement accuracy of Fischer Sigmascope conductivity gauge of 60 kHz is ≤ 1 %.
Hardness of all the samples was measured using the Rockwell C scale. Results are given in Table 3.2\(^1\). Samples AR and SHT were measured using the Rockwell B scale and converted into Rockwell C scale. Each listed value is the average of 5 hardness measurements.

**Table 3.2** Rockwell C-scale hardness (HRC) of the Inconel 718 samples\(^1\).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Hardness HRC</th>
<th>Standard deviation in 5 measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td>AR</td>
<td>11.4 ± 1</td>
<td>0.3*</td>
</tr>
<tr>
<td>SHT</td>
<td>9.0 ± 1</td>
<td>0.1</td>
</tr>
<tr>
<td>UA1</td>
<td>27.5 ± 1</td>
<td>0.4</td>
</tr>
<tr>
<td>UA2</td>
<td>41.1 ± 1</td>
<td>0.3</td>
</tr>
<tr>
<td>PA</td>
<td>42.9 ± 1</td>
<td>0.4</td>
</tr>
<tr>
<td>OA1</td>
<td>26.1 ± 1</td>
<td>0.5</td>
</tr>
<tr>
<td>OA2</td>
<td>20.4 ± 1</td>
<td>0.2</td>
</tr>
</tbody>
</table>

*Values measured in Rockwell B-scale and converted to C-scale (HRC). Hardness measurements taken from experimental samples produced estimated uncertainty of ±1.0 \(R_c\).

Figure 3.1\(^1\) shows a plot of both the measured values a) bulk conductivity and b) Rockwell hardness for each sample. Samples are shown in order of increasing hardness.
Figure 3.1 Rockwell C-scale hardness (HRC) and bulk conductivity of the heat-treated Inconel 718 samples in ascending order of hardness from left to right\(^1\).

Note that samples AR and SHT are essentially identical since both consist of solution-heat-treated material. The bulk conductivity of the samples tends to increase with hardness, except for UA1 which is less conducting but is harder than the solutionized samples AR and SHT. The general trend of increasing bulk conductivity with hardness observed in the other samples is attributed to the formation of secondary phase precipitates, which results in the removal of solute atoms from the matrix. This reduces electron scattering by solute atoms in the solid solution and therefore results in higher conductivities of the aged samples (UA2, PA, OA1 and OA2) than in the solutionized ones (AR and SHT).
The low conductivity measured for sample UA1 is somewhat surprising. One possible explanation for the low conductivity is that sample UA1 consists of a high density of fine, coherent secondary phase precipitates such as would be present in the early stages of nucleation before substantial growth has occurred. Such precipitates can be expected to be coherent with the matrix in the initial stages, and if the interparticle spacings are comparable to the electron mean free path, the precipitates could scatter conduction electrons and reduce the conductivity. If the precipitates are coherent, they cause internal lattice strain that leads to lower conductivity. To answer these questions, a detailed examination of the microstructure is needed.

3.2 Microstructure

Although all the sample material studied was delivered in the solution-heat-treated condition, it was not known upon initial receipt of the material exactly how effective the as-received solution heat treatment was in producing a homogeneous structure. Therefore, a second solution heat treatment was carried out on all the material except that designated Sample AR. Samples were then selected and heat treated in accordance with Table 2.1. Before detailed examination of microstructure was conducted using SEM and TEM, optical microscopy images were taken of the polished samples and analyzed to determine mean grain size.

3.2.1 Grain Size

The grain size averaged over all samples is 6.1 ± 0.5 ASTM grain size number, and grain size measurements corresponding to each condition are tabulated in Table 3.3. The quoted uncertainty is the standard deviation of five repeated measurements.
Table 3.3 Grain size measurements of Inconel 718 samples measured using linear intercept method.

<table>
<thead>
<tr>
<th>Sample</th>
<th>ASTM grain size number</th>
</tr>
</thead>
<tbody>
<tr>
<td>AR</td>
<td>6.1 ± 0.2</td>
</tr>
<tr>
<td>SHT</td>
<td>6.1 ± 0.4</td>
</tr>
<tr>
<td>UA1</td>
<td>6.1 ± 0.1</td>
</tr>
<tr>
<td>UA2</td>
<td>6.0 ± 0.3</td>
</tr>
<tr>
<td>PA</td>
<td>6.2 ± 0.2</td>
</tr>
<tr>
<td>OA1</td>
<td>6.1 ± 0.2</td>
</tr>
</tbody>
</table>

Note that this is the grain size determined for the Ni- γ (FCC) matrix phase in all samples. As precipitates nucleate and grow due to heat treatments they will, of course, form their own grains which (presumably) is simply the size of the precipitate.

3.2.2 Phase Identification
An SEM electron micrograph of sample AR in the as-received condition is shown in Figure 3.2. Sample AR was found to have a homogeneous solutionized structure of γ phase Inconel 718 without any detectable secondary phase precipitates, which is the goal of a solution heat treatment. Thus, the as-received material appears to have received an adequate solution heat treatment.
Figure 3.2 SEM micrograph obtained from Sample AR showing the absence of any secondary phase precipitates except a few Nb-rich and Ti-rich particles\(^1\).

Sample SHT was also found to have a solutionized structure (Fig. 3.3a)\(^1\) of γ phase Inconel 718 without any detectable secondary phase precipitates, except for a few Nb- and Ti-rich particles (as detected using energy dispersive spectroscopy, Fig. 3.3b)\(^1\) that are presumably carbides\(^4\). The presence of carbides might be due to the solutionizing condition used for this sample. Thus, it is possible that instead of ensuring adequate heat treatment the further solution heat treatment has resulted in carbide formation. However, the more likely scenario is that a small amount of carbides still exists in all the solution heat treated samples but due to their low density they are rarely seen.
Figure 3.3 Sample SHT a) SEM image. Micron-sized particles rich in Nb and a small amount of Ti, b) EDS of Sample SHT indicating the presence of particles (Nb rich and smaller Ti rich).
Figure 3.4 TEM image with selected area electron diffraction pattern (inset) of Sample SHT$^1$.

Figure 3.4 shows bright field TEM micrograph obtained from Sample SHT. The inset shows the diffraction pattern used to obtain the bright field. This micrograph clearly indicates the absence of any secondary phase precipitates (no superlattice points in diffraction pattern)$^1$.

Sample UA1 is the first sample that was heat treated to produce precipitation. As shown in Table 3.2, Sample UA1 is harder than the solutionized samples, and was expected to consist of a high density of fine hardening phase precipitates. This is confirmed in Figure 3.5, which shows bright field and dark field TEM micrographs of Sample UA1, respectively, obtained using g= (100).
Figure 3.5 Sample UA1 a) Bright field image. Precipitation is so fine it is difficult to distinguish between γ' and γ", b) SAD pattern showing the presence of uniform secondary precipitates. c) Dark field image obtained using SAD pattern (red circle) shown in b. The presence of uniform secondary precipitates γ" of size 2-3nm.
Figures 3.6a and 3.6c show the bright field and dark field micrographs (obtained using \( g = \{110\} \)) of Sample UA2, respectively. The heat treatment of Sample UA2 was chosen in such a way that large amounts of \( \gamma' \) and \( \gamma'' \) precipitates are expected to form as mentioned in Table 2.2. Both \( \gamma' \) and \( \gamma'' \) precipitates were present in the sample as revealed through extra reflections present in diffraction patterns taken from the structure (shown in Figure 3.6b). A dark-field image (Figure 3.6c) was formed using the super lattice reflections corresponding to the secondary precipitate \( \gamma'' \). The sizes of the precipitates were roughly 5 nm, which we can see in Figure 3.6 with the scale bar of 20 nm.

**Figure 3.6** Sample UA2 a) Bright field image shows the presence of small uniform secondary precipitates \( \gamma' \) and \( \gamma'' \), b) SAD pattern showing the presence of uniform secondary precipitates, c) Dark field image using diffraction pattern circled in red shown in the Figure 3.6b reveals \( \gamma'' \).
Figure 3.7 Sample PA a) Bright field image shows the presence of small uniform secondary precipitates $\gamma'$ and $\gamma''$, b) SAD pattern showing $\{1 1 0\}_{\gamma'}$ type superlattice reflections, c) Dark field TEM image using diffraction pattern circled in red shown in the Figure 3.7b reveals $\gamma''$ precipitates.
For Sample PA, bright field TEM image showing the presence of secondary phase and dark-field TEM images formed using the \{1 1 0\}γ” type superlattice reflections revealed acicular γ” precipitates (Fig. 3.7a and Fig.3.7c)\(^1\).

The SAD pattern which was used in obtaining the dark field image (Figure 3.7c) using \{1 1 0\}γ’ type superlattice reflections is shown in Figure 3.7b\(^1\).

