2007

Residual stress assessment for shot peened nickel based superalloy by eddy current technique

Yuping Shen
Iowa State University

Follow this and additional works at: https://lib.dr.iastate.edu/rtd

Part of the Materials Science and Engineering Commons, and the Mechanical Engineering Commons

Recommended Citation
https://lib.dr.iastate.edu/rtd/15988

This Dissertation is brought to you for free and open access by the Iowa State University Capstones, Theses and Dissertations at Iowa State University Digital Repository. It has been accepted for inclusion in Retrospective Theses and Dissertations by an authorized administrator of Iowa State University Digital Repository. For more information, please contact digirep@iastate.edu.
Residual stress assessment for shot peened nickel based superalloy by eddy current technique

by

Yuping Shen

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:
R. Bruce Thompson, Major Professor
Brian Gleeson
Ralph E. Napolitano
Norio Nakagawa
John R. Bowler
Joseph N. Gray

Iowa State University
Ames, Iowa
2007

Copyright © Yuping Shen, 2007. All rights reserved.
DEDICATION

To my wife Ling, my mother Yuzhen, my father Jinhai, my sister Cuiping and my brother Guoping for their support and encourager during my doctoral program study.
TABLE OF CONTENTS

List of Tables ........................................................................................................................................ vi

List of Figures ......................................................................................................................................... viii

Abstract .................................................................................................................................................. xiii

CHAPTER 1. OVERVIEW .................................................................................................................. 1

References: ........................................................................................................................................... 6

CHAPTER 2. SWEPT HIGH-FREQUENCY EDDY CURRENT INSPECTION SYSTEM ................................................................. 8

2.1 Introduction.................................................................................................................................. 8

2.2 Experimental Setup .................................................................................................................. 10

2.3 Validation of Experimental Setup and Inversion Procedure ................................................. 12

References: ........................................................................................................................................... 20

CHAPTER 3. CONDUCTIVITY PROFILE DETERMINATION BY EDDY CURRENT FOR SHOT PEENED SUPERALLOY SURFACE ........ 22

3.1 Introduction.................................................................................................................................. 22

3.2 Experimental Procedure ............................................................................................................ 23

3.3 Eddy Current Inversion Models ............................................................................................... 27

3.3.1 Conductivity profile of shot-peened metal plate ............................................................... 27

3.3.2 Eddy current model of multi-layered half space ............................................................... 28
APPENDIX A. PIEZORESISTIVITY THEORY FOR TEXTURED MATERIALS WITH CYLINDRICAL SYMMETRY

82

APPENDIX B. RELATIONSHIP BETWEEN ODC \( W_{400} \) AND XRD PEAK INTENSITY RATIO

90

APPENDIX C. PERPENDICULAR STRAIN AS A FUNCTION OF X-RAY DIFFRACTION INDEX

93

References: .................................................................................................................. 96

ACKNOWLEDGEMENT ....................................................................................................... 97
List of Tables

Table 2.1 Configurations of simulated layer specimens used in the validation of the conductivity profile in forward model (Stage 1) and inversion procedure (Stage 2 to Stage 4)........................................................................12

Table 2.2 Summary of the results for Stages 2, 3 and 4 inversion with the following fixed input parameters: the bulk conductivity of IN718 block \( \sigma_{\text{IN718}} = 1.38\% \text{IACS} \), and the measured thicknesses of the IN718 and NiCr foils are 103 \( \mu \text{m} \) and 133 \( \mu \text{m} \), respectively. Also shown are the conductivities of the IN718 and NiCr foils measured using the DCPD method. ................................................................................20

Table 3.1 The shot peening parameters and the surface roughness values of the unpeened (Sample 1) and peened samples (Sample 2 to 5). 1 PSI = 6894 Pa; * \( S_a \) = Arithmetic mean of the deviations from the mean;

** \( S_q \) = Quadratic mean of the deviations from the mean; + \( S_p \) = Highest peak of the surface; ++ \( S_v \) = Deepest valley of the surface. .............23

Table 3.2 The EC probe parameters for the bridge coil.................................................................31

Table 3.3 Rietveld refinement of X-ray diffraction data of IN718 Sample 5.........................46

Table 4.1 Texture profile measured from partial X-ray diffraction polar figure of shot peened Inconel 718 by layer removal.................................................................53
Table 4.2  Inverted parameters..........................................................................................65

Table 5.1  Fitted XRD 2-theta peak positions of selected Miller index for shot peened Inconel 718 after different layer removal. The removed layer thicknesses are listed in Table 4.1. ........................................................................74
List of Figures

Figure 1.1  Residual stress and cold work profile for IN100 measured by destructive XRD........................................................................................................................................2

Figure 2.1  (a) Illustrations of simulated layer specimen configurations (not to the scale): (a) The “single-layer” configuration that consists of (a1) the top layer, (a2) the air gap and (a3) the substrate. (b) The “double-layer” configuration that consists of the two metal layers [(b1), (b3)], the two air gaps [(b2), (b4)] and the substrate [(b5)]. Summary of the results for Stages 2, 3 and 4 inversion with the following fixed input parameters: the bulk conductivity of IN718 block $\sigma_{IN718} = 1.38\%IACS$, and the measured thicknesses of the IN718 and NiCr foils are 103 $\mu$m and 133 $\mu$m, respectively. Also shown are the conductivities of the IN718 and NiCr foils measured using the DCPD method. ........................................................................................................9

Figure 2.2  Illustration of the EC probes fabricated on a PCB........................................10

Figure 3.1  (a) and (b) Secondary electron images and (c) the EDS spectra obtained from the Inconel 718 sample shot peened at 6A. The white spots (e.g. (b)) each surrounded by dark halos were identified by EDS as shot residues embedded into the sample surface. The EDS spectrum (refer to (c)) obtained from the white spot exhibits strong
Zr, Si and Al peaks corroborating the shot composition of 60-70wt% ZrO₂, 28wt% SiO₂ and < 10wt% Al₂O₃.

**Figure 3.2** The experimental V-component signals up to 50 MHz and the error bars. Experiments 1 through 4 refer to measurements from the Inconel 718 specimens shot peened at Almen intensities of 6A, 9A, 13A, and 17A, respectively.

**Figure 3.3** Illustration diagram of a solenoid coil placed above a laterally uniform multi-layered half space metallic alloy.

**Figure 3.4** The experimental V-component signals up to within 20 MHz and the corresponding computed signals obtained from the inverted parameterized conductivity depth profiles (Eq. 3.1) with 31 layers.

**Figure 3.5** Inverted conductivity deviation profiles as function of depth from the surface. They are parameterized via Eq. 3.1, discretized into 31 layers, and then determined by the EC inversion from the swept frequency V-component signals for each of the shot-peened samples.

**Figure 3.6** Convergence test of the inverted relative conductivity profiles against the number of fitting parameters.

**Figure 3.7** The inverted and modified relative conductivity depth profiles, used to demonstrate the strong sensitivity of the calculated V-component signals against small conductivity profile deviations in the near-surface region (see Figure 3.8 below).
Figure 3.8  The V-component signals computed from the modified profile (Fig. 7), in comparison with the experimental and inverted V-components. The large deviations outside the error bars show that the candidate profile shown in Figure 3.7 could not account for the experimental V component.

Figure 3.9  Insensitivity of the conductivity profile inversion to built-in lift off variation. Perturbing the built-in lift off values between 60 μm and 150 μm affects the maximum variation of the inverted relative conductivity only by 10^{-4} or less, demonstrating that the built-in lift off noise is highly suppressed in our inverse procedure.

Figure 3.10  Sensitivity of the inverted relative conductivity to the additional lift off value, as demonstrated by the variations among those computed from the inverted relative conductivity with 3 different additional lift off values. The actual additional lift off used in the measurements is 29.5 μm.

Figure 3.11  Surface morphology profiles of Sample 5 in a 2mm-by-2mm area (a) before and (b) after shot peening as measured by a laser profilometer.

Figure 3.12  Arithmetic (Sa) and quadratic (Sq) surface roughness values of the samples at several different Almen intensities, as measured by a laser profilometer.
Figure 3.13  Estimated upper bounds of the surface roughness effect onto the V-component signal as a function of frequency.................................44

Figure 3.14  Rietveld refinement of X-ray diffraction data of Sample 5 before (top) and after (bottom) shot peening... .................................................................45

Figure 4.1  XRD pattern of shot peened Inconel 718 at the surface and after a 96 μm thick surface layer was removed... .................................................................54

Figure 4.2  Depth profile of the ratio of the fitted integrated Bragg intensities of [111] peak over [022] peak.................................................................55

Figure 4.3  Schematic diagram of the mass flow due to shot peening and the corresponding geometrically necessary dislocations.............................................56

Figure 4.4  (a) Schematic diagram showing the coordinate rotation from the imaged cross-section to the peened surface of the Inconel 718 sample. (b) Inverse polar figure of grains from OIM measurements after rotation of coordinates.................................................................57

Figure 4.5  (a) Inverse polar figure of the first 50 μm thick peened surface layer of Fig. 4.4 (b). (b) Inverse polar figure of the region 60 μm to 150 μm below the peened surface.................................................................59

Figure 4.6  Depth profile of orientation distribution coefficient $W_{400}$....................63

Figure 4.7  Measured and best fitted V-component of SHFEC signals........................66

Figure 4.8  Inverted conductivity profiles with and without including the texture and roughness effect.................................................................66
Figure 4.9  Inverted residual stress profile compared to experimental stress profile measured by the $\sin^2\psi$ method (by Lambda Research, Inc.).............67

Figure 5.1  Fitted and experimental XRD theta-2theta data of diffraction indexes $[111]$ (above) and $[022]$ (below)...............................................................................................................76

Figure 5.2  The linear dependence of perpendicular strain of shot peened surface on index function $f(h,k,l)$ ........................................................................................................77

Figure 5.3  Measured residual stress profile by theta-2theta method compared to the one by Sine-Squared-Psi method from Lambda Research Corporation... .................................................................77

Figure A1  Euler rotation for a single crystal from an arbitrary orientation to the status with $[hkl]$ perpendicular to the sample surface..........................82
Abstract

Surface enhancement treatment by shot peening has been widely used in industrial applications, especially for aircraft engine components. Typical peening processes use small shots of a few hundred micrometer in diameter blasted on component surfaces, resulting in residual stress near the surface in the 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, by either replacing or re-treating the part. X-ray and neutron diffraction methods are the only two standard methods considered the most reliable. However, conventional XRD methods can achieve relatively low penetration depth (<10 μm for most metals), and hence 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. Neutron diffraction method has also a practical limitation in terms of its cost and resulting radioactivity.

In this dissertation, we developed a swept high frequency eddy current (SHFEC) measurement methodology for conductivity characterization of shot peened nickel based alloys. A model-based, eddy current inversion method is presented and applied to the SHFEC data obtained from a series of shot peened nickel based alloys to determine the depth profiles of actual conductivity up to 400 μm below the samples’ surfaces. By developing a modified piezo-resistivity theory that includes the effect of texture on stress-induced conductivity
changes, the residual stress profile of a shot peened IN718 sample is obtained from eddy current data. The obtained residual stress depth profile agrees with that measured by the standard layer removal XRD method. Texture profile of the shot peened IN718 sample is demonstrated by an XRD partial pole figure and orientation image microscopy (OIM). A new procedure of analyzing conventional $\theta - 2\theta$ XRD data is also developed in this dissertation for determining residual stresses in shot peened surfaces assuming an isotropic plane stress state. Collectively, this work lays foundation to the eddy current technique to assess residual stress in shot peened nickel based alloys that have extensive applications in aircraft engines.
CHAPTER 1. OVERVIEW

Quantitative nondestructive evaluation tests a component's ability to be operated safely, and prevents it from unexpected catastrophic failures. The research project, part of which is described in this dissertation, has the goal of improving the non-destructive evaluation of residual stress due to shot peening in order to justify the life extension of the engine components of the Air Force fleet for the economical benefits.

Jet engines are among the most critical and expensive parts of an airplane. Some jet engine components such as rotors are strengthened by the process called shot peening. Large amounts of small beads with diameters usually less than 1 mm are shot at the surface of the part during the fabrication, which creates compressive residual stresses that impede potential crack growth. Figure 1.1 shows the results of a series of measurements of compressive stress depth profiles and cold work for a nickel-based alloy IN100 due to the different shot peening levels (measured in, so called Almen intensities). Here the cold work is defined as percent thickness decrease after rolling that has equivalent full width at half maximum to the measured sample from X-ray diffraction $\theta$-2$\theta$ scan. However, this residual stress protection diminishes at high temperatures and under tensile stresses during operation of the jet engine. When the surface compressive residual stress is diminished, the chance of failure of the component in service increases and thus the component is required to be either shot peened again or replaced for safety reasons. Thus, knowing the state of the stress is important in
Figure 1.1  Residual stress and cold work profile for IN100 measured by destructive XRD.
determining how much of the component life remains in order to fully exploit the benefits of shot peening on extending the service life of components.

There is a long history of developing residual stress characterization methods such as X-ray diffraction (XRD), neutron diffraction, hole drilling, sectioning, ultrasonic, electromagnetic-acoustic and eddy current (EC) methods. Among them, X-ray and neutron diffraction methods are the only two standard methods considered the most reliable. However, conventional XRD methods can achieve relatively low penetration depth (<10 μm for most metals), and hence 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 possible alteration of the stress state by layer removal also needs to be considered by the X-ray diffraction results. The expense associated with this measurement is relatively high. Furthermore, there are limitations on sample geometry where XRD is applicable. Neutron diffraction method also has a practical limitation in terms of its cost and resulting radioactivity. Other approaches have been tried with varying degrees of success. Among them, the eddy current method is gaining increasing attention particularly in nickel-base superalloys, thanks to its advantage of being non-destructive and economical.

Groups of researchers reported observable correlations between EC signals and residual stress changes for shot-peened alloys, attributing the results to the piezoresistivity effect which refers to stress-induced changes in electrical conductivity. Recently, Blodgett, Nagy and Yu showed that the apparent conductivity of nickel-based alloy increased after shot peening. This observation has cast new light on the feasibility of using EC method for quantitatively measuring residual stress of nickel-based alloys, which are extensively used in
aviation industry. Yu and Nagy\textsuperscript{12,13} have developed empirical models to relate the measured apparent electric conductivity change (AECC) to the residual stress.

There could be several possible mechanisms that can affect the electric conductivity change measured by eddy current technique. Namely, (a) piezo-resistivity; (b) magnetic phase; (c) dislocation density; (d) grain surface resistivity and (e) second phase with different conductivity. Regarding (b), Blodgett et al.\textsuperscript{9} mentioned that no magnetic phase was detected for shot peened Inconel 718 samples. Also, for most magnetic materials, the magnetic resonant frequency is far less than 1 MHz. Thus for frequency above 1 MHz, we may ignore the possible magnetic phase effect on eddy current measurement. For the possible mechanism (c), since nickel based alloys are very hard materials, shot peening will increase the dislocation density only by a small amount, leading to a minor effect if any at all. Moreover, as being added free electron scatterers, the dislocations tend to decrease the AECC rather than increasing, contrary to the experimental observation. The mechanism (d) is not likely if the grain size is greater by about 50 times than the mean free path of the conductivity electron. This critical grain size is about 1 μm for Inconel 718 sample, while the average grain size of shot peened Inconel 718 is larger than this value as we will show in Chapter 4 below. The piezoresistivity effect [(a)] is the remaining mechanism accepted by the previous authors who used it to explain the increased AECC for shot peened superalloys.

