DETECTION OF SURFACE IMPERFECTIONS IN METALS BY A NONCONTACTING THERMOELECTRIC METHOD

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Hector Carreon

M.S., Institute of Metallurgic Research, Universidad Michoacana, Mexico 1997
B.S., Engineering Mechanics, Universidad Michoacana, Mexico 1995

Committee Chair: Prof. Peter B. Nagy
ABSTRACT

A variety of different physical principles have been exploited for nondestructive detection, localization, and characterization of material imperfections in metals. The most popular techniques rely on ultrasonic, eddy current, X-ray radiographic, magnetic, thermal, and microwave principles. A common feature of these conventional methods is that they are sensitive to both intrinsic material (e.g., electrical and thermal conductivity, permeability, elastic stiffness, density, etc.) and spurious geometrical (e.g., size, shape, surface roughness, etc.) parameters. Unfortunately, these two classes of properties are often very difficult to separate, which sets the ultimate limit for the detectable smallest or weakest material imperfection. It was recently discovered that inclusions and other types of imperfections can be nondestructively detected by thermoelectric measurements in a noncontact way by using high-sensitivity magnetometers to sense the thermoelectric currents generated around the affected area when the specimen is subjected to directional heating and cooling. This novel noncontacting thermoelectric inspection technique offers the following distinct advantages over conventional methods: (a) high sensitivity to subtle variations in material properties, (b) complete insensitivity to the size, shape, surface roughness and other geometrical features of the specimen, (c) noncontacting nature with a substantial stand-off distance, and (d) probing deep into the material and penetration through thick, multiple-layer structures. The potential applications of this method cover a very wide range from detection of metallic inclusions, segregations, inhomogeneities, to characterization of hardening, fatigue, texture, and residual stresses.

This dissertation presents evidence of these potential applications of a novel NDE technique based on theoretical and experimental data for the magnetic field produced by thermoelectric currents generated around imperfections when a temperature gradient is established in the specimen. This effort lays the groundwork for a new branch in the nondestructive characterization field and demonstrates that owing to recent technological advances in the development of high-sensitivity magnetic sensors, such as Giant Magneto-Resistive (GMR) detectors, fluxgates and, especially, high-temperature Superconductive QUantum Interference Device (SQUID) magnetometers, it has become feasible to adapt the noncontacting magnetic thermoelectric inspection to many applications of great interest in NDT.
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LIST OF SYMBOLS

\( V \) Thermoelectric voltage
\( S \) Absolute thermoelectric power
\( T \) Temperature
\( E \) Electrical field
\( T_h \) Preset (hot) temperature junction
\( T_c \) Room (cold) temperature junction
\( x, y, z \) Cartesian coordinate system
\( \xi_1, \xi_2, \xi_3 \) Physical coordinate system
\( j \) Electrical current density
\( h \) Thermal flux density
\( \Phi \) Electrical potential
\( \sigma \) Electrical conductivity
\( \kappa \) Thermal conductivity for zero electrical field
\( k \) Thermal conductivity for zero electrical current
\( \varepsilon \) Thermoelectric coupling coefficient
\( B \) Magnetic flux density
\( H \) Magnetic field strength
\( F \) Universal spatial distribution function
\( F(\xi) \) Universal spatial distribution function for spherical inclusions
\( a \) Inclusion radius
\( \Gamma \) Universal thermoelectric contrast
\( W \) Half-width of the bipolar signature
\( \theta \) Rotation angle
\( b \) Baseline distance
\( g \) Apparent lift-off distance
<table>
<thead>
<tr>
<th>Symbol</th>
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<tr>
<td>e</td>
<td>Sensing element length of the fluxgate</td>
</tr>
<tr>
<td>d</td>
<td>Sensing element diameter of the fluxgate</td>
</tr>
<tr>
<td>n</td>
<td>Protective case length of the fluxgate</td>
</tr>
<tr>
<td>( h_0 )</td>
<td>Undisturbed heat flux</td>
</tr>
<tr>
<td>( \dot{Q} )</td>
<td>Heat flow rate from a source</td>
</tr>
<tr>
<td>m</td>
<td>Distance between source and sink</td>
</tr>
<tr>
<td>p</td>
<td>Orthogonal polarization direction</td>
</tr>
<tr>
<td>( \ell )</td>
<td>Bar length</td>
</tr>
<tr>
<td>w</td>
<td>Bar width</td>
</tr>
<tr>
<td>t</td>
<td>Bar thickness</td>
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<td>( H_x )</td>
<td>Normal component of the magnetic field</td>
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<tr>
<td>( H_y )</td>
<td>Tangential component of the magnetic field</td>
</tr>
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<td>( c_x )</td>
<td>Inhomogeneity in the width-direction</td>
</tr>
<tr>
<td>( c_y )</td>
<td>Inhomogeneity in the thickness-direction</td>
</tr>
<tr>
<td>d</td>
<td>Depth below the surface</td>
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<tr>
<td>( C_{max} )</td>
<td>Maximum compressive stress</td>
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<tr>
<td>( T_{max} )</td>
<td>Maximum tensile stress</td>
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CHAPTER I
INTRODUCTION

1.1 OVERVIEW

The thermoelectric technique, based on the well-known Seebeck effect that is commonly used in thermocouples to make temperature measurements, has been in use for many years. This conventional technique has also found application as a useful tool for investigating different material characteristics and properties such as metal sorting, flaw detection, thickness gauging of layers, quality testing and microscopic analysis.[1-5] In general, the thermoelectric technique monitors the thermoelectric power of conductor materials, which is sensitive to small changes caused by material imperfections. In order to apply the conventional thermoelectric technique for NDE material characterization, a conductor with well-defined properties (reference probe) and a known temperature difference along the probe is used to evaluate the properties of a second conductor (test sample) from changes in the test sample thermoelectric power. However, these changes in the thermoelectric power can be influenced by numerous parameters, and these will therefore influence the outcome of the thermoelectric measurements. The most important effects are those associated with contact and volumetric properties.[6] The volumetric effect is closely related to the thermoelectric phenomena by the kinetics of the diffusion of electrons throughout the volume of the material. This effect is mainly dependent upon material properties such as chemical composition, different heat treatment, anisotropy, hardening, texture, residual stress, fatigue, etc. On the other hand, contact effects are related to imperfect contact between the test sample and the reference probe, with factors such as amount of pressure applied to the probe, temperature of hot and cold junctions and probe material influencing it.

A variety of different physical principles have been exploited for nondestructive detection, localization, and characterization of material imperfections in metals. The most
popular techniques rely on ultrasonic, eddy current, x-ray radiographic, magnetic, thermal, and microwave principles. A common feature of these conventional methods is that they are sensitive to both intrinsic material (e.g., electrical and thermal conductivity, permeability, elastic stiffness, density, etc.) and spurious geometrical (e.g., size, shape, surface roughness, etc.) parameters. Unfortunately, these two classes of properties are often very difficult to separate, which sets the ultimate limit for the detectable smallest or weakest material imperfection. Conventional thermoelectric techniques, that have been used in nondestructive materials characterization for several decades, are essentially free from the previously mentioned geometrical limitations, i.e., they are solely sensitive to intrinsic material variations only regardless of the size, shape, and surface quality of the specimen to be tested. In spite of its obvious advantages over other methods, thermoelectric testing is rarely used in NDT because of the requirement that a metallic contact be established between the specimen and the reference electrode.

This dissertation presents a novel noncontacting thermoelectric technique based on magnetic detection of local thermoelectric currents around imperfections when a temperature gradient is established in the specimen. The self-referencing, noncontacting, nondestructive inspection technique to be presented will offer the following advantages over conventional methods: (a) high sensitivity to subtle variations in material properties, (b) noncontacting nature with a substantial stand-off distance and, (c) complete insensitivity to the size, shape, and other geometrical features of the specimen, in particular, surface topography, that often hinder other more conventional NDE techniques in the characterization of material imperfections. The potential applications of this novel technique cover a very wide range of material imperfections from detection of metallic inclusions, segregations, inhomogeneities, anisotropy and tight cracks to characterization of hardening, fatigue, localized texture, residual stresses and fretting.
1.2 LITERATURE REVIEW

1.2.1 SEEBECK EFFECT

The phenomenon of thermoelectricity was first observed by Seebeck in 1826, who found that an electromotive force is produced when two junctions between dissimilar metals forming a closed circuit are kept at different temperatures, and is called the Seebeck effect. Today, the main application of the Seebeck effect is to measure temperature by using thermocouples which consist of two dissimilar metal wires soldered together forming a loop. A thermoelectric voltage is produced between junctions A and B, provided by the temperature difference between two points as shown in Fig. 1.1. The voltage \( V = V_a - V_b \) is the thermoelectric voltage developed by the thermocouple and the thermoelectric power is given by

\[
S_{AB} = S_A - S_B = \lim_{\Delta T \to 0} (\Delta V / \Delta T). \tag{1.1}
\]

In order to have a better understanding of this phenomenon, we are going to show in detail the relationships that describe the working of the thermocouple of Fig. 1.1. Let us assume that the points of terminals a and b are at the same temperature, while junctions c and d are at different temperatures \( T_2 \) and \( T_1 \), respectively. We define the absolute thermoelectric power \( S \), which is a physical material property, by the following relation

\[
E = S \nabla T, \tag{1.2}
\]

where \( E \) is the electric field in the material, \( \nabla T \) is the temperature gradient, and we assumed that the conductor materials A and B of Fig. 1.1 are isotropic (have cubic symmetry). From Eq. (1.2) we have that
Thus,

$$V = V_a - V_b = (V_a - V_d) + (V_d - V_c) + (V_c - V_b)$$

$$= \int dV + \int dV + \int dV$$

$$= \int S_B dT - \int S_A dT - \int S_B dT + (\int S_B dT - \int S_B dT)$$

$$= \int S_B dT - \int S_A dT$$

$$= \int S_{AB} dT.$$

Finally,

$$V = \frac{T_2}{T_1} \int S_{AB}(T) dT. \quad (1.4)$$

The thermoelectric voltage $V$ produced by a thermocouple consisting of two conductors A and B is a function of the thermoelectric material properties and the temperature difference between the two junctions.[7,8]
Figure 1.1 A schematic diagram of the Seebeck effect.
**1.2.2 PRINCIPLE OF THERMOELECTRICITY**

Thermoelectricity is a conversion of heat into electrical energy or vice versa, in conductor materials by means of different phenomena such as the Seebeck effect, the Peltier effect and the Thomson effect. In order to understand these phenomena, in particular the Seebeck effect, consider a conductor material that is heated at one side and cooled at the other. When a small temperature gradient $\nabla T$ is established across the conductor, the electrons at the hot side have higher energy than those at the cold side. Therefore the heat is carried from the hot region to the cold region by a net diffusion of electrons. In general, the diffusion rate is a function of electron energy and thus, a net electron current will result.

The flow of electrons leaves behind exposed positive metal ions (charges) in the hot region and piles up electrons in the cold region. So the higher energy electrons at the hot region are able to lower their energies by diffusing to the cold region. This situation prevails until the electric field produced by the electron current opposes the further flow of electrons from the hot to cold region. Therefore the cold side becomes negatively charged, while the hot side is positively charged and, as a result, a thermoelectric voltage is induced along the conductor. This potential difference $\Delta V$ across the conductor caused by a temperature gradient $\nabla T$ is called the Seebeck effect. The magnitude of this effect is given by the Seebeck coefficient that is defined as the thermoelectric voltage developed per unit temperature difference in a conductor

$$S = -\frac{dV}{dT}. \quad (1.5)$$
1.3 CONVENTIONAL THERMOELECTRIC TECHNIQUE

Thermoelectricity is caused by coupled transport of heat and electricity in metals, that leads to a number of interesting phenomena, some of which can be exploited for nondestructive testing (NDT) and materials characterization. Essentially all existing thermoelectric NDE methods are based on the well-known Seebeck effect that is commonly used in thermocouples to measure temperature at the junction between two different conductors. Figure 1.2 shows a schematic diagram of the thermoelectric measurements as most often performed in nondestructive materials characterization. One of the reference electrodes is heated by electrical means to a preset temperature of \( T_h \), pretty much like the tip of a temperature-stabilized soldering iron while the other electrode is left cold at room temperature \( T_c \). The measurement is done quickly in a few seconds to assure (i) that the hot reference electrode is not cooled down perceivably by the specimen and (ii) that the rest of the specimen beyond the close vicinity of the contact point is not warmed up perceivably. Then, the measured thermoelectric voltage is given by (from Eq. (4) by substituting \( S \rightarrow A, R \rightarrow B, T_h \rightarrow T_2 \), and \( T_c \rightarrow T_1 \))

\[
V = \frac{T_h}{T_c} \int \left[ S_S(T) - S_R(T) \right] dT = \frac{T_h}{T_c} \int S_{SR}(T) dT, \tag{1.6}
\]

where \( T \) is the temperature, and \( S_S \) and \( S_R \) denote the absolute thermoelectric powers of the specimen and the reference electrode, respectively. Any variation in material properties can affect the measured thermoelectric voltage via \( S_{SR} = S_S - S_R \), which is the relative thermoelectric power of the specimen to be tested with respect to the reference electrode. In most cases, the temperature-dependence of the thermoelectric power can be neglected over the range of operation and the thermoelectric voltage can be approximated as \( V = (T_h - T_c) S_{SR} \). Ideally, regardless of how high the temperature difference between the junctions is, only thermocouples made of different materials, or more precisely, materials of different
thermoelectric power, will generate a thermoelectric signal. This unique feature makes the simple thermoelectric tester one of the most sensitive material discriminators used in nondestructive inspection. The thermoelectric power of metals is sensitive to a variety of material properties that can affect the measurement. Clearly, chemical composition exerts the strongest effect on the thermoelectric properties and accordingly the basic application of thermoelectric materials characterization is metal sorting.[5,9] However, it is well known that under special conditions materials of identical chemical composition can also produce an efficient thermocouple as a result of different heat treatment, hardening, texture, residual stress, fatigue, etc., which can be further exploited for nondestructive testing of materials.[5,10-12]

As we mentioned above, the fundamental assumption in nondestructive thermoelectric testing is that, although the measured voltage might be somewhat uncertain because of inevitable variations in the contact temperature, identical materials do not produce any thermoelectric voltage. Consequently, small differences in the thermoelectric power can be unequivocally detected by using a reference electrode that is very similar to the materials to be tested. However, the inherently imperfect contact between the specimen and the reference electrode can produce a significant thermoelectric signal even if the measuring electrodes are made from the very same material as the specimen.[11,12] This thermoelectric offset can be reduced, but not entirely eliminated, by decreasing the thermal and electrical resistance between the specimen and the reference electrode (e.g., via cleaning or imposing higher contact pressure). Ultimately, the presence of this imperfect contact limits the detectability of small variations in material properties by the conventional thermoelectric technique. When subtle local variations such as texture, hardening, fatigue damage, or weak impurities are to be detected, the best sensitivity can be achieved by using the surrounding intact material as the reference electrode. This so-called self-referencing method not only provides an ideal reference material, but also automatically eliminates the above mentioned spurious thermoelectric offset caused by having a less than perfect artificial interface between the part to be tested and the surrounding intact reference material.
Figure 1.2  A schematic diagram of conventional thermoelectric measurement as most often used in nondestructive materials characterization.
1.4 NONCONTACTING NDE THERMOELECTRIC TECHNIQUE

Ordinary thermocouples based on the Seebeck effect can not operate in a noncontacting way partly because they need strong conduction-type thermal coupling with the specimen to be tested and partly because they need direct electrical coupling with the measuring electronics. However, material imperfections naturally form such thermocouples in the specimen itself and in the presence of an externally induced temperature gradient. These innate thermocouples produce thermoelectric currents around the imperfections that can be detected by magnetic sensors from a significant lift-off distance between the tip of the magnetic sensor and the specimen surface, even when the material imperfections (segregations, residual stresses, inhomogeneity) are rather deep below the surface.