![Sample PA Scanning TEM image showing the presence of γ” precipitates. A line scan of EDS showed that the precipitates (indicated by the arrows in the plot) have composition close to Ni\(_3\)Nb\(^1\).](image)

**Figure 3.8** Sample PA Scanning TEM image showing the presence of γ” precipitates. A line scan of EDS showed that the precipitates (indicated by the arrows in the plot) have composition close to Ni\(_3\)Nb\(^1\).

The identity of the γ” precipitates were also confirmed by energy-dispersive spectroscopy (EDS). A line scan was conducted across the sample (shown in red in Fig. 3.8) that encompassed both the matrix and two precipitates revealed by dark field imaging. Analysis of the resultant EDS scan (Fig. 3.8) showed that the precipitates have a composition close to Ni\(_3\)Nb (Fig.3.8).
Sample OA1 has an over-aged microstructure (Fig. 3.9b) characterized by coarser γ'' particles (imaged using the SAD pattern from Figure 3.9a) compared with those observed in Samples UA2 and PA, and the presence of what is believed to be δ phase along the grain boundaries. SEM micrographs (Fig 3.10) show coarse γ'' precipitates present in Sample OA1 condition. The size of the precipitates in this sample is so large that they can easily be imaged using SEM rather than requiring TEM.

Figure 3.9 Sample OA1 a) SAD pattern showing the presence of secondary phase precipitates, b) Dark field TEM micrograph using diffraction pattern circled in red shown in the Figure 3.91 reveals the presence of coarse γ'' precipitates.
In Sample OA2, γ'' precipitates were again found in the grain interior; however, the number is greatly reduced. In this case the structure is dominated by the presence of inter- and intra-granular δ platelets (Fig. 3.11). The reduction in number of γ'' precipitates mostly likely are due to Nb depletion that occurs from δ phase formation. Sample OA2, similar to sample OA1, has precipitates of a size that can easily be imaged using SEM rather than requiring TEM. Precipitation in both samples (OA1 and OA2) has coarsened to such an extent that distinct morphologies are now exhibited.
Figure 3.11 SEM micrograph obtained from Sample OA2 shows a) δ platelets and b) γ” in grain interior.
3.2.3 Phase Quantification

The micrographs taken using SEM and TEM were processed using the commercial software package ImagePro. The contrast and brightness of the images was enhanced, and then the secondary precipitates alone were highlighted. The results obtained are shown below in Table 3.4. The volume fraction of $\gamma'$ and $\gamma''$ precipitates are calculated for sample conditions UA2 and PA, $\gamma''$ and $\delta$ precipitates for OA1, and $\delta$ precipitates for OA2. The precipitate sizes in UA2 and PA are so small that the volume fraction can be calculated only using TEM micrographs whereas precipitates size in OA1 and OA2 are big enough that volume fractions can be calculated using SEM micrographs.

Table 3.4 Measured volume fraction (%) of secondary precipitates present.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Volume fraction (%)</th>
<th>Imaging Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>SHT</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>AR</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>UA2</td>
<td>9 ± 2</td>
<td>TEM micrographs</td>
</tr>
<tr>
<td>PA</td>
<td>17 ± 3</td>
<td>TEM micrographs</td>
</tr>
<tr>
<td>OA1</td>
<td>11 ± 4</td>
<td>SEM micrographs</td>
</tr>
<tr>
<td>OA2</td>
<td>6 ± 1</td>
<td>SEM micrographs</td>
</tr>
</tbody>
</table>

As seen from Figure 3.5, the size of the precipitates in sample UA1 was so small that it proved impossible to distinguish between them and the background in any meaningful way using the computer software. Therefore, results for this sample are omitted from Table 3.4.

When considering the values reported in Table 3.4, several things should be noted. Firstly, precipitation in the UA samples and PA was so small that required the use of TEM images prevents a reliable bulk analysis of precipitation from being conducted.
The numbers reported, while reflected a number of TEM measurements, still have only come from a very small amount of material, orders of magnitude smaller than what is possible in the OA samples where SEM imaging could be used. Secondly, while the volume percentage of precipitates seems to be decreasing as aging proceeds, this is most likely not the case as this would require Nb to go back into solution in the matrix. Close observation of the possible error associated with the measurements from OA1 and OA2 show that the volume percentage of precipitate can essentially be considered as remaining constant, with only the size of the precipitates changes as is evidenced in Figures 3.9 and 3.10

3.3 Stress Measurements using X-ray Diffraction
Figures 3.12-3.18 show the XRD measured surface residual stresses for samples AR, SHT, UA1, UA2, PA, OA1 and OA2 (sample conditions described in Table 3.1), respectively. Stress was measured on each of the four different regions, namely pristine (unpeened) and shot peened at Almen intensities 4A, 8A and 12A as shown in Figure 2.2. Due to the nature of XRD stress determination, the values shown represent only the surface residual stresses to a depth of approximately 20 microns. Note that all the samples were measured in the as-peened condition. As such, surface roughness due to peening might have affected the scattering of the X-ray beam slightly.

In each figure, $\sigma_1$ and $\sigma_2$ correspond to the principal stress values of the samples as shown in Figure 2.6. An average of both stress values is then calculated. For all samples, $\sigma_1$ values were higher than the $\sigma_2$ values. This is suspected to be due to residual stress present in the material in the as-received condition. For example, rolling is known to produce anisotropic residual stresses in the rolling direction versus the transverse direction. However, in this study, shot peening is used which typically produces isotropic stresses in the plane of the peened layer.
All these samples were polished before shotpeening to obtain a mirror finish. The samples were not subjected to any mechanical polishing after shotpeening and the residual stresses present in the samples after shotpeening were measured. It is possible that the slight polishing before shot peening altered the as-received residual state of the material.

Surface residual stress seems to vary from sample to sample. For all the sample conditions, 0A and 4A are two halves of a single piece of metal while the 8A and 12A samples are two halves of a second piece, as shown in Figure 2.2. All the samples were mechanically polished (to remove machining effects and obtain smooth mirror finish surfaces) prior to swept frequency eddy current measurements. This mechanical polishing presumably is the reason for the stress present in the samples that are unpeened (0A), which should be stress-free samples.

From Figure 3.12, we observe that for Sample AR, the as-received (solutionized) state, after shot peening the residual stress is fairly constant for the three Almen intensities used and is in the range 450-500 MPa compressive. That the value does not change significantly with increasing intensity is expected since shot peening is known to introduce compressive stresses up to a limit of half the yield strength of the material. Yield strength of Inconel 718 aged at 540 °C is 1060 MPa.
Figure 3.12 Surface residual stresses present on four quarters of Sample AR namely, pristine (0A), shot peened at 4A, shot peened at 8A and shot peened at 12A (A- Almen intensity).

Figure 3.13 Surface residual stresses present on four quarters of Sample SHT namely, pristine (0A), shot peened at 4A, shot peened at 8A and shot peened at 12A (A- Almen intensity).
Similarly from Figure 3.13 we observe that for Sample SHT, which is solutionized and has a similar microstructure to sample AR, has a fairly constant residual stress averaging between 450 and 550 MPa compressive. This is not surprising since they are essentially the same microstructure. The slight difference between them can be taken as a measure of the sample-to-sample scatter for the same level of shot peening.

Figure 3.14 Surface residual stresses present on four quarters of Sample UA1 namely, pristine (0A), shot peened at 4A, shot peened at 8A and shot peened at 12A (A- Almen intensity).

From Figure 3.14 we observe that Sample UA1 shot peened at 4A seems to have a higher surface compressive stress compared to the ones shot peened at 8A and 12A respectively, being roughly 150-200 MPa higher. This is a considerable deviation from what was seen for Samples AR and SHT and is greater than the sample-to-sample variation that one might expect by comparing samples AR and SHT.
Figure 3.15 Surface residual stresses present on four quarters of Sample UA2 namely, pristine (0A), shot peened at 4A, shot peened at 8A and shot peened at 12A (A- Almen intensity).

From Figure 3.15, we observe that for Sample UA2 the compressive stress seen increases slightly in the order 4A, 8A and 12A, an orderly progression that might be expected. However, if one considers the variation present between Sample AR and SHT, it might be concluded that the differences seen between the shot peening levels in Sample UA2 are within sample-to-sample variation.

From Figure 3.16 we observe that for sample PA the 8A sample has a higher stress as compared to both 4A and 12A, although not remarkably higher than 12A but considerably higher than 4A.
Figure 3.16 Surface residual stresses present on four quarters of Sample PA namely, pristine (0A), shot peened at 4A, shot peened at 8A and shot peened at 12A (A- Almen intensity).