Two issues stand out in the above mentioned prior publications, which are addressed in this dissertation. First, the apparent conductivity, as measured by the calibration-based method,\textsuperscript{9-13} shows frequency dependence. This dependency arises from the nontrivial depth profile of the true conductivity deviation near the surface. The concept of AECC may be inadequate to describe this behavior. AECC is defined as the conductivity of a hypothesized
half space metal with uniform conductivity and permeability, which generates the same coil impedance as the examined sample does at the same operation frequency and coil lift-off. Since the metal after shot peening does not have uniform conductivity, the AECC will vary with frequency. Secondly, the AECC description with isotropic piezo-resistivity does not completely explain the observation. As a case in point, the maximum residual stress of shot peened samples is almost 3 times lower than the value estimated from the experimental piezo-resistivity values when applying isotropic piezo-resistivity theory\textsuperscript{10}. The discrepancy is sufficiently significant to require proper consideration of actual conductivity depth profile, as well as taking into account of additional phenomena. This work proposes a texture effect as a possible explanation, unlike the previous work where the texture effect was not considered.

In this dissertation, we describe an eddy current methodology for obtaining the actual near surface conductivity profile of shot peened nickel based superalloys, and a model that incorporates the effect of shot-induced texture on the piezo-resistivity effect. Specifically, we first developed a swept high frequency eddy current measurement system operational to 50 MHz. This high level operational frequency is desirable as it offers a higher depth resolution of the near surface conductivity because of the smaller skin depth at a high frequency. The details of the measurement system and the results of the validation study using layered specimens are described in Chapter 2. Second we employed the eddy current inversion technique to obtain the actual conductivity profiles of a series of Inconel 718 samples shot peened at various Almen intensities. The details of this conductivity inversion procedure are described in Chapter 3. Finally, we developed a piezo-resistivity effect model to convert the inverted conductivity depth profile to a residual stress profile, which is the primary result of this dissertation. Our model takes into account the effect of stress-induced
texture on macroscopic piezoresistivity as a possible mechanism to account for the apparent discrepancy between the measured conductivity change by inverting EC data, and the values estimated from residual stress using isotropic piezoresistivity constants. The residual stress profile inverted from eddy current technique using the new model agrees with the standard XRD stress data. The details of the stress profile inversion are described in Chapter 4. Chapter 5 demonstrates a new method of analyzing experimental theta-2theta XRD data to determine isotropic plane stresses. The residual stress profile determined for a shot peened IN718 sample was compared with that obtained by the standard Sine-Squared-Psi method. Finally, we draw overall conclusions of the dissertation work in Chapter 6.

References
1. Data taken from a document prepared by Air Force Research Laboratory.


CHAPTER 2. SWEPT HIGH-FREQUENCY EDDY CURRENT INSPECTION SYSTEM

2.1 Introduction

A high-sensitivity swept high-frequency eddy current (SHFEC) system with operating frequency up to 50MHz has been developed and validated. This SHFEC system was 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 IACS. In this approach, we regard shot-peened surfaces as modified surface layers of varying conductivity, and determine the conductivity deviation profile by inversion of the SHFEC data. The SHFEC measurement system 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 the smallest skin depth of 80 μm for nickel-based superalloys. We devised a conductivity profile inversion procedure based on the laterally uniform multi-layer theory of Cheng, Dodd and Deeds, and performed validation studies. Namely, the forward and inverse
models were validated against measurements on artificial layer specimens consisting of metal films with different conductivities placed on a metallic substrate. The inversion determined the film conductivities which were found to agree with those measured using the direct current potential drop (DCPD) method.

Conventional eddy current measurements are performed under 10 MHz with the smallest penetration depth of 200 µm for typical 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. We have therefore built up a SHFEC measurement system that can operate up to 50MHz. Swept-frequency eddy current measurements have been applied to coating measurements\textsuperscript{10-12}, particularly for conductivity profile measurements. In our research, we swept the frequency in the range of 0.2-50MHz. The corresponding penetration depth for Waspaloy ranging approximately from 1.25mm down to 80µm over this frequency range. For the measurements in this range, there exist a number of technical challenges such as probe design, electrical

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure.png}
\caption{(a) Illustrations of simulated layer specimen configurations (not to the scale): (a) The “single-layer” configuration that consists of (a1) the top layer, (a2) the air gap and (a3) the substrate. (b) The “double-layer” configuration that consists of the two metal layers [(b1), (b3)], the two air gaps [(b2), (b4)] and the substrate [(b5)].}
\end{figure}
instruments selection, and the distributed stray elements. Below, we present solutions to these problems, based on using printed circuit board (PCB) probe fabrication and laboratory-grade instrumentation, and by the use of impedance ratios where the parasitic effects can be cancelled out. For calculating multi-layer eddy current signals, we used the Cheng-Dodd-Deeds model\textsuperscript{13}, which allows us to calculate the impedance of a coil placed on a multi-layer specimen, as illustrated in the Figure 2.1 (a). The model is the extension of the well-known Dodd-Deeds model, corresponding to the case where there is a single layer [Figure 2.1(b)].

![Illustration of the EC probes fabricated on a PCB.](image)

\textbf{Figure 2.2} Illustration of the EC probes fabricated on a PCB.

\subsection{2.2 Experimental Setup}

The probe design is a critical step of the instrumentation development. Instead of the conventional wire-winding approach, we selected to use the PCB fabrication, so that high performance at high frequencies can be achieved (Figure 2.2). Our data indicate that the PCB coils exhibit superiority over the conventional coils, both in sensitivity and in high-frequency performance. The features include: (A) a better bridge balance owing to the precisely matching coil pair, (B) a small lift off value (~50\textmu m), and (C) short, rigid, and balanced wiring. In fact, the bridge itself is integrated with the probe coils on the same PCB, with surface-mount balancing resistors.
For probe drive and signal detection, we used laboratory-grade instrumentation. As stated above, the probe PCB actually contains a pair of solenoid coils forming an AC bridge with two resistors. The bridge is driven directly by a signal generator (Agilent 33250A), while the balanced bridge output is picked up by a differential amplifier (LeCroy DA1855A). The
differential amplifier output is amplified by a gain of 25, and is then finally detected by a high-frequency lock-in amplifier (SRS 844A). All the instruments are connected to a personal computer via GPIB for controlled data acquisition. This combination of the instruments and the PCB probe allows us to operate in the range of 0.2-50MHz.

2.3 Validation of Experimental Setup and Inversion Procedure

In order to validate the SHFEC measurements and the inversion procedure, a four-stage study on simulated layer specimens was performed. The artificially made specimens consist of alloy foils placed on metal substrate as illustrated schematically in Figure 2.1, where the foils have known thicknesses with slightly different conductivities from the substrate, in order to simulate modified surface layers of shot-peened

| TABLE 2.1. Configurations of simulated layer specimens used in the validation of the conductivity profile in forward model (Stage 1) and inversion procedure (Stage 2 to Stage 4). |
|---|---|---|---|
| **Stage** | **Top-most layer** | **2nd layer** | **Substrate** |
| 1 | 25μm Cu foil | None | Al Block |
| 2 | 103μm IN718 foil | None | IN718 Block |
| 3 | 133μm NiCr foil | None | IN718 Block |
| 4a | 133μm NiCr foil | 103μm IN718 foil | IN718 Block |
| 4b | 103μm IN718 foil | 133μm NiCr foil | IN718 Block |
components. The foil materials and thicknesses are listed in Table 2.1. The purposes of the validation procedure were to (1) calibrate and evaluate the accuracy of the SHFEC measurement system, and (2) validate the EC inversion procedure, i.e. solving conductivity profile perpendicular to a stratified alloy plate.

For a given test sample, we actually perform three sets of SHFEC measurements, in order to obtain what is termed vertical (V) component signals that can be directly compared with theoretical values in our inversion procedure. Explicitly, the experimental vertical-component signal $V_{EX}$ is defined as

$$V_{EX} = \text{Im}\left( \frac{V_T^O - V_R^O}{V_L^O - V_R^O} \right).$$

(2.1)

In Eq. (2.1), $V_R^O$ denotes the reference voltage output, namely the lock-in amplifier output when the as-polished area of the test specimen is placed on the sensing coil. Similarly, $V_L^O$
denotes the lift-off signal, namely the lock-in output voltage from the same as-polished specimen surface except with an extra lift off (29.5 μm in our measurements) caused artificially by a plastic film insertion. Finally, \( V_T^O \) is the test signal, namely the lock-in output voltage when the shot-peened sample surface is placed on the sensing coil. Figure 2.4 shows one of the several pairs of samples used in experiment. In available eddy current model we employed, impedance is the finally computed quantity while in experiment voltage signal is what we measured. If the voltage signal deviation from liftoff \( (V_L^O - V_R^O) \) and conductivity \( (V_T^O - V_R^O) \) are small enough such that the changes are linear, the ratio \( V_{EX} \) of this 2 quantities would be dimensionless. Thus this ratio can be compared to its counterpart from eddy current model. The advantage of using the V-component signal (Eq. (2.1)) is two-fold: First, the V-component by definition is free of lift-off noise because, by taking the imaginary part, we only use the signal component perpendicular to the lift-off direction. In contrast, the horizontal component (i.e. the real part) is lift-off noise prone, and thus not used in our subsequent analyses. Second, the V-components provide a means to compare experiment and theory directly without the influence of the transfer function effect. To see this explicitly, we similarly introduce the “theoretical” vertical-component signal \( V_{TH}^V \) as

\[
V_{TH}^V \equiv \text{Im} \left( \frac{Z_T - Z_R}{Z_L - Z_R} \right) \tag{2.2}
\]

where \( Z_R, Z_L, \) and \( Z_T \) are the coil impedance values for the three aforesaid configurations, i.e., the reference, lift off, and test configurations, respectively. (We call \( V_{TH}^V \) as theoretical because it can be computed by theoretical models. See Section 3.3, Chapter 3) Now, given a
small impedance deviation (e.g. $Z_T - Z_R$), the output voltage will undergo a correspondingly small deviation (e.g. $V_T^O - V_R^O$) which is linearly proportional to the impedance deviation with an instrumentation-dependent but material-independent proportionality constant (i.e. the multiplicative transfer function). It is clear that, if the instrumentation conditions are kept fixed, the transfer function cancels out in the ratios. It hence follows that $V_{ex} = V_{th}$. As a corollary, possible parasitic element effects, including the wire-to-wire capacitive couplings within the coils, are suppressed in $V_{ex}$ [Eq. (2.1)] because they are a part of the transfer function. The equality $V_{ex} = V_{th}$ plays the key role in our model-based inversion where measured and computed signals are equated. (See Section 3.3, Chapter 3) The V-component measurements were repeated for each sample at least five times to obtain the average V-components and the error bars.

For validation of the forward model (Stage 1, refer to Table 2.1), the conductivity profile was considered known; and the measured and computed V components as a function of frequency were compared. For Stages 2 through 4, which were aimed at validating the inverse model, the NiCr and IN718 foil conductivities were determined by both inversion from SHFEC and direct measurements using the direct current potential drop (DCPD) method. It should be pointed out that our EC model takes into account the air gap between two adjacent surfaces which is indeed present in the simulated layer specimens. The existence of the effective air gaps is due to the imperfect matching of the two surfaces, which is inevitable at our surface flatness tolerance. Another possibility could be due to the surface oxidation.
In applying the inversion algorithm, we typically treated the foil conductivity and the air gap thickness as fitting parameters when minimizing the difference between $V_{\text{EX}}$ and $V_{\text{TH}}$ over the entire frequency range. The initial guess for the NiCr and IN718 foil conductivities were set to their nominal bulk values, and that for the air gaps was estimated by the root-mean-square roughness of the shot peened surfaces as measured by an optical profilometer (Solarius™ Optical System). This gave typical initial values of the air gap from 10 to 20 $\mu$m. The inclusion of the air gaps affects only low-frequency predictions and its fitted values are typically of the order of 10 $\mu$m for Stages 1-4 (Table 2.2). In the inversion process, the initial value of the air gap was set as 1 $\mu$m and that of the foil conductivity was set as the bulk conductivity. The norm of the tolerance vector was $1/1000$ of the initial step vector.

Results of the four stages of the validation study are shown in Figure 2.3. In general, the computed and measured V components (with variance of $\sim10\%$) show agreement in all four stages, validating the forward model. As explained above, the experimental H component is prone to lift-off noise and could be significantly different from the theoretical value (e.g. Figure 2.3(a)). The H-component is therefore unusable in validation. The results of all the four test stages are summarized in Table 2.2, including the inverted foil conductivities where applicable. The inverted conductivities of the NiCr and IN718 foils agree with those measured by the DCPD method. The present results show that (1) our inverse procedure functions properly for our SHFEC measurement setup and the frequency range. (2) Our forward and inversion modeling procedures work adequately, with 12% error at worst in terms of fitted relative norm over the entire frequency range of 1-50 MHz.
(c) measured V-component, fitted V-component

(d) measured V-component, fitted V-component
Figure 2.5  (a) The calculated and experimental H and V components vs. frequency for Stage 1. Note the agreement in the V components and the disagreement in the H components. (b) through (e) The calculated (based on inversion of eddy current data) and experimental V components vs. frequency for Stages 2, 3, 4a, and 4b, respectively. The relative large noise between 10 MHz to 20 MHz in Figure 2.5(e) is found most likely due to the internal resonant effect of the phase lock-in amplifier.
TABLE 2.2. Summary of the results for Stages 2, 3 and 4 inversion with the following fixed input parameters: the bulk conductivity of IN718 block $\sigma_{\text{IN718}} = 1.38\%\text{IACS}$, and the measured thicknesses of the IN718 and NiCr foils are 103 $\mu$m and 133 $\mu$m, respectively. Also shown are the conductivities of the IN718 and NiCr foils measured using the DCPD method.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Stage-2</th>
<th>Stage-3</th>
<th>Stage-4a</th>
<th>Stage-4b</th>
<th>DCPD</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Inversion output parameters</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\sigma$ of IN718 foil (%IACS)</td>
<td>1.348</td>
<td>—</td>
<td>1.352</td>
<td>1.352</td>
<td>1.342</td>
</tr>
<tr>
<td>$\sigma$ of NiCr foil (%IACS)</td>
<td>—</td>
<td>1.629</td>
<td>1.595</td>
<td>1.626</td>
<td>1.562</td>
</tr>
<tr>
<td>Air gap 1 ($\mu$m)</td>
<td>15.0</td>
<td>12.9</td>
<td>10.0</td>
<td>13.2</td>
<td></td>
</tr>
<tr>
<td>Air gap 2 ($\mu$m)</td>
<td>—</td>
<td>—</td>
<td>17.0</td>
<td>13.5</td>
<td></td>
</tr>
<tr>
<td>Relative fitting error (%)</td>
<td>3.7</td>
<td>9.2</td>
<td>11.5</td>
<td>11.5</td>
<td></td>
</tr>
</tbody>
</table>

**References**


CHAPTER 3. CONDUCTIVITY PROFILE DETERMINATION
BY EDDY CURRENT FOR SHOT PEENED
SUPRALLOY SURFACE

3.1 Introduction

This chapter describes the application of the swept high-frequency eddy current methodology to determine the near-surface conductivity deviation profiles of shot-peened superalloy surfaces. A newly developed model of the piezoresistivity effect that converts the inverted conductivity profile into a residual stress profile will be described in detail in Chapter 4. A series of shot peened Inconel 718 block specimens were prepared and SHFEC measurements were performed on the samples from 0.4MHz to 50MHz. The conductivity depth profiles of the samples under various shot peening intensities were obtained by inversion based on the Cheng-Dodd-Deeds model. Several sensitivity and consistency test results are given to evaluate the reliability of the inverted conductivity profiles. The extreme near-surface regions (10-20 μm) of the shot-peened surfaces were also examined by various microstructural characterization methods. Implications of the observed shot-induced microstructural changes on SHFEC signals are discussed in Section 3.5.
3.2 Experimental Procedure

Five Inconel 718 block specimens of dimensions 50 mm × 75 mm × 12.7 mm (2” × 3” × 0.5”) were used. The top and bottom surfaces of the samples were polished to a mirror-like finish with 600 grit sandpapers. One sample was kept as-polished for use as a reference in all EC measurements. The other four samples were shot-peened at various air pressures.
Figure 3.1. (a) and (b) Secondary electron images and (c) the EDS spectra obtained from the Inconel 718 sample shot peened at 6A. The white spots (e.g. (b)) each surrounded by dark halos were identified by EDS as shot residues embedded into the sample surface. The EDS spectrum (refer to (c)) obtained from the white spot exhibits strong Zr, Si and Al peaks corroborating the shot composition of 60-70wt% ZrO₂, 28wt% SiO₂ and < 10wt% Al₂O₃.
from 29 PSI to 150 PSI (1 PSI = 6894 Pa), as summarized in Table 3.1, by the use of in-house equipment. Zirconia-based ceramic beads (Zirshot Z850®, Saint-Gobain ZirPro) of a nominal 900 μm diameter, made of a mixture of ZrO₂ (nominally 60-70wt%), SiO₂ (28-33wt%) and Al₂O₃ (<10wt%), were used. The shot-peening angle was 90°. The corresponding Almen intensities were determined by the standard Almen A-strip measurements on an Almen gage (Table 3.1). Study of the shot-peened surfaces by scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS) revealed that they were somewhat contaminated with embedded shot residues (Figure 3.1). The lateral dimensions of the contamination are nominally tens of micrometers. Possible contamination effects on EC signals are discussed in Section 3.5 below.