It was recently demonstrated that self-referencing thermoelectric measurements can be also done in an entirely noncontact way by using high-sensitivity magnetic detectors to sense the weak thermoelectric currents around inclusions and other types of inhomogeneities when the specimen to be tested is subjected to directional heating or cooling. Figure 1.3 shows a schematic diagram of the self-referencing thermoelectric measurement with noncontact magnetic sensing. External heating or cooling is applied to the specimen to produce a substantial temperature gradient in the region to be tested. As a result, different points on the boundary between the host material and the inclusion will be at different temperatures, and therefore also at different thermoelectric potentials. These potential differences will produce opposite thermoelectric currents inside and outside the inclusion. The thermoelectric current forms local loops that run in opposite directions on the two sides of the inclusion relative to the prevailing heat flux, as indicated in Fig. 1.3.

When the specimen is scanned with a sensitive magnetometer, such as a fluxgate or SQUID detector, the magnetic field of these thermoelectric currents can be detected even when the inclusion is buried below the surface and the sensor is far away from the specimen. Since the surrounding intact material serves as the “reference” electrode and there is no artificial
interface between the host and the imperfect region to be detected, the detection sensitivity to
variations in material properties could be very high and subtle effects such as local plastic
deformation, fretting, residual stress, localized texture, cold work, intrinsic anisotropy,
inghomogeneity can also be detected.\cite{18-24,64}

1.5 MAGNETIC SENSORS

The feasibility of such self-referencing thermoelectric measurements by noncontact
magnetic sensing crucially depends on the sensitivity of the magnetometer. Fortunately, as a
result of recent technological advances in high-sensitivity magnetic sensors, state-of-the-art
magnetometers such as Giant Magnetoe-Resistive (GMR) detectors, spin dependent tunneling
(SPD) detectors, fluxgates and, especially, Superconductive QUantum Interference Device
(SQUID) magnetometers, it has become feasible to achieve very low equivalent noise levels
between 1 and 10 pT over a detection bandwidth of 0.1 and 20 Hz. Figure 1.4 shows the typical
noise spectra between 0.1 and 20 Hz for various magnetic sensors currently available on the
market. Of course, the strength of the magnetic field to be detected greatly depends on the
physical nature and dimensions of the imperfection.

In comparison, under modest ($\approx$ 1-10°C/cm) temperature gradients most
inhomogeneities produce magnetic flux densities between 10 pT and 1000 nT.\cite{13-24,27,28,64} At
such sensitivities, noncontacting sensing of thermoelectric currents will undoubtedly lead to
numerous NDE applications. Although the feasibility of this brand new technique in all these
applications has not been studied yet with sufficient scientific detail and very few analytical or
experimental results have been published yet in the open literature, there is plenty of evidence
that, even in its infancy, the proposed technique works very well in all these demanding
applications.
Figure 1.3  A schematic diagram of noncontacting detection of material imperfections by magnetic monitoring of thermoelectric currents.
The experimental results presented in this dissertation were obtained by a Bartington Instruments Mag-03 fluxgate that has a noise-limited detection threshold of \( \approx 2.5 \text{ pT/} \sqrt{\text{Hz}} \) over a bandwidth of 0.1 to 20 Hz. In comparison, the measured peak magnetic flux densities varied between 0.5 nT and 250 nT, i.e., the sensitivity of the fluxgate magnetometer was quite sufficient. The maximum output voltage of the sensor is limited to \( \pm 12 \text{ V} \), corresponding to a maximum detectable field strength of \( \pm 120 \text{ \mu T} \) giving a sensor sensitivity of \( (10 \text{ \mu T/V}) \).

Besides the fluxgate sensor, only a SQUID can provide field sensitivities in a lower range of about \( \approx 0.1 \text{ pT/} \sqrt{\text{Hz}} \) without unacceptably lowering the spatial resolution. GMR sensors provide a better spatial resolution but their field sensitivity is too low. Although SQUID sensors provide most of the desirable characteristics, they are rather expensive and require special cooling.

On the other hand, fluxgates have some advantages (high field sensitivity, high linearity, ruggedness, dynamic range) over other sensors available on the market, therefore they are used in a wide range of applications such as measurements in outer space (satellites), naval and terrestrial applications, e.g., detection of submarines and geomagnetic observation. Fluxgate sensors measure the absolute strength of a surrounding magnetic field in a frequency between DC and a few 100 Hz. The sensor itself consists of a high-permeability ferromagnetic core with a primary coil winding which periodically saturates the probe core. The response field is detected by a compensation coil winding in a direction opposite to the primary coil. The fluxgate uses a filtered signal of the probe core excitation frequency. This signal, which is low-pass filtered, synchronously detects and controls the feedback current flowing in the field compensation coil, resulting in a voltage proportional to this current that is digitized as a measure of the external magnetic field along the magnetic axis of the sensor.[25]
Figure 1.4  Typical noise spectra between 0.1 and 20 Hz for different magnetic sensors.
1.6 EXPERIMENTAL SET UP

Figure 1.5 shows a schematic diagram of the experimental setup used for measuring the magnetic field produced by the thermoelectric currents due to material imperfections such as inclusions, anisotropy, inhomogeneity, fretting and residual stresses using the noncontacting thermoelectric technique. In this case, a pair of fluxgate gradiometer was used to sense the magnetic flux density produced by the thermoelectric currents due to these imperfections in a host medium under two-dimensional directional heating and cooling. In general, the fluxgate gradiometer is a transducer that converts a magnetic field into an electric voltage that is then processed in order to obtain a meaningful signal. The signal is then displayed using a LeCroy 9310A oscilloscope and finally acquired by a computer for further analysis. Numerous steps were taken to process the sought magnetic signal (these measures are discussed in great detail in chapter II “Elimination of External Magnetic Sources”).

1.7 ACHIEVEMENT OF THE DISSERTATION

In spite of their unique sensitivity to subtle variations in intrinsic material properties, conventional thermoelectric techniques are rarely used in nondestructive testing (NDT) because of the requirement that a metallic contact be established between the specimen and the reference electrode. According to the proposed technique, the surrounding intact material serves as the “reference” electrode and there is no artificial interface between the host and the imperfect region to be detected, therefore the detection sensitivity could be very high assuming that the absolute sensitivity of the magnetometer is sufficient to pick up the weak magnetic field produced by the thermoelectric currents in the specimen. The purpose of this dissertation is to demonstrate the potential applications of this new method in the field of nondestructive materials characterization and to lay down the groundwork for its further development.
Figure 1.5 A schematic diagram of the experimental set up used to detect material imperfections using the noncontacting thermoelectric technique.


1.8 ORGANIZATION OF THE DISSERTATION

The dissertation is organized in the order of the research sequence, i.e., each following chapter is proposed and developed from the problems encountered in or conclusions derived from the previous chapters. Chapter II presents experimental data for the magnetic field produced by thermoelectric currents around surface-breaking spherical tin inclusions in copper under external thermal excitation for different lift-off distances between the sensor tip and the specimen surface. These results were compared with previously published theoretical predictions.\cite{15} Chapter III provides an analytical method developed by Nayfeh and Nagy for calculating the magnetic field produced by thermoelectric currents in anisotropic materials under two-dimensional directional heating and cooling. Experimental results from a textured Ti-6Al-4V titanium-alloy plate are presented and compared to the predictions of this analytical model.\cite{17} Chapter IV investigates the spurious magnetic signature produced by the simplest type of macroscopic inhomogeneity when the material properties exhibit a linear spatial variation in the cross-section of a slender bar. An analytical method has been developed by Nayfeh and Faidi for calculating the normal and tangential magnetic fields produced by the resulting thermoelectric currents. Experimental results from a Ti-6Al-4V titanium-alloy bar are presented and compared with the predictions of this analytical model.\cite{36} Chapter V presents a noncontacting thermoelectric method that can be used to characterize the prevailing residual stress in shot-peened specimens.\cite{64} Finally, Chapter VI offers a summary of the work done in the whole dissertation including conclusions and recommendations for the further development of this very promising NDT method for thermoelectric detection and characterization of inclusions and other types of imperfections in metals.
CHAPTER II
DETECTION OF SURFACE-BREAKING SPHERICAL TIN INCLUSIONS IN COPPER

2.1 INTRODUCTION

Inclusions and other types of imperfections in metals can be nondestructively detected by noncontacting magnetic measurements that sense the thermoelectric currents around such flaws when the specimen is subjected to directional heating and cooling. The noncontacting thermoelectric technique is very sensitive to the presence of foreign body inclusions, when the thermoelectric power of the affected region is significantly different from that of the surrounding medium.[13-16,22-24] This chapter presents experimental data for the magnetic field produced by thermoelectric currents around surface-breaking spherical tin inclusions in copper under external thermal excitation for different lift-off distances between the sensor and the surface of the specimen. The diameter of the inclusions and the lift-off distance varied from 2.4 to 12.7 mm and from 12 to 20 mm, respectively. A fairly modest 0.7°C/cm temperature gradient in the specimen produced peak magnetic flux densities ranging from 1 to 250 nT. The experimental results were found to be in good agreement with recently published theoretical predictions.[15,24]

The main goal of the current research effort is to help lay down the groundwork necessary to develop this new field of nondestructive testing and materials characterization based on noncontacting magnetic detection of thermoelectric currents. Achieving this goal will require closely related theoretical and experimental efforts that will lead to better understanding of the underlying physical phenomena, the development of new, predictive analytical models, more sensitive experimental procedures, and, ultimately, increased probability of detection (POD) for small inclusions and weak material imperfections. Although the best experimental tool for such studies is undoubtedly a SQUID-based magnetometer, we managed to use an
ordinary fluxgate to provide experimental evidence of the theoretical predictions through the example of surface-breaking spherical tin inclusions of varying diameter in copper. First, we are going to present a brief review of the analytical model of Ref. 15, then we will proceed by describing the experimental procedure and, finally, discuss the experimental results and compare them to the analytical predictions.

2.2 CONSTITUTIVE RELATIONS AND FIELD EQUATIONS

Thermoelectricity is a result of intrinsically coupled transport of electricity and heat in metals, that can be expressed by the following constitutive relationship[26]

$$\begin{bmatrix} j \\ h \end{bmatrix} = \begin{bmatrix} \sigma & \sigma S \\ \sigma S T & \kappa \end{bmatrix} \begin{bmatrix} -\nabla \Phi \\ -\nabla T \end{bmatrix}, \quad (2.1)$$

where \( j \) is the electrical current density, \( h \) is the thermal flux density, \( \Phi \) is the electric potential, \( T \) is the temperature, \( \sigma \) denotes the electrical conductivity measured at uniform temperature, \( \kappa \) is the thermal conductivity for zero electrical field, and \( S \) is the absolute thermoelectric power of the material. Assuming weak thermoelectric coupling, steady-state solutions can be obtained by requiring that \( \nabla \cdot h = 0 \).[15,24] In addition, Maxwell's law requires that \( \nabla \cdot j = 0 \).

The determinant of the matrix in Eq. (2.1) is \( \sigma (\kappa - \sigma S^2 T) = \kappa \), where \( \kappa \) is the thermal conductivity for zero electrical current. Noting that \( \sigma \kappa \neq 0 \), \( \nabla \cdot h = 0 \) and \( \nabla \cdot j = 0 \) require that the Laplacians of \( T \) and \( \Phi \) vanish individually, i.e.,

$$\nabla^2 T = 0 \quad \text{and} \quad \nabla^2 \Phi = 0. \quad (2.2)$$
2.3 REVIEW OF THE ANALYTICAL MODEL

In order to illustrate the magnetic field produced by thermoelectric currents flowing around a surface-breaking spherical inclusion, we will present some examples of the numerical results obtained by the analytical method described in Refs. 15 and 24. To simplify the numerical calculations, all spatial coordinates were normalized to the radius of the spherical inclusion as \( \xi = x / a \). The magnetic field can be also written in a normalized form as \( \mathbf{H} = H_0 \mathbf{F}(\xi) \), where

\[
H_0 = -a \nabla T \sigma (S' - S) \Gamma
\]  

(2.3)

combines the size of the inclusion \( a \), the externally enforced temperature gradient \( \nabla T \), the electrical conductivity of the host \( \sigma \), the relative thermoelectric power of the inclusion with respect to the host \( S' - S \), a universal spatial distribution function for all spherical inclusions \( \mathbf{F}(\xi) \), while \( \Gamma \) is a universal thermoelectric contrast that is determined by the ratio between the electrical and thermal conductivities of the inclusion (with prime) and the host medium (without prime) and is given by

\[
\Gamma = \frac{3}{(1 + 2 \frac{\sigma}{\sigma'}) (2 + \frac{\kappa}{\kappa'})}.
\]  

(2.4)

Figure 2.1 shows the two-dimensional distributions of the normal component of the magnetic field \( F_1(\xi_2, \xi_3) \) taken in planes parallel to the free surface at four different lift-off distances \( (\xi_1 = 0, 0.5, 1, \text{ and } 2) \) for a semi-spherical inclusion with its center lying on the surface. As it could be expected qualitatively from Fig. 1.3, the thermoelectric currents flow in opposite directions along two loops on the opposite sides of the inclusion relative to the direction of the heat flux, therefore the magnetic field is asymmetric with respect to this
direction. The characteristic bipolar shape produced by the positive and negative peaks in the magnetic field distribution are very typical in noncontacting thermoelectric detection of inclusions and can be easily exploited by digital image processing and feature extraction techniques to increase the probability of detection for inclusions. The larger the lift-off distance, the lower the peak magnetic field and the wider the field distribution. The latter can be quantitatively characterized by the lateral distance of the peaks from the center of the inclusion, $W$, which we are going to call the half-width of the bipolar signature.

### 2.3.1 PRINCIPAL PARAMETERS OF THE FIELD DISTRIBUTION

Since the field distribution remains very similar regardless of the lift-off distance, it is reasonable to choose only two parameters, namely the peak magnetic field and the half-distance between the peaks, to qualitatively characterize the whole field distribution. Figure 2.2 shows these two parameters as functions of the normalized lift-off distance. The solid lines are numerical results while the dashed lines are far field asymptotes. At small lift-off distances, the peaks are located directly above the circumference of the inclusion, i.e., $W \approx 1$, and their magnitude approaches 0.5. At large distances, the field distribution spreads out so that the peaks occur at $\theta \approx 45^\circ$, i.e., $W \approx \xi_1 / \sqrt{2}$, and their magnitude is inversely proportional to the square of the normalized lift-off distance.[15,24]
Figure 2.1 Two-dimensional distributions of the normal component of the magnetic field taken in four different planes parallel to the free surface at different lift-off distances for a semi-spherical inclusion with its center lying on the surface (normalized scale 6 × 6, the peak normalized flux density $F_1$ is indicated for comparison).
Figure 2.2  The two principal parameters of the field distribution, namely the peak of the normalized magnetic field and the half-distance between the peaks, as functions of the normalized lift-off distance. The solid lines are numerical predictions while the dashed lines are far-field asymptotes.
2.4 EXPERIMENTAL METHOD

In the following, we will describe the experimental setup and procedure used to verify that the analytical model of Ref. 15, which has been summarized in the previous section, truthfully captures the main features of the thermoelectrically generated magnetic field and accurately predicts its magnitude over a wide range of inclusion sizes and lift-off distances. Figure 2.3 shows a schematic diagram of the experimental arrangement. We prepared a series of semi-spherical pure tin inclusions embedded in two pure copper bars of $12.7 \text{ mm} \times 38.1 \text{ mm} \times 500 \text{ mm}$ dimensions. First, we prepared the semi-spherical holes by milling, then we heated the specimens to approximately $300^\circ\text{C}$ and filled the holes with molten tin, and finally milled the surface flat after the specimen has cooled down. The diameter of the inclusions varied from $2.38 \text{ mm}$ to $12.7 \text{ mm}$ and the center of each inclusion was at the level of the specimen surface. The distance between inclusions was approximately $75 \text{ mm}$ to avoid interference between their individual magnetic fields.