Figure 3.17 Surface residual stresses present on four quarters of Sample OA1 namely, pristine (0A), shot peened at 4A, shot peened at 8A and shot peened at 12A A- Almen intensity).
For Sample OA1 (Figure 3.17), 8A is again higher than 4A and 12A, only this time a greater difference is seen between 8A and 12A.

![Condition OA2](image)

**Figure 3.187** Surface residual stresses present on four quarters of Sample OA2 namely, pristine (0A), shot peened at 4A, shot peened at 8A and shot peened at 12A (Almen intensity).

The sequence again changes when looking at the data for Sample OA2 (Figure 3.18). In this instance the stress for 4A is higher when compared to 8A and 12A, significantly so in both cases but especially so as compared to 12A.

The surface XRD measurements are of value for two different reasons: a) they give an indication of the stress state in the region that is subsequently ignored by SFEC testing and b) they provide a sense of the sign of the residual stress (i.e. tensile or compressive) that might be expected. Since the XRD stress is only measured down to a depth of approximately 20 microns, these results cannot be compared to eddy current measurements, which sample depths on the millimeter scale. True bulk stress
measurements using XRD would require chemically etching the surface serially followed by repeating the XRD measurements. The initial surface stress data shown in Figures 3.12- 3.18 have large amounts of scatter in the measurements (i.e. between $\sigma_1$ and $\sigma_2$) within samples and from sample to sample. Scatter on the unpeened samples was due to the mechanical polishing, while scatter on the peened samples was due to the combination of shotpeening introducing stress and the surface roughness produced by shotpeening. Unfortunately, the large amount of scatter makes the measured surface stress values (Figures 3.12 -3.18) useful only in a qualitative sense. Ideally future studies could employ neutron diffraction methods to obtain through thickness residual stresses of the exact same sample used for eddy current measurements.

A single bulk XRD measurement was obtained from sample condition OA2, shotpeened at 12A shown in Figure 3.19. These results were obtained by the commercial firm of Proto Manufacturing, Ltd., who employed XRD and successive removal of the top layer using etching.

Figure 3.19 Depth profile of residual stress on sample condition OA2 shot-peened at 12A (A- Almen intensity).
While the commercial residual stress measurement most likely relaxes some of the stress as layers are being removed, it does give an excellent qualitative assessment of the trend in the residual stress profile. Since the commercial results show that the surface stresses are qualitatively similar to those measured on campus at ISU, it was assumed that all the samples probably possessed a stress profile similar to sample OA2. The commercial results show that the residual stresses penetrate to a depth of ~400 microns, much deeper than the surface damage produced by shot peening.

3.4 Surface Roughness Measurements

Surface roughness has been shown to affect the eddy current responses from copper samples when the surface roughness of the samples is above 5 microns\textsuperscript{8}. To determine the extent of surface roughness due to shot peening in this study, surface roughness measurements of the sample surfaces were made using laser profilometry. Figure 3.20 shows the roughness of all the samples as a function of measured hardness in increasing order, while Figure 3.21 shows roughness as a function of Almen intensity.

From Figures 3.20 and 3.21, we see at low hardness values (between 10 and 30 R\textsubscript{C}, corresponding to Samples AR, SHT, UA1, OA1 and OA2), there was no clear trend relating surface roughness to Almen intensity. The 12A samples generally had a higher roughness but not remarkably so. The 8A and 4A samples traded places as having the second highest roughness, but given possible scatter between individual samples the roughness of the two are essentially the same. Surface roughness of the unpeened samples is considerably less than the peened samples as might be expected, and varied from 0.11–0.56 microns. A discernible difference is seen in surface roughness of the samples at peak hardening (> 40R\textsubscript{C}). At this value the material can be expected to undergo less plastic deformation, which leads to lower surface roughness. The shot peened roughness under this condition has an average value of 1.00 ± 0.5 microns.
Figure 3.20 Surface roughness of samples with pristine surface (0A) and shot peened surfaces at different intensities at 4A, 8A and 12A (A- Almen intensity).

Figure 3.21, a comparison of sample surface roughness as a function of Almen intensity, makes it clear that roughness increased substantially as expected due to shot peening. Samples AR and SHT (which are essentially identical) are close in roughness values and again give some measure of the sample-to-sample variation that can be expected. Sample UA1, which has low hardness shows the highest roughness of all samples at Almen intensities 4A and 8A. Samples UA2 and PA (which is in the peak aged condition) show greatly varying surface roughness while overaged Samples OA1 and OA2 are more similar.
Figure 3.21 Surface roughness of pristine surface (0A) and shot peened surfaces at different intensities 4A, 8A and 12A (A-Almen intensity).

At the peak hardening condition (sample PA) the surface roughness falls below that measured for all other samples. This might be expected since a higher hardness will produce less plastic deformation upon shot-peening, presumably resulting in less roughness. UA2 and OA1 show similar roughness profiles, both being higher than PA. The outlier in the data is the UA1 condition, which had a higher surface roughness than any of the other samples. This may be due to the resistance to plastic deformation produced by the small coherent precipitates observed using TEM that appear in this sample.
3.5 Swept Frequency EC Signals

Figures 3.22 -3.28 show the V-component EC signals measured for all the samples (AR, SHT, UA1, UA2, PA, OA1 and OA2) at different shot peening intensities, namely, 4A, 8A and 12 A (A-Almen intensity) with reference to the pristine surface of the corresponding heat treatments. The signals measured in the two frequency bands (100 kHz to 4 MHz and 1 MHz to 50 MHz) using different sets of coils (14 turns, 12 mm diameter fabricated on a printed circuit board for measurements from 1 MHz to 50 MHz and an air-cored pancake coil (244 turns) for measurements from 100 kHz to 4 MHz) show a continuous curve over the entire frequency range in accordance to Eq. 2.4, as the diameters of the detection coils (>12 mm) are substantially larger than all other length parameters such as the maximum skin depth of 1.8 mm at 100 kHz and the additional coil lift off of 25 μm. Note that the data shown are normalized for lift-off using the measurement technique described in Chapter 2. Abu-Nabah et al 8,9 stated that lift-off problems at high frequencies such as 50 MHz make the eddy current technique less reliable for use at these frequencies. This was overcome by the lift-off normalized technique mentioned in Chapters 1 and 2. After shot peening, the test V-component signals become more positive than the baseline signals and the signal increase is larger for higher Almen intensity. As shown in Figures 3.22 -3.28 the shot-peening-induced changes in V-component EC signals vary from sample to sample. Nevertheless, a general trend of decrease in amount of eddy current change can still be identified by sorting the results in ascending order of sample hardness (i.e., SHT, AR, OA2, OA1, UA1, UA2, and PA).
**Figure 3.22** Liftoff-normalized V-component EC signals measured after shot peening with different Almen intensities (4A, 8A, 12A) versus frequency for Sample AR with pristine surface as reference.

**Figure 3.23** Liftoff-normalized V-component EC signals measured after shot peening with different Almen intensities (4A, 8A, 12A) versus frequency for Sample SHT with pristine surface as reference.
**Figure 3.24** Liftoff-normalized V-component EC signals measured after shot peening with different Almen intensities (4A, 8A, 12A) versus frequency for UA1 with pristine surface as reference.

**Figure 3.25** Liftoff-normalized V-component EC signals measured after shot peening with different Almen intensities (4A, 8A, 12A) versus frequency for Samples UA2 with pristine surface as reference.
Figure 3.26 Liftoff-normalized V-component EC signals measured after shot peening with different Almen intensities (4A, 8A, 12A) versus frequency for Sample PA with pristine surface as reference

Figure 3.27 Liftoff-normalized V-component EC signals measured after shot peening with different Almen intensities (4A, 8A, 12A) versus frequency for Samples OA1 with pristine surface as reference
For the softest samples, Samples AR and SHT, which have a solutionized structure, the V-component signals increase after shot peening, and the increase in signals is larger for higher Almen intensity. For Samples OA2, OA1, and UA1, samples aged at less than peak hardness, shot peening tends to increase the signals, although the signal changes are significantly smaller than those observed in Samples AR and SHT. For the two hardest samples, UA2 and PA, shot peening tends to reduce the signals at high frequencies (above ~8 MHz), which become increasingly negative as the Almen intensity increases.

3.6 References


CHAPTER 4: CONSIDERATION OF EXPERIMENTAL FACTORS

It has been observed that shot-peened, nickel-based superalloys exhibit an apparent increase in eddy current conductivity at increasing shotpeening intensities\(^1\). It may be possible to exploit this increase for nondestructive residual stress assessment of subsurface residual stresses if the additional factors that contribute to the eddy current signal can be separated from the stress component. The large number of additional factors that need to be considered (such as microstructure, surface roughness, instrumental factors etc.) makes this a difficult task. Therefore, while the main objective of this thesis was to take initial steps at separating out the effects of microstructure in order to discover whether determination of residual stress using the eddy current technique is viable, the effect of several of these other factors was necessarily considered first. In this chapter factors that can affect the quality and utility of an eddy current measurement are considered and accounted for first to provide a sound basis for further analysis of the eddy current results. The measured eddy current results are then discussed in light of these factors.