Swept-frequency EC measurements between 0.4 MHz and 50 MHz were performed on the samples before and after the shot peening treatment. The PCB bridge coils were placed with their faces up on an insulator plate, and driven by a function generator. The reference sample (Sample 1 in Table 3.1) with the polished surface down was kept on the reference coil. The test samples were placed similarly, with the test surface down, on the sensing coil. The bridge output is detected by a differential amplifier and a lock-in amplifier with 28 dB amplification in between.¹²

A remark is in order regarding possible bulk conductivity variations and their compensation. Before shot peening the samples, we examined the possible bulk conductivity difference between the two polished surfaces of each sample, by taking baseline eddy current measurements. The same V-component measurement procedure as described above was applied, except that, in this case, one of the polished surfaces was used for the reference and lift-off measurements, and the other was used for the test measurement. Ideally, we expect
null results, and therefore the observed non-vanishing V-component signals are attributable to the bulk conductivity deviations. Figure 3.2 shows the un-subtracted experimental V-component data up to 50 MHz with error bars. In practice, the base line signals, or in other words, the signal differences between the polished reference sample and the polished test samples before shot peening is not always zero within measured frequency. We have

Figure 3.2. The experimental V-component signals up to 50 MHz and the error bars. Experiments 1 through 4 refer to measurements from the Inconel 718 specimens shot peened at Almen intensities of 6A, 9A, 13A, and 17A, respectively.
subtracted these baseline signals from the subsequent experimental V-component data obtained after shot peening.

3.3  **Eddy Current Inversion Models**

### 3.3.1 Conductivity profile of shot-peened metal plate

In our multi-layer model, the conductivity deviation of a shot-peened surface is assumed uniform in the surface directions, and varies perpendicularly to the surface. Among various conceivable forms of profile functions, we have chosen an empirical conductivity depth profile function that is a product of an exponentially decaying function and a polynomial function. The exponentially decaying function captures the overall conductivity behavior that can deviate significantly near the surface but rolls back smoothly into the bulk value with increasing depth as the shot-induced residual stress and the resulting conductivity deviation diminish. The polynomial function captures the fine structure of the profile around the exponential function. All the practically conceivable conductivity profile forms arising from shot peening can be represented by this parameterization. Explicitly, our conductivity deviation profile takes the parametric form,

$$\frac{\sigma - \sigma_0}{\sigma_0} = e^{-x/\lambda} \sum_{i=0}^{N} a_i x^i, \quad (3.1)$$

where $x$ is the depth below surface, $\sigma_0$ is the bulk conductivity while $\lambda$ and $a_i (i = 0$ to $N$) are fitting parameters. In the actual computation, this continuous conductivity profile is discretized into laterally uniform discrete multi-layers for use in the multi-layer Cheng-
Dodd-Deeds model. The thickness of each discretized layer is chosen adaptively according to the area of interest. In the fitting process, it was found that six parameters (i.e. $N = 4$ in Eq. (3.1)) are enough for the model accuracy. Section 3.4 has more discussion about this optimal choice for striking a balance between the model accuracy and computational efficiency.

![Figure 3.3](image.png)

Figure 3.3. Illustration diagram of a solenoid coil placed above a laterally uniform multi-layered half space metallic alloy.

### 3.3.2 Eddy current model of multi-layered half space

For the forward model calculation, we use the multi-layer model of Cheng, Dodd and Deeds. For a coil placed on a multi-layer specimen as illustrated in Figure 3.3, the impedance of the coil is given by
\[ Z = j \omega K \int_0^\infty \frac{I^2(\alpha, r_1, r_2)}{\alpha^3 \alpha_0^3} \left[ 2(e^{-\alpha_0 L} + \alpha_0 L - 1) + A(\alpha_0) \frac{V_1(n+1,1)}{V_2(n+1,1)} \right] d\alpha. \quad (3.2) \]

Here, the prefactor \( K \) is given by

\[ K = \frac{\pi \mu_0 n_t^2}{L^2 (r_2 - r_1)^2} \quad (3.3) \]

and

\[ \alpha_0 \equiv \sqrt{\alpha^2 - \omega^2 \mu_0 \varepsilon_0} \quad (3.4) \]

defined in terms of the angular frequency \( \omega \), the vacuum permeability \( \mu_0 \), and the vacuum permittivity \( \varepsilon_0 \), while \( n_t, L, r_1 \), and \( r_2 \) are the coil parameters, i.e. the number of turns, the height, the inner and outer radii, respectively. The radial dimensions of the coil are used in the prefactor \( K \) and in the arguments of the function \( I \) which is defined as

\[
I(\alpha, r_1, r_2) = \int_{\alpha r_1}^{\alpha r_2} x J_1(x) dx = \frac{1}{2} \pi \alpha \left[ J_1(x) S_0(x) - J_0(x) S_1(x) \right]_{x=\alpha r_1}^{x=\alpha r_2} \quad (3.5)
\]

where \( J_0 \) and \( J_1 \) are the zero-th and first order Bessel functions, and where \( S_0 \) and \( S_1 \) are the zero-th and first order Struve functions, respectively. The effects of the lift off and length of the coil are expressed primarily through the function \( A \), which is given by

\[ A(\alpha_0) = e^{-2\alpha_0 h_2} + e^{-2\alpha_0 h_1} - 2e^{-2\alpha_0 (h_1 + h_2)} \quad (3.6) \]
where \( h_1, h_2 \) is the height of the lower and upper surfaces of the coil, respectively. The properties of the sample are incorporated into the factor \( \frac{V_1(n+1,1)}{V_2(n+1,1)} \), where \( n \) is the number of layers as shown in Figure 3.3. \( V_1(n+1,1) \) and \( V_2(n+1,1) \) can be recursively computed from the following matrix equations:

\[
\begin{bmatrix}
V_1(n+1,1) \\
V_2(n+1,1)
\end{bmatrix} = \begin{bmatrix}
T_{11}(n+1,n) & T_{12}(n+1,n) \\
T_{21}(n+1,n) & T_{22}(n+1,n)
\end{bmatrix} \begin{bmatrix}
V_1(n,1) \\
V_2(n,1)
\end{bmatrix}
\]

(3.7)

where the lowest-order factors are

\[
\begin{bmatrix}
V_1(2,1) \\
V_2(2,1)
\end{bmatrix} = \frac{1}{\beta_2} \begin{bmatrix}
\beta_2 - \beta_1 \\
\beta_2 + \beta_1
\end{bmatrix}.
\]

(3.8)

The transfer matrix is

\[
T_{i,j}(n+1,n) = [1 + (-1)^{i+j} (\beta_n / \beta_{n+1})] \exp[(-1)^i \alpha_n d_n], \quad i, j = 1, 2
\]

(3.9)

where \( \alpha_n = \sqrt{\omega^2 + j \omega \mu_n \sigma_n - \omega^2 \mu_n \varepsilon_n}, \quad \beta_n = \alpha_n / \mu_n \)

(3.10)

with \( \mu_n, \sigma_n, \varepsilon_n \) and \( d_n \) denoting the \( n \)-th layer permeability, electrical conductivity, permittivity and thickness, respectively.

It is convenient to compute the impedance relative to the substrate impedance \( Z_{hsp} \) where the self-inductance term cancels out. The impedance difference reads
Two special cases are needed for computing the V-component signals via Eq. (2.2) (see Section 2.3). One is the impedance change for a given conductivity deviation profile resulting from shot peening, expressed as

\[
\Delta Z = Z - Z_{\text{imp}} = j\omega K \int_0^\infty \frac{I^2(\alpha, r_1, r_2)}{\alpha^3} \frac{A(\alpha_0)}{\alpha_0^3} \left[ \left( \frac{V_1(n+1,1)}{V_2(n+1,1)} \right) - \left( \frac{V_1(2,1)}{V_2(2,1)} \right) \right] d\alpha
\]  

(3.11)

where the primed terms involve conductivity deviations. We also need the impedance formula for calculating the lift off effect, which reads

\[
\Delta Z_\sigma = Z' - Z = j\omega K \int_0^\infty \frac{I^2(\alpha, r_1, r_2)}{\alpha^3} \frac{A(\alpha_0)}{\alpha_0^3} \left[ \left( \frac{V_1'(n+1,1)}{V_2'(n+1,1)} \right) - \left( \frac{V_1(n+1,1)}{V_2(n+1,1)} \right) \right] d\alpha
\]  

(3.12)
\[ \Delta Z_i = Z_i - Z = j \omega K \int_0^\infty \frac{I^2(\alpha, r_1, r_2)}{\alpha^3} \frac{A_i(\alpha_0) - A(\alpha_0)}{\alpha_0^3} \frac{V_i(n+1,1)}{V(n+1,1)} d\alpha \] (3.13)

where the subscript “l” denotes the lift off. The coil parameters are given in Table 3.2.

### 3.3.3 Inversion algorithm

We have developed a software code to perform the conductivity profile inversion from the swept frequency EC data. We chose to use six fitting parameters \((\lambda, a_i, \text{ where } i = 0 \text{ to } 4)\) in the profile parameterization [Eq. (3.1)]. Dependence of the inverted conductivity profile on the number of fitting parameters is discussed in Section 3.4. When applying the multi-layer model, the continuous conductivity profile was discretized into 31 discrete layers with adapted thickness for each layer. The coil impedance deviations were calculated from this discretized profile by Eq. (3.12). Similarly, the lift off effect was calculated by Eq. (3.13). The computed impedance values have been inserted into Eq. (2.1’) to yield the theoretical V-component signals that can be compared to the experimental V-component signals [Eq. (2.1)]. Hooke and Jeeves’s \(^{14-16}\) search algorithm was employed to obtain the best fit parameters by minimizing the RMS difference between the measured and calculated V-component signals. This algorithm is frequently used in many optimization problems,\(^ {16}\) and indeed it consistently exhibits the most efficient convergence for our inversion problem, among several direct search optimization algorithms we tried. Technically, it is based on the principle of a guided walk through an \(n\)-dimensional parameter space, \(n\) being the number of unknown parameters (e.g. those in Eq. (3.1)), to search for the minimum RMS value. Each i-
th iteration starts with the i-th update of the \( n \) parameters representing a point in the parameter space. Around this point in the same space, we generate additional \( 2n \) points by moving the point along each of the \( n \) axes in either the positive or negative direction, with adapted step sizes. Correspondingly, we obtain the total of the \( (2n+1) \) parameter sets, for each of which we evaluate the forward model and compare the output with the experimental data, to find the minimal root-mean-square difference between the experimental and calculated values. If the minimal one is the start point itself, then the search process is repeated with decreased step sizes until a new minimum is found. Given the new minimal parameter set, we then determine the \( (i+1) \)-th update of the unknown parameters such that the newly found minimum point coincides with the average point between the i-th and \( (i+1) \)-th updates. The iteration process stops when all the \( (2n+1) \) parameters sets converge within the preset tolerance. The final figure of merit of the searched conductivity profile is determined by re-calculating the theoretical V-component signals from the fitted conductivity profile, and by comparing them directly with the experimental V-component signals.
Figure 3.4. The experimental V-component signals up to within 20 MHz and the corresponding computed signals obtained from the inverted parameterized conductivity depth profiles (Eq. 3.1) with 31 layers.

3.4 Inverted Conductivity Profiles and their Sensitivity Studies

Figure 3.4 shows the experimental and V-component signals based on the inversion procedure for the four shot-peened samples at Almen intensities of 6A, 9A, 13A, and 17A, plotted against the frequency. The inverted V-components fall within the experimental errors for all the samples. Figure 3.5 shows the corresponding inverted conductivity profiles for the same shot-peened samples, as function of the depth. The plots show that shot peening
affected the finite surface layer (up to 350 - 400 μm deep), beyond which the conductivity is equal to the nominal bulk value. The conductivity values increase gradually for decreasing depth, peaking at a maximum of 3% increase from the bulk value. The peak positions range from around 30 to 60 μm below the surface. Both the peak location and intensity increase systematically, as the Almen intensity increases. The behaviors around the peaks are consistent with those arising from the expected residual stress profiles via the piezoresistivity effect. In contrast, the layer conductivity decreases within about 10 to 20 μm from the sample surface, and becomes lower than the bulk value at the surface. The near-surface low

Figure 3.5. Inverted conductivity deviation profiles as function of depth from the surface. They are parameterized via Eq. 3.1, discretized into 31 layers, and then determined by the EC inversion from the swept frequency V-component signals for each of the shot-peened samples.
conductivities indicate contributions from other material deviations competing with the effect of compressive residual stress.

Various consistency and sensitivity tests were conducted to support our results on conductivity deviation profile inversion. For instance, the positive values of the V-components in Figure 3.4 show that the affected surface layer becomes more conductive than the bulk after shot peening. This trend is consistent not only with the previous observations,\textsuperscript{8-10} but also with independent measurements taken with an EC instrument (Nortec NDT-19) and a 2 MHz (corresponding to a skin depth of about 400 $\mu$m for Inconel

\[
\frac{\sigma - \sigma_0}{\sigma_0} = e^{-x/\lambda} \left( a_0 + a_1 x + a_2 x^2 + a_3 x^3 + \ldots \right)
\]

Fitting variables: $\lambda$ and $a_i$

\textbf{Figure 3.6.} Convergence test of the inverted relative conductivity profiles against the number of fitting parameters.
EC probe, which repeatedly showed a higher conductivity on the shot-peened side than on the polished side.

Dependency of the inverted relative conductivity profiles on the number of the fitting parameters of the assumed conductivity derivation profile has been tested. The inverted profiles were calculated for three to six parameters. The specimen 17A profiles, for example, are plotted in Figure 3.6, to show the degree of their variation. From these tests, we have

![Figure 3.7](image.png)

**Figure 3.7.** The inverted and modified relative conductivity depth profiles, used to demonstrate the strong sensitivity of the calculated V-component signals against small conductivity profile deviations in the near-surface region (see Figure 3.8 below).
concluded that the inverted conductivity profile becomes stable after the number of unknowns reaches 5.

To test the sensitivity of the inversion results to the low surface conductivity at depths below 15 μm as indicated in Figure 3.5, we hypothesized another conductivity profile as shown in Figure 3.7, which is identical to the inverted profile of Figure 3.5 almost everywhere except for the near-surface layer, so that the conductivity remains larger than the bulk value in that range. We then calculated the corresponding V-component signals from the hypothesized conductivity profile. The resulting V-component signals, as well as the experimental and inverted V-components are shown in Figure 3.8.