Both ends of the copper bar were perforated by a series of holes and equipped with sealed heat exchangers to facilitate efficient heating and cooling and then mounted on a non-magnetic translation table for scanning. The ends of the copper bar were simultaneously heated and cooled by running water to temperatures of $\approx+10^\circ\text{C}$ and $\approx+40^\circ\text{C}$, respectively. The actual temperature difference between the ends of the bar was monitored during the measurements by thermocouple thermometers and the temperature gradient was kept at $0.7^\circ\text{C/cm}$, which is more than sufficient to produce detectable magnetic signals in high-conductivity materials like copper and tin. It should be mentioned that most structural metals exhibit much lower electrical conductivity than copper. For example, in Ti-6Al-4V, the most popular aerospace titanium alloy, the electrical conductivity is only $\sigma \approx 5.8 \times 10^5 \text{ A/Vm}$, i.e., two orders of magnitude lower than that of pure copper $\sigma \approx 59.7 \times 10^6 \text{ A/Vm}$, therefore the expected magnetic signals are also proportionally lower as shown by Eq. (2.3).
Figure 2.3  A schematic diagram of the experimental arrangement for the magnetic detection of surface breaking tin inclusion embedded in copper.
2.4.1 PHYSICAL PROPERTIES OF PURE COPPER AND TIN

The relevant physical properties of pure copper and tin are listed in Table I. These parameters were taken from standard references with the exception of the absolute thermoelectric power of tin at room temperature, which was measured by a thermoelectric instrument on the largest inclusion itself since it was found to be significantly affected by melting and subsequent recrystallization during sample preparation (the measured value was approximately 20% lower than the tabulated data). The absolute thermoelectric power of the largest tin inclusion was measured by a Koslow TE-3000 thermoelectric instrument. This instrument is a conventional thermoelectric alloy tester used in NDT, that provides relative readings with arbitrary units. Therefore it was first calibrated by materials of known absolute thermoelectric power such as pure copper and standard thermocouple alloys like alumel and chromel as shown in Fig 2.4. in order to get the corresponding thermoelectric power value.

2.4.2 ELIMINATION OF EXTERNAL MAGNETIC SOURCES

In order to better separate the sought magnetic signals of truly thermoelectric origin from potentially much stronger spurious artifacts, we had to adopt a series of protective measures. Since the Earth's static magnetic field is about 50 µT, i.e., orders of magnitude stronger than the fields generated by thermoelectric currents around metallic inclusions, absolute dc measurements are rendered completely useless. In the experiments, we used ac coupling with a high-pass filter of very low cut-off frequency at 0.01 Hz. Since sufficiently fast alternating heating could not be implemented because of the inherently sluggish thermal response of the specimen, all measurements were done under steady-state thermal condition that was achieved in a few minutes after starting the heating and cooling. The pseudo-dynamic magnetic signals required for ac detection were produced by laterally (normal to heat flux) scanning the specimen at a speed of ≈20 mm/s.
<table>
<thead>
<tr>
<th></th>
<th>copper (host)</th>
<th>tin (inclusion)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\sigma$ $[10^6 \text{ A/Vm}]$</td>
<td>59.7</td>
<td>8.31</td>
</tr>
<tr>
<td>$\kappa$ $[\text{VA/m}^0\text{C}]$</td>
<td>399</td>
<td>62.4</td>
</tr>
<tr>
<td>$S$ $[10^{-6} \text{ V/}^0\text{C}]$</td>
<td>1.72</td>
<td>-1.73</td>
</tr>
</tbody>
</table>

Table I  Physical properties of pure copper and tin at room temperature.
Figure 2.4 Relative readings of the absolute thermoelectric power of pure copper, alumel, chromel, and tin as measured by a Koslow TE-3000 thermoelectric instrument.
As we have previously mentioned, the magnetic field is asymmetric to the principal direction of heating, therefore the signal to be detected does not exhibit a significant dc component. In addition to the relatively fast lateral scanning in the “line” direction, we also scanned the specimens at a much lower rate in the axial “frame” direction. In this way, a 76.2 mm × 76.2 mm scan of 200×200 grid points took about 30 minutes. In order to reach the high sensitivity required in these measurements, non-stationary extraneous magnetic fields also have to be rejected. Most of the ac magnetic interference occurs at the 60 Hz mains frequency and its upper harmonics. These signals were effectively eliminated by a very sharp (six-pole) low-pass filter of 20 Hz cut-off frequency. These methods based on the frequency spectra of the spurious magnetic signals cannot be applied to reduce extraneous signals in the very frequency range were the thermoelectric signals are expected, namely between 0.01 Hz and 20 Hz. In this range, we can separate the sought magnetic signals from unwanted background signals based on their spatial rather than temporal dependence. It should be mentioned that, because of the limited bandwidth of the measurement, the scanning velocity (20 mm/s), dimension (76.2 mm), and resolution (200 points/line) can affect the images. For the parameters used, the effective sampling rate of the image digitized is about 50 Hz, and the Nyquist condition is satisfied. Significantly higher or lower scanning speed would shift the spectrum of the signal to be detected above or below, respectively, of the limited detection bandwidth thereby distorting the obtained images. All these measures taken to eliminate the adverse effects of magnetic interference from the surroundings cannot assure that the specimen itself does not produce a spurious magnetic signature caused by ferromagnetic contamination or high susceptibility of the material itself. In order to positively identify magnetic signals of purely thermoelectric origin, we always recorded a “reference” scan without external heating and cooling. In addition, the experimental data were always obtained as the difference between measurements taken at opposite heating directions. As it is indicated in Fig. 2.3, alternating the heating and cooling directions was achieved simply by changing the hot and cold water supplies connected to the heat exchangers and waiting a few minutes until steady-state conditions were reached.
2.4.3 GRADIOMETRIC CONFIGURATION

Generally, the feasibility of self-referencing thermoelectric measurements by noncontact magnetic sensing crucially depends on the sensitivity of the magnetometer. As we mentioned before, as a result of recent technological advances in high-sensitivity magnetic sensors, it is now feasible to achieve the very high sensitivity levels that are necessary in these applications. Of course, the strength of the magnetic field to be detected greatly depends on the physical nature and dimensions of the imperfection, but for modest ($\approx 1-10^\circ$ C/cm) temperature gradients and for thermoelectric power of most metals in the $\pm 10 \mu$V/$^\circ$C range the magnetic flux density is expected to be between 10 pT and 1000 nT.[13-24,27,28]

The experimental results presented in this thesis were obtained by a Bartington Instruments Mag-03 fluxgate that has a noise-limited detection threshold of $\approx 2.5$ pT/$\sqrt{\text{Hz}}$ over a bandwidth of 0.01-20 Hz. In comparison, the measured peak magnetic flux densities varied between 0.5 nT and 250 nT, i.e., the sensitivity of the fluxgate magnetometer was quite sufficient and the incoherent electrical noise produced by the sensor and the preamplifier had no significant effect on the accuracy of the measurements. The signals to be detected are generated in the close vicinity of the magnetometer therefore they significantly vary from point to point, i.e., they exhibit strong gradients. Extraneous signals typically originate at larger distances from the magnetometer, therefore they smoothly vary from point to point, i.e., they exhibit relatively small gradients. In order to exploit this difference, we used a pair of detectors in a gradiometric arrangement. The primary sensor closer to the specimen picks up a much stronger signal from the inclusion than the secondary sensor further away, while the two sensors exhibit essentially the same sensitivity for sources at large distances. The baseline distance ($b$ in Figure 2.3) was chosen to be 28.6 mm in our case (generally, baseline optimization depends on the spatial distribution of the magnetic field to be measured). Further reduction of the baseline distance would improve the rejection of extraneous signals but would also reduce the sensitivity to the thermoelectric signals to be detected.
2.4.4 THERMOELECTRIC BACKGROUND SIGNATURE IN PURE COPPER BARS

Like most other methods used in NDT, the noncontacting thermoelectric method is ultimately limited by temporally coherent material noise rather than temporally incoherent electrical noise that could be easily eliminated by simple time-averaging. Strictly speaking, material “noise” is really unwanted background “signal” that is often called noise only because it interferes with, and often conceals, the flaw signals to be detected. The main sources of such adverse background signals in thermoelectric NDT are macrostructural features such as case hardening, cold work, texture-induced anisotropy, residual stress, etc., while small-scale microstructural features such as grains are less important because of the lack of sufficient spatial resolution.

The peak magnetic flux density of the background signature in pure copper bar stock was ≈ 4 nT, which is actually larger than the signals produced by the smallest tin inclusions used in our experiments. This background signature is due to case hardening and axial texture caused by cold rolling during manufacturing of the bar stock. These effects are much smaller in a bar cut from a larger plate (≈ 1 nT), but can be more or less eliminated (< 0.5 nT) only by appropriate annealing (30 minutes at 700°C in a vacuum furnace) as shown in Fig. 2.5. Since this signature is essentially the same everywhere along the length of the bar, its adverse effect on flaw detection can be significantly reduced by subtraction. The copper specimens used in the following experiments were all cut from a 12.7-mm-thick plate, but instead of the rather troublesome annealing process we chose to simply subtract the reference material signature whenever it reached above 10 % of the signal from the inclusion.
Figure 2.5 Case hardening induced background signature in pure copper bars ($\nabla T \approx 0.5°C/cm$, 2 mm lift-off distance, 3”×3” scanning dimension).
2.5 EXPERIMENTAL RESULTS

2.5.1 COMPARISON BETWEEN EXPERIMENTAL AND THEORETICAL RESULTS

Figure 2.6 shows examples of the magnetic images obtained from 6.35- and 9.53-mm-diameter surface-breaking semi-spherical tin inclusions embedded in copper. These pictures were taken at $g = 2$ mm distance above the surface (see Fig. 2.3). This apparent lift-off distance is the actual gap between the tip of the magnetometer probe and the specimen. However, the sensing element of the fluxgate is an $e = 15$ mm long ferromagnetic rod of $d = 2$ mm diameter centered in an $n = 25$ mm long case. It should be emphasized that, besides its much lower sensitivity with respect to a SQUID magnetometer, a fluxgate detector also suffers from its larger size that adversely affects the absolute accuracy of measurements for fields that vary over the sensor’s volume. The geometric center of the fluxgate is approximately 12.5 mm below the tip of the case, i.e., the 2-mm apparent lift-off corresponds to a much larger 14.5-mm actual lift-off distance. This crude approximation, however, will not be sufficient for the purposes of quantitative comparison to the analytical predictions, therefore later we are going to use a more accurate method. The main goal is only to establish an empirical relation between the peak value and the half-width of the bipolar magnetic signature on one side and the inclusion diameter and lift-off distance on the other side.

The measured magnetic field distributions are similar in shape to the analytical predictions previously shown in Figure. 2.1. As expected, the characteristic bipolar lobes change sign when the direction of the temperature gradient in the specimen is reversed. These lobes get larger and the magnitude of the magnetic flux decreases when the lift-off distance is increased. Figure 2.7 shows how the peak magnetic flux density changes with the apparent lift-off distance between 1 and 8 mm for six inclusions of different diameters between 2.38 mm and 12.7 mm.
Figure 2.6  Magnetic images of surface-breaking semi-spherical tin inclusions on the top of a copper host ($\nabla T \approx 0.7^\circ C/cm$, 2 mm lift-off distance, 76.2 mm $\times$ 76.2 mm scanning dimension, the peak magnetic flux density is indicated for comparison).
Figure 2.7 Comparison between the experimentally measured and theoretically predicted magnetic flux densities plotted as functions of the lift-off distance for surface-breaking tin inclusions in copper (\(\nabla T \approx 0.7^\circ\text{C/cm}\)).
The solid lines represent the analytical predictions based on the material properties listed in Table I. For each inclusion diameter and lift-off distance, the theoretical values were calculated at two sensor positions corresponding to the primary and secondary fluxgates of the gradiometer and the values were subtracted. In most cases, subtraction of the magnetic field measured by the secondary sensor has a negligible effect on the result, but in the case of small inclusions and large lift-off distances the effect can be as high as 15-20%. The lift-off distance was corrected for the depth of the sensing element below the surface of the probe, but no other adjustments were made. Because of the decay of the magnetic field with increasing lift-off distance, the effective center of the sensing element is closer to the surface than its geometrical center. We tried to compensate the resulting inherent underestimation in the analytically predicted magnetic field by best fitting the numerical results to the experimental data using the unknown effective depth of the sensing element as the variable parameter. The least-mean-square fitting process yielded an optimal depth of 11.04 mm, slightly lower than the previously described rough approximation based on the geometrical center of the sensing element, as one would expect. With this reasonable adjustment, the standard deviation between the theoretical predictions and the experimental results is only 6.3% (based on the geometrical center of the sensor, i.e., without any adjustable parameter, the corresponding error is approximately 18%).

Considering the rather crude approximations used in the theoretical model, the inherent uncertainties of the large number of independent material parameters involved in the phenomenon, and the potential experimental errors associated with the measurement, the agreement over a range of two orders of magnitude is surprisingly good, possibly even fortuitous to some degree.

Another parameter that can be readily used to compare the analytical predictions and experimental observations is the half-width of the bipolar magnetic signature, which was defined as half of the lateral separation between the positive and negative peaks. The theoretical curve for the normalized half-width as a function of the normalized lift-off distance was shown previously in Figure 2.2. We simply compared the experimental results to the predicted values
at the level of the primary sensor. Finally, Fig. 2.8 shows the theoretical predictions (solid line) and experimental results (symbols) for the half-width of the magnetic signature as a function of lift-off. There is a good agreement between the theoretical and experimental data except when the normalized lift-off distance exceeds 10.
Figure 2.8  Theoretical predictions (solid line) and experimental results (symbols) for the half-width of the magnetic signature as a function of lift-off.
2.6 SUMMARY

We conducted an experimental investigation of the magnetic field produced by thermoelectric currents around surface-breaking spherical tin inclusions in copper bars under external thermal excitation. The diameter of the inclusions and the lift-off distance varied from 2.38 to 12.7 mm and from 12 to 20 mm, respectively. We enforced a constant 0.7 °C/cm temperature gradient in the specimen. The resulting peak magnetic flux densities ranged from 1 to 250 nT and could be measured by a commercial fluxgate magnetometer. The main goal of this chapter was to verify the recently published theoretical predictions of Ref. 15. The experimental results were found to be in good agreement with the predictions of this model concerning both the peak value and the normalized half-width of the thermoelectric magnetic field. The results clearly indicate that inclusions and other types of imperfections in metals can not only be nondestructively detected by noncontacting magnetic measurements, but can also be characterized using the previously developed analytical model.