4.1 Microstructure

Microstructure effects are difficult to account for due to the various factors that can affect the eddy current response. These factors include

a. Grain size of the material
b. Dislocation substructure
c. Secondary phases present, their relative amounts and sizes.

For example, in a single-phase material a large-grained sample will have a response different from a fine-grained sample\(^4\). Similarly, a large-grained, single-phase sample with a high dislocation density will have a different response from the same grain size material that is dislocation free. Similarly, a two-phase material consisting of large regions of each phase will have a different response from the same two-phase material
having the same percentage of phases but with one phase present as a fine dispersion of precipitates within the matrix of the second phase\textsuperscript{5,6}. These microstructural considerations and the results obtained and described in Chapter Three are discussed in turn below in regard to how they might possibly have affected the eddy current response.

4.1.1 Grain Size Measurements
Kahrobaee et al mentioned that different grain sizes affects the eddy current signals differently\textsuperscript{4}. Grain size measurements in bulk regions shown in Table 3.3 reveal that before shot peening all samples have the same matrix grain size.

Shot peening can distort and disturb the grain structure due to the plastic deformation it causes, and this possibility must be considered. While near-surface measurements of any type are almost certainly affected, bulk measurements will not be. There is clear evidence that this is true since grain sizes of the samples measured after shot peening lie within the error bar of initial grain size measurements (grain size measurements shown in Table 3.3). Thus, while grain size has an effect, for this study the effect was assumed to be constant for all the samples.

4.1.2 Dislocation density
Dislocation density is another factor that can affect the eddy current response of the sample. TEM images obtained from the shot peened samples (see, for example, figures 3.4- 3.9) showed no significant difference in the dislocation structure within the samples. However, these samples were prepared from the bulk region of material and not the near surface peened region. Thus, although the TEM examination did not indicate the increase or presence of dislocations in the bulk of the material, it cannot be said that this effect didn't occur in the near-surface regions. In fact, given that shot peening produces plastic deformation as a means of inducing a residual compressive stress, it is certain that the dislocation density is higher than the bulk.
No detailed study was carried out to measure dislocation density in the near-surface regions. However, the damage in the surface layer is taken into consideration while obtaining the conductivity profile, as detailed in section 5.1. Thus, the measurement technique used provides a method for compensating for near-surface changes in dislocation density without actually having to measure and document the change.

4.1.3 Phase Assemblage, Size and Distribution
Electron micrographs obtained from samples subjected to various heat treatments such as AR, SHT, UA1, UA2, PA, OA1 and OA2 (see Figures 3.2- 3.11) show the expected microstructures with various quantities of secondary phase precipitates like γ', γ" and δ. Samples AR and SHT have uniform homogenous microstructures free of secondary phase precipitates like γ', γ" and δ. The changing nature of the microstructure that resulted due to heat treatment produced significant changes in the measured conductivities of the samples.

Electron micrographs obtained from UA1 (Figure 3.5) show small coherent precipitates with sizes around 3 nm as expected. For this sample, the conductivity of the sample decreased below the solutionized condition while the hardness increased over that of the solutionized condition. These results are explained by a consideration of the strain created by the small coherent precipitates. Coherent precipitation has been shown to decrease conductivity as seen in Ag-Pd and Cu-Pd systems due to the increasing coherency strains and abnormal scattering of zones created when the precipitation reaches a critical dimension comparable with the wavelength of free electrons.

Electron micrographs obtained from UA2 (Figure 3.6) show the presence of secondary phase precipitates like γ' and γ" approximately 5 nm in size. Hardness of the sample increased due to the presence of these precipitates since they can be expected to act as dislocation pinning points. The bulk conductivity of the sample increases in the heat
treatment condition where the precipitates are larger in diameter. The increased boundary area can be expected to decrease conductivity; however this is offset by the increase in conductivity that results by removing atoms from solution.

Electron micrographs from the PA sample (Figures 3.7 and 3.8) show the presence of secondary phase precipitates at peak-aged condition with 7 nm approximate diameter. The bulk conductivity of sample reaches its maximum value at this heat treatment condition due to uniform precipitation that occurs throughout the sample, which helps to remove solute atoms from the matrix. Hardness of the samples (Figure 3.1) also reaches its peak value compared to all the other heat treatment conditions.

Secondary phase precipitates such as δ start growing at the OA1 condition at the expense of γ" (observed from the electron micrographs of Fig. 3.9 and Fig. 3.10) and also γ" grows in diameter compared to the PA condition. The δ phase is observed as the predominant secondary phase in OA2 condition while γ" also present within the grains. As the type and amount of phases present in the material change, it can be expected that the bulk conductivity will also change in relation to the individual conductivities of the respective phases. Information concerning the individual conductivities of γ’, γ" and δ does not appear to exist in the literature. However, from observation of the bulk conductivity (Table 3.1) and hardness (Table 3.2) as a function of heat treatment, we can see that both increase until the peak-aged condition of sample PA is reached after which both drop again for the over-aged conditions. As the precipitates (γ’ and γ") start growing in UA1, increase in size in UA2 and reach the maximum size in PA, we see that hardness increases gradually, whereas the conductivity dips in UA1 due to the small size of the precipitates, and starts increasing in UA2 and reaches the peak in PA. As the δ precipitates start growing at the expense of γ" in OA1, we see that conductivity starts dropping and drops further in OA2 where the precipitates are mostly δ.
The unit cell volume of $\delta^8$ (a= 0.424 nm, b=0.512 nm and c= 0.453 nm) is 0.09834 nm$^3$ while the cell volume of $\gamma''^8$ (a= 0.362 nm, b=0.362 nm and c= 0.741 nm) is 0.09710 nm$^3$. Since the atomic volumes of both precipitates are so equal to each other, we can assume that differences in total precipitate volume fraction as the material transforms from $\gamma''$ to $\delta$ are negligible. Since the conductivities of both $\gamma''$ and $\delta$ precipitates are believed similar$^7$, it appears that the change in conductivity is predominantly due to the overall change in volume fraction of precipitates in various heat treatments as described in detail in later section 5.2.

### 4.2 Physical Parameters

Various parameters inherent to the sample can also affect the ability to successfully measure an eddy current response and relate it to the residual stress present in the sample. These include such factors as the magnetic properties of the sample itself and the physical nature of the surface being examined. These effects are discussed below.

#### 4.2.1 Magnetic Permeability

Magnetic factors must be considered when evaluating the effectiveness of typical eddy current measurements; they pose significant problems for iron-based alloys$^9$. While many Ni-based alloys$^{10}$ are ferromagnetic and subject to the same problems, Inconel 718 is a non-ferromagnetic nickel-based superalloy$^{11}$; thus, the effect of ferromagnetism need not be considered when accounting for the changes in eddy current signals.

#### 4.2.2 Surface Roughness Measurements

Surface roughness primarily affects eddy current signals by increasing the chance of problems associated with varying or unknown lift-off. Surface roughness of all samples was determined, both in the as-heat-treated and shot-peened states, and shown in Figures 3.20 and 3.21. It was expected that the shot-peened samples would have a higher surface roughness, and this was indeed seen to be the case. The measured
roughness values varied from a minimum of 0.11 microns to 2.04 microns maximum (Figure 3.20), with the unpeened samples having an average rms roughness of $0.38 \pm 0.2$ microns, while the shot peened samples have average rms roughness of $1.58 \pm 0.4$ microns.

Given that surface roughness does exist, the question is to what extent does the varying roughness affects the eddy current signal. The answer is very little; the inversion model chosen allows us to overcome this surface roughness variation, where the eddy current signals corresponding to the top 10 microns of the sample are subtracted and the rest of the signals are inverted. The inversion model will be discussed in more detail in section 5.2. But by employing the combination of swept frequency eddy current measurement used, which has been shown effective in minimizing the effects of lift-off, with use of the inversion model, it is believed that the surface roughness effect can be discounted.

To check this hypothesis a simple experiment was conducted and the result is shown in Figure 4.1. To take into account the roughness effect, which might act as additional lift-off, we compared the effect of 25 µm and 31 µm liftoff values (a difference of 6 µm, approximately three times the value of the most severe surface roughness) to see the signal change. Figure 4.1 compares the SFEC signals obtained for the UA1 condition shot peened at 12 A with lift-offs of a) 25 µm and b) 31 µm. Condition UA1 was chosen as it has larger surface roughness compared to other sample conditions. From the figure, it can be seen that for an additional lift-off of 6µm, the change in signal is 10%. Since the surface roughness measured in this study is a maximum of 2 µm, the maximum change that surface roughness might expect to add to the signal is thus less than 2% to the signal. Therefore, the roughness effects in this study are expected to be minor.
Figure 4.1 SFEC signals obtained for UA1 condition comparing additional lift-off of 25 μm and 31 μm on shotpeeneed sample at 12A.