**Figure 3.8.** The V-component signals computed from the modified profile (Fig. 7), in comparison with the experimental and inverted V-components. The large deviations outside the error bars show that the candidate profile shown in Figure 3.7 could not account for the experimental V component.
Figure 3.9. Insensitivity of the conductivity profile inversion to built-in lift off variation. Perturbing the built-in lift off values between 60 μm and 150 μm affects the maximum variation of the inverted relative conductivity only by $10^{-4}$ or less, demonstrating that the built-in lift off noise is highly suppressed in our inverse procedure.

Experimental and the best-fit V-component signals from the inversion, are plotted in Figure 3.8. It can be seen that, unlike the inverted signals, the modified V-component output from the hypothesized conductivity profile falls outside the experimental error bars (of one
standard derivation). This test shows that no experimental artifact is likely to explain the near-surface low conductivities found by the inversion.

Since the lift-off-noise-free V-components are employed, the profile inversion result is insensitive to the built-in lift off, which, in our setup, arises from the recess fabricated to hold the PCB coil on the plastic plate. The nominal, built-in coil lift-off is measured to be 100 μm. For testing the sensitivity, we intentionally perturbed the built-in lift off values from 60 μm to 150 μm in 10 μm steps in our fitting calculations, with all the other input parameters being fixed. Figure 3.9 shows the maximum variations of the inverted conductivity profiles against this artificial perturbation. It can be seen that the maximum change of the inverted conductivity is of the order of 10^{-4} of the bulk conductivity, being negligible compared to the shot-peening effect.

However, the EC inversion result is very sensitive to the value of the additional lift off that we use when measuring or calculating the denominator of Eq. (2.1) or Eq. (2.1'), respectively. Experimentally, we inserted a 25 μm thick plastic film between the test sample and coil, and then measured the actual vertical rise of the coil. The measured additional lift-off value is 29.5 μm in average in our measurements. (The difference of 4.5 μm is consistent with the measured values of sample surface roughness of a few micrometers.) For testing the sensitivity of the EC inversion results, we again perturbed the additional lift-off value around the measured values of 29.5 μm to 24 μm and 34 μm in the fitting calculations. Figure 3.10 shows the resulting sensitivity against the perturbation. Due to this somewhat strong sensitivity, it is important to measure the additional lift-off value explicitly and accurately, as we practiced.
The surface roughness effect was also estimated and proven unimportant for the PCB coil used in this work. Here, our analysis is based on another effective layer model, where the rough surface is effectively replaced by a thin layer of uniform thickness and modified conductivity. To verify the uniformity of the effective layer in the lateral directions, we first measured the shot-peened surface morphology by a laser profilometer. Figure 3.11 shows the surface profile of a 2mm × 2mm area for the shot peened Sample 5 (17A) before and after shot peening. The various roughness parameters were obtained and included in Table 3.1.

Figure 3.10. Sensitivity of the inverted relative conductivity to the additional lift off value, as demonstrated by the variations among those computed from the inverted relative conductivity with 3 different additional lift off values. The actual additional lift off used in the measurements is 29.5 μm.
Figure 3.11. Surface morphology profiles of Sample 5 in a 2mm-by-2mm area (a) before and (b) after shot peening as measured by a laser profilometer.
The crater-like dents caused by the shots are visible in the image, and it is likely that they are the dominant microstructural sources of the roughness signals. Approximately 20 of the dents are counted in the image area (4 mm$^2$), and therefore the PCB coil area (310 mm$^2$) correspondingly contains a statistically significant number (approximately 1500) of the dents. Since the probe field averages over that many micro-defects, and because the peening treatments are performed sufficiently uniformly (as evidenced, e.g. by the images in Figs. 3.1 and 3.11), it is reasonable to assume that the effective roughness layer behaves as a uniform layer, just as our effective shot-peened layer does. We next estimated the thickness of the effective layer. Figure 3.12 shows the arithmetic and quadratic surface roughness values of the samples as a function of the Almen intensity. For example, the 17A sample has the quadratic mean roughness of about 2.4 μm. Given the effective roughness layer, we can estimate the bounds of the V-component roughness signals from the two extreme cases, one being when the 2.4 μm layer is entirely empty, and the other case is when the 2.4 μm layer is fully occupied by the bulk material. The resulting roughness signal bounds are plotted in Figure 3.13 in terms of the maximum relative V-component error $ΔV/V$ as a function of frequency. We thus estimated that, even for the sample of the highest peen intensity, the maximum roughness effect on the relative V-component does not exceed 2.5% for frequencies up to 20 MHz, which are small compared to typical experimental error bars of ~10%.
Figure 3.12. Arithmetic (Sa) and quadratic (Sq) surface roughness values of the samples at several different Almen intensities, as measured by a laser profilometer.

Figure 3.13. Estimated upper bounds of the surface roughness effect onto the V-component signal as a function of frequency.
3.5 Near-Surface Residual Stress and Inverted Conductivity Profile

The shot peening process increases near-surface material irregularities (both atomistic-scale dislocations and meso-scale grain boundary formations) in addition to generating the residual stress. Irrespective of their orientations, the shot-induced material irregularities always increase the conduction electron scattering and thus the resistivity.\textsuperscript{17,18}

\textbf{Figure 3.14.} Rietveld refinement of X-ray diffraction data of Sample 5 before (top) and after (bottom) shot peening.
Therefore, the observed conductivity increase after shot peening is likely attributable to the residual stress via the piezoresistivity effect. However, we have found, by inversion, evidences for near-surface conductivity reductions for depth below 16 μm. Microstructural characterization on the shot peened surface by using several available methods was therefore performed in order to investigate the possible physical mechanisms which relate the conductivity and residual stress profiles. Figure 3.14 shows the Rietveld refinement of the X-

<table>
<thead>
<tr>
<th>Lattice Constant</th>
<th>Space group</th>
<th>Before shot peening</th>
<th>After shot peening</th>
<th>ZrO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fm3m</td>
<td>Fm3m</td>
<td>P21/C</td>
<td></td>
</tr>
<tr>
<td>a (Å)</td>
<td>3.60811 (17)</td>
<td>3.61541 (38)</td>
<td>5.15186 (198)</td>
<td></td>
</tr>
<tr>
<td>b (Å)</td>
<td>3.60811 (17)</td>
<td>3.61541 (38)</td>
<td>5.20769 (205)</td>
<td></td>
</tr>
<tr>
<td>c (Å)</td>
<td>3.60811 (17)</td>
<td>3.61541 (38)</td>
<td>5.30984 (199)</td>
<td></td>
</tr>
<tr>
<td>α (deg)</td>
<td>90</td>
<td>90</td>
<td>90</td>
<td></td>
</tr>
<tr>
<td>β (deg)</td>
<td>90</td>
<td>90</td>
<td>99.0210 (274)</td>
<td></td>
</tr>
<tr>
<td>γ (deg)</td>
<td>90</td>
<td>90</td>
<td>90</td>
<td></td>
</tr>
<tr>
<td>March preferred</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>orientation [hkl]</td>
<td>011</td>
<td>111</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>March preferred</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>orientation value</td>
<td>0.65786 (223)</td>
<td>0.82701 (269)</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Rp (%)</td>
<td>10.99</td>
<td>10.15</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rwp (%)</td>
<td>14.60</td>
<td>13.25</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Table 3.3.** Rietveld refinement of X-ray diffraction data of IN718 Sample 5.
ray diffraction data ($\theta$-2$\theta$ scans) taken from Sample 5 (17A) before and after shot peening. The quantitative refinement results are tabulated in Table 3.3. Besides confirming the SEM and EDS indications of the shot material (ZrO$_2$) contamination, the Rietveld refinement shows no new alloy phase generation by the shot peening process within the XRD detection limit. However, it reveals an important fact, i.e. an anisotropic material response to shot peening. Specifically, the fitted March preferred orientation $^{19}$ is 0.66 for the peak index [011] and 0.83 for the index [111]. The preferred orientation before shot peening is presumably a remnant of the forging process of the sample. The preferred orientation of the {111} plane on the surface after shot peening can be quantified by the intensity of the [111] peak relative to the other peaks. This may be important experimental evidence for explaining the piezoresistivity effect that results in the increased conductivity.

Let us estimate the near-surface residual stress from this XRD measurement. First, notice that the lattice constant changes from the Rietveld refinement yield the averaged strain $\varepsilon_3$ perpendicular to the surface via $\varepsilon_3 = (d - d_0)/d_0$ where $d_0$ and $d$ are the averaged plane separation before and after shot peening. This is because each peak position corresponds to the plane separation of \{hkl\} that is parallel to the surface.$^3$ Second, let us assume that the shot peened surface is in a plane stress state where the two principal stresses, $\sigma_1$ and $\sigma_2$, are equal (i.e. $\sigma_1 = \sigma_2 = \sigma$). Then, there is the standard relationship between $\varepsilon_3$ and the stress values in the two in-plane directions,

$$\varepsilon_3 = -\frac{\nu}{E}(\sigma_1 + \sigma_2) = -\frac{2\nu}{E}\sigma.$$ (3.14)
Substituting the averaged plane separations from Table 3.3, and using the nominal Young’s modulus \((E = 211 \text{ GPa})\) and Poisson’s ratio \((\nu = 0.29)\), we find that \(\sigma = -736 \text{ MPa}\) on the surface.

This surface stress estimate is consistent with the expectation that shot peening will create compressive stress parallel to the surface. It shows, however, that a simple scalar piezoresistivity coefficient does not consistently explain the near-surface negative conductivity changes obtained by the inversion (Figure 3.5). This apparent discrepancy indicates that other microstructural material deviations, in addition to the scalar piezoresistivity effect, are contributing to the conductivity deviation, particularly in the extreme vicinity of the surface. The possible physical mechanisms under considerations include 1) anisotropy of the piezoresistivity coefficients suggested by the XRD measurements, and 2) shot-induced near-surface material irregularities (i.e. generalized roughness that may consist of sharp penetrating microcracking and/or grain boundary irregularities) acting as additional electron scatterers causing higher resistivity. In Chapter 4, we will present further experimental evidence of shot-induced texture, and a new model that incorporates the texture effects on the piezoresistivity coefficients and offers a possible physical mechanism to convert the inverted conductivity profile to the residual stress whereas the use a simple scalar piezoresistivity coefficient fails to do so.

### 3.6 Conclusions

In conclusion, we have demonstrated a swept-frequency eddy current methodology that can determine near-surface conductivity deviation profiles of shot-peened superalloy
surfaces. A series of shot peened Inconel 718 block specimens have been prepared and examined by the proposed EC technique. The conductivity profiles of the samples under various shot peening intensities have been obtained by model-based inversion of the swept high frequency EC measurement data, as given in Figures 3.4 and 3.5 in Section 3.4. Several sensitivity and consistency tests of our experimental and inversion procedures were conducted, and the results are presented in Section 3.4 (Figures 3.7 to 3.10) to support the reliability of the inverted conductivity profile results. We also examined the extreme near-surface regions (10-20 μm) of the shot-peened surfaces, by using various microstructural characterization methods such as SEM, EDS, laser profilometry, and X-ray diffraction (Figures 3.1, 3.11, 3.12, and 3.14). These microstructural analysis results not only verify the quality of our in-house shot-peening process, but also give insight to the microstructural responses of the shot peened materials, for example, to the possible anisotropy of the piezoresistivity effect (Section 3.5).

Our ultimate goal is to determine the residual stress profile, and the conductivity profile results presented here provide the key input to the final stress profile determination. Our data nevertheless show that the piezoresistivity effect alone is insufficient to convert the inverted conductivity profile into the residual stress profile if we applying the experimental piezo-resistivity constants in reference 8. Indeed, we have shown evidences indicating that there are competing processes, other than the residual stress, which contribute to the conductivity deviations, particularly at extreme near-surface regions. It is microstructural material models that can reconcile the nontrivial relationship between the conductivity and stress profiles. In the next Chapter we present a modified piezoresistivity model that includes the shot-induced texture effect for residual-stress profile determination.
References


CHAPTER 4. RESIDUAL STRESS PROFILE ASSESSMENT BY EDDY CURRENT FOR SHOT PEENED NICKEL SUPERALLOY

4.1 Introduction

In this chapter, we report a study of the connection among electrical conductivity, residual stress and texture by developing a macroscopic piezo-resistivity theory for polycrystalline materials with texture. The theory was applied to analyze the swept high frequency eddy current data obtained from a shot peened Inconel 718 sample, which was found to exhibit shot-induced texture in the near surface region using XRD and orientation imaging microscopy (OIM). The residual stress profile of the peened sample was inverted from eddy current data, and was found to agree with independent experimental residual stress profiles measured using the standard layer removal XRD technique (the $\sin^2 \psi$ method).

4.2 Experimental Details
Sample 5, which was shot peened to an Almen intensity of 17A (Table 3.1, Section 3.2 of Chapter 3), was used in this work. Depth profile $\theta$-2 $\theta$ XRD study was carried out on a small coupon of dimensions 10 mm $\times$ 10 mm $\times$ 3.1 mm cut from the specimen, with one of the 10 mm $\times$ 10 mm surfaces being shot peened. XRD $\theta$-2$\theta$ scan was conducted on the peened coupon surface for $\theta$ from 30$^\circ$ to 140$^\circ$ with a step of 0.02$^\circ$ using a Cu $K_{\alpha}$ source. The integration time of each step is 2 seconds. XRD scans were repeated as we manually removed the surface layer by layer. The small coupon was gently polished using sand paper with grits from 400 to 1000 until desired depth. The thicknesses of the removed layers are listed in Table 4.1, showing 10 consecutive depth values at which XRD scans were performed.

Residual stress depth profile of the shot peened sample was also measured using a two-angle $\sin^2 \psi$ technique (Lambda Research, Inc$^{17}$, in accordance with SAE HS-784), employing the diffraction of Mn $K_{\alpha}$ radiation from the (311) planes of the fcc structure of the Inconel 718. The depth profiles of both the residual stress and cold-work (the half-width of XRD pole figures) are presented in Table 4.1.

**Table 4.1** Texture profile measured from partial X-ray diffraction polar figure of shot peened Inconel 718 by layer removal.

<table>
<thead>
<tr>
<th>depth ($\mu m$)</th>
<th>0</th>
<th>30</th>
<th>44</th>
<th>56</th>
<th>74</th>
<th>96</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R_2 = I_{[111]} / I_{[022]}$*</td>
<td>3.38466</td>
<td>2.14417</td>
<td>1.41563</td>
<td>1.08556</td>
<td>0.42427</td>
<td>0.45066</td>
</tr>
<tr>
<td>$W_{400}$</td>
<td>-0.00859</td>
<td>-0.00573</td>
<td>-0.00270</td>
<td>-0.00064</td>
<td>0.00627</td>
<td>0.00588</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>depth ($\mu m$)</th>
<th>129</th>
<th>175</th>
<th>230</th>
<th>288</th>
<th>355</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R_2 = I_{[111]} / I_{[022]}$</td>
<td>0.50457</td>
<td>0.79427</td>
<td>0.83906</td>
<td>0.88434</td>
<td>0.92128</td>
</tr>
<tr>
<td>$W_{400}$</td>
<td>0.00512</td>
<td>0.00179</td>
<td>0.00137</td>
<td>0.00096</td>
<td>0.00064</td>
</tr>
</tbody>
</table>
of the (311) diffraction peak) were measured to a depth of 399 μm.