The copper specimens used in the experiments were actually cut from a 1/2”-thick plate instead of using commercially available bar stock because we found that the latter produced a substantial background signature that interfered with the accurate measurement on some of the smaller inclusions and even prevented the detection of the smallest ones. This background signature is due to case hardening and axial texture caused by cold rolling during manufacturing of the bar stock. The measured magnetic field in the normal (thickness) direction under axial heating and cooling is produced by variations in material properties along the lateral (width) direction. This variation is much smaller in a bar cut from a larger plate, but can be completely eliminated only by appropriate annealing. Of course spatially incoherent material noise caused by local variations in material properties are much more difficult to eliminate or correct for than the more or less uniform material signature associated with case hardening and texture.
CHAPTER III
ROLE OF ANISOTROPY

3.1 INTRODUCTION

Recently, Nagy and Nayfeh developed an analytical model to predict the magnetic field produced by thermoelectric currents around surface-breaking and subsurface spherical inclusions in a homogeneous host material under external thermal excitation.[15] These predictions were subsequently experimentally verified using surface-breaking spherical tin inclusions of varying diameter in copper.[16] These experiments also revealed that the detectability of small inclusions and subtle imperfections by this novel method is ultimately limited by the intrinsic thermoelectric anisotropy and inhomogeneity of the material to be inspected. The probability of detection (POD) of a given material flaw is determined by the resulting signal-to-noise ratio rather than by the absolute magnitude of the signal itself. With the exceptions of a few special cases, most sensors used in NDE are not limited by temporally incoherent electrical noise but rather by temporally coherent material noise that cannot be eliminated by time-averaging. Material “noise” is really unwanted background “signal” that is called noise only because it interferes with, and often conceals, the flaw signal to be detected. Such material signature is produced by microstructural features (e.g., grains, second phases, or precipitations) and macrostructural features (e.g., heat-affected, work-hardened, strained, and textured regions). Let us consider an example how this material signature limits the detectability of weak imperfections by the thermoelectric method.

The noncontacting thermoelectric method has been recently adapted to the characterization of weak surface and near-surface imperfections caused by localized plastic deformation that is usually very difficult to detect by conventional NDE methods because they are effectively hidden by the accompanying surface roughness.[20] The same approach can also be adopted to the characterization of near-surface material damage on fretted Ti-6Al-4V
specimens. This titanium alloy has very poor wear resistance and is highly susceptible to fretting that is produced when two pieces of the very same material are rubbed against each other. This fretting damage produces subtle changes in the material properties via hardening, localized texture and residual stresses in addition to surface roughness. As an example, Fig. 3.1 shows magnetic scans of two Ti-6Al-4V specimens taken at $\nabla T \approx 13^\circ\text{C/cm}$ temperature gradient for two opposite heating directions with the noncontacting thermoelectric technique. The specimen without fretting damage exhibits a background signature of $\approx 7\ \text{nT}$ peak-to-peak amplitude that is caused by intrinsic anisotropic texture. After substantial fretting damage was induced over a spot of $\approx 1/2''$ diameter at the center (as indicated schematically by the superimposed circle), the magnetic signature essentially doubled to $\approx 14\ \text{nT}$. The main advantage of the thermoelectric method is its sensitivity to the “material” effects (residual stress, local texture, and increased dislocation density) of fretting and its insensitivity to the “geometrical” by-product, i.e., the rough surface topography.[20] However, the detectability of weak imperfections is obviously adversely affected by the presence of the rather strange looking background signature in the intact material, that can be reduced only by annealing above the recrystallization temperature, which would also completely change the existing microstructure. The appearance of the above described background signature in noncontacting thermoelectric inspection is not surprising at all. For example, it has been known for a long time that texture induced anisotropy can lead to significant thermoelectric signals between regions of different orientation.[11,29] Generally, the anisotropy is partly due to partial alignment of the crystallographic orientations of the neighboring grains and partly to morphological features such as preferred orientation of elongated grain boundaries, dislocations, slip bands, etc. It should be mentioned that, in contrast to mechanical properties, thermoelectric properties of cubic materials (aluminum, copper, nickel, steel, etc.) do not exhibit crystallographic anisotropy. The few structural metals of great practical importance that preferentially crystallize in noncubic (hexagonal) symmetry, therefore do exhibit thermoelectric anisotropy, are titanium and its alloys.[30]
Figure 3.1 Magnetic scans of a Ti-6Al-4V specimen before and after fretting ($\nabla T \approx 13^\circ$C/cm temperature gradient, $3" \times 3"$).
Another important effect that might contribute to the thermoelectric material signature is the substantial stress-dependence of the thermoelectric power that could result in significant point-to-point variations as well as orientation dependence when residual or externally induced stresses are present in the material.[31]

The present goal is to study how thermoelectric anisotropy causes the characteristic background signature shown in Fig. 3.1a in order to reliably estimate the POD of certain material flaws. From this point of view, all the above mentioned effects (i.e., hardening, texture, residual stresses) are clearly negative as far as they adversely affect the detectability of flaws. On the other hand, it is also clear that the unique sensitivity of the thermoelectric method to these effects can be readily exploited in the future for the detection and quantitative characterization of these subtle material variations.

3.2 BACKGROUND

3.2.1 ANISOTROPIC TEXTURE

Most solid materials have a polycrystalline structure, which means that they are composed of many crystallites or "grains" of different size, shape and crystallographic orientations. Any preferred orientation of the grains, either crystallographic or morphological, is called texture. Texture can cause anisotropy in the physical properties of a polycrystalline material. Anisotropy means that the material properties depend on the direction in which they are measured. As an example Fig. 3.2 shows the effect of crystallographic anisotropy in a noncubic material. This figure illustrates that the thermoelectric power of zinc is very different when measured parallel and perpendicular to the hexagonal axis.[29]

In general, the exact mechanisms by which texture evolve is not completely understood, although empirical validation is sufficient for many processes in order to take advantage of the
Figure 3.2  Thermoelectric power of zinc parallel and perpendicular to the hexagonal axis (crystallographic anisotropy).
influence of texture on the material properties. In most cases, crystallographic orientation of the
grains can be induced naturally or artificially by special processing, e.g., rolling, forging,
pressing, extruding or drawing. Figure 3.3 shows schematic drawings of (a) a texture-free
material (equiaxed grains with no orientation preference) and (b) a textured material (elongated
grains with orientation preference). It has been reported that the preference of certain
crystallographic orientations (texture) affects the electrical, magnetic and mechanical properties
of the material by as much as 20-50% of their nominal values.[32] Most metals develop a
preferred crystallographic orientation during the rolling process, giving, as a result, an
anisotropic behavior of the rolled material. So the material properties are different, depending
on whether they are measured perpendicular or parallel to the rolling direction.[33] The
influence of the texture on material properties can be exploited in order to design materials with
certain characteristics or behavior, depending on their crystallographic orientation. Therefore
the characterization of texture is of fundamental importance, especially in material technology
and design.

Nowadays, several techniques are available for the analysis of texture in the materials
including, X-ray and neutron diffraction, known as macrotexture techniques. The most common
method to measure texture is by X-ray diffraction. This technique provides information about
the volume fraction of a particular crystallographic orientation in the specimen from the
intensity of X-ray diffraction in certain directions. The evaluation of the texture is given by
conventionally accepted formats that provide the data in a quantitative way, such as pole figures
(see Chapter V “XRD Localized Sub-Surface Texture”). The principle of the pole figure
measurement by diffraction technique is based on Bragg's law for reflection of X-rays at the
crystal lattice planes. It is important to mention that the X-ray technique does not provide
information about how the grains are distributed throughout the sampled volume of the
material. This technique only gives information about the average texture in the sampled
volume.[34]
Figure 3.3  Schematic drawings of (a) a texture-free material and (b) a textured material (crystallographic and morphological anisotropy).
3.3 REVIEW OF THE ANALYTICAL MODEL

In this section we present a briefly summary of the analytical model developed by Nayfeh and Nagy in order to predict the magnetic background signature in homogeneous anisotropic media by using the classical coupled transfer theory of thermoelectricity.[26] For a homogeneous isotropic medium, $\sigma$, $\kappa$, and $S$ are scalar quantities that do not depend directly on the spatial coordinates, though generally they do depend on temperature, that can introduce an indirect spatial variation. For purely thermal excitation in an isotropic medium, the local electrical field $E = -\nabla \Phi$ is strictly parallel to the temperature gradient $\nabla T$ and the electrical current density can be shown to vanish identically everywhere. This means that, regardless of the size, shape, and material properties of a homogeneous isotropic specimen, no thermoelectric current will be generated by any type of heating or cooling. In an anisotropic medium, $\sigma$, $\kappa$, and $S$ are second-order tensor quantities and the local electrical field is not necessarily parallel to the temperature gradient. Though the divergence of the electrical current density $\nabla \cdot j = 0$ is still identically zero everywhere by virtue of Maxwell's theorem, the curl of the electrical current density $\nabla \times j \neq 0$ does not necessarily vanish. Therefore, specimens made of anisotropic materials can produce a nonvanishing thermoelectric current distribution, and an associated nonvanishing magnetic field, even when the material is completely homogeneous. The nonvanishing thermoelectric current distribution in anisotropic media is caused by nonuniform heat flow that can be due to either nonuniform heating and cooling or the irregular shape of the specimen, e.g., a rivet hole that forces the heat flux to go around it.

In order to illustrate the magnetic field produced by the thermoelectric currents due to anisotropy in homogeneous media, we will present some examples of the numerical results obtained by the analytical method described in Refs. 17 and 18. The sought magnetic field can be obtained from Maxwell's equation of $\nabla \times H = j$ by integration. Numerous steps were taken to derive the magnetic field produced by thermoelectric currents in anisotropic materials under two-dimensional directional heating and cooling (these calculations were discussed in
great detail in Refs. 17 and 18). It was found, that a line source along the \( x_3 \) axis of a \((x_1, x_2, x_3)\) cartesian coordinate system produces a magnetic field parallel to the source itself, that can be written as \( H_3 = H_0 F(x_1, x_2) \), where \( H_0 \) is proportional to the heat flow rate \( \dot{Q} \) of the source over a unit length and the degree of thermoelectric anisotropy, and \( F \) is a universal spatial distribution

\[
F(x_1, x_2) = \frac{x_1 x_2}{x_1^2 + x_2^2}.
\]

(3.1)

\( H_0 \) is a fairly complicated function of the material properties, but it does vanish when the degree of anisotropy diminishes, i.e., when \( \sigma, \kappa, \) and \( \varepsilon \) all approach unity (\( \varepsilon \) is a thermoelectric coupling coefficient that can be expressed by the absolute thermoelectric power \( S \) of the material as \( \varepsilon = \sigma S \)).\[17,18\] From Eq. (3.1) it is clear that the magnetic field depends only on the angular coordinate of the point of observation with respect to the principal directions, but not on the distance from the source. In contrast to the electrical current density and heat flux, which are inversely proportional to the distance from the source, the magnetic field produced by an infinite line source exhibits no loss associated with the spreading of the heat. It should be mentioned that \( F(x_1, x_2) \) vanishes on the principal axes \((x_1 = 0 \text{ or } x_2 = 0)\) where there is no skewing between \( \nabla \Phi \) and \( \nabla T \).

### 3.3.1 INFINITE DIPOLE ALONG A PRINCIPAL DIRECTION

In order to illustrate the main features of the analytical predictions for the anisotropic thermoelectric effect in the case of simultaneous heating and cooling, we will consider the case of a dipole consisting of a line source and a sink separated by a unit distance \( m = 1 \) as it is shown in Fig. 3.4. Because of the cancellation effect between the source and the sink, far away from the dipole the magnetic field decreases as the inverse of the distance from the dipole. Now, the principal directions of the anisotropic material \((x_1, x_2)\) are rotated by an arbitrary
angle of $\theta$ with respect to the dipole orientation along the $x_3 = \xi_3$ axis. Using superposition, the magnetic field of the dipole can be written in the physical coordinate system $(\xi_1, \xi_2, \xi_3)$ as

$$H_3 = H_0 F_d(\xi_1, \xi_2) ,$$

where

$$F_d(\xi_1, \xi_2) = F_r(\xi_1, \xi_2 + 0.5) - F_r(\xi_1, \xi_2 - 0.5).$$

Here, $F_r(\xi_1, \xi_2)$ denotes the rotated distribution function of a line source, that can be easily calculated from the previously determined approximate spatial distribution function $F(x_1, x_2)$ by rotation from the material coordinate system $(x_1, x_2)$ into the physical coordinate system $(\xi_1, \xi_2)$, i.e., by substituting $x_1 = \xi_1 \cos \theta + \xi_2 \sin \theta$ and $x_2 = -\xi_1 \sin \theta + \xi_2 \cos \theta$. For simplicity, in Eq. (3.2) all coordinates are normalized to the separation distance $m$ between the source and the sink.

Figures 3.5a and 3.5b show the normalized magnetic field distributions $F_d(\xi_1, \xi_2)$ of the dipole shown in Fig. 3.4 for $\theta = 0^\circ$ and $\theta = 45^\circ$, respectively. When the dipole is aligned with the principal directions of the material ($\theta = 0^\circ$), two large asymmetric lobes of opposite signs appear on the two sides of the dipole direction. Since the main heat flux is along the dipole direction from the source towards the sink, this type of material signature is similar to the lobes produced by inclusions. When the dipole is oriented along the bisector between the principal directions of the material ($\theta = 45^\circ$), both main lobes split into two twin peaks and the distribution becomes symmetric with respect to the dipole direction. This type of material signature is rather unique and quite different from the typical pattern produced by inclusions. As it was mentioned earlier, far away from the dipole the source and the sink increasingly cancel each other and the magnetic field is inversely proportional to distance.
Figure 3.4 A schematic diagram of a dipole consisting of a line source and a sink separated by a unit distance \( m = 1 \).
Figure 3.5 Normalized magnetic field distributions of the dipole shown in Fig. 3.4 in an anisotropic material for (a) $\theta = 0^\circ$ and (b) $\theta = 45^\circ$. 
3.4 EXPERIMENTAL VERIFICATION FOR A COLD-ROLLED TI-6AL-4V PLATE

3.4.1 DETERMINATION OF THE PRINCIPAL MATERIAL DIRECTION BY CONVENTIONAL NDE TECHNIQUES

In order to verify qualitatively the previous described analytical model, we conducted a series of experiments on a 1.9-mm-thick cold-rolled Ti-6Al-4V plate. First, we determined the principal material directions of the plate by ultrasonic velocity measurements in the through-thickness direction. The main effect of texture induced anisotropy on the velocity of ultrasonic waves is to slow it down in one direction and make it faster in another. Shear wave transducers can be used to determine the degree of anisotropy in a material from measurements in one direction via birefringence, which means the refraction of the ultrasonic wave propagating in a given direction into two waves of slightly different velocities (fast and slow shear waves) and mutually orthogonal polarizations ($p_A$ and $p_B$).[35] Figure 3.6 shows the geometrical configuration of birefringence measurements used to determine the principal material directions in the cold-rolled Ti-6Al-4V plate. The $x_1$ and $x_2$ principal directions were chosen to coincide with the polarization directions of the “fast” and “slow” shear waves, respectively, which were found to differ by $\approx 4.6\%$ on the average over the entire surface of the 305-mm-diameter circular disk.

Next, we used an elliptical eddy current coil of 5:1 aspect ratio to determine the principal directions and anisotropy factor of the electrical conductivity in the plate between 30 kHz and 300 kHz. We found that $\sigma = \sigma_1 / \sigma_2 \approx 1.023$, i.e., the degree of anisotropy was approximately 2.3% (the direction of high electrical conductivity was parallel to the polarization direction of the fast through-thickness shear wave).