4.3 Instrumental Parameters
Any time an instrument is employed to gather data there is always the possibility that the instrument was used incorrectly, was not calibrated, etc. Even if used correctly there is a certain amount of variability and error in any measurement. Possible contributions to the eddy current signal obtained due strictly to instrumental factors are briefly discussed below.

4.3.1 Lift off
As discussed above in relation to the surface roughness discussion, the potential lift-off problem has been circumvented by the use of the liftoff-normalized vertical component EC signal $V_{\text{EXP}}$ (V-component signal). The use of $V_{\text{EXP}}$ helps to suppress the liftoff noise, while avoiding mismatch due to variation across different instruments used in frequency range studied as explained in Section 1.5.
4.3.2 Instrument calibration

Coils used for the swept frequency eddy current measurements were calibrated using samples of known conductivity. The reproducibility of the data was ensured by repeating each measurement procedure five times. Any data found to be irreproducible was not included in the analysis.

4.3.3 Instrument hardware

Measured eddy current signals obtained can be affected by being performed near edges of the samples. This kind of error might cause potential error in the analysis. Hence, swept frequency eddy current measurements in this study were performed by placing the coil far from the edges of the samples and always measured from the center of the sample.

4.4 References


CHAPTER 5: ANALYSIS OF EDDY CURRENT SIGNALS

Having discussed potential problems associated with conducting eddy current measurements on the samples used in this thesis attention will now be focused on the actual measurements themselves. Evaluation of the measurements in relation to both microstructure and expected residual stress measurements are considered.

5.1 Conductivity Deviation Profiles

As stated in Section 3.5, conductivity profiles were obtained using a matched filter approach by inverting the swept frequency signals measured from the corresponding samples. When fitting the data to obtain a functional form for the conductivity profile \( \frac{\Delta \sigma (z)}{\sigma_{\text{Ref}}} \), the following three factors are considered:

a) Possible shifts in conductivity when comparing the reference to the sample. When using a reference of different conductivity, we expect to see a constant shift in conductivity profile, which represents the conductivity difference between the reference and test sample alone.

b) Surface damage on or in the sample. If there is any surface damage present, that might produce a layer where the conductivity will be lower than that of the bulk; this needs to be taken into account. This can include damage that extends into the sample, such as deformation slip lines, rather than just surface deformation.

c) A peak in residual stress with exponential decay. Residual stress can result from a number of causes. In all cases, residual stress does not remain constant throughout the bulk of a part but is expected to have a peak value and decay, depending on
location in the sample, as the entire part seeks to balance the residual stress in any one specific location.

The above mentioned factors were considered in obtaining the conductivity profile fits to the SFEC signals (presented in Figure 3.22-3.28) by computing the theoretical V-component signals. From the SFEC signals, theoretical V-component signals $V_{TH}$ are computed via the approximate formula i.e., when the relative conductivity changes are sufficiently smaller than unity, and when the outer diameter of the detection coil (assumed a cylindrical air-cored coil) is much larger than any other length parameters, such as the skin depth $\delta$ or coil liftoff $l$, $V_{TH}$ can take an approximate form shown in Equation 5.1 where $\frac{\Delta \sigma (z)}{\sigma_{Ref}}$ denotes the small conductivity deviation $\Delta \sigma (z) = \sigma(z) - \sigma_{Ref}$ relative to the reference conductivity $\sigma_{Ref}$ as a function of depth $z$.

$$V_{TH}(f) = -\frac{1}{l} \text{Im} \int_{0}^{\infty} \exp \left[-(1+j) \left(\frac{2z}{\delta}\right)\right] \frac{\Delta \sigma(z)}{\sigma_{Ref}} dz$$  \hspace{1cm} 5.1

To obtain the conductivity profile fit, the three considerations mentioned above labeled a-c must be accounted for. How this was done, and the assumptions made in doing so, are detailed as follows:

**Consideration a: Possible shift in the conductivity when comparing the reference to the sample.**

Possible shift in the conductivity depends on the difference between the reference and test sample, which is a constant, given by the functional form represented in equation 5.2.

$$\frac{\Delta \sigma(z)}{\sigma_{Ref}} = g(a_1) = a_1$$  \hspace{1cm} 5.2
where \( \alpha_1 \) is a fitting parameter used for this consideration in obtaining the conductivity profile.

![Figure 5.1 Conductivity deviation profile due to possible shift in conductivity.](image)

By using this functional form, we can quantify a conductivity deviation profile \( \left( \frac{\Delta \sigma(z)}{\sigma_0} \right)^{(1)} \) (Eq. 5.3), which is due to the effect of change in conductivity \( \alpha_0 \) on the EC signals and the conductivity profile is shown in Figure 5.1.

\[
\left( \frac{\Delta \sigma(z)}{\sigma_0} \right)^{(1)} = \alpha_0
\]

**Consideration b: Surface damage**
Surface damage creates a layer of low conductivity of a given fixed depth. Both of these factors, two factors, namely, the depth or thickness of the damaged layer and the conductivity of the layer, can be determined, the first through microstructural observations and the second through eddy current measurements. We can assume the functional form for surface damage has the form,

\[
\frac{\Delta \sigma(z)}{\sigma_{Ref}} = g(a_2, \xi) = a_2 \cdot \theta(\xi)
\]

where \( \xi = \frac{z}{d} \). The fitting parameters for equation 5.4 are \( a_2 \) and \( d \).

Observation of polished samples shows that surface damage (in this case, slip damage) is observed to be present within 20 to 30 \( \mu m \) of the surface of the shot-peened samples. This damage was observed in all shotpeened samples. An example of this is shown in Figure 5.2.

Figure 5.2 SEM micrograph of shot peened surface showing the presence of slip damage region that corresponds to the layer of lower conductivity than the bulk.
Figure 5.3 Conductivity deviation profile due to surface damage.

The effect of surface damage on the SFEC is quantified by introducing a conductivity deviation profile (Equation 5.5) that exists within the damaged layer (i.e., $a_0 \leq 30 \, \mu m$) and shown in Figure 5.3.

\[
\left( \frac{\Delta \sigma(z)}{\sigma_0} \right)^{(II)} = \alpha_0 + \beta_0 \begin{cases} 1 \\ 0 \end{cases} \text{ when } \begin{cases} z \leq a_0 \\ z \geq a_0 \end{cases}
\]

By combining the equations 5.2 - 5.5, the V-component EC data are computed as $V_{TH}^{(II)}$ (Equation 5.6) that is used to fit the experimental data.

\[
V_{TH}^{(II)} = \frac{\delta}{4l} \left\{ \alpha_0 + 2\beta_0 \left[ 1 - e^{-\xi} \right] \right\}
\]

where, $\alpha_0$ is the depth corresponding to the maximum damaged layer and $\beta_0$ is the strength of the damaged-layer effect.
Consideration c: A peak in residual stress with exponential decay

After subtraction of the fitted damaged layer effect, the residual EC signals are processed further to consider a residual stress peak with exponential decay. This consideration depends on factors like the depth to which residual stress can potentially reach (i.e., $a \leq 500\mu m$) and the $\varepsilon$-ratio of peak height to the length of decay, and can be written in functional form (Equation 5.7) as

\[
\frac{\Delta \sigma(z)}{\sigma_{Ref}} = g(a_3, \varepsilon, \xi) = a_3 \frac{\xi^\xi}{\varepsilon^2 + \xi^2} e^{-\xi}
\]

where, $\xi \equiv \frac{2a}{d}$, $0 \leq \varepsilon \leq 0.5$ and $a_3$ is a fitting parameter.