### 4.3 Evidence of Shot-induced Texture in IN718

Results of the XRD θ-2θ scans show that the intensity ratio of [111] reflection peak to [022] peak has the largest change with depth among all peak ratios. Figure 4.1 shows the XRD θ-2θ pattern for these two peaks for a shot peened IN718 plate, at the peened surface (i.e. depth of removed layer = 0) and after a 96 μm thick surface layer was removed. It can be

![XRD pattern](image)

**Figure 4.1** XRD pattern of shot peened Inconel 718 at the surface and after a 96 μm thick surface layer was removed.
seen that, at the depth of 96 μm, the [111] peak is suppressed while the [022] peak becomes stronger compared to the peened surface. This shows a change in preferred grain orientation, namely a difference in texture, between these two depth points. The intensity and peak position of every peak of the spectra were determined by fitting the individual peak profile using Rietica\textsuperscript{16} and the final values were taken for $K_{\alpha_1}$ reflection. As a measure of the texture profile, the intensity ratio of peak [111] and [022] is plotted as a function of depth in Figure 4.2. Within about the first 60 μm, the XRD data show a preferred [111] orientation over [022] or [011] orientation as the main phase of Inconel 718 is face centered crystallite (fcc). Below that depth, [011] orientation gradually dominates over [111] orientation and reaches the maximum value at about 75 micron. The texture gradually diminishes beyond

![ratio of intensity [111]/[022]](image)

**Figure 4.2** Depth profile of the ratio of the fitted integrated Bragg intensities of [111] peak over [022] peak.
that maximum point, as suggested by the intensity ratio which approaches an asymptotical value of 1.58 corresponding to the value obtained before shot-peening.

The observed texture profile can be explained schematically as shown in Figure 4.3, by considering geometrically necessary dislocations resulting from shot peening. Suppose that a shot hits the surface into the maximum depth. To geometrically adapt the shape of the surface, material near the bottom of the shot needs to be pushed down. This process is most likely realized by movement of edge dislocations from the surface into the bulk. Since the major phase (gamma phase) of Inconel 718 is fcc, the easiest sliding plane is [111] and sometimes [211] while the easiest sliding direction is [011]. For those grains without perfect orientation with sliding plane and sliding direction parallel to the shear stress, the grains are subject to rotation. It is this kind of rotation below the bottom of the single shot that leads to preferred [111] and [211] zone axis perpendicular to the peened surface and \{011\} plane parallel to the peened surface. It is notable that the maximum grain rotation may happen not exactly at the contact point of the shot bottom, but slightly below the lowest contact point.

![Figure 4.3](image-url)  

**Figure 4.3**  Schematic diagram of the mass flow due to shot peening and the corresponding geometrically necessary dislocations.
This can be understood from the approximated Hertzian contact theory where both the shot and the sample are pure elastic and the stress is maximum at a depth about a few times of the shot radius below the lowest contact point\textsuperscript{18}.

Plastic deformation of the materials surrounding the shot impact involves mass flow parallel to the surface (Figure 4.3). After many shots hitting the surface, the average mass flow at both sides of every shot is parallel to the surface and mass flow perpendicular to the surface is negligible. Thus the mass flow at the near surface region is different from the mass

\[\text{(a)}\]

\[\text{(b)}\]

\textbf{Figure 4.4} (a) Schematic diagram showing the coordinate rotation from the imaged cross-section to the peened surface of the Inconel 718 sample. (b) Inverse polar figure of grains from OIM measurements after rotation of coordinates.
flow in the deeper region. It follows that there exists a certain depth below the peened surface where the texture changes. Below that depth, the texture is expected to be dominated by [111] and [211] zone axis perpendicular to the material surface and {011} plane parallel to the surface, while above that depth the texture changes to {111} and {211} plane parallel to and [011] zone axis perpendicular to the surface. This texture change is manifested in the change in XRD [111]-to-[022] peak intensity ratio from a higher value in the peened surface and a lower ratio below the lowest shot contact point compared to an unpeened fcc material surface.

In order to verify the XRD results and the analysis above, orientation image microscopy (OIM) texture measurement was independently conducted. Another sample was cut from the shot peened specimen at the 17A Almen intensity. This sample was polished and etched to reveal the microstructure by OIM measurement. One of the surfaces perpendicular to the shot peened surface is hereby named the cross-section to be examined. For the purpose of comparing these results to XRD data, we need to change the view direction of the grain inverse pole figure from the normal of the cross-section to the normal of the peened surface. Figure 4.4 (a) to show schematically the rotation of the coordinates by 90 degree anticlockwise about the y axis. This leads to the Euler’s angles in Bunge’s notation from $(\phi, \theta, \psi)$ uniquely to $(\phi', \theta', \psi')$ with the following relationship:

\[
\begin{align*}
\cos \theta' &= \cos \phi \sin \theta \\
\sin \phi' &= \sin(\phi) \sin(\theta) / \sin(\theta') \\
\cos \phi' &= -\cos \theta / \sin \theta' \\
\sin \psi' &= (-\cos \phi \cos \theta \sin \psi - \sin \phi \cos \psi) / \sin \theta' \\
\cos \psi' &= -(\cos \phi \cos \theta \cos \psi - \sin \phi \sin \psi) / \sin \theta'
\end{align*}
\]

(4.1)
We modified the Euler’s angles of every scanned point according to Eq. (4.1) to obtain the OIM image of the cross-section viewed from the peened surface normal as shown in Figure 4.4 (b). The dark points in Figure 4.4 (b) that are not represented in the scale bar mean signal with confidence index (or image quality) below the acceptable level and were not used in the

Figure 4.5 (a) Inverse polar figure of the first 50 μm thick peened surface layer of Fig. 4.4 (b). (b) Inverse polar figure of the region 60 μm yo 150 μm below the peened surface.
texture analyses. The typical grain diameters are from 10 μm to 80 μm. The transition of the color inside some big grains means distortion after rotation of the grains due to shot peening.

Figure 4.5 (a) shows the inverse pole figure of the first 50 μm of the peened surface shown in Figure 4.4(b). This area corresponds to the horizontal mass flow area in Figure 4.3 and the (111) planes dominate over (011) planes in Figure 4.2. The inverse pole figure in Figure 4.5(a) also shows dominance of [211] and [111] over [011], with the highest intensity close to [211] direction (the Miller direction index represents the plane with the same index since IN718 has a fcc structure). This result confirms the XRD results of the texture within the area. Figure 4.5 (b) shows the inverse pole figure of the region from 60 to 150 μm below the peened surface. This area corresponds to the vertical mass flow area in Figure 4.3 and the (011) planes dominate over (111) planes as suggested by the XRD results in Figure 4.2. Dominance of [011] over [111] and [211] can be seen in Figure 4.5(b). Again, the OIM result confirms the XRD results of the texture within this area.

4.4 Piezoresistivity Theory and Orientation Distribution Coefficients (ODCs) Profile of Shot-peened Inconel 718 plate

For crystallite metal with cubic symmetry, the conductivity is isotropic in space. If the crystallite is under stress, the cubic symmetry will be perturbed and the conductivity is no longer isotropic. The first order approximation of this perturbation of the resistivity or conductivity due to stress is called the linear piezo-resistivity effect. For cubic crystallites with random texture under isotropic (hydrostatic) stress, the macro-conductivity is still isotropic due to the non-texture status. However, for polycrystalline with texture and under
any kind of stress, the conductivity space distribution is no longer the same as that with random texture.

For shot peened Inconel 718 with fcc crystal symmetry and cylindrical sample symmetry along the normal to the peened surface, we derived a texture-influenced piezoresistivity relation [see also Eq. (A29) of APPENDIX A] which includes the effect of texture on the relationship between the relative conductivity change and the residual stress, namely,

$$\frac{\Delta \sigma}{\sigma_0} = -(\pi_{11}' + \pi_{12}') \cdot \tau = (\pi_{11}' + \pi_{12}')_{\text{no texture}} \cdot \left[ 1 - \frac{48\sqrt{2}\pi^2}{77} \cdot p \cdot W_{400} \right] \cdot (-\tau)$$  \hspace{1cm} (4.2)

where $\Delta \sigma$ is the absolutely conductivity change, $\sigma_0$ is the bulk conductivity, $\pi_{11}'$ and $\pi_{12}'$ are the polycrystalline piezoresistivity constants, $W_{400}$ is the orientation distribution coefficients, $\tau$ is the plane residual stress (in MPa), and

$$p \equiv \frac{\frac{1}{4} (\pi_{11} - \pi_{12} - \pi_{44})}{\frac{1}{11} (4\pi_{11} + 6\pi_{12} + \pi_{44})}$$  \hspace{1cm} (4.3)

is the ratio of two normalized combinations of the piezo-resistivity constants $\pi_{11}$, $\pi_{12}$ and $\pi_{44}$ of single crystal. Blodgett and Nagy reported the following polycrystalline piezoresistivity constants for Inconel 718 alloy (assumed to have no texture) \cite{8}

$$\pi_{11}'_{\text{no texture}} = 4.0870 \times 10^{-12} \text{ Pa}^{-1}$$

$$\pi_{12}'_{\text{no texture}} = 3.1739 \times 10^{-12} \text{ Pa}^{-1}$$  \hspace{1cm} (4.4)

Substituting these isotropic macroscopic piezo-resistivity constants into equation (4.2) leads to the relative conductivity change as,

$$\frac{\Delta \sigma}{\sigma_0} = -(\pi_{11}' + \pi_{12}') \cdot \tau = 0.00726 \times 10^{-9} \cdot \left[ 1 - \frac{48\sqrt{2}\pi^2}{77} \cdot p \cdot W_{400} \right] \cdot (-\tau)$$  \hspace{1cm} (4.5)
It should be noted that $\Delta \sigma$, $\tau$ and $W_{400}$ are functions of depth $z$ below the peened surface.

Orientation distribution functions and coefficients are typically determined from pole figures measured by XRD or OIM. For shot peened surface with cylindrical symmetry along the plane normal, there are only two linearly independent ODC’s, namely $W_{000}$ (nontexture) and $W_{400}$ (texture). In this work, we derived equations which relate the plane-normal orientation distribution $q_i(\chi, \eta)$ of the $i$th Miller index (please see APPENDIX B) as a function of ODC $W_{400}$ as shown in equation (A38) and (A39), namely,

\[ q_{[111]} = \frac{1}{4\pi} - \frac{7\sqrt{2}\pi}{6} W_{400} \]  
\[ q_{[022]} = \frac{1}{4\pi} + \frac{9\sqrt{2}\pi}{8} W_{400} \]

Let $R = \frac{I_{[111]}}{I_{[022]}}$, where $I$ is the integrated Bragg intensity which can be experimentally assessed by XRD. If there is no texture,

\[ R_i \equiv \frac{I_{[111]}}{I_{[022]}} \bigg|_{w_{000}=0} = \frac{q_{[111]}}{q_{[022]}} \bigg|_{w_{000}=0} \int_0^{2\pi} \int_0^{2\pi} \int_{-1}^{1} I_{[111]}(\phi, \theta) w(\phi, \theta, \psi) d\xi \xi d\eta d\psi - \int_0^{2\pi} \int_0^{2\pi} \int_{-1}^{1} I_{[022]}(\phi, \theta) w(\phi, \theta, \psi) d\xi \xi d\eta d\psi \bigg|_{w_{000}=0} \]  

For cylindrically symmetric texture (which shot peened samples are assumed to exhibit),
where the triple integration of the integrated Bragg intensities are the same for both non-texture and texture cases. Solving the above equations for \( W_{400} \) as function of the ratio \( R_2 / R_1 \) leads to,

\[
W_{400} = \frac{3\sqrt{2}}{\pi^2} \left( 1 - \frac{R_2}{R_1} \right) \frac{1}{28 + 27 \cdot \frac{R_2}{R_1}}
\]  

\[ (4.10) \]
where \( R_2 / R_1 \) can be experimentally measured. The depth profile of the ratio \( R_2 / R_1 \) is shown in Figure 4.2. The calculated \( W_{400} \) profile is plotted in Figure 4.6. The magnitude of this ODC is in the similar order of rolled thin copper and aluminum\(^{19-21} \). The typical \( W_{400} \) values for rolled Cu and Al are in the order of magnitude of \( 10^{-3} \) while our measured maximum \( W_{400} \) for shot peened IN718 are -0.00859 and 0.00627.

### 4.5 Near-Surface Residual Stress Profile Inversion

We will closely follow the eddy current model and inversion procedure in Chapter 3 (Section 3.3)\(^{15} \). The residual stress profile \( \tau \) is assumed to have the functional form as

\[
\tau = e^{-x/\lambda} \sum_{i=0}^{2} a_i x^i
\]  

(4.11)

where \( x \) is the depth below surface while \( \lambda \) and \( a_i (i = 0 \text{ to } 2) \) are fitting parameters. This empirical function is chosen to capture conceivable residual stress profiles. The conductivity profile is computed from equation (4.5) and then discrete into 94 layers as described in the previous chapter\(^{15} \).

A parameter was introduced to account for the possible roughness effect on electrical conductivity. Here the conductivity of a thin layer of shot peened surface is set to be 0 and the thickness of this layer \( \delta \) is a fitting parameter. At each frequency, values of coil impedance \( Z_R \), \( Z_L \), and \( Z_T \) were calculated from the conductivity profile based on the Cheng-Dodd-Deeds model\(^{16} \) for the three EC measurement configurations, i.e. the unpeened...
(reference) sample surface, liftoff and the peened surface, respectively. The theoretical V-component signal was then calculated as

\[ V_{TH} = \text{Im} \left( \frac{Z_L - Z_R}{Z_L - Z_R} \right) \]  

(4.12)

and compared with the measured V-component signal as defined by Eqn. (2.1). The thickness of zero-conductivity surface layer \( \delta \) and the piezo-resistivity constant ratio \( p \) as well as \( \lambda \) and \( a_i \) in equation (4.11) are the fitting parameters. The best fit values of these parameters were determined by minimizing the rms difference between the measured and calculated V-component signals over the frequency range of 0.4 MHz to 20 MHz. The measured and best fitted V-components are plotted as Figure 4.7. The inverted conductivity profiles with and without including the texture are plotted as Figure 4.8. \( \delta \) is inverted to be 20 \( \mu \)m in the figure. This value is reasonable when compared to the measured profile of the peened surface, which show a rms roughness value of 2.4 \( \mu \)m and a deepest valley of 80.8 \( \mu \)m\(^2\). Figure 4.9 is the inverted residual stress profile as compared to the residual stress profile measured using the \( \sin^2 \psi \) method (Lambda Research, Inc.\(^2\)). It can be seen that the inverted residual stress profile matches the experimental results very well. The inverted conductivity profile parameters are listed in Table 4.2.

<table>
<thead>
<tr>
<th>Table 4.2</th>
<th>Inverted parameters.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Names</td>
<td>( \delta ) (( \mu )m)</td>
</tr>
<tr>
<td>Values</td>
<td>20</td>
</tr>
</tbody>
</table>
Figure 4.7. Measured and best fitted V-component of SHFEC signals.

Figure 4.8. Inverted conductivity profiles with and without including the texture and roughness effect.
We developed XRD partial pole figure technique for texture profile measurement of shot peened Inconel 718 samples. The measured rich texture profile is independently confirmed by OIM measurement. A texture-influenced piezoresistivity theory has been developed to relate the conductivity profile to the residual stress and the only ODC $W_{400}$ profiles. By assuming a stress profile functional form and by employing eddy current inversion technique, we obtained the inverted residual stress profile which agrees with the direct experimental data taken by Lambda Research very well. The agreement between these two profile data

**Figure 4.9.** Inverted residual stress profile compared to experimental stress profile measured by the $\sin^2\psi$ method (by Lambda Research, Inc.).

**4.6 Conclusions**

We developed XRD partial pole figure technique for texture profile measurement of shot peened Inconel 718 samples. The measured rich texture profile is independently confirmed by OIM measurement. A texture-influenced piezoresistivity theory has been developed to relate the conductivity profile to the residual stress and the only ODC $W_{400}$ profiles. By assuming a stress profile functional form and by employing eddy current inversion technique, we obtained the inverted residual stress profile which agrees with the direct experimental data taken by Lambda Research very well. The agreement between these two profile data
indicates that the texture can play a very important role in conductivity change in shot peened superalloys. The significant texture effect also suggests that there is a strong correlation between the texture change and the residual stress state (thus conductivity profile) for shot peened Inconel 718.