Finally, the absolute thermoelectric power of the cold-rolled Ti-6Al-4V plate was measured by a calibrated ATS-6044 alloy ThermoSorter (Walker Scientific, Inc.).
instrument is a thermoelectric alloy tester used in NDT, that provides relative readings with arbitrary units, therefore it was first calibrated by materials of known absolute thermoelectric power such as pure copper and standard thermocouple materials like alumel and chromel as shown in Fig. 3.7. Figure 3.8 shows the results of the absolute thermoelectric power measurement at different angles of $\theta$ in the cold-rolled Ti-6Al-4V plate. We found that the absolute thermoelectric power changed from $-5.01 \, \mu V/{}^\circ C$ to $-4.86 \, \mu V/{}^\circ C$ with orientation in the plane of the plate so that $S_1 / S_2 \approx 1.031$ (in our notation then $\varepsilon = \varepsilon_1 / \varepsilon_2 \approx 1.055$). In this case, our limited goal is simply to qualitatively verify the theoretical model by comparing the rather strange spatial distributions of the analytical predictions and experimental observations, therefore the less easily measurable anisotropy factor of the thermal conductivity was not determined at this point.
transducer

shear transducer

cold-rolled Ti-6Al-4V plate

\[ x_1 \cdot x_2 = \text{principal material directions} \]
\[ p_A \cdot p_B = \text{orthogonal polarization directions} \]

Figure 3.6 A schematic representation of determining the principal material directions in the plane of the plate by shear wave birefringence measurements in the through-thickness direction.
Figure 3.7 Relative readings of the absolute thermoelectric power in pure copper, standard thermocouple materials like alumel and chromel and the corresponding value of the cold-rolled Ti-6Al-4V plate measured by an ATS-6044 alloy Thermo-Sorter (Walker Scientific, Inc.).
Figure 3.8  Degree of anisotropy of the thermoelectric power in the cold-rolled Ti-6Al-4V plate.
3.4.2 EXPERIMENTAL ARRANGEMENT

Figure 3.9 shows a schematic diagram of the experimental arrangement used to map the spatial distribution of the magnetic signature. A pair of fluxgate magnetic sensors was utilized in gradiometric arrangement to detect the thermoelectric signals from the specimen. Since the generated magnetic field is perpendicular to both the heat flux in the specimen (parallel to the surface) and the gradient of the material property (tangential to the surface), the magnetometer was polarized in the normal direction. The apparent lift-off distance between the magnetic sensor (differential fluxgate) and the specimen surface was \( g = 2 \) mm. Numerous steps were taken to separate the sought magnetic signal of truly thermoelectric origin from potentially much stronger spurious artifacts (these measures are discussed in great detail in Chapter II “Elimination of External Magnetic Sources”). The experimental data presented in this section is actually the difference between measurements taken at opposite heating directions and then divided by two. As it is indicated in Figure 3.9, alternating the heating and cooling directions was achieved simply by changing the hot and cold water supplies connected to the heat exchangers and waiting a few minutes until steady-state conditions were reached. In order to improve heat conduction from the 12-mm-diameter copper conductor rods to the specimen, a heat conducting silicone compound was applied between them. The centers of the heating and cooling spots were \( m = 50 \) mm apart and the temperatures there were kept constant at 50°C and 10°C, respectively.

Because of its particular importance in interpreting the measured data, we should mention that, in order to eliminate the strong magnetic field of the Earth, we used ac coupling with a high-pass filter of very low cut-off frequency at 0.01 Hz. The pseudo-dynamic magnetic signals required for ac detection were produced by laterally (normal to the main heat flux) scanning the specimen at a speed of \( \approx 20 \) mm/s. In addition to the relatively fast lateral scanning in the “line” direction, we also scanned the specimens at a much lower rate in the axial “frame” direction, i.e., parallel to the main heat flux. In this way, a 203 mm × 203 mm scan of 200×200
grid points took about 45 minutes. Whenever the magnetic field is asymmetric to the direction of heating, the resulting bipolar line signal does not exhibit a significant dc component and will be recorded without substantial distortion. However, if the magnetic field is symmetric to the direction of heating, the resulting unipolar line signal loses its dc component and will be significantly distorted.
Figure 3.9 A schematic diagram of the experimental arrangement used to map the spatial distribution of the magnetic signature in the cold-rolled Ti-6Al-4V plate.
3.5 EXPERIMENTAL RESULTS

3.5.1 COMPARISON BETWEEN EXPERIMENTAL AND THEORETICAL RESULTS

Figures 3.10 - 3.16 shows the comparison between the analytical and experimental results for the two-dimensional distribution of the magnetic signature produced by an anisotropic material at different angles of \( \theta \). In the case of the experimental results the measured peak magnetic flux density is also listed while in the case of the analytical results the peak is always unity. Whenever \( \theta \) is close to either 0° or 90°, i.e., the principal material directions are aligned with the heating/cooling direction, two large asymmetric lobes of opposite signs appear on the two sides and the resulting line scans are bipolar in nature and are therefore well reproduced by the experimental data. However, when the dipole is aligned along the bisectors between the principal directions of the material \( (\theta = 45^\circ) \), both main lobes split into twin peaks and the distribution becomes symmetric with respect to the dipole direction. As we indicated above, these distributions are inherently distorted by the necessity of using ac coupling. This distortion effectively eliminates the average signal in each line scan thereby producing virtual peaks and valleys of opposite sign with respect to the dominating principal features. As a result, the measured distributions exhibit additional secondary bumps not predicted by the theory. In spite of this distortion, the good correlation of the experimental data with the analytical predictions is still quite obvious. Finally, it should be mentioned that the spatial resolution of the experimental images is inherently lower than that of the theoretical predictions. This effect is only partially due to the approximately 3 mm diameter of the fluxgate sensor itself and it is mainly caused by the finite thickness of the plate and the significant \( \approx \)12-mm effective lift-off of the sensors.\textsuperscript{[15]}

Except for the close vicinity of the one-sided heating and cooling areas, the heat flux and electrical current distribution within the thin plate are essentially the same as in the infinite
medium assumed in the analytical model. However, the magnetic field produced by the
thermoelectric currents in a plate of finite thickness should be calculated using the Biot-Savart
integration technique that leads to a loss of lateral resolution comparable to the lift-off distance,
which is actually much larger than the diameter of the sensor.[15] It is probable that the
observed drop in the measured peak magnetic flux density at around $\theta = 45^\circ$ is mainly caused
by this insufficient resolution to capture the sharp twin peaks that occur at this orientation.
Considering the inherently lower resolution and ac distortion of the experimental images, the
agreement with our theoretical predictions is very good and clearly indicates that the suggested
analytical model truthfully captures the main features of the anisotropic magnetic signature of
textured materials.
(a) Theoretical
\[ \theta = 0^\circ \]

(b) Experimental
\[ \theta = 0^\circ \ (\approx 22 \text{ nT}_p) \]

Figure 3.10 Comparison between the (a) analytical and (b) experimental results for the two-dimensional distribution of the magnetic signature produced by an anisotropy material at \( \theta = 0^\circ \). The measured peak magnetic flux density is also listed for the experimental results while in the case of the analytical results the peak is always unity due to normalization.
Figure 3.11 Comparison between the (a) analytical and (b) experimental results for the two-dimensional distribution of the magnetic signature produced by an anisotropic material at $\theta = 15^\circ$. The measured peak magnetic flux density is also listed for the experimental results while in the case of the analytical results the peak is always unity due to normalization.
Figure 3.12  Comparison between the (a) analytical and (b) experimental results for the two-dimensional distribution of the magnetic signature produced by an anisotropic material at $\theta = 30^\circ$. The measured peak magnetic flux density is also listed for the experimental results while in the case of the analytical results the peak is always unity due to normalization.
Figure 3.13  Comparison between the (a) analytical and (b) experimental results for the two-dimensional distribution of the magnetic signature produced by an anisotropic material at $\theta = 45^\circ$. The measured peak magnetic flux density is also listed for the experimental results while in the case of the analytical results the peak is always unity due to normalization.
Figure 3.14  Comparison between the (a) analytical and (b) experimental results for the two-dimensional distribution of the magnetic signature produced by an anisotropic material at $\theta = 60^\circ$. The measured peak magnetic flux density is also listed for the experimental results while in the case of the analytical results the peak is always unity due to normalization.
Figure 3.15  Comparison between the (a) analytical and (b) experimental results for the two-dimensional distribution of the magnetic signature produced by an anisotropic material at $\theta = 75^\circ$. The measured peak magnetic flux density is also listed for the experimental results while in the case of the analytical results the peak is always unity due to normalization.
(a) Theoretical

\[ \theta = 90^\circ \]

(b) Experimental

\[ \theta = 90^\circ \approx 28 \text{ nT}_p \]

Figure 3.16 Comparison between the (a) analytical and (b) experimental results for the two-dimensional distribution of the magnetic signature produced by an anisotropic material at \( \theta = 90^\circ \). The measured peak magnetic flux density is also listed for the experimental results while in the case of the analytical results the peak is always unity due to normalization.
3.6 SUMMARY

This chapter presented an analytical method for calculating the magnetic field produced by thermoelectric currents in anisotropic materials under two-dimensional directional heating and cooling. The results clearly indicate that the earlier observed strange background signatures in textured specimens can be attributed to the thermoelectric anisotropy of the material. Whenever $\theta$ is close to either 0° or 90° (the principal material directions) the thermoelectric currents change in sign. This fact demonstrates that the thermoelectric properties of noncubic materials are different depending on the orientation at which they are measured. In such specimens the best flaw detectability can be achieved by rotating the heating/cooling direction so that the anisotropic effect averages out. Only noncubic materials such as titanium alloys exhibit crystallographic anisotropy, though a much weaker morphological anisotropy can be also exhibited by textured cubic materials. Experimental results from a textured Ti-6Al-4V titanium-alloy plate were shown to be in very good qualitative agreement with the predictions of the analytical model. The results of this study can be used to optimize thermoelectric inspection procedures. Furthermore, they also indicate that noncontacting thermoelectric inspection can be used to characterize the macroscopic texture of materials by evaluating their magnetic signatures under external heating and cooling.
CHAPTER IV
ROLE OF MATERIAL PROPERTY GRADIENTS

4.1 INTRODUCTION

It was recently demonstrated that the noncontacting thermoelectric technique can be used to detect various imperfections in conducting metals, including foreign body inclusions and more subtle local property variations caused by service or manufacturing related effects such as cold work, localized texture, residual stress, excess heat, fatigue damage, fretting, etc.[13-24,27,28,36] As we have discussed earlier, the detection sensitivity of the noncontacting thermoelectric method is ultimately limited by temporally coherent material noise rather than temporally incoherent electrical noise, which could be easily eliminated by simple time averaging. The main sources of such adverse background signals in thermoelectric NDE are macrostructural features such as case hardening, cold work, texture induced anisotropy, residual stress, etc., while small-scale microstructural features such as grains are less important because of the lack of sufficient spatial resolution. For example, in untextured polycrystalline materials, that are spatially incoherent over distances larger than the average grain size, the detection sensitivity is ultimately limited by random grain noise. In many cases, e.g., in textured materials like extruded rods and rolled plates, the structural features are correlated over relatively large distances and, in addition to the spatially incoherent microstructural noise, a spatially coherent macrostructural background signature is also evident.

In Chapter III, we studied the effect of anisotropic texture in a homogeneous material by comparing an analytical model developed by Nayfeh and Nagy, that is capable of quantitatively predicting the resulting thermoelectric signature for simple inspection geometries.[17] In this chapter, the main goal is to experimentally verify an analytical model capable of predicting the thermoelectric background signature caused by weak material inhomogeneity for the simplest and most common inspection geometry, namely, in the case of an axially heated slender
rectangular bar. The analytical method for calculating the magnetic field produced by thermoelectric currents due to such material inhomogeneity was developed by Nayfeh and Faidi.

Of course, real specimens often exhibit both anisotropy and inhomogeneity, therefore the measured magnetic signature is due to a combination of both effects. Whether the actual signature is dominated by anisotropy or inhomogeneity can be established on a case by case basis by comparing the magnetic signatures recorded after rotating the specimen around its principal (length, width, and thickness) axes.[36] Since anisotropic properties are invariant for 180º-rotations, the true source of the magnetic signature can be always established by repeated measurements at different orientations of the bar. This technique will be discussed in more details in the experimental part of this chapter. At this point, it is sufficient to say that in many cases we found that the shape of the background signature essentially remained the same but flipped its sign when the specimen was rotated by 180º around the direction of heat propagation, which clearly indicates that the observed signal cannot originate from the anisotropy of the specimen unless it is also inhomogeneous.[17,18] It will be also shown that this rotational symmetry can be exploited not only to separate anisotropic effects from those of inhomogeneity, but also to further separate the two principal inhomogeneity components in the thickness- and width-directions from each other.

4.2 BACKGROUND

Since the mechanical, electrical and magnetic properties of a polycrystalline material are related to its atomic arrangement, it is not surprising that the material microstructure can be influenced by metal-forming and manufacturing processes. This type of mechanical work process is generally applied after the metal is cast, so it is important to understand that this process interacts with the microstructure and might induce inhomogeneity in the manufactured material. One of the most common metal-forming processes used to reduce material
imperfections due to casting and to change material properties, is rolling. Most metals like titanium develops microstructures in which the grains “rotate” during the rolling process giving, as a result, crystallographic directions that are aligned in the principal rolling direction. This crystallographic alignment can result in anisotropic behavior of the material so that the material properties are different depending on whether they are measured perpendicular or parallel to the rolling direction.[29,32,33]

Figure 4.1 shows a schematic drawing of the metal-forming process that occurs during rolling. This metal working process is used to reduce the thickness of the workpiece by compressing it between two rollers. When this process occurs above the recrystallization temperature of the metal, it is called hot rolling, whereas when the rolling is carried out below this temperature, it is called cold rolling. Figure 4.1 demonstrates how preferred orientation is developed through the effects of the rolling process on the grains of an unprocessed cast material and also shows the difference in microstructure that could be produced by rolling a metal, that depends on various factors such as rolling conditions, the amount of reduction, prior processing and chemical composition of the material, etc. It is well known that rolling conditions are highly significant for texture and texture-dependent properties. Cold rolling increases the dislocation density and alters the shape, but not the average grain size. This process may produce an inhomogeneous texture distribution along the plate width and through the thickness.[37] This material inhomogeneity can hide or reduce dramatically the detection and characterization of subtle imperfections in the material.[16-18,36]
Figure 4.1 A schematic drawing of rolling.
The noncontacting thermoelectric technique loses its ability to detect small and weak defects due to the intrinsic anisotropy and inhomogeneity in the material. As an example, Refs. 16 and 23 compared analytical and experimental results of the magnetic field produced by thermoelectric currents around different surface breaking spherical inclusions under external thermal excitation. The comparison between analytical and experimental results shows a better match on bigger inclusions than on smaller ones. The reason for this difference is the peak magnetic flux density of the background signature in a large plate (≈1 nT) from which the specimens were cut, which is actually larger than the signals produced by the smallest inclusion used in the experiments. Such background signals are due to case hardening and axial texture caused by the manufacturing process the material underwent. In comparison, these effects are much larger (≈4 nT) in a pure copper bar stock, but can be more or less eliminated (< 0.5 nT) by choosing an appropriate annealing condition for the specimen.