Figure 5.4 Conductivity deviation profile due to a peak in residual stress with exponential decay.
Including all three of these considerations and by combining the equations 5.2 - 5.7, the assumed conductivity profile (Eq. 5.8) obtained is given as

\[
\left( \frac{\Delta \sigma(z)}{\sigma_0} \right)^{\text{(III)}} = \alpha + \beta \frac{z}{a} \left[ 1 + \left( \frac{z}{a} \right)^2 \right]^{-1} \exp \left( -\varepsilon \frac{z}{a} \right) \tag{5.8}
\]

and shown in Figure 5.4. In practice, the residual stress data were matched against the computed \( V^{(\text{III})}_{\text{TH}} \) (Eq. 5.9) that involves the exponential integral function \( E_1 \) defined in Eq. 5.10.

\[
V^{(\text{III})}_{\text{TH}} = \frac{\delta}{4i} \left\{ \alpha - \beta \xi \text{Im} \left[ e^{i\theta} E_1(j\theta) + e^{-i\theta} E_1(-j\theta) \right] \right\} \tag{5.9}
\]

where \( \theta \equiv (1 + j)\xi + \varepsilon_2, \quad \xi \equiv \frac{2a}{\delta} \)

\[
E_1(z) = \int_z^{\infty} t^{-1} \exp(-t) \, dt \tag{5.10}
\]

By using Equation 5.8, conductivity profiles were obtained by fitting theoretical curves to the raw data via the nonlinear least-squares minimization process. Doing this for the raw data presented in Figures 3.22 – 3.28 produces the results shown in Figure 5.6. In the process of nonlinear least-squares minimization, the strength of the damaged-layer effect is constrained to be less than 20% (i.e., \( |\beta_0| \leq 0.2 \)) in addition to the constraints \( \alpha_0 \leq 30 \mu m \) (depth corresponding to maximum damaged layer), \( 30 \mu m \leq \alpha \leq 500 \mu m \) (depth range corresponding to residual stress produced by shot peening), and \( 0 \leq \varepsilon \leq 0.5 \). All the above mentioned parameters are summarized in Table 5.1.
Table 5.1 Summary of parameters used for analysis.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Constraints</th>
</tr>
</thead>
<tbody>
<tr>
<td>$a_0$ (depth of damaged layer)</td>
<td>$\leq 30 \mu m$</td>
</tr>
<tr>
<td>$\beta_0$ (strength of damaged layer)</td>
<td>$\leq 0.2$</td>
</tr>
<tr>
<td>$a$ (depth of residual stress)</td>
<td>$30 \mu m \leq a \leq 500 \mu m$</td>
</tr>
<tr>
<td>$\varepsilon$ (ratio of peak height to length decay)</td>
<td>$0 \leq \varepsilon \leq 0.5$</td>
</tr>
</tbody>
</table>

By examining Figure 5.5, one can see that the estimated conductivity deviations (i) are nominally of the same order of magnitude among all samples, (ii) all show positive changes in the sequence according to the Almen intensities of 4A, 8A, and 12A, and (iii) exhibit weak hardness effects unlike the V-component EC signals. Of note is that the estimated conductivity changes are still above 1% in most cases, being larger than those expected from the piezoresistivity effect (see section 1.4.3). This implies there must be another competing factor (e.g. microstructure) which needs to be considered for residual stress measurements.

Figure 5.5 shows that almost all the theoretical fits to the raw data presented in Figures 3.22-3.28 have $R^2$ close to 0.98 except for the PA and UA2 condition. It is interesting (and perhaps unexpected) that while both conditions AR and SHT (Figures 3.2 and 3.3) are similar in microstructure, their response to shot peening is different (Figure 5.5), producing different deviation profiles. The conductivity fits for these samples both have $R^2$ value 0.9836 or above. One possible explanation for their difference might be that the additional heat treatment given SHT has resulted in subtle changes to the structure that is not readily apparent when compared to sample AR. A slightly larger grain size, slightly lower residual stress, dislocation density, etc., all might combine to produce the observed differences.
Figure 5.5 Conductivity deviation profiles of the shot-peened Inconel 718 samples estimated from the EC data by the described procedure for all samples AR, SHT, UA1, UA2, PA, OA1 and OA2.
Samples UA2 and OA1 show smaller conductivity deviations than the remaining samples. Although the microstructures of these samples (Figures 3.6 and 3.9) show the predominant phases in each sample are different (UA2 has γ' and γ" precipitates, while OA1 has γ" and δ precipitates), the volume fraction of these precipitates and bulk conductivities of these samples (Figure 5.7) are similar. Two conclusions can be drawn from these observations. 1) The conductivities of a mixture of γ' / γ" precipitates versus γ" / δ precipitates appear similar, and 2) it is clear that microstructure plays a distinct role in changing the conductivity of the samples as both samples are near the peak-aged condition yet show large conductivity deviations from that condition.

Given the deviation profiles of Figure 5.5 the next task is to attempt to separate out the stress effects from other competing factors, the principal one being microstructure. This is discussed in the next section.

5.2 Determination of Near Surface Conductivity Variation

By comparing theoretically calculated eddy current V-component signal to the experiment V-component signal obtained using the difference between the reference and test sample, we can obtain the near surface conductivity variation. This, in turn, can be related to variation in precipitate density.

Conductivity variation observed in eddy current (V-component) signals due to precipitates was determined by the following steps:

1) Measurement of the bulk conductivities as a function of bulk precipitate fraction
2) Determination of volume fraction of precipitate as a function of depth
3) Combining 1 and 2, estimation of conductivity as a function of depth
4) Using step 3, calculation of theoretical eddy current (V-component) signal
5) Comparison of calculated and experimental eddy current (V-component) signals
A flow-chart of the entire process described above is provided in Figure 5.6.

**Figure 5.6** Flowchart describing the calculation of near surface conductivity variation.

**Step 1: Bulk conductivity as a function of precipitate volume fraction**

To obtain an estimate of the bulk conductivity for any given precipitate density, a calibration curve was obtained by plotting the bulk conductivity (Table 3.1) as a function of the measured bulk precipitate density (Table 3.4) obtained by examination of all of the samples as described in section 2.6. These data are shown in Figure 5.7. By using a linear fit line these data can be used as a calibration curve to estimate conductivity as a function of any given level of precipitation.
In obtaining this calibration curve, the following assumptions are made. As no literature reports are available on the conductivities of γ’, γ” and δ phases separately\(^7\), the conductivities of all the phases are assumed the same. It is also assumed that crystallographic orientation of the precipitates plays little or no role in the measured conductivities. These assumptions may not be true. For example, UA2 mainly has γ’ and γ” precipitates while OA1 has γ” and δ precipitates; it is interesting that the value recorded for UA2 is the reading that varies most substantially from the best fit line. However, considering that the phase assemblage and microstructure varies substantially when moving from SHT, through the under-aged region, to peak aged then to over-aged, the deviation is relatively minor and the assumptions are believed to be fairly accurate.

**Figure 5.7** Calibration curve of bulk conductivity as a measure of volume fraction of secondary precipitates.
Step 2: Volume fraction of precipitates as a function of depth

Volume fraction of precipitates as a function of depth was examined to determine whether any variation or distribution of secondary precipitates existed. For this examination sample OA2 condition was chosen since, being in the overaged condition, the phases would be most easily observed.

SEM micrographs for sample OA2 were obtained from three different regions, those being the upper shot-peened region (Figure 5.8a), the middle (bulk) region (Figure 5.8b), and the bottom unpeened region (i.e. the opposite side from the shot peened surface) (Figure 5.8c). Details of this analysis are described in detail under Experimental Procedure in section 2.6. Precipitate volume fractions were calculated from these three different regions, which are assumed to represent precipitation that might be expected in a typical worked region, the bulk of the sample, and on a free surface. This procedure allowed observation of any variation in the precipitate density between these regions and enabled the precipitate density as a function of depth with respect to the shot-peened region to be determined.

The measured volume fractions obtained from Sample OA2 are shown in Table 5.2. Reference measurements were obtained from a different sample piece, which was subjected to the same heat treatment as Sample OA2 but not shot peened and was used as a reference in swept frequency eddy current measurements. Note that the stated volume fractions at each depth are an average of five measurements taken across the width of the sample, the variation associated with each measurement depth being ±3%.
Figure 5.8 Representative SEM micrograph obtained from sample OA2, a) shot-peened region, b) bulk region, and c) free surface.

For both shot peened and unpeened regions, the distances recorded in the Table 5.2 are taken from the corresponding surfaces. In other words, for column 1, the distances recorded are measured from the shot peened surface and extend into the sample. For column 2, the distances were measured from the opposite side of the specimen piece (see Section 2.6 and Figure 2.6). The reference surface corresponds to the reference
sample used for swept frequency eddy current measurements, the measurements being made within the bulk of the sample as described by Figure 2.6

Table 5.2 Depth profiles of volume fraction of precipitates for Sample OA2 condition.