References


CHAPTER 5. RESIDUAL STRESS MEASUREMENT FOR SHOT PEENED NICKEL BASE ALLOY BY THETA-2THETA XRD METHOD

Abstract: In this chapter, a new scheme for analyzing θ-2θ XRD data for residual stress measurement, particularly for shot peened surfaces which can be considered as an isotropic plane stress problem, is presented. The standard XRD residual stress measurement is the Sine-Squared-Ψ method which can be used to assess triaxial stress status. In the course of our study of the shot-induced texture in an Inconel 718 sample using XRD, we devised a procedure to analyze conventional θ – 2θ XRD data for residual stress assessment by taking the advantage of cylindrical symmetry of the sample. This method can also give the anisotropy ratio of the nickel based superalloy IN718, which was found to be close to the value obtained from ultrasound measurements.

As a byproduct of the partial XRD pole figure measurement, in which we used the intensities of diffraction peaks, we employed the peak positions of all available peaks from the step theta-2theta scans to obtain the residual stress profile. The strain $\varepsilon_{33}'$ perpendicular to a shot-peened surface, which is assumed to have isotropic residual stresses, is given by equation (A52) in APPENDIX C as a function $f(h,k,l)$ of the diffraction (Miller) indices $h,k,l,$
\[ \varepsilon_{33}' = 2\tau'[S_{12} + (S_{11} - S_{12} - S_{44}/2) \cdot f(h,k,l)] \]  
(5.1)

where

\[ f(h,k,l) = \frac{(h^2k^2 + k^2l^2 + l^2h^2)}{(h^2 + k^2 + l^2)^2}, \]  
(5.2)

\( \tau' \) is the plane stress, and \( S_{ij} \) are the components of the compliance tensor.

This strain can be experimentally measured by XRD, namely,

\[ \varepsilon_{33}' = \frac{d_{hkl}^0 - d_{hkl}^0}{d_{hkl}^0} = \frac{d_{hkl}^0}{d_{hkl}^0} - 1 = \frac{\sin \theta_{hkl}^0}{\sin \theta_{hkl}^0} - 1 \]  
(5.3)

where \( d_{hkl}^0 \) and \( d_{hkl} \) are the spacing between parallel lattice planes with Miller indices \((hkl)\) before and after peening, respectively. \( \theta_{hkl}^0 \) and \( \theta_{hkl} \) are the diffraction angles before and after peening, respectively. Equating equation (5.1) and (5.3) will give us the plane stress by,

\[ \frac{\sin \theta_{hkl}^0}{\sin \theta_{hkl}^0} - 1 = 2\tau'[S_{12} + (S_{11} - S_{12} - S_{44}/2) f(h,k,l)] \]  
(5.4)

If \( \theta_{hkl} \) deviates from \( \theta_{hkl}^0 \) by a small amount, equation (5.4) can be further simplified in the first order approximation as,

\[ \frac{\sin \theta_{hkl}^0}{\sin \theta_{hkl}^0} - 1 = -\cot \frac{\theta_{hkl}^0 + \theta_{hkl}^0}{2} \cdot (\theta_{hkl} - \theta_{hkl}^0) = 2S_{12} \cdot \tau' + 2\tau'(S_{11} - S_{12} - S_{44}/2) \cdot f(h,k,l) \]  
(5.5)

After plotting the left hand side of (5.5) as a function of \( f(h,k,l) \), the interception of the y axis will give the value of the 2 times of the plane stress multiplied by \( S_{12} \), which can be independently measured from other experimental methods such as XRD and ultrasound technique.
We collect the XRD data of an IN718 sample before and after shot-peening at 17A. Seven diffraction indexes were collected from $2\theta = 30^\circ$ to $140^\circ$ as [111], [200], [220], [311], [222], [400] and [331]. Let $\Delta \theta_0$ and $\Delta \theta$ be the sample zero shifts before and after peening of the collected 2-theta pattern, respectively. Equation (5.5) can be written as,

$$
- \cot \frac{\theta_{hkl}^m}{2} + \cot \frac{\theta_{hkl}^0}{2} - (\Delta \theta_0 + \Delta \theta) \left[ \left( \theta_{hkl}^m - \theta_{hkl}^0 \right) + (\Delta \theta_0 - \Delta \theta) \right] = 2S_{12} \cdot \tau' \cdot 2\tau' (S_{11} - S_{12} - S_{44} / 2) \cdot f(h,k,l) 
$$

(5.6)

where the superscript $m$ denotes the measured values. Since the sample zero shifts are very small compared to the diffraction angles, the above equation can be approximated by,

$$
- \cot \frac{\theta_{hkl}^m}{2} + \cot \frac{\theta_{hkl}^0}{2} \left[ \left( \theta_{hkl}^m - \theta_{hkl}^0 \right) + (\Delta \theta_0 - \Delta \theta) \right] = 2S_{12} \cdot \tau' \cdot 2\tau' (S_{11} - S_{12} - S_{44} / 2) \cdot f(h,k,l) 
$$

(5.7)

or in the matrix form,

$$
\hat{y}_{hkl} = \begin{bmatrix} 1 & f(hkl) & \beta_{hkl} \end{bmatrix} \begin{bmatrix} \hat{x}_1 \\ \hat{x}_2 \\ \hat{x}_3 \end{bmatrix}
$$

(5.8)

where

$$
\hat{y}_{hkl} = - \cot \frac{\theta_{hkl}^m}{2} + \cot \frac{\theta_{hkl}^0}{2} \cdot (\theta_{hkl}^m - \theta_{hkl}^0), \quad \beta_{hkl} = - \cot \frac{\theta_{hkl}^m}{2} + \cot \frac{\theta_{hkl}^0}{2};
$$

(5.9)

$$
\hat{x}_1 = 2S_{12} \cdot \tau', \quad \hat{x}_2 = 2\tau' (S_{11} - S_{12} - S_{44} / 2), \quad \hat{x}_3 = (\Delta \theta - \Delta \theta_0)
$$

(5.10)

Once we obtain the coefficients of $\hat{y}_{hkl}$, $f(hkl)$ and $\beta_{hkl}$ for the 7 indexes, the $\hat{x}_i$ can be solved from the linear regression, i.e.

$$
\{ \hat{\beta} \} = \left[ C^T C \right]^{-1} C^T \{ \hat{y} \}
$$

(5.11)

where
Table 5.1  Fitted XRD 2-theta peak positions of selected Miller index for shot peened Inconel 718 after different layer removal. The removed layer thicknesses are listed in Table 4.1.

<table>
<thead>
<tr>
<th>Miller index</th>
<th>[111]</th>
<th>[002]</th>
<th>[022]</th>
<th>[113]</th>
<th>[222]</th>
<th>[004]</th>
<th>[331]</th>
<th>Anisotropy</th>
</tr>
</thead>
<tbody>
<tr>
<td>(h^2k^2 + k^2l^2 + l^2h^2) / (h^2 + k^2 + l^2)^2</td>
<td>1/3</td>
<td>0</td>
<td>1/4</td>
<td>19/121</td>
<td>1/3</td>
<td>0</td>
<td>99/361</td>
<td>-</td>
</tr>
<tr>
<td>2θ₀ₘ (degree)</td>
<td>43.384</td>
<td>50.339</td>
<td>74.219</td>
<td>89.913</td>
<td>95.355</td>
<td>116.737</td>
<td>136.957</td>
<td>-2.53</td>
</tr>
<tr>
<td>2θ₁ₘ (degree)</td>
<td>43.474</td>
<td>50.408</td>
<td>74.268</td>
<td>89.959</td>
<td>95.372</td>
<td>116.634</td>
<td></td>
<td>-2.10</td>
</tr>
<tr>
<td>2θ₂ₘ (degree)</td>
<td>43.520</td>
<td>50.453</td>
<td>74.302</td>
<td>89.980</td>
<td>95.402</td>
<td>116.585</td>
<td>136.904</td>
<td>-2.19</td>
</tr>
<tr>
<td>2θ₃ₘ (degree)</td>
<td>43.227</td>
<td>50.165</td>
<td>74.016</td>
<td>89.704</td>
<td>95.138</td>
<td>116.291</td>
<td></td>
<td>-2.07</td>
</tr>
<tr>
<td>2θ₄ₘ (degree)</td>
<td>43.541</td>
<td>50.446</td>
<td>74.315</td>
<td>89.978</td>
<td>95.425</td>
<td>116.532</td>
<td>136.893</td>
<td>-2.23</td>
</tr>
<tr>
<td>2θ₅ₘ (degree)</td>
<td>43.437</td>
<td>50.354</td>
<td>74.238</td>
<td>89.917</td>
<td>95.320</td>
<td>116.542</td>
<td>136.900</td>
<td>-2.30</td>
</tr>
<tr>
<td>2θ₆ₘ (degree)</td>
<td>43.424</td>
<td>50.341</td>
<td>74.220</td>
<td>89.907</td>
<td>95.328</td>
<td>116.496</td>
<td>136.879</td>
<td>-2.32</td>
</tr>
<tr>
<td>2θ₇ₘ (degree)</td>
<td>43.466</td>
<td>50.389</td>
<td>74.253</td>
<td>89.961</td>
<td>95.397</td>
<td>116.564</td>
<td>136.908</td>
<td>-2.34</td>
</tr>
<tr>
<td>2θ₈ₘ (degree)</td>
<td>43.530</td>
<td></td>
<td>74.329</td>
<td>90.051</td>
<td>95.453</td>
<td>116.710</td>
<td>137.028</td>
<td>-2.28</td>
</tr>
<tr>
<td>2θ₉ₘ (degree)</td>
<td>43.443</td>
<td>50.435</td>
<td>74.272</td>
<td>90.018</td>
<td>95.389</td>
<td>116.8</td>
<td>137.002</td>
<td>-2.25</td>
</tr>
<tr>
<td>2θ₁₀ₘ (degree)</td>
<td>43.542</td>
<td>50.52</td>
<td>74.361</td>
<td>90.139</td>
<td>95.495</td>
<td>116.975</td>
<td>137.123</td>
<td>-2.39</td>
</tr>
</tbody>
</table>
\[ [C] = [1, f(hkl), \chi_{hkl}] \]  

The residual stress is expressed as,

\[ \tau'^1 = \frac{\hat{x}_1}{2S_{12}} \]  

The anisotropy ratio is obtained from,

\[ \frac{S_{12}}{S_{11} - S_{12} - S_{44}/2} = \frac{\hat{x}_1}{\hat{x}_2} \]  

where the compliance constant \( S_{12} \) can be obtained from other independent experimental data so that the plane stress can be determined from this theta-2theta XRD technique. In practice, the number of collected diffraction index could be less than 7, but must be more than or equal to 3.

The compliance constant \( S_{12} = -0.2585 \times 10^{-11} \text{ Pa}^{-1} \) of IN718 used in this study was obtained from the stiffness constants measured for an Inconel 718 polycrystallite coupon by ultrasound technique\(^1\). The values of \( f(h,k,l) \) and the measured XRD peak positions of the selected Miller index \([hkl]\) as well as the best fitted anisotropy ratio \( S_{12}/(S_{11} - S_{12} - S_{44}/2) \) are listed in Table 5.1.

Figure 5.1 shows 2 examples of the fitted and experimental XRD \( \theta-2\theta \) data. Figure 5.2 shows that the linearity of equation (5.5) does not have noticeable change by employing the linear regression method discussed above, thus validating the approximation in equation (5.7). The measured raw residual stresses of every layer are corrected\(^2,3\) to compensate the effect due to the layer material removal. Figure 5.3 shows the measured small coupon residual stress profile using \( \theta-2\theta \) method as well as the residual stress profile measured by Lambda Research Inc. using the standard \( \sin^2 \psi \) method. Our measurement of the small
coupon stress profile deviates somehow from that of Lambda Research. These could be due to the bending effect of the small coupon, the perturbation of the plane stress status when cutting the small coupon from the plate or the value of $S_{12}$ we employed here.

Figure 5.1  Fitted and experimental XRD theta-2theta data of diffraction indexes [111] (above) and [022] (below).
**Figure 5.2.** The linear dependence of perpendicular strain of shot peened surface on index function $f(h,k,l)$.

**Figure 5.3**  Measured residual stress profile by theta-2theta method compared to the one by Sine-Squared-Psi method from Lambda Research Corporation.
References


CHAPTER 6. CONCLUSIONS AND FUTURE WORK

In conclusion, this dissertation describes three main areas of work that, when combined, establish an eddy current NDE method for residual stress characterization on shot peened nickel-based alloy surfaces. Our first technical tool is a high frequency eddy current measurement system especially useful for multiple-frequency measurements (Chapter 2). The second area of work is a physics-model-based eddy current inversion method that has been developed and applied to the actual conductivity assessment from eddy current data (Chapter 3). Rich conductivity profiles are obtained within the nominal depth of 400 μm for shot peened nickel base alloy samples. The third and the most critical development of this dissertation has to do with the texture-influenced piezo-resistivity theory and its application (Chapter 4). By developing and applying the piezo-resistivity theory with texture, the residual stress profile of a shot peened Inconel 718 sample is obtained from eddy current data. The resulting residual stress depth profile agrees with the data taken by the standard destructive XRD method very well. It is found that the texture effect is indeed playing a very important role in eddy current assessment of residual stress. Texture profile of shot peened nickel base alloy has been obtained from XRD partial pole figure and orientation image micrograph (OIM) data. In making independent XRD stress profile measurements for comparison, we also developed an approach to analyzing $\theta - 2\theta$ XRD data for residual
stress, as described in Chapter 5 of this dissertation, as an alternative to the conventional sine-squared-psi method for measuring residual stresses in shot peened nickel based alloys. All of this work together cast promising light on the ability of eddy current technique on residual stress assessment of shot peened nickel base alloys that have extensive application in aircraft engine.