From a microstructural point of view, a bar stock is more cold worked at the edges than in the core of the cross-section and so the thermal and electrical conductivities and the thermoelectric coupling coefficients will not be homogeneous throughout the cross-section of the bar. It can be shown that the variation in the material properties due to cold work is significant and can be as large as 10-15% for the electrical conductivity[38] and 1-2% for the thermoelectric coupling coefficients.[39] On the other hand, for a bar cut from a larger plate, the inhomogeneous cold work (case hardening) throughout the plate width will lead to spatially dependent material properties in the bar. Though the spatial variation of the material properties in such bar is smaller than it is in the bar stock, it will still be responsible for the thermoelectric background signal. Due to such spatial dependence of the bar's material properties. Specimens made of such inhomogeneous materials can produce a nonvanishing thermoelectric current distribution and an associated nonvanishing magnetic field even when the material is completely isotropic.[15,17,18,36]
4.3 REVIEW OF THE ANALYTICAL MODEL

In this section we present a summary of the analytical model developed by Nayfeh and Faidi using the classical coupled transfer theory of thermoelectricity, that predicts the magnetic background signature caused by linear inhomogeneity in an isotropic bar.[36] As we mentioned earlier, for a homogeneous isotropic medium, $\sigma$, $\kappa$, and $S$ are scalar quantities that do not depend directly on the spatial coordinates, though generally they do depend on temperature, which can introduce an indirect spatial variation. For purely thermal excitation in a homogeneous isotropic medium, regardless of the size, shape, and material properties of the specimen, no thermoelectric current will be generated by any type of heating or cooling. However, in the presence of material inhomogeneity the thermoelectric current does not necessarily vanish in the specimen, a condition that can be easily detected by external noncontacting magnetic sensors.

4.3.1 LINEAR SPATIAL DEPENDENCE OF THE MATERIAL PROPERTIES

Nayfeh and Faidi considered a slender bar of rectangular cross-section with length $\ell$ much larger than its two other dimensions $w$ and $t$. The bar is aligned with the $z$-direction of a cartesian coordinate system $(x,y,z)$ as illustrated in Fig. 4.2. In the simplest first-order approximation of inhomogeneity, the spatial dependence of the material properties can be assumed to follow linear profiles

\[
\kappa \approx \kappa_0 (1 + a_x x + a_y y + a_z z + \ldots) \ , \tag{4.1a}
\]

\[
\sigma \approx \sigma_0 (1 + b_x x + b_y y + b_z z + \ldots) \ , \tag{4.1b}
\]
Here, the subscripts \((0)\) refer to the average values of the material properties, while \(a\), \(b\) and \(c\) are property gradients characterizing the relative material inhomogeneity. In the case under consideration, material variations along the length of the bar can be neglected partly because the technological effects responsible for the development of linear inhomogeneity (e.g., cold work, residual stress, etc.) do not cause significant axial variations in the material properties and partly because only material variations normal to the direction of heat propagation cause thermoelectric currents. In order to further simplify the analytical model, it will be assumed that property variations exist along the \(y\)-direction only.

\[
S = S_0(1 + c_x x + c_y y + c_z z + \ldots).
\]  

(4.1c)

where the \(y\) subscript is dropped for brevity. Later, experimental evidence will be presented that lateral variations in both thickness- and width-directions of the bar contribute to the observed magnetic signatures. However, this more general two-dimensional inhomogeneity can be easily handled by the one-dimensional model using superposition.
Figure 4.2 The geometrical arrangement of the rectangular bar and the cartesian coordinate system used in the analytical calculations.
4.3.2 MAGNETIC FIELD

Generally, the magnetic field produced by the thermoelectric currents can be calculated using the Biot-Savart law

\[
H(x) = \frac{\int_{-l/2}^{l/2} \int_{-w/2}^{w/2} j(X) \times (x - X) dX dY dZ}{4 \pi |x - X|^3},
\]

(4.3)

where \( x \) and \( X \) are coordinate vectors of the point of observation and the differential volume of the bar, respectively. Numerous steps were taken to derive the magnetic field produced by the thermoelectric currents due to linear inhomogeneity in an isotropic medium under two-dimensional directional heating and cooling (these calculations were discussed in great detail in Ref. 36). Finally, the normal and tangential components of the magnetic field can be calculated by

\[
H_x(x, y) = H_0 \left[ F_x(x + w/2, y, Y)_{t/2}^{l/2} - F_x(x - w/2, y, Y)_{-t/2}^{l/2} \right],
\]

(4.4a)

\[
H_y(x, y) = H_0 \left[ F_y(x + w/2, y, Y)_{t/2}^{l/2} - F_y(x - w/2, y, Y)_{-t/2}^{l/2} \right],
\]

(4.4b)

where, after some algebraic simplifications\(^{[36]}\)

\[
F_x(\xi, y, Y) = \xi y \ln[1 + (\frac{Y - Y}{\xi})^2] + \xi Y + (Y^2 - y^2 + \xi^2) \tan^{-1}[\frac{Y - Y}{\xi}],
\]

(4.5a)
\[ F_y(\xi, y, Y) = \frac{Y^2 - y^2}{2} + \xi^2 \ln[1 + (\frac{y - Y}{\xi})^2] - yY - 2\xi_y \tan^{-1}[\frac{y - Y}{\xi}]. \] (4.5b)

Here,

\[ \xi = x \pm w / 2 \] (4.6a)

and

\[ H_0 = \frac{j_0}{4\pi} = \frac{\sigma_0 S_0 h_0 c}{4\pi \kappa_0} \] (4.6b)
4.4 EXPERIMENT METHOD

In this section, we will describe the experimental setup and procedure used to verify the previously introduced analytical model that predicts the normal and tangential components of the magnetic field produced by thermoelectric currents induced in an inhomogeneous specimen when it is subjected to an external temperature gradient. A Ti-6Al-4V titanium-alloy specimen of length $\ell = 203$ mm, width $w = 12.7$ mm, and thickness $t = 6.35$ mm was cut from a cold-rolled plate as shown in Fig. 4.3. The manufacturing process produced some microstructural material changes including bands of preferentially oriented elongated grains parallel to the rolling direction. Figure 4.4 shows the metallographic pictures taken after sectioning, polishing and etching a specimen from the cold-rolled plate in order to reveal the microstructure at two different orientations (top and side) as shown in Fig. 4.3. The metallographic microstructure clearly exposes the rolling direction along the side-view (Fig 4.4b), in which a preferential orientation of elongated grains is revealed, whereas in the top-view (Fig 4.4a) the metallographic micrograph shows an equiaxed grain structure with no preferential orientation (bimodal condition, consisting of colonies of $\approx 60\%$ equiaxed primary $\alpha$ within $\approx 40\%$ lamellar $\alpha + \beta$).

In order to characterize the material inhomogeneity produced by the cold-rolling process and to verify the analytical model for calculating the normal and tangential magnetic fields produced by thermoelectric currents due to such material inhomogeneity, we decided to further investigate this Ti-6Al-4V specimen. The top, left, bottom, and right sides of the bar were arbitrarily marked as A, B, C, and D, respectively as it is shown in Fig. 4.3. Then, any other orientation can be easily identified by the order of the edges progressing in a counter-clockwise direction starting from the top. For example, CBAD, ADCB, and CDAB correspond to $180^\circ$-rotations around the $x$, $y$, and $z$ axes, respectively.
Figure 4.3  A schematic drawing of the Ti-6Al-4V titanium-alloy specimen that was cut from a cold-rolled plate.
Figure 4.4 Microstructure of the Ti-6Al-4V specimen at 200x. (a) the top-view reveals equiaxed grains and no preferential orientation, while (b) the side-view reveals bands of elongated grains with preferential orientation parallel to the rolling direction.
4.4.1 ELECTRICAL CONDUCTIVITY AND THERMOELECTRIC POWER MEASUREMENTS

In order to establish whether the specimen was strongly inhomogeneous and/or anisotropic, we measured the electrical conductivity and absolute thermoelectric power on the four sides of the specimen. First, we used a US-450 (UniWest Corp.) eddy current instrument with a 1.5-mm-diam probe coil at 1 MHz to measure the electrical conductivity. The system was calibrated on reference materials of know electrical conductivity, 100 readings were taken on each side and then averaged to get a representative value. The measured data are shown in Fig. 4.5 as a histogram of the probability distributions for four sides (A, B, C & D) in the rectangular Ti-6Al-4V specimen. On sides A (0.587 MS) and C (0.590 MS) the electrical conductivity was found to be significantly higher than on sides B (0.572 MS) and D (0.570 MS). The ≈3% difference between sides A&C versus sides B&D was well above the standard deviation (0.003 MS or 0.5%), which indicates the presence of significant anisotropy in electrical conductivity. On the other hand, the small difference between sides A and C or sides B and D was less than the standard deviation, therefore we can conclude that conventional eddy current inspection could not unequivocally verify the presence of a significant inhomogeneity in electrical conductivity.

Secondly, we measured the absolute thermoelectric power with a Koslow TE-3000 alloy sorter (Koslow Scientific Company) in an attempt to establish the presence of thermoelectric anisotropy and/or inhomogeneity. Again, the equipment was calibrated on reference materials of know absolute thermoelectric power and 100 readings were taken on each side of the Ti-6Al-4V specimen and then averaged to get a representative value. The measured data are shown in Fig. 4.6 as a histogram of the probability distributions for four sides (A, B, C & D) in the rectangular Ti-6Al-4V specimen. On sides A (-5.09 µV/°C) and C (-5.11 µV/°C) the thermoelectric power was found to be of significantly higher magnitude than on sides B (-4.69 µV/°C) and D (-4.72 µV/°C).
Figure 4.5  Electrical conductivity probability distributions for the rectangular Ti-6Al-4V specimen.
The ≈ 8% difference between sides A&C versus sides B&D was again above the standard deviation (0.15 μV/°C or 3%), which indicates the presence of a significant anisotropy in thermoelectric power. As before, the small difference between sides A and C or sides B and D was less than the standard deviation, therefore we can conclude that conventional thermoelectric inspection could not unequivocally verify the presence of significant thermoelectric inhomogeneity in the specimen.

4.4.2 TWO DIMENSIONAL INHOMOGENEITY OF THE SPECIMEN

In spite of the apparently stronger effect of anisotropy, the thermoelectric background signature of a slender bar could be still dominated by the inhomogeneous contribution because the heat flux is forced to be parallel to the axis of the bar. This could be easily verified by rotating the bar around its longitudinal (z) axis by 180°, upon which the signature should not change at all if it were caused by anisotropy. We found that the signature of the specimen was essentially reversed upon such rotation, therefore it was primarily due to inhomogeneity. When the specimen was rotated around the transverse (x and y) axes, the observed change in the signature depended on how the 180° rotation was executed. The obvious reason for this is that, in contrast with the simple analytical model, the specimen exhibited property gradients in both width- and thickness-directions. Figure 4.7 illustrates the true two-dimensional inhomogeneity of the specimen (a) and how addition and subtraction of different profiles can be used to eliminate either the (b) width- or the (c) thickness-component of the inhomogeneity.
Figure 4.6  Thermoelectric power probability distributions for the rectangular Ti-6Al-4V specimen.
Figure 4.7 A schematic drawing of the two-dimensional inhomogeneity of the specimen (a) and how addition and subtraction of different profiles can be used to eliminate either the (b) width- or the (c) thickness-component of the inhomogeneity.
4.4.3 EXPERIMENTAL ARRANGEMENT

Figure 4.8 shows a schematic diagram for the experimental arrangement used to study the different thermoelectric signatures produced by the bar. The fluxgate magnetometer can be polarized either tangential or normal to the top surface in order to measure the \(x\) and \(y\) components of the magnetic field, respectively. The specimen was mounted on two copper supports that also acted as heat exchangers to facilitate efficient heating and cooling and the whole assembly was mounted on a non-magnetic translation table for scanning. In order to improve the heat transfer, a layer of heat conducting silicone grease compound was applied between the specimen and the copper heat exchangers, which were heated and cooled to temperatures of 85°C and 5°C, respectively. The actual temperature of the Ti-6Al-4V specimen was monitored by thermocouples at two points and the temperature gradient along the center part of the bar was kept at 4.5°C/cm in all the measurements.

A pair of fluxgate sensors configured in a differential arrangement was used to detect the thermoelectric signals from the specimen. The primary sensor, which is closer to the specimen, measures a stronger signal than the secondary sensor, while the two sensors exhibit essentially the same sensitivity for external sources at larger distances, which are rejected accordingly. In each case the so called lift-off distance, i.e., the gap between the plastic case of the primary sensor and the surface of the specimen was \(g = 2\) mm. Because of the differential experimental arrangement, the magnetic field must be calculated from the analytical model at the positions of both the primary and the secondary sensors and then subtracted. However, when comparing the experimental results to the model calculations, the actual distance between the probes and the surface of the specimen should be used. The sensing element of the fluxgate is an \(e=15\)-mm-long ferromagnetic rod of \(d=2\) mm diameter inside an \(n = 25\)-mm-long protective case. For more details about the differential fluxgate sensor configuration (see chapter II “Gradiometric Configuration”).
The specimen was scanned with both normal and tangential sensor polarizations. The total distance between the effective center of the sensor and the surface of the specimen were respectively $g_{pn} = 13$ mm for the primary fluxgate and $g_{sn} = 41$ mm for the secondary fluxgate at normal polarization and $g_{pt} = 6$ mm and $g_{st} = 14$ mm for tangential polarization. The magnetic signals were detected by scanning horizontally the specimen at the center of the bar in a direction normal to the heat flux. Fig. 4.8 shows also how the normal and tangential magnetic signatures can be recorded when scanning in the “width-direction” of the specimen. Since the translation table can move only horizontally, but not vertically, similar scans in the thickness-direction of the specimen were taken by rotating the specimen 90° around its axis and laying it on its thinner side. As we described above, in order to separate the “width” and “thickness” components of the inhomogeneity, we have to record the magnetic signature at four specimen orientations. Altogether, sixteen independent signatures can be measured using eight specimen orientations and two sensor polarizations. However, all magnetic signatures were recorded with both positive and negative temperature gradient and then subtracted to separate the truly thermoelectric component from spurious contributions of external sources. Therefore, the experimental data to be presented later represent thirty-two measurements. It should be mentioned that numerous additional measures were taken to assure that the magnetic signatures were recorded with minimal distortion (these measures were described in detail in Chapter II “Elimination of External Magnetic Sources”).
Figure 4.8 Schematic diagram for the experimental arrangement for the characterization of the width and thickness inhomogeneities in a Ti-6Al-4V rectangular bar.
4.5 EXPERIMENTAL RESULTS

In order to compare the measured spatial distribution of the magnetic field due to the assumed inhomogeneity profile, we used the known geometrical dimensions of the specimen. The relevant physical parameters of Ti-6Al-4V that are needed for substitution into the analytical model are $\kappa_0 = 7.3$ W/m°C, $\sigma_0 = 5.7 \times 10^5$ A/mV, and $S_0 = -4.9$ µV/°C. Two free parameters, that measure the inhomogeneity in the width $c_x$ and thickness $c_y$ directions, were used to find the best fit between the model predictions and the four experimentally determined signatures for each case. The average of the four best fitting values of $c_x$ can be considered as the best estimate for the actual degree of inhomogeneity in the width-direction, while the same is true for the average of the our best fitting values of $c_y$ and the inhomogeneity in the thickness-direction of the specimen. Though these values cannot be independently verified at this point, the fact that the four values are reasonably close to each other in both cases lends some confidence to the obtained values.