<table>
<thead>
<tr>
<th>Depth from Corresponding Surface (μm)</th>
<th>Shot peened region</th>
<th>Unpeened (free) region</th>
<th>Reference surface</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Volume fraction (%)</td>
<td>Volume fraction (%)</td>
<td>Volume fraction (%)</td>
</tr>
<tr>
<td>22</td>
<td>13.3 ± 8.6</td>
<td>12.4 ± 2.4</td>
<td>6.0 ± 1.5</td>
</tr>
<tr>
<td>44</td>
<td>11.8 ± 4.1</td>
<td>9.4 ± 3.2</td>
<td>6.1 ± 1.1</td>
</tr>
<tr>
<td>66</td>
<td>10.3 ± 4.1</td>
<td>11.6 ± 5.5</td>
<td>8.5 ± 3.7</td>
</tr>
<tr>
<td>88</td>
<td>10.5 ± 3.6</td>
<td>10.8 ± 3.7</td>
<td>7.5 ± 3.2</td>
</tr>
<tr>
<td>109</td>
<td>8.7 ± 3.8</td>
<td>9.3 ± 2.1</td>
<td>5.3 ± 2.6</td>
</tr>
<tr>
<td>131</td>
<td>7.8 ± 3.2</td>
<td>8.0 ± 3.4</td>
<td>9.0 ± 3.3</td>
</tr>
</tbody>
</table>

**Step 3: Conductivity as a Function of Depth**

By combining the results of Steps 1 and 2, it should be possible to determine conductivity as a function of depth in the event precipitate density is changing. The initial impression one obtains when viewing the images of Figure 5.8 is that precipitate density does vary as a function of depth in shot-peened samples. However, close observation of the data reveals several points. Firstly, there is a large amount of uncertainty associated with the measurements, so large in fact that statistically they can all be considered equivalent. Secondly, there is a considerable variation in the precipitate amount observed on the unpeened side of the sample as compared to the reference surface of the unpeened sample used for swept frequency eddy current measurements. These large uncertainties prevent a clear determination of whether precipitate density is changing as a function of shot peening, and a much more extensive study would need to be completed to statistically validate this supposition. However, for the sake of completeness, the subsequent discussion will deal with the two possibilities, namely,
A. Including the uncertainty, the average value of precipitate fraction remains constant for all depths.

B. Excluding the uncertainty, the average value of precipitate fractions change with depth.

For the second scenario, Table 5.3 gives us the conductivity as a function of depth for condition OA2 in three different regions. Depth profiles of volume fraction of precipitates as a function of depth using the calibration curve (Figure 5.7) is given by Figure 5.9. Note the large error bars (c.f. scenario A).

Table 5.3 Depth profiles of conductivities for OA2 condition estimated using calibration curve of Figure 5.7.

<table>
<thead>
<tr>
<th>Depth (μm)</th>
<th>Shot peened region</th>
<th>Unpeened (free) region</th>
<th>Reference surface</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Conductivity (% IACS)</td>
<td>Conductivity (% IACS)</td>
<td>Conductivity (% IACS)</td>
</tr>
<tr>
<td>22</td>
<td>1.48 ± 0.07</td>
<td>1.48 ± 0.02</td>
<td>1.43 ± 0.01</td>
</tr>
<tr>
<td>44</td>
<td>1.47 ± 0.03</td>
<td>1.45 ± 0.02</td>
<td>1.43 ± 0.01</td>
</tr>
<tr>
<td>66</td>
<td>1.46 ± 0.03</td>
<td>1.47 ± 0.04</td>
<td>1.45 ± 0.03</td>
</tr>
<tr>
<td>88</td>
<td>1.46 ± 0.03</td>
<td>1.47 ± 0.03</td>
<td>1.44 ± 0.02</td>
</tr>
<tr>
<td>109</td>
<td>1.45 ± 0.03</td>
<td>1.45 ± 0.02</td>
<td>1.42 ± 0.02</td>
</tr>
<tr>
<td>131</td>
<td>1.44 ± 0.03</td>
<td>1.44 ± 0.03</td>
<td>1.45 ± 0.03</td>
</tr>
</tbody>
</table>
Step 4: Calculation of theoretical V-component

Conductivity deviation $\Delta \sigma(z)$ as function of depth was given by equation 5.11.

$$\Delta \sigma(z) = \sigma_T(z) - \sigma_R(z)$$ \hspace{1cm} 5.11a

Where, $\sigma_T(z)$ – Test conductivity, $\sigma_R(z)$ - Reference conductivity. Equation 5.11a can be rewritten as:
\[ \Delta \sigma(z) = \sigma_T(z) - \sigma_{Tb} - (\sigma_R(z) - \sigma_{Rb}) + (\sigma_{Tb} - \sigma_{Rb}) \quad 5.11b \]

Where, \( \sigma_{Tb} \) - Test bulk conductivity, \( \sigma_{Rb}(z) \) - Reference bulk conductivity.

The difference between test conductivity and test bulk conductivity \( (\sigma_T(z) - \sigma_{Tb}) \) gives test surface conductivity \( \sigma_{Ts}(z) \) and the difference between reference sample conductivity and reference sample bulk conductivity \( (\sigma_R(z) - \sigma_{Rb}) \) gives reference surface conductivity \( \sigma_{Rs}(z) \). Similarly, the difference between test bulk conductivity and reference bulk conductivity \( (\sigma_{Tb}(z) - \sigma_{Rb}) \) will give the bulk conductivity difference \( \Delta \sigma_b \). By substituting these values into Equation 5.11b, we get,

\[ \Delta \sigma(z) = \Delta \sigma_b + \sigma_{Ts}(z) - \sigma_{Rs}(z) = \Delta \sigma_b + \Delta \sigma_s(z) \quad 5.11c \]

Where, \( \Delta \sigma_b \) - Bulk conductivity difference, \( \Delta \sigma_{Ts}(z) \) - Test surface conductivity, \( \Delta \sigma_{Rs}(z) \) - Reference surface conductivity, \( \Delta \sigma_s(z) \) - Surface conductivity difference. The V-Component in Equation 2.1 can be written as \( V = V_b + V_s \), where \( V_b \) gives the vertical component signal due to the bulk and is given by equation 5.12:

\[ V_b = -\frac{1}{l} \text{Im} \int_0^\infty e^{-pz} \frac{\Delta \sigma_b}{\sigma_0} dz = \frac{\delta}{4l} \frac{\Delta \sigma_b}{\sigma_0} \quad 5.12 \]

\( V_s \) gives the vertical component signal due to the precipitates and is given by equation 5.13:

\[ V_s = -\frac{1}{l} \text{Im} \int_0^\infty e^{-pz} \frac{\Delta \sigma_s(z)}{\sigma_0} dz \xrightarrow{\delta \to \infty} \text{const} \quad 5.13 \]

By substituting the values of \( \Delta \sigma_s(z) \) and \( \Delta \sigma_b \) into equations 5.12 and 5.13, we calculate \( V_b \) and \( V_s \), and the results are shown in Figure 5.10. The summation of \( V_b \) and \( V_s \) gives us the theoretical vertical component signal.
Figure 5.10 Theoretical V-component signal obtained by the summation of $V_b$ (V-comp signal due to bulk) and $V_s$ (V-comp signal due to precipitates) for a) OA2 condition, Free surface and b) Shot peened at 12A (bottom).
The theoretical vertical component signal (red line in Fig. 5.10) is given by the summation of $V_b$, the vertical component signal due to bulk (shown in blue in the figure) and $V_s$, the vertical component signal due to precipitates (given by green line).

**Step 5: Comparison of Experimental and Theoretical Data**

The actual experimental values (Figure 5) obtained are given by $V_{exp\text{-}high}$ and $V_{exp\text{-}low}$, the vertical component signal in the frequency ranges 1 MHz to 50MHz and 100 kHz to 4MHz, respectively. Figure 5.11 shows the comparison of theoretical and experimental $V$-component signals for sample OA2. The results from the free surface (i.e., as-heated-treated condition) are shown in Fig. 5.11a, while the results from the shot-peened surface are shown in Fig. 5.11b.

While neither figure 5.11 a or b shows a perfect match between theoretical and experimental vertical component signals, it must be remembered that factors such as surface damage in the samples and residual stress due to shot peening are yet to be added. However, we can clearly see that the red theoretical line, which includes adding the vertical component signal due to precipitates, has a reasonable match to the $V_{exp\text{-}low}$ experimental data, much better than simply considering the vertical component signal due to the bulk alone.
Figure 5.11 Comparison of theoretical V-component signal against experimental V-component signal for a) free surface and b) peened surface of condition OA2. The shot peened level shown was 12A. Experimental data points are joined by straight lines that serve only as a guide to the eye.
Figure 5.12 shows us the comparison of two scenarios mentioned earlier, namely whether precipitation density remains constant or is increasing. Note that only the peened surface is examined in this analysis.

![Graph showing comparison of theoretical V-component signal against experimental V-component signal for peened surface of condition OA2 shotpeened at 12A, a) assuming precipitate density increases, b) constant average precipitate density.](image)

**Figure 5.12** Comparison of theoretical V-component signal against experimental V-component signal for peened surface of condition OA2 shotpeened at 12A, a) assuming precipitate density increases, b) constant average precipitate density.
By comparing top and bottom figures in Figure 5.12 it can be seen that slight changes in precipitate density have little effect on the final calculation of eddy current signals.