There is a number of areas to consider for future work, among which we only give a few considerations here: For example, the experimental and theoretic work in this dissertation is applicable, strictly speaking, only to the nickel base alloys right after shot peening but before being put in service. During service, shot peened surfaces will be exposed to complex mechanical processes, as well as undergoing high temperature exposures. The underlying material conditions such as stress and texture will respond differently to the mechanical processes and the heat exposures. The texture effect, for instance, could be different after some time in service (in particular at elevated operating temperatures) from that found from newly shot peened components. It is thus desirable to have a material response model that may be capable of comprehensively predicting the changes of stress, cold work, texture, and/or other microstructural states against given environmental influences such as shot peening, mechanical loading, and heat exposure. Judging from our work to date, there appears to be a strong correlation between the texture and the residual stress (and thus conductivity) for nickel base alloy, right after shot peening. This suggests existence of an underlying common mechanism to cause stress and texture state changes simultaneously. A comprehensive study of texture under shot peening is called for, in order to uncover the underlying physical mechanisms, by way of either mechanical models or experimental benchmark measurements. For example, a compilation of the texture profile changes under
different Almen intensities will be extremely beneficial to eddy current nondestructive residual stress profile characterization.
APPENDIX A. PIEZORESISTIVITY THEORY FOR TEXTURED MATERIALS WITH CYLINDRICAL SYMMETRY

For 3-D anisotropic crystal, Ohm's law is expressed in the generalized form as,

\[ E_i = \rho_{ik} J_k \]  \hspace{1cm} (A1)
where \( E^e \) is the electric field, \( j \) is the current density, \( \rho \) is the resistivity and the subscripts \( i \) and \( k \) indicate the 3 Cartesian coordinates and hereby repeated subscript is the Einstein convention, summing over all coordinate components. For cubic crystallite, the resistivity matrix \( [\rho_{ik}] \) free of stress (strain) reduces to only 1 constant\(^1\), i.e.

\[
\begin{bmatrix}
E_i^e \\
E_2^e \\
E_3^e
\end{bmatrix} =
\begin{bmatrix}
\rho_0 & 0 & 0 \\
0 & \rho_0 & 0 \\
0 & 0 & \rho_0
\end{bmatrix}
\begin{bmatrix}
j_1 \\
j_2 \\
j_3
\end{bmatrix}
\]

or

\[
E_i^e = \rho_0 j_i
\]

When the crystal is strained, the resistivity is perturbed and can be expressed as,

\[
\begin{bmatrix}
E_i^e \\
E_2^e \\
E_3^e
\end{bmatrix} =
\begin{bmatrix}
\rho_0 & 0 & 0 \\
0 & \rho_0 & 0 \\
0 & 0 & \rho_0
\end{bmatrix} + \begin{bmatrix}
\Delta \rho_{11} & \Delta \rho_{12} & \Delta \rho_{13} \\
\Delta \rho_{21} & \Delta \rho_{22} & \Delta \rho_{23} \\
\Delta \rho_{31} & \Delta \rho_{32} & \Delta \rho_{33}
\end{bmatrix}
\begin{bmatrix}
j_1 \\
j_2 \\
j_3
\end{bmatrix}
\]

The resistivity change matrix \( [\Delta \rho_{ij}] \) is symmetric under the Onsager’s principle\(^1\). Therefore the resistivity change matrix only has at most 6 independent components. Since the strain is typically small (in the order of magnitude of 1% or less), as a first order approximation, the resistivity tensor change can be expressed as being linearly depending on the strain tensor,

\[
\Delta \rho_{ij} = \xi_{ijklmn} \cdot \rho_{kl} \cdot \epsilon_{mn}
\]

where \( \epsilon_{mn} \) are the strain tensor component and \( \xi_{ijklmn} \) are the components of a \( 3 \times 3 \times 3 \times 3 \) tensor. The tensor \( [\xi_{ijklmn}] \) is hereby named strain-resistivity tensor. For cubic crystallites, \( \rho_{kl} \) has only one component and as a result,

\[
\Delta \rho_{ij} = \rho_0 \xi_{ijklmn} \cdot \epsilon_{mn}.
\]
It is well known that the strain tensor has only 6 independent components and so does the resistivity change tensor as we stated above. The symmetry of tensors $\Delta\rho_y$ and $\varepsilon_{mn}$ lead to only 3 independent coefficients $\xi_{ijmn}$. It is convenient to express the relationship between the independent components of the resistivity change tensor and the strain tensor contractedly,

$$\frac{\Delta\rho_L}{\rho_0} = \xi_{ij} \cdot \varepsilon_{ij}.$$  \hspace{1cm} (A7)

Employing Voigt’s notation for the independent strain components we have the following explicit strain-resistivity matrix form for a single crystal with cubic symmetry:

$$\begin{bmatrix} \Delta\rho_1 \\ \Delta\rho_2 \\ \Delta\rho_3 \\ \Delta\rho_4 \\ \Delta\rho_5 \\ \Delta\rho_6 \end{bmatrix} = \begin{bmatrix} \xi_{11} & \xi_{12} & 0 & 0 & 0 \\ \xi_{12} & \xi_{11} & 0 & 0 & 0 \\ \xi_{12} & \xi_{11} & \xi_{12} & 0 & 0 \\ 0 & 0 & \xi_{44} & 0 & 0 \\ 0 & 0 & 0 & \xi_{44} & 0 \\ 0 & 0 & 0 & 0 & \xi_{44} \end{bmatrix} \begin{bmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \varepsilon_3 \\ 2\varepsilon_4 \\ 2\varepsilon_5 \\ 2\varepsilon_6 \end{bmatrix}.$$  \hspace{1cm} (A8)

Similarly, the piezoresistivity tensor can be expressed as,

$$\begin{bmatrix} \Delta\rho_1 \\ \Delta\rho_2 \\ \Delta\rho_3 \\ \Delta\rho_4 \\ \Delta\rho_5 \\ \Delta\rho_6 \end{bmatrix} = \begin{bmatrix} \pi_{11} & \pi_{12} & \pi_{12} & 0 & 0 & 0 \\ \pi_{12} & \pi_{11} & \pi_{12} & 0 & 0 & 0 \\ \pi_{12} & \pi_{11} & \pi_{12} & 0 & 0 & 0 \\ 0 & 0 & \pi_{44} & 0 & 0 & 0 \\ 0 & 0 & 0 & \pi_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & \pi_{44} \end{bmatrix} \begin{bmatrix} \tau_1 \\ \tau_2 \\ \tau_3 \\ \tau_4 \\ \tau_5 \\ \tau_6 \end{bmatrix}.$$  \hspace{1cm} (A9)

where $\tau_j$ are the independent stress components and $\pi_{ij}$ are the so-called piezo-resistivity constants. Note that $\xi_{ij}$ are dimensionless and are related to $\pi_{ij}$ by the elastic stiffness tensor or the elastic compliance tensor,

$$[\pi] = [\xi] [S],$$  \hspace{1cm} (A10)
and

$$[\xi] = [\pi][s]^{-1} = [\pi][c].$$

(A11)

where $[s]$ is elastic compliance tensor and $[c]$ is the elastic stiffness tensor.

This leads to

$$\begin{align*}
\pi_{11} &= \xi_{11}s_{11} + 2\xi_{12}s_{12} \\
\pi_{12} &= \xi_{11}s_{12} + \xi_{12}(s_{11} + s_{12}) \\
\pi_{44} &= \xi_{44}s_{44}
\end{align*}$$

or

$$\begin{align*}
\xi_{11} &= \pi_{11}c_{11} + 2\pi_{12}c_{12} \\
\xi_{12} &= \pi_{11}c_{12} + \pi_{12}(c_{11} + c_{12}) \\
\xi_{44} &= \pi_{44}c_{44}
\end{align*}$$

(A12)

We shall now derive the macroscopic piezoresistivity constants as a function of orientation distribution coefficients (ODC’s) for fcc poly-crystallite in a shot-peened surface. Due to the surface plane stress condition, the near surface stress has tetragonal symmetry in every sliced thin layer parallel to the surface. The macroscopic piezoresistivity tensor of each layer also has tetragonal symmetry. The residual stress generated from random shot-peening process is a plane stress possessing cylindrical symmetry, which is higher order symmetry than tetragonal symmetry.

We shall now derive the macro-piezoresistivity (polycrystalline) constants from the micro-piezoresistivity (single crystal) constants. In Figure A1, let the original coordinates $(x_1, y_1, z_1)$ be along the crystalline axes of a grain and the rotated coordinates $(x_3, y_3, z_3)$ lie in the sample surface plane with $x_3$ and $y_3$ parallel to while $z_3$ is perpendicular to the sample surface.

Let $A$ be the rotation transform matrix from $(x_1, y_1, z_1)$ to $(x_3, y_3, z_3)$ with 3 Euler rotations respect to $z, y_2$ and $z_3$ coordinates in sequence as shown in Figure A1. The final rotation matrix is given by
The resistivity change tensors after $[\Delta \rho']$ and before $[\Delta \rho]$ rotation can be related to each other as,

$$
\begin{bmatrix}
    \Delta \rho'_1 \\
    \Delta \rho'_2 \\
    \Delta \rho'_3
\end{bmatrix} = 
\begin{bmatrix}
    l_1 & l_2 & l_3 \\
    m_1 & m_2 & m_3 \\
    n_1 & n_2 & n_3
\end{bmatrix}
\begin{bmatrix}
    \Delta \rho_1 & \Delta \rho_2 & \Delta \rho_3 \\
    \Delta \rho_4 & \Delta \rho_5 & \Delta \rho_6 \\
    \Delta \rho_7 & \Delta \rho_8 & \Delta \rho_9
\end{bmatrix}
\begin{bmatrix}
    l_1 & l_2 & l_3 \\
    m_1 & m_2 & m_3 \\
    n_1 & n_2 & n_3
\end{bmatrix}^T
$$

(A13)

Suppose the polycrystalline is under a uniaxial stress $\tau'$ along $x_3$ of the sample coordinate systems, the stress field tensors before $[\tau]$ and after $[\tau']$ rotation are related to each other by

$$
\begin{bmatrix}
    \tau \\
    \tau'
\end{bmatrix} = 
\begin{bmatrix}
    \gamma \\
    0 \\
    0
\end{bmatrix}
\begin{bmatrix}
    \tau \\
    \tau'
\end{bmatrix}
\begin{bmatrix}
    \gamma \\
    0 \\
    0
\end{bmatrix}
$$

(A14)

Solving equations (A9), (A14) and (A15) lead to the relationship between the stress and resistivity change after rotation,

$$
\frac{1}{\rho_0} [\Delta \rho']_{6 \times 1} = [\pi']_{6 \times 6} [\tau']_{6 \times 1}
$$

(A16)

This gives the component of $\pi'_{ij}$ as,

$$
\pi'_{11} = \pi_{11} - 2k <r_1>, \quad r_1 = l_1^2 l_3^2 + l_2^2 l_3^2 + l_1^2 l_2^2
$$

$$
\pi'_{12} = \pi_{12} + k <r_2>, \quad r_2 = l_1^2 m_1^2 + l_2^2 m_2^2 + l_3^2 m_3^2
$$

(A17)
where \( k \equiv \pi_{11} - \pi_{12} - \pi_{44} \) and,

\[
\begin{align*}
l_1 &= -\sin \psi \sin \phi + \cos \psi \cos \phi \cos \theta \\
l_2 &= -\sin \psi \cos \phi - \cos \psi \sin \phi \cos \theta \\
l_3 &= \sin \theta \cos \psi \\
m_1 &= \cos \psi \sin \phi + \sin \psi \cos \phi \cos \theta \\
m_2 &= \cos \psi \cos \phi - \sin \psi \sin \phi \cos \theta \\
m_3 &= \sin \theta \sin \psi \\
n_1 &= -\sin \theta \cos \phi \\
n_2 &= \sin \theta \sin \phi \\
n_3 &= \cos \theta
\end{align*}
\]

(A18)

\[
< r_1 > = \int \int \int r_1 \cdot w(\phi, \cos \theta, \psi) \cdot d\phi \cdot d\cos \theta \cdot d\psi
\]

(A19)

\[
< r_2 > = \int \int \int r_2 \cdot w(\phi, \cos \theta, \psi) \cdot d\phi \cdot d\cos \theta \cdot d\psi
\]

where \( w(\phi, \cos \theta, \psi) \) is the weight function satisfying

\[
\int \int \int w(\phi, \cos \theta, \psi) \cdot d\phi \cdot d\cos \theta \cdot d\psi = 1.
\]

(A20)

Expanding the crystallite orientation distribution function \( w(\phi, \cos \theta, \psi) \) and \( r(\phi, \cos \theta, \psi) \) into spherical harmonic series gives,

\[
w(\phi, \cos \theta, \psi) = \sum_{l=0}^{\infty} \sum_{m=-l}^{l} \sum_{n=-l}^{l} W_{lmn} \cdot \exp(-im\phi) \cdot Z_{lmn}(\cos \theta) \cdot \exp(-in\psi)
\]

(A21)

\[
r(\phi, \cos \theta, \psi) = \sum_{l=0}^{\infty} \sum_{m=-l}^{l} \sum_{n=-l}^{l} R_{lmn} \cdot \exp(-im\phi) \cdot Z_{lmn}(\cos \theta) \cdot \exp(-in\psi)
\]

where \( r \) represents \( r_1 \) or \( r_2 \), \( Z_{lmn} \) are the normalized generalization of the associated Legendre functions defined by Roe\(^27\). The \( W_{lmn} \) are given as,

\[
W_{lmn} = \frac{1}{4\pi^{2}} \int \int \int w(\phi, \cos \theta, \psi) \cdot \exp(im\phi) \cdot Z_{lmn}(\cos \theta) \cdot \exp(in\psi) \cdot d\phi d\cos \theta d\psi.
\]

(A22)
and a similar expression holds for $R_{lmn}$. Substituting Equations (A22) into Equations (A19) we found that,

\[
<r> = \int_0^{2\pi} \int_0^{\pi} r(\phi, \cos \theta, \psi) \cdot w(\phi, \cos \theta, \psi) \cdot d\phi \cdot d\cos \theta \cdot d\psi = 4\pi^2 \sum_{l=0}^{\infty} \sum_{m=-l}^{l} \sum_{n=-l}^{l} R_{lmn} W_{lmn}.
\]

(A23)

For cubic crystallites with cylindrical symmetry, which is exactly the case of shot peened Inconel 718, Roe\textsuperscript{2-4} and Bunge\textsuperscript{5, 6} shown that only $W_{000}$ and $W_{400}$ are linearly independent for $l \leq 4$. This leads to,

\[
<r_1> = \frac{1}{5} - \frac{6}{35} \sqrt{2}\pi^2 W_{400}
\]

\[
<r_2> = \frac{1}{5} + \frac{4}{35} \sqrt{2}\pi^2 W_{400}.
\]

(A24)

For randomly distributed (no texture) cubic crystallites,

\[
W_{400} = 0, \quad <r_1> = <r_2> = \frac{1}{5}
\]

(A25)

where the values of $<r_1>$ and $<r_2>$ come from normalization of the weighting function.

This leads to the explicit expressions of the macroscopic piezo-resistivity constants,

\[
\pi_{11}' = \frac{3\pi_{11} + 2\pi_{12} + 2\pi_{44}}{5} + \frac{12\sqrt{2}}{35} \pi^2 \cdot W_{400} \cdot (\pi_{11} - \pi_{12} - \pi_{44})
\]

\[
\pi_{12}' = \frac{\pi_{11} + 4\pi_{12} - \pi_{44}}{5} + \frac{4\sqrt{2}}{35} \pi^2 \cdot W_{400} \cdot (\pi_{11} - \pi_{12} - \pi_{44})
\]

(A26)

Thus the resistivity change of shot peened sample under plane residual stress has the form
\[
\frac{\Delta \rho}{\rho_0} = (\pi_{11}' + \pi_{12}') \cdot \tau \\
= \left( \frac{4\pi_{11} + 6\pi_{12} + \pi_{44}}{5} \right) \cdot \left[ 1 - \frac{48\sqrt{2}\pi^2}{77} \cdot \left( \frac{\pi_{11}}{3} - \frac{\pi_{12}}{3} - \frac{\pi_{44}}{3} \right) \cdot W_{400} \right] \cdot \tau \\
= \left( \pi_{11}' + \pi_{12}' \right)_{\text{no texture}} \cdot \left[ 1 - \frac{48\sqrt{2}\pi^2}{77} \cdot p \cdot W_{400} \right] \cdot \tau \\
\]

where

\[
p \equiv \left( \frac{\pi_{11}}{3} - \frac{\pi_{12}}{3} - \frac{\pi_{44}}{3} \right) \left/ \frac{4\pi_{11}}{11} + \frac{6\pi_{12}}{11} + \frac{\pi_{44}}{11} \right\} \\
\]

is the ratio of normalized combinations of piezo-resistivity constants (namely \(\pi_{11}, \pi_{12}\) and \(\pi_{44}\)) of single crystals. The conductivity change of shot peened sample under plane stress has the form,

\[
\frac{\Delta \sigma}{\sigma_0} = -\frac{\Delta \rho}{\rho_0} = -(\pi_{11}' + \pi_{12}') \cdot \tau \\
= \left( \pi_{11}' + \pi_{12}' \right)_{\text{no texture}} \cdot \left[ 1 - \frac{48\sqrt{2}\pi^2}{77} \cdot p \cdot W_{400} \right] \cdot (-\tau) \\
\]
APPENDIX B. RELATIONSHIP BETWEEN ODC $W_{400}$ AND XRD PEAK INTENSITY RATIO