4.5.1 COMPARISON BETWEEN EXPERIMENTAL AND THEORETICAL RESULTS

Figure 4.9 shows the comparison between the experimental and theoretical thermoelectric magnetic signatures (thickness-inhomogeneity, width-scan). The width-signature at normal polarization (Fig. 4.9a) corresponds to $H_y(x, y_{pn}) - H_y(x, y_{sn})$, where $y_{pn} = g_{pn}$ + $t/2$ and $y_{sn} = g_{sn}$ + $t/2$ are the constant distances of the primary and secondary fluxgates from the $x$-axis at normal polarization. It was noticed that the relatively sharp peaks on the theoretical curves were always somewhat blurred on the experimental results by the finite size of the active element that reduces the spatial resolution of the measurement. Therefore, the theoretical curves were smoothened by averaging over a distance equal to the size of the fluxgate ($\pm 1$ mm for normal polarization and $\pm 7.5$ mm for tangential polarization) in order to simulate the reduced
resolution of the experimental data. With this correction, the agreement between the shapes of the theoretical and experimental signatures is very good. The width-signature at tangential polarization (Fig. 4.9b) corresponds to \( H_x(x, y_{tp}) = H_x(x, y_{ts}) \), where \( y_{tp} = g_{tp} + t/2 \) and \( y_{ts} = g_{ts} + t/2 \) are the constant distances of the primary and secondary fluxgates from the x-axis at tangential polarization. In a similar way, Figure 4.10 shows the comparison between the experimental and theoretical thermoelectric magnetic signatures for thickness-inhomogeneity and scanning in the thickness-direction. It should be emphasized that the very good qualitative agreement between the experimental and theoretical data shown in Figs. 4.9 and 4.10 is partially due to the fact that each curve was individually best fitted by changing the thickness-inhomogeneity parameter \( c_y \) (these values are listed on each figure). The average of the four \( c_y \) values was 22.1 m\(^{-1}\), with a standard deviation of 2.7 m\(^{-1}\), which indicates that the accuracy of the thickness-inhomogeneity assessment is \( \approx \pm 12\% \).

Using the symmetry relationships shown in Fig. 4.7, the width-inhomogeneity of the specimen can be evaluated separately. Figures 4.11 and 4.12 show the comparison between the experimental and theoretical thermoelectric magnetic signatures caused by width-inhomogeneity for scanning along the width- and thickness-directions, respectively. The average value of the four \( c_x \) values was 3.4 m\(^{-1}\), with a standard deviation of 0.4 m\(^{-1}\), which indicates that the accuracy of the width-inhomogeneity assessment is also \( \approx \pm 12\% \). Considering the rather crude approximations used in the theoretical calculations to account for the finite size of the sensor elements, the fact that the secondary sensor is partially shielded by the primary sensor, and the experimental errors associated with the measurement, the agreement between the experimental and theoretical thermoelectric magnetic signatures is surprisingly good. These results verify that the observed magnetic signatures are indeed caused by a linear thermoelectric current distribution of the form \( j_z = j_{0x} x + j_{0y} y \) in the slender bar. However, the magnitude of the observed signatures raises serious doubts about the actual source of the thermoelectric currents.
Figure 4.9 Comparison between the experimental and theoretical thermoelectric magnetic signatures (thickness-inhomogeneity, width-scan).
Figure 4.10  Comparison between the experimental and theoretical thermoelectric magnetic signatures (thickness-inhomogeneity, thickness-scan).
Figure 4.11 Comparison between the experimental and theoretical thermoelectric magnetic signatures (width-inhomogeneity, width-scan).
Figure 4.12 Comparison between the experimental and theoretical thermoelectric magnetic signatures (width-inhomogeneity, thickness-scan).
The $c_x w = 0.043$ and $c_y t = 0.14$ products represent our estimates for the total relative variation of the thermoelectric power through the width and thickness of the slender bar, respectively. These numbers are obviously far too large and cannot be reconciled with the independent experimental measurements quoted earlier.

4.6 SUMMARY

This chapter presented an analytical method developed by Nayfeh and Faidi for calculating the normal and tangential magnetic fields produced by thermoelectric currents due to the presence of weak inhomogeneity in a slender rectangular bar under axial heating and cooling.\cite{36} Experimental results from a Ti-6Al-4V titanium alloy bar were shown to be in very good qualitative agreement with the predictions of the analytical model. These results clearly indicate that the observed background signatures on the four sides of the specimen can be attributed to a linear thermoelectric current distribution in the cross-section of the slender bar. However, the high magnitude of the observed signatures essentially excludes the possibility that the actual source of these thermoelectric currents is simply a minor inhomogeneity of the otherwise isotropic material. Even without positively identifying the physical source of the thermoelectric signature, we can conclude that in such specimens the best flaw detectability can be achieved by rotating the specimen and measuring both normal and tangential magnetic signatures so that even arbitrary two-dimensional inhomogeneity could be averaged out. The results of this study can be used to optimize thermoelectric inspection procedures for flaw detection as well as to develop techniques capable of quantitatively assessing the thermoelectric inhomogeneity of metals. It is clear that thermoelectric NDE methods offer a unique opportunity to evaluate otherwise hidden variations in material properties related to fatigue, plasticity, hardness, or the presence of residual stresses. This unique sensitivity originates from the inherent susceptibility of the underlying physical phenomenon, i.e., differential electron diffusion induced by the presence of a temperature gradient, to subtle variations occurring at the
atomic level. Unfortunately, this very origin might very well result in spurious sensitivity to intrinsic variations that have no or negligible effect on the macroscopic mechanical behavior of the material, therefore represent unwanted material noise in the measurements. Further investigation of the underlying physical phenomenon could clarify the relationship between the strength of the thermoelectric signature and the microstructural and chemical features that affect the thermoelectric behavior of the material, thereby facilitating the wider application of this promising NDE method.
CHAPTER V
EVALUATION OF RESIDUAL STRESS IN SHOT-PEENED METALS

5.1 INTRODUCTION

Nondestructive evaluation (NDE) of the existing residual stress in the shallow subsurface layer of shot- and shock-peened components could be very beneficial during manufacturing to monitor and minimize process variations. Even more importantly, NDE is absolutely necessary after extended service if residual stresses were to be taken credit for in fatigue life predictions of critical components because of the very significant and highly variable level of stress release that might occur at elevated operating temperatures. However the nondestructive evaluation of the prevailing compressive residual stress in the shallow subsurface layer is complicated by the adverse effects of shot peening, such as surface roughness and cold work that manifests itself through increased dislocation density and localized texture.

Currently, the only reliable NDE method for residual stress assessment is based on X-ray diffraction measurement that is limited to an extremely thin (≈ 1 mil) surface layer, which is approximately one order of magnitude less than the typical penetration depth of compressive residual stresses produced by shot peening. It has been found that conventional ultrasonic and eddy current NDE methods are simply too sensitive to surface roughness to quantitatively assess the relatively weak variations in mechanical and electrical properties that are caused by the near-surface compressive residual stresses in shot-peened components. On the other hand, it is well known that thermoelectric techniques are very unique among all other NDE methods in that they are solely sensitive to intrinsic material variations regardless of the size, shape, and surface topography of the specimen to be tested. It is also well-known that the absolute thermoelectric power of metals depends on stress.\textsuperscript{[7,31,40,41]} This chapter presents a noncontacting thermoelectric method that can be used to characterize the prevailing residual
stress in shot-peened specimens. This novel method is based on magnetic detection of local thermoelectric currents in the compressed near-surface layer of metals produced by shot-peening when a temperature gradient is established throughout the specimen. Besides the primary residual stress effect, the thermoelectric method is also sensitive to the secondary “material” effects of shot peening (local texture, increased dislocation density, hardening), but it is entirely insensitive to its “geometrical” by-product, i.e., the rough surface topography. With this notable advantage over conventional NDE methods, the noncontacting thermoelectric technique is a step forward towards the characterization of subsurface residual stresses produced by surface treatments.

5.2 BACKGROUND

It is well known that surface properties play a major role in determining the overall performance and, in particular, the fatigue resistance of structural components. Shot peening, one of the most popular surface improvement methods, induces compressive stresses in the surface layers of metallic parts via bombarding the surface with a stream of high-velocity shots as shown schematically in Fig. 5.1. As the plastically deformed surface layer tries to expand relative to the intact interior of the specimen, residual compressive stress develops parallel to the surface at shallow depths, while beneath this layer a reaction-induced tensile stress results. Generally, the compressive stress at the surface may be several times greater than the subsurface tensile stress. This near-surface compressive stress offsets any service-imposed tensile stress, retards fatigue crack nucleation and growth, and ultimately extends the fatigue life of the part. Figure 5.2 shows a comparison between peened and unpeened 7075 aluminum specimens on the basis of average life. [42] Shot peening can enhance fatigue life by as much as ten-fold and increase the endurance limit, i.e., the stress below which fatigue damage accumulation does not occur, by ≈20%. As Fig. 5.1 illustrates, in addition to the primary residual stress effect, shot peening also causes an adverse geometrical side effect by roughening
the surface and certain relatively subtle variations in material properties, such as increased hardness and texture, that are consequences of the significant plastic deformation through cold work. Figure 5.3 shows a typical profile of the sub-surface residual stress produced by shot-peening and its most important parameters.

In the rare cases when full credit is taken in the design process for the imparted near-surface residual stress, very expensive and time-consuming manufacturing controls must be put in place to ensure that the part is properly peened. Nondestructive evaluation of the specimen would often be desirable to verify the actual level of residual stress, though the process itself is simple and reliable enough so that, under well-controlled conditions, there could be little doubt that the protective surface layer of compressive residual stress is there. Still, manufacturers and users often do not take credit for shot peening because there is a fear that even properly peened parts might lose their protection via unpredictable stress release during service if the component is exposed to high temperatures. NDE of residual stress could eliminate these concerns and would permit the user to take full advantage of shot peening. The potential increase in endurance limit and fatigue life are comparable to the advantages that can be gained only through the development of new alloys, thus the value of a reliable nondestructive residual stress assessment method can be compared to the cost of new alloy development.
Figure 5.1  A schematic diagram of shot peening and its three major effects.
Figure 5.2 The effect of shot peening on fatigue life in 7075 aluminum.
Figure 5.3  Typical residual stress profile produced by shot peening and its most important parameters.

\[ d_0 = \text{nominal penetration depth} \]
\[ C_{max} = \text{maximum compressive stress} \]
\[ T_{max} = \text{maximum tensile stress} \]
5.3 STATE-OF-THE-ART

Shot peening can be reliably controlled and optimized by measuring the subsurface residual stress distributions produced by the surface treatment. The stress distribution produced by shot peening depends upon the properties of the material being shot-peened, prior processing, and the specific peening parameters used during the surface treatment. Residual stress analysis by nondestructive methods is a highly developed field using a great variety of different physical principles, ranging from radiography to ultrasonics, electromagnetism, and ferromagnetism, to assess the absolute level and relative distribution of elastic stresses prevailing in the material.[43] The present research is necessary because none of the existing methods can reliably and accurately measure residual stresses in the shallow layer below shot-peened surfaces. The most advanced techniques for measuring residual stress in crystalline materials are based on X-ray and neutron diffraction, that measure changes in atomic interplanar spacing to determine the magnitude of the prevailing elastic strain (stress). Neutrons can penetrate many millimeters into most engineering materials, while X-rays are typically absorbed within a surface layer of 5-20 µm. In comparison, the crucial compressive part of typical stress profiles ranges from 50 µm to 500 µm. Therefore, residual stress assessment by X-ray diffraction (XRD) is nondestructive only within a very shallow surface layer.

Figure 5.4 shows characteristic residual stress profiles in shot-peened 7075-T351 aluminum for different Almen intensities as measured by the XRD method.[44] The increasing peening intensity affects mainly the penetration depth of the compressive stress, but barely has any noticeable effect on the maximum stress level. It is interesting to note that, due to work hardening at the surface, the peak residual stress, which is always slightly below the actual yield strength of the material, is somewhat higher than the original yield strength of the material before shot peening (≈ 48 ksi). To obtain such profiles, successive layers are removed, usually through etching or electropolishing, i.e., in a destructive manner. The removal of material also alters the stress field, and thus requires theoretical corrections of the measured values.
Furthermore, since the method probes only the surface, the results can be easily skewed by spurious effects in the extremely shallow top layer. In spite of the troublesome and destructive sectioning required by the low penetration depth, XRD is probably the most accurate and reliable method for residual stress assessment in surface-treated metals. One of the main reasons for this is that XRD methods are not significantly influenced by additional variations in material properties such as hardness, plastic strain, or texture.

In recent years, several candidate methods for nondestructive stress evaluation were identified, including neutron diffraction, magnetic methods (Barkhausen noise and magnetostriction), thermal methods, ultrasonics, and eddy current methods. As we mentioned above, the neutron diffraction method is based on the same physical principle as the XRD method, but the penetration depth of neutron beams, depending on the material, could be as high as a few centimeters, i.e., approximately three orders of magnitude larger than that of X-rays. In contrast to the common XRD method, neutron diffraction can be used to map the residual stress profile below shot-peened surfaces in a truly nondestructive way with a depth resolution of ≈100 µm and accuracy of ≈50 MPa and it can be used to measure the average compressive stress with an even better accuracy.[43] Among the biggest obstacles to employing neutron diffraction in nondestructive inspection are the lack of availability of appropriate neutron sources, the scarcity of “beam time,” bulkiness, very high expense, and hazards associated with neutron beam sources. On the other hand, magnetic methods are applicable only to ferromagnetic materials, which excludes most engineering materials of interest to the aerospace industry such as aluminum, titanium, stainless steel, and nickel-base superalloys. For many years, eddy current and ultrasonic methods have been considered to be the most promising NDE candidates. However, one important disadvantage of shot peening is that it makes the surface of the specimen rough, which not only negates some of the positive effects of compressive residual stresses via unwanted stress concentrations, but also prevents the accurate assessment of the prevailing stresses.
Figure 5.4 Typical residual stress profiles in shot-peened 7075-T351 aluminum specimens of different Almen intensities as measured by X-ray diffraction.
In ductile materials such as copper, the root-mean-square (rms) surface roughness and the correlation length typically increase from 2 µm to 10 µm and from 50 µm to 200 µm, respectively, as the peening intensity increases from Almen 2 to Almen 16. As a general rule, the penetration depth of the compressive residual stress is roughly three times the correlation length of the spurious surface roughness. In most cases of practical importance this unwanted but inherent surface roughness is the primary reason why conventional NDE techniques cannot be exploited for residual stress assessment in shot-peened specimens.

For a long time, the characteristic dependence of ultrasonic velocity on stress has been thought to be very promising for residual stress measurements in materials,[45-47] though these expectations have remained largely unfulfilled as far as shot-peened specimens are concerned. The main problem in the practical utilization of most ultrasonic techniques is the difficulty of separating the effects of stress on the surface wave velocity from those of surface roughness [48-50] and from those of cold work such as localized texture[51] and increased dislocation density.[52] Although a few methods have been proposed to separate texture and residual stress effects,[53-56] these methods are limited to finding a difference between principal stresses in the surface plane of the sample. However, the residual stresses created by shot peening are the same in every direction in the plane of the surface, so these methods would not be helpful in determining their absolute value. To assess the sought residual stress, each factor must be identified and separated from the overall measurement. Unfortunately, among all the different effects of shot peening, the primary residual stress effect is often the least persistent and the first to disappear, or at least substantially decay, under normal operation conditions. Some of the secondary material effects might persist much longer and can be completely eliminated only by full recrystallization, which usually cannot occur even under the most severe operational conditions. Finally, the most adverse side effect of shot peening, namely, the rough surface topography, is the most persistent of all. It is unaffected at best or even exacerbated by extended service and cannot be eliminated without mechanically polishing the surface.
Theoretically, dispersion of Rayleigh-type surface waves in the ultrasonic frequency range could be exploited to characterize the residual stress distribution below shot-peened surfaces. However, in most materials the stress dependence of the ultrasonic velocity (the so-called acoustoelastic effect) is rather weak and the relative velocity increase under bi-axial compressive stress is less than 0.5% even at levels equal to the yield strength of the material. At the same time, for corresponding values of surface roughness and correlation length, the relative drop of the surface wave velocity is typically 2-3 times larger. Correction for this much larger and opposite effect of surface roughness is essentially impossible, since the ratio between the residual stress-induced velocity increase and the surface roughness-induced velocity decrease becomes worse and worse during service as a result of thermally induced stress release. In addition, near-service localized texture will also decrease the surface velocity at increasing inspection frequencies, which further complicates the problem.