5.3 References

CHAPTER 6: DISCUSSION

It is clear from the conductivity profiles shown in Figure 5.5 that the eddy current response changes both as a function of heat treatment and of shot peening. Both of these effects are related to microstructure.

6.1 Effect of heat treatment on Eddy Current

In the case of heat treatment, the precipitation of second phases produces the change in conductivity. Due to annealing of the structure brought about by the elevated temperatures used to produce precipitation, these samples can be expected to be free from any residual stresses, and the effect should be purely related to microstructure. In this case one might expect a relatively straightforward relationship to exist between measured hardness and eddy current response.

Figure 6.1 shows the measured eddy current responses obtained from all samples subjected to various heat treatments. The reference sample used for eddy current has a bulk conductivity of 1.39% IACS. As we can see, each heat treatment has different responses. Eddy current signals from heat treated samples (AR, SHT and UA1) that have lower conductivity than the reference samples fall below zero in the figure, while heat treated samples which have higher conductivity than the reference sample fall above zero in the figure.
Figure 6.1 V-Component signals obtained from all samples subjected to various heat treatments.

Both samples AR and SHT are in the solutionized state. In these samples the larger amount of lattice strain due to the atoms in solid solution leads to lower conductivity of these samples as explained in section 3.1. Conductivity drops further for Sample UA1, the first heat-treated sample which has secondary phase precipitates. Although atoms are being removed from solid solution, which should result in an increase in conductivity, the formation of extremely small (~3nm) precipitates (similar to GP zones in aluminum precipitate alloys)\(^1\), leads to an increase in strain, which decreases conductivity. Though the hardness increases compared to AR and SHT, there is an overall drop in conductivity, which lies well below the AR and SHT results in Figure 6.1.
As the precipitates continue to grow they can be expected to change from coherent with the matrix to incoherent\(^1\). In Sample UA2, where the precipitates have reached an average size of 5nm, removal of solute atoms from the matrix has a greater effect than the creation of new boundaries associated with the precipitates. Accordingly, UA2 has higher conductivity than that of UA1. Sample PA, which is in peak hardened condition, has the highest conductivity of all the samples with a mean precipitate size of 7nm. Conductivity decreases slightly again for both OA1 and OA2 conditions, where coarsening has produced larger average precipitate sizes. When considering the error associated with the measurements, the increased conductivity associated with sample PA is clearly different from that of samples UA2, OA1, and OA2. From this, we can deduce that heat treatment, which is the primary source of producing secondary phase precipitates, plays a large role in affecting the eddy current signal response.

### 6.2 Effect of Shot peening on Eddy Current

Separating and understanding the effects produced due to shot peening is considerably more complex. Since shot peening involves plastic deformation of the surface, the outermost surface layer will exhibit physical roughness, and the near-surface region will consist of a deformed grain structure with an expected increase in dislocation density due to the plastic deformation. Compressive residual stresses result due to relaxation of the elastic component being constrained by the surrounding, deformed material. Taken together, separating microstructure effects from residual stress effects becomes a difficult challenge.

From Figures 3.22 – 3.28 the effect produced due to shot peening is clearly seen in the vertical spread of the plotted data. In all cases as shot peening intensity increases, the conductivity deviation profile also increases. If one assumes that precipitate density is not changing in the shot peened layer (see discussion in section 5.2), then the change in conductivity can be expected to primarily be a result of increased residual stress and damage to the microstructure, which will include surface roughness effects, increased
dislocation densities in the damaged layer and increased boundary scattering due to the formation of slip lines, shear bands, etc. Surface roughness caused by shot peening can act as additional lift-off. In this investigation, both the effect of surface roughness and microstructural damage can be discounted by use of the inversion model discussed in sections 4.2.2 and 5.1, respectively, which essentially discounts contributions to conductivity from the top 30 microns. Thus, the only component unaccounted for should be the residual stress present in the sample, apart from the microstructural effects. Once we account for the microstructure, we can deduce the residual stress, which should be present in the material down to depths as great as 500µm.

6.3 Combined effect of heat treatment and shot peening on eddy current signals

Eddy current signals are affected by a combination of both heat treatment and shot peening. When considering the combined effects of these two factors, it is useful to examine how the signal changes as a function of hardness, since both shot peening and heat treatment produce hardness changes.

The change in eddy current response follows the trend of hardness change shown in Figure 3.1. It was observed for samples with a solutionized, underaged or predominantly δ-phase structure that shot peening raises the apparent near-surface conductivity, and the EC responses are generally stronger for higher Almen intensities. As the sample hardness increases, the shot-peening induced changes in the EC response diminish in magnitude. To induce the same magnitude of residual stress in hardened material as is seen for soft samples, higher Almen intensity shot peening is required.

Figure 5.11a shows a reasonable fit between the experimental and calculated theoretical vertical component signal for the free surface, although it is clear that the
theoretical value is overestimated. In the calculation of the theoretical V-component signal, variations due to factors such as surface finish or other anomalies were not included in the analysis; precipitation alone was considered. Factors such as surface finish, damage, or other anomalies can be expected to affect the measured signal. This is evident in Figure 5.11b, where the fit as compared to the shot-peened surface is much worse. In this case the theoretical vertical component signal is underestimated, as compressive stress induced by shot peening and surface damage were not included in the calculation.

Comparison of theoretical and experimental V-component signals obtained from the OA2 condition sample show that while surface conditions may play a small role in the measured signal the residual stress profile is critical in obtaining an accurate fit between experimental and theoretical values. Residual stress profiles can be obtained by XRD and destructive layer removal and/or neutron diffraction for validation purposes. Once obtained the vertical component signal \( V_R \) due to shot peening can be calculated. By adding the vertical component signal \( V_R \) due to shot peening to the theoretical vertical component \( (V_b + V_s) \), a better fit should be possible. However, we can clearly see that adding the vertical component signal due to precipitates produces a better fit of theoretical vertical component signals to the experiment vertical component signals, as compared to the vertical component signal due to the bulk alone.

6.4 References
CHAPTER 7: CONCLUSION

Swept frequency eddy current (SFEC) measurements were performed in two different but partially overlapping frequency bands using different sets of instrumentation and detection coils from 100 kHz to 50 MHz with an additional coil liftoff of 25 μm from the sample surface. For each frequency band, baseline and test V-component EC signals were measured from the Ni-based alloy 718 on samples that had been heat-treated to produce precipitation hardening followed by shot peening. Samples were examined to obtain the preexisting and post-peening conductivity profiles, respectively. Examination of the conductivity data in relation to the microstructure as determined using SEM and TEM observations yields the following conclusions.

• The change of EC response with heat treatment was clearly seen to relate to the amount of precipitation, which was observed directly and monitored indirectly by measuring the hardness of the samples. Bulk conductivity was related to total amount of precipitation but appeared to be independent of precipitate type, i.e., γ', γ" or δ. This would suggest that the individual conductivities of these phases are similar.

• Shot peening is known to result in residual compressive stresses with the amount of residual stress increasing with increasing peening intensity. In this study, swept frequency eddy current signals obtained from identical samples that had been subjected to varying Almen intensities were seen to increase corresponding to the amount of peening received. This suggests that swept frequency eddy current signals can be used to determine residual stress if all the other factors that might affect the signal (e.g., surface roughness, damage, lift-off, etc.) can be accounted for.

• Change in eddy current signals due to variation in microstructure (precipitates) was determined theoretically. Comparison of this theoretically calculated (V-
component) signal to the experimental (V-component) signals obtained by SFEC measurements clearly reveals the microstructure dependency of the eddy current signals.

Comparison of theoretical signals with experimental eddy current (V-component) signals show that while surface conditions may play a small role in the measured signal the residual stress profile is critical in obtaining an accurate fit between experimental and theoretical values. By accounting for all the other factors (surface damages, microstructure), residual stress present in the sample can be calculated.
CHAPTER 8. RECOMMENDATIONS FOR FUTURE WORK

1. Residual stress profiles of shot-peened samples could be obtained by neutron diffraction without any destructive layer removal as an independent study and compared with the experimental data obtained using eddy current measurement to enable exact inference of the actual stress profile for the given microstructure.

2. Near-surface conductivity variation due to secondary phase precipitates could be studied in more detail for all conditions. By doing this, a complete data profile could be obtained that will aid in development of a model that could be used for predicting the residual stress profile for samples with different microstructures and shot peening intensities without employing a destructive layer model.

3. A calibration curve of residual stress profiles as a function of shot-peening intensity can be obtained as follows: a) Shot peen the sample with different intensities b) obtain the residual stress profile by surface layer removal / neutron diffraction techniques c) using these data, develop a calibration curve of stress induced versus depth as a function of peening intensity.
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