We shall now derive the expression of the only ODC parameter $w_{400}$ as function of the measurable XRD peak intensities. The distribution function of all crystallites in the sample can be represented by $w(\phi, \theta, \psi)$, where $-\phi$, $-\theta$ and $-\psi$ are the 3 Euler angles with respect to the sample coordinate system. The normalization condition is,

$$
\int_{-\pi}^{\pi} \int_{-1}^{1} \int_{-\pi}^{\pi} w(\phi, \theta, \psi) \cdot d\phi \cdot d\theta \cdot d\psi = 1 \quad \text{(A30)}
$$

Suppose there is a crystallite which rotates from the sample coordinate characterized by Euler rotation angles of $(-\phi, -\theta, -\psi)$. For a particular Miller index $(hkl)$ of this crystallite, the plane-normal orientation vector will uniquely characterize it. In another word, only the first 2 Euler angles $(-\phi, -\theta)$ will fully determine the Miller index $(hkl)$. It is convenient to use polar and azimuthal angles in this case. Let $\chi_i$ and $\eta_i$ be the polar and azimuthal angles of the $i$th $[hkl]$ plane normal to the reference coordinates. Let $I(\chi_i, \eta_i)$ be the corresponding integrated intensity of the diffracted X-ray. The plane-normal orientation distribution $q_i(\chi_i, \eta_i)$ is then obtained by normalization of the intensity function.

$$
q_i(\chi_i, \eta_i) = \frac{I(\chi_i, \eta_i)}{\int_{0}^{2\pi} \int_{-1}^{1} \int_{-\pi}^{\pi} I(\phi, \theta) w(\phi, \theta, \psi) d\xi d\eta d\psi} \quad \text{(A31)}
$$
Expand \( q_i(\chi_i, \eta_i) \) and \( w(\phi, \theta, \psi) \) in a series of spherical harmonics and generalized spherical harmonics, respectively:

\[
q_i(\chi_i, \eta_i) = \sum_{l=0}^{\infty} \sum_{m=-l}^{l} \mathcal{Q}_{lm}^i \mathcal{P}_l^m(\cos \chi_i) e^{-im\eta_i}, \tag{A32}
\]

and

\[
w(\xi, \psi, \phi) = \sum_{l=0}^{\infty} \sum_{m=-l}^{l} \sum_{n=-l}^{l} \mathcal{W}_{lmn} \mathcal{Z}_{lmn}^i (\cos \theta) e^{-im\psi} e^{-in\phi}. \tag{A33}
\]

Here \( \mathcal{P}_l^m(\cos \theta) \) is the normalized associated Legendre function, and \( \mathcal{Z}_{lmn}(\cos \theta) \) is a generation of the associated Legendre function. Solving the equations (A31) to (A33) and applying the orthogonal property of Legendre function leads to the relationship between the plane normal orientation distribution coefficients \( \mathcal{Q}_{lm}^i \) and the ODCs \( \mathcal{W}_{lmn} \),

\[
\mathcal{Q}_{lm}^i = (2\pi)^{1/2} \left( \frac{2}{2l+1} \right)^{1/2} \sum_{n=-l}^{l} \mathcal{W}_{lmn} \mathcal{P}_l^m(\cos \theta) e^{in\phi}. \tag{A34}
\]

For shot peened metal plate, taking the only non-texture term \( W_{000} \) and the only texture term \( W_{400} \) into account for the plane normal orientation distribution coefficients leads to,

\[
\mathcal{Q}_{00}^i = 2\sqrt{2} \pi W_{000} P_0^{\theta} (\cos \theta) = 2\sqrt{2} \pi \cdot \frac{1}{8\pi^2} \cdot \sqrt{\frac{2}{2}} = \frac{1}{4\pi}, \tag{A35}
\]

\[
\mathcal{Q}_{40}^i = \frac{2\sqrt{2} \pi}{3} W_{400} P_4^{\theta} (\cos \theta). \tag{A36}
\]

Substituting equations (A35) into equation (A32) leads to,

\[
q_i(\xi, \eta_i) = \mathcal{Q}_{00}^i P_0^{\theta}(\cos \theta) P_0^{\chi}(\cos \chi_i) + \mathcal{Q}_{40}^i P_4^{\theta}(\cos \theta) P_4^{\chi}(\cos \chi_i)
= \frac{1}{4\pi} + \frac{2\sqrt{2} \pi}{3} W_{400} P_4^{\theta}(\cos \theta) P_4^{\chi}(\cos \chi_i). \tag{A36}
\]
For typical $\theta - 2\theta$ X-ray diffraction scans of shot peened IN718 plate, $\chi_i = 0$ holds for every reflection index. Furthermore, we have the following relationships for particular reflection indexes $[hkl]$ with cubic crystallite symmetry:

$$
\cos \theta_i = \frac{l}{\sqrt{h^2 + k^2 + l^2}}, \quad \cos \theta_{[111]} = \frac{1}{\sqrt{3}}, \quad \cos \theta_{[022]} = 0 \quad (A37)
$$

These lead to,

$$
q_{[111]} = \frac{1}{4\pi} + \frac{2\sqrt{2}\pi}{3} W_{400} P_4^0 (1/\sqrt{3}) P_4^0 (1) = \frac{1}{4\pi} - \frac{7\sqrt{2}\pi}{6} W_{400} \quad (A38)
$$

$$
q_{[022]} = \frac{1}{4\pi} + \frac{2\sqrt{2}\pi}{3} W_{400} P_4^0 (0) P_4^0 (1) = \frac{1}{4\pi} + \frac{9\sqrt{2}\pi}{8} W_{400} \quad (A39)
$$

where

$$
P_4^0 (x) = \frac{1}{8} \left( 35 \cdot x^4 - 30 \cdot x^2 + 3 \right) \frac{\sqrt{2}}{3}, \quad P_4^0 (0) = \frac{9}{16} \sqrt{2}, \quad P_4^0 (1) = \frac{3\sqrt{2}}{2}, \quad P_4^0 (1/\sqrt{3}) = -\frac{7}{12} \sqrt{2} \quad (A40)
$$
APPENDIX C. PERPENDICULAR STRAIN AS A FUNCTION OF X-RAY DIFFRACTION INDEX

We will derive the strain perpendicular to a surface as a function of diffraction indexes for fcc polycrystallite materials under an isotropic plane stress, such as Inconel 718 after shot-peening.

In Figure A1, let the original coordinates be along the crystalline axes and the rotated coordinates (denoted by $x_3$, $y_3$ and $z_3$) lie in the sample surface plane with $x_3$ and $y_3$ while $z_3$ is perpendicular to the surface.

Let $A$ be the rotation transform matrix for $[hkl]$. For an Euler rotation respect to $z$, $y_2$ and $z_3$ coordinates in sequence as shown in Figure A1, the final rotation matrix is given by

$$A = \begin{bmatrix}
  a_{11} & a_{12} & a_{13} \\
  a_{21} & a_{22} & a_{23} \\
  a_{31} & a_{32} & a_{33}
\end{bmatrix}$$

$$\equiv \begin{bmatrix}
  \cos \psi \cos \theta \cos \phi - \sin \psi \sin \phi & \cos \psi \cos \theta \sin \phi + \sin \psi \cos \phi & -\cos \psi \sin \theta \\
  -\sin \psi \cos \theta \cos \phi - \cos \psi \sin \phi & -\sin \psi \cos \theta \sin \phi + \cos \psi \cos \phi & \sin \psi \sin \theta \\
  \sin \theta \cos \phi & \sin \theta \sin \phi & \cos \theta
\end{bmatrix}$$

(A41)

As shown in the Figure A1, we have the following geometry relationship,
\[
\begin{align*}
\phi &= \arccos \frac{\cos \alpha_i}{\sin \psi}, \\
\theta &= \alpha_3, \\
\psi &= \text{arbitrary angle}
\end{align*}
\]  

(A42)

where \( \alpha_i \) are the angles between the \([hkl] \) direction and the 3 original coordinates. For fcc crystallite, the direction cosine between crystal axes and \([hkl] \) can be expressed as,

\[
\cos \alpha_3 = \frac{h}{\sqrt{h^2 + k^2 + l^2}}, \quad \cos \alpha_2 = \frac{k}{\sqrt{h^2 + k^2 + l^2}}, \quad \cos \alpha_1 = \frac{l}{\sqrt{h^2 + k^2 + l^2}}. 
\]  

(A43)

The strain field tensors after and before rotation from the crystal coordinates to the sample coordinates can be related to each other as,

\[
\varepsilon' = \begin{bmatrix} \varepsilon'_{11} & \varepsilon'_{12} & \varepsilon'_{13} \\ \varepsilon'_{21} & \varepsilon'_{22} & \varepsilon'_{23} \\ \varepsilon'_{31} & \varepsilon'_{32} & \varepsilon'_{33} \end{bmatrix} = \begin{bmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{bmatrix} \begin{bmatrix} \varepsilon_{11} & \varepsilon_{12} & \varepsilon_{13} \\ \varepsilon_{21} & \varepsilon_{22} & \varepsilon_{23} \\ \varepsilon_{31} & \varepsilon_{32} & \varepsilon_{33} \end{bmatrix}^T.
\]  

(A44)

Let’s consider the situation where the sample is under an isotropic plane stress. i.e.,

\[
\tau'_{ij} = \tau' \quad \text{for } ij = 11 \text{ and } 22, \quad \text{and } = 0 \quad \text{otherwise.}
\]

The stress field tensors before and after rotation are given by

\[
\tau = A^T \tau' A = \begin{bmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{bmatrix} \begin{bmatrix} \tau_{11} & 0 & 0 \\ 0 & \tau'_{22} & 0 \\ 0 & 0 & \tau_{33} \end{bmatrix} \begin{bmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{bmatrix}.
\]  

(A45)

In Voigt’s notation, the strain-stiffness relationship before rotation is expressed by,

\[
\begin{bmatrix} \varepsilon_{11} \\ \varepsilon_{22} \\ \varepsilon_{33} \\ 2\varepsilon_{12} \\ 2\varepsilon_{23} \\ 2\varepsilon_{31} \end{bmatrix} = \begin{bmatrix} S_{11} & S_{12} & S_{13} & 0 & 0 & 0 \\ S_{12} & S_{11} & S_{12} & 0 & 0 & 0 \\ S_{13} & S_{12} & S_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & S_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & S_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & S_{44} \end{bmatrix} \begin{bmatrix} \tau_{11} \\ \tau_{22} \\ \tau_{33} \\ \tau_{12} \\ \tau_{23} \\ \tau_{31} \end{bmatrix}.
\]  

(A46)
where \( S_{ij} \) are compliance constants. Solving equation (A44), (A45) and (A46) lead to the strain-stiffness relationship after rotation as,

\[
\left[ \varepsilon' \right] = [N][S'][N]^T \left[ \tau' \right]
\]

where

\[
[N] = \begin{bmatrix}
a_{11}^2 & a_{12}^2 & a_{13}^2 & a_{12}a_{13} & a_{11}a_{13} & a_{11}a_{12} \\
a_{21}^2 & a_{22}^2 & a_{23}^2 & a_{22}a_{23} & a_{21}a_{23} & a_{21}a_{22} \\
a_{31}^2 & a_{32}^2 & a_{33}^2 & a_{32}a_{33} & a_{31}a_{33} & a_{31}a_{32} \\
2a_{21}a_{31} & 2a_{22}a_{32} & 2a_{23}a_{33} + a_{22}a_{32} & a_{21}a_{33} + a_{23}a_{31} & a_{21}a_{32} + a_{22}a_{31} & 2a_{12}a_{32} \\
2a_{11}a_{31} & 2a_{12}a_{32} & 2a_{13}a_{33} + a_{12}a_{32} & a_{11}a_{33} + a_{13}a_{31} & a_{11}a_{32} + a_{12}a_{31} & 2a_{12}a_{32} \\
2a_{11}a_{12} & 2a_{12}a_{22} & 2a_{13}a_{33} + a_{12}a_{22} & a_{11}a_{23} + a_{13}a_{21} & a_{11}a_{22} + a_{12}a_{21} & 2a_{12}a_{32} \\
\end{bmatrix}
\]

(A47)

Solving equation (A47) for \( \varepsilon_{33} \) results in

\[
\varepsilon_{33} = r \left[ 2S_{12} + 2(S_{11} - S_{12} - S_{44}) / 2 \right] \left( a_{31}^2 a_{32}^2 + a_{32}^2 a_{33}^2 + a_{33}^2 a_{31}^2 \right)
\]

(A49)

or in terms of Euler’s rotational angles,

\[
\varepsilon_{33} = r \cdot \left( 2S_{12} + 2(S_{11} - S_{12} - S_{44}) / 2 \right) \sin^2 \theta (\cos^2 \vartheta + \sin^2 \vartheta \sin^2 \phi \cos^2 \phi)
\]

(A50)

or in terms of direction cosines,

\[
\varepsilon_{33} = 2r' \left[ S_{12} + (S_{11} - S_{12} - S_{44}) / 2 \right] \sin^2 \alpha_3 \left[ \cos^2 \alpha_3 + \sin^2 \alpha_3 \frac{\cos^2 \alpha_1 \cos^2 \alpha_2}{(\cos^2 \alpha_1 + \cos^2 \alpha_2)^2} \right]
\]

(A51)

where the constraint \( \cos^2 \alpha_1 + \cos^2 \alpha_2 + \cos^2 \alpha_3 = 1 \) is used. The same relationship can also be expressed in terms of the \([hkl]\) index by employing equation (A43),

\[
\varepsilon_{33} = 2r' \left[ S_{12} + (S_{11} - S_{12} - S_{44}) / 2 \right] \left( \frac{h^2 k^2 + k^2 l^2 + l^2 h^2}{(h^2 + k^2 + l^2)^2} \right)
\]

(A52)
References:
5. H.J. Bunge, Z. Metalk. 56 872(1965)
ACKNOWLEDGEMENT

I would like to express my sincerely gratitude to my major professor Dr. R. Bruce Thompson, who has been encouraging, supporting and guiding my research work. With his broad knowledge and deep wisdom, Dr. R. Bruce Thompson always has a wide picture of my research and can always quickly point out what are the major issues to be solved and usually gives me the key references in remarkably short time. I got some very important suggestions and references in the process of solving the eddy current inversion problem and deriving the formula of texture effect on piezoresistivity in nickel-based alloy. I would also like to thank him for carefully revising my proposal, paper and dissertation, sometimes happened in plane due to his very tight schedule.

My sincerely thanks also go to the project manager, Dr. Norio Nakagawa for his guidance, support and patience during my research toward my Ph.D. I learned a lot about eddy current from Dr. Norio Nakagawa, who is definitely an expert with a lot experience in this area. Dr. Norio Nakagawa introduced a very important transfer function—V component—in high frequency eddy current measurement, which is the key for reliable high frequency eddy current experimental data in my dissertation. I would also like to convey my
deep appreciation for his thorough and careful revision of all of my written research documents.

Many thanks go to Dr. Chester Lo for all of his help on the experimental results, revision of my research documents and some interesting research references periodically sent to me. Dr. Chester Lo involved all of the experimental part in this dissertation who always can get experimental data with the least error bar due to his remarkable patience and creativity.

I am very grateful to Dr. Frank Margetan for his patience and comments in the group research meeting, Dr. Changqing Lee for designing the PCB coils and setting up the high frequency eddy current measurement system, which greatly moved the project forward on which I were working. I am also very grateful to Dr. Anatoli Frishman for his comments in the group research meeting and revision on my proposal, Dr. Seong-Jae Lee for his comments in my rehearsal. I acknowledge Dr. Matthew Kramer and Dr. Francis Laabs of Ames Laboratory for their help on the OIM experiment.

Thank all the committee members for donating their time to make my dissertation a better document. Thank Dr. Ralph Napolitano for his careful revision of my proposal.

I also want to express my thanks to my classmates Anxiang Li and Fangwei Fu for much fun time spent with them on discussing on materials science, eddy current, class projects and how to fish in Saylorville Lake.