During the last few years, researchers at UTRC conducted an extensive feasibility study to establish whether the stress-depth profile in shot-peened specimens can be determined from the frequency dependence of the measured surface wave velocity as previously suggested in the literature.[44,57] This study has shown that the residual stress effects are usually entirely overshadowed by the surface roughness-induced dispersion and, to a lesser degree, by texture. For example, in smooth 7075-T351 Aluminum specimens, the surface wave velocity was found to increase approximately 0.5% under a maximum external compressive stress equal to the yield strength of the material, but the surface wave velocity actually measured on shot-peened specimens always decreased with increasing peening intensity. What is more, the velocity difference between low-stress-ground and shot-peened parts of the same specimen was essentially unaffected by annealing, which clearly indicates that the observed dispersion is dominated by surface roughness scattering. These negative results were recently confirmed by noncontacting laser ultrasonic methods working in a very wide frequency range as well.[58]

Eddy current conductivity measurements were found to suffer from essentially the same limitation. This is not surprising since eddy currents exhibit similar “surface hugging” behavior
as Rayleigh-type surface waves (though the penetration depth is inversely proportional to the square root of frequency rather than to frequency as in the case of surface waves). The pressure variation of electrical conductivity was the focus of intensive research as early as in the late 1930s [59] and was more recently suggested as a practical solution to the residual stress assessment problem. [60] In paramagnetic materials, such as aluminum and titanium alloys, the electrical conductivity is known to increase by approximately 1% under a maximum compressive stress equal to the yield strength of the material, i.e., by roughly twice as much as the previously discussed surface wave velocity. However, the electrical conductivity measured on shot-peened specimens typically decreases with increasing peening intensity, often as much as 2-3%. [57,61] Again, the relative conductivity difference between low-stress-ground and shot-peened parts of the same specimen is essentially unaffected by annealing, which clearly indicates that the observed phenomenon is due mainly to surface roughness, which increases the path length of surface-hugging eddy currents.

The spurious surface roughness effect, produced by the cold work in shot-peened parts, renders both conventional residual stress characterization methods, that have been found to be quite effective on smooth surfaces, essentially useless on shot-peened surfaces. Furthermore, because of the diminishing contribution of the slowly fading residual stress effect during long-term service, any NDE method that is expected to reliably characterize the remaining residual stress must be entirely insensitive to the persistent surface roughness effect. Therefore, we proposed the development of a novel noncontacting thermoelectric method based on magnetic detection of local thermoelectric currents in metals when a temperature gradient is established throughout the specimen. In the following we present experimental evidence that suggests that this method can detect and quantitatively assess the weighted average of the compressive residual stress within the shallow surface layer of shot-peened specimens.
5.4 **EFFECT OF COLD WORK AND STRESS-DEPENDENCE ON THE THERMOELECTRIC POWER**

It is well known that when a material is cold worked by any means (surface treatment, rolled, forged, extruded, pressed etc.), its properties will change, e.g., hardness and electrical resistivity increase with the degree of cold work while ductility decreases. These changes in the material properties depend on several factors such as the degree of cold work, prior manufactured process, chemical composition, etc. From this point of view, it will be very interesting to find some information about the relation between the thermoelectric power and the corresponding change in material properties that in the particular case of the shot peening will be the residual stress and the cold work induced localized texture and hardness. In the case of copper, some information is already available in the scientific literature on both the stress-dependence of the thermoelectric power and the effect of cold work.

Figure 5.5a shows the absolute thermoelectric power of copper at different stress levels. Except at very low temperatures, the thermoelectric power is linearly proportional to temperature and, at room temperature its value is $\approx 1.7 \mu V/°C$. Figure 5.5b shows the thermoelectric voltage produced between junctions at $T_h = 100 °C$ and $T_c = 0 °C$ at different hydrostatic stress levels. These curves were obtained by Bundy\(^{[31]}\) who measured the thermoelectric voltage of a thermocouple formed between two identical wires when one of them was subjected to compressive pressure, a technique originally developed by Bridgman\(^{[40]}\).

In relative terms, the stress-dependence of the thermoelectric power is similar to those of the ultrasonic velocity and electrical conductivity as they all change about 1% when the stress approaches the yield strength of the material. However, this particular physical property of the material, i.e., the thermoelectric power, can be easily measured without any interference from geometrical variations such as surface roughness, therefore lends itself much easier to nondestructive assessment of near-surface residual stresses in shot-peened metals.
As for the effect of cold work on the thermoelectric power of copper, Kropschot and Blatt measured the thermoelectric voltage produced by a thermocouple made from the same cold-drawn pure copper wire after annealing one leg to eliminate the existing texture and hardening.\cite{39} Because of the thin diameter of the wire, there cannot be significant residual stresses present even in the cold-drawn wire. Figure 5.6 shows the thermoelectric voltage as a function of the hot junction temperature when the cold junction was kept at $T_c = -269 \, ^\circ C$. For our purposes, the important part is the slope of the curve at room temperature, which is about 0.02 $\mu V/^\circ C$ and represents roughly 1% relative variation with respect the absolute thermoelectric power of pure copper. This result indicates that extreme levels of cold work found in thin cold-drawn wires could produce variations in the thermoelectric power comparable to those produced by high residual stresses around the yield point of the material. However, shot peening usually results in lower levels of cold work than cold-drawing of wires, therefore the relative roles of these competing mechanisms should be established on typical shot-peened specimens.
Figure 5.5  The absolute thermoelectric power of copper (a) in a wide temperature range and the thermoelectric voltage (b) between junctions at $T_h = 100 \, ^\circ C$ and $T_c = 0 \, ^\circ C$ for different hydrostatic stress levels.
Figure 5.6  Thermoelectric voltage produced by a thermocouple made of annealed and cold-drawn pure copper wires (the cold junction was kept at $T_c = -269^\circ C$).
5.5 THERMOELECTRIC DETECTION OF PLASTIC DEFORMATION

In spite of the relatively strong stress-dependence of the thermoelectric power of most metals, the main advantage of the proposed nondestructive residual stress assessment technique is not its high absolute sensitivity to the parameter (stress) to be measured but rather its uniquely low sensitivity to surface topography, the very artifact that renders conventional NDE methods essentially useless in the characterization of shot-peened specimens. So the crucial question arises whether mere plastic deformation of the material can produce a perceivable thermoelectric contrast with respect to the surrounding intact host.

To answer this question, Figure 5.7 compares the magnetic scans of two apparently similar 0.375"-diameter surface holes in copper. Figure 5.7a corresponds to a semi-spherical hole produced by low-stress milling which is expected to generate only negligible hardening and residual stress below the machined surface. In this case, no detectable magnetic field was observed from a 2-mm lift-off distance at $\nabla T \approx 0.5^\circ\text{C/cm}$ temperature gradient. In comparison, Figure 5.7b shows the results from an otherwise similar semi-spherical indentation produced by pressing a stainless steel ball into the material in a manner that simulates a single impact during shot peening. As a result of plastic deformation, the surrounding material below and around the indentation is substantially hardened and supports significant residual stresses. The resulting peak magnetic flux was measured at $\approx 35$ nT, i.e., approximately 60 dB above the noise-limited detection threshold of the fluxgate magnetometer used in these experiments. The main concern in connection with shot-peened surfaces is not necessarily the presence, degree, and penetration depth of the produced residual stress directly after surface treatment, but rather whether the stress have been significantly reduced or even eliminated by thermally induced stress release after a long time spent in service. In order to demonstrate that the proposed thermoelectric method could readily detect this change, Figure 5.7c and 5.7d show the magnetic images of the same two specimens after annealing in a vacuum furnace for 30 minutes at 700 °C. All the effects of plastic deformation during indentation, as well as the much weaker manufacturing
Figure 5.7  Comparison between (a) a semi-spherical hole produced by low-stress milling and (b) an otherwise similar semi-spherical indentation produced by pressing a stainless steel ball into the material. The scans at the bottom (c and d) show the magnetic pictures of the same holes after annealing in a vacuum furnace for 30 minutes at 700 °C ($\nabla T \approx 0.5 ^\circ C/cm$, 2 mm lift-off distance, 3"×3").
texture found in the original bar stock, are gone. As a result, the thermoelectric currents are also eliminated and the measured magnetic field is essentially zero.

5.6 THERMAL STRESS RELEASE

In this experimental work, we conducted a detailed investigation of the surface roughness, residual stress and cold work, which are the main materials properties affected by shot peening, in order to establish how they individually and collectively affect the recorded magnetic signatures obtained by thermoelectric measurements and to verify that the residual stress effect dominates the outcome of the final results. It is particularly important to mention the interesting role that cold work could play in shot peening and on the final interpretation of our results in order to properly assess the residual stress by the noncontacting thermoelectric technique. For this reason, it was essential to take into consideration all physical phenomena that could affect the outcome of the thermoelectric results. It was initially postulated in this study that the penetration depth and the particular depth-profile of the residual stress are primarily determined by material and manufacturing process variables and will change accordingly during thermally activated stress release. Different surface treatments produce not only different residual stress amplitudes and penetration depths, but, even more important, different cold-work amplitudes and penetration depths, which cause very different thermal relaxation behaviors. Generally, the rate of thermally induced relaxation towards equilibrium is proportional to the existing deviation from equilibrium at that particular location and at that particular instance of time.

In some cases of thermo-mechanical stress relaxation, the penetration depth and the particular depth-profile of the residual stress will not change significantly during thermally activated stress release. Figure 5.8 shows a schematic illustration of such proportional stress relief (solid lines), which does not affect the normalized depth distribution of the residual stress, though it significantly reduces its magnitude. However, this model neglects that certain types of
surface treatments, especially shot peening, impart a very strong cold work to the material, that substantially reduces the stress relaxation temperature at shallow depth below the surface. Therefore, in many cases, the relaxation directly at and below the surface is much stronger than at larger depths, and the depth profile of the residual stress significantly distorts during relaxation. Since the thermoelectric method measures a weighted average of the material variation below the surface, absolute calibration of the technique is very difficult if not impossible. Instead, as in almost all other cases of nondestructive materials characterization, we have to rely on empirical calibration curves that are obtained separately for different materials, different surface treatments, and possibly even different levels of surface treatment.

When a polycrystalline metal is cold worked, it produces a significant change in almost all its mechanical, electrical and physical properties. It is also well known that the principal change in the hardness occurs simultaneously with the matrix-material recrystallization. On actual shot-peened specimens the residual stress and cold work effects can be best modified by appropriately chosen heat treatment that simulates thermally activated stress release during service. So we decided to anneal a series of cold-rolled C11000 copper bars at different temperatures for 30 minutes and measure the microhardness after the heat treatment. In the absence of substantial surface hardening, microhardness, which is particularly easy to measure, can be used to characterize the degree of cold work. Figure 5.9 shows the measured Rockwell F hardness of cold-rolled C11000 copper as a function of annealing temperature in a vacuum furnace for 30 minutes. Figure 5.9 also shows the two stages of the annealing heat treatment process, recovery and recrystallization. Theoretically, in the recovery stage, any change in the mechanical, electrical and physical properties due to the cold work recover their original values, while in the recrystallization stage all the cold work effects (hardening, localized texture, etc.) and residual stress effects vanish owing to the material-matrix recrystallization. As it is shown, there is a sharp drop at around 440 °C where the hardness decreases rapidly and simultaneous recrystallization occurs.
Figure 5.8  A schematic illustration of proportional stress relief that does not affect the normalized depth distribution of the residual stress and enhanced stress relief due to near-surface cold work.
It is expected that on shot-peened specimens the residual stress and cold work effects start to decay well below this temperature because of the higher degree of cold work directly below the shot-peened surface. Based on this observation, we chose annealing at 315 °C for 30 minutes to induce stress release without recrystallization, i.e., approximately 125 °C below the measured transition temperature. Unquestionably, this somewhat arbitrary choice of the stress release temperature leaves much to be desired. These results are intended only as demonstrations of the much more rigorous procedure to be followed in the next stage, when stress release will be accomplished in several steps and in each step destructive X-ray diffraction measurements will be carried out to determine the remaining residual stress level as well as the local anisotropic texture as characterized by pole figures.

5.7 CHARACTERIZATION OF SHOT-PEENED SPECIMENS BY THERMOELECTRIC NDE

In order to verify the feasibility and potential of the proposed thermoelectric NDE method for the characterization of residual stress in shot-peened specimens. Fig. 5.10a shows the schematic diagram of the noncontacting thermoelectric method as used for the characterization of shot-peened specimens.[19,20,21,64] The main purpose behind showing these results is to supply information about the magnetic sensor position (polarization) with respect to the surface and the shape of the magnetic signature obtained from these measurements. In this case, since the generated magnetic field is perpendicular to both the heat flux in the specimen (parallel to the surface) and the gradient of the material property (normal to the surface), the magnetometer was polarized in the tangential direction. The apparent lift-off distance between the magnetic sensor (differential fluxgate) and the shot-peened area (surface imperfection) was 2 mm in all thermoelectric measurements.
Figure 5.9  Rockwell F hardness versus annealing temperature in cold-rolled C11000 copper (in a vacuum furnace after 30 minutes).
Figure 5.10b shows an example of the characteristic magnetic profiles that were obtained from shot-peened copper specimens at $\nabla T \approx 2.5 \, \degree C/cm$ temperature gradient and 2 mm lift-off distance. This unipolar magnetic profile, that originated from the surface treatment, is very typical in noncontacting thermoelectric measurements. It is important to mention that the unique characteristic magnetic profile of the thermoelectric measurements could supply information about the presence of inhomogeneity in a material depending on the signal profile obtained (unipolar or bipolar) from textured polycrystalline materials.\[36] Because of the relatively low level of the magnetic signatures to be measured, special precautions were taken to eliminate spurious effects of the Earth magnetic field, magnetic interference from the electrical power system and surrounding instruments, and other extraneous magnetic sources. These protective measures were discussed in great detail in Chapter II “Elimination of External Magnetic Sources.”
Figure 5.10  a) A schematic diagram of the modified noncontacting thermoelectric method as used for the characterization of shot-peened specimens and b) the characteristic magnetic profile obtained from a shot-peened copper specimen.
5.8 EXPERIMENTAL RESULTS

5.8.1 RESULTS ON SHOT-PEENED COPPER SPECIMENS WITH PREEXISTING COLD WORK IN THE C11000 BARS

As a first tentative step, in order to characterize shot-peened surfaces, one series of four copper specimens were shot-peened with preexisting cold work in C11000 bars at different Almen intensities ranging from Almen 2 to Almen 14 over areas of 25.4 mm × 25.4 mm by Metal Improvement Co. of Cincinnati. The peening was done by commercially available Z-600 ceramic beads of 0.6-0.85 mm in diameter to a nominal coverage of 100%. All specimens were inspected by the noncontacting thermoelectric technique described earlier in this chapter. Figure 5.11 presents the combined experimental data for the peak-to-peak magnetic flux density produced by thermoelectric currents for shot-peened surfaces of different Almen intensities in copper before stress release, after partial stress release, and after recrystallization.

A number of important conclusions can be drawn from these results. First, the magnetic signature produced by shot peening is proportional to the peening intensity, which is very favorable from the point of view of quantitative residual stress assessment. Second, even the lowest intensity (Almen 2) is well above the detection threshold of the noncontacting thermoelectric measurement, therefore the absolute sensitivity of the technique seems to be sufficient for most practical applications. Third, most of the drop in the thermoelectric signature occurred after the first heat treatment (i.e., after stress release) below the recrystallization temperature. So, at this point in the experimental work, the contribution of residual stresses to the measured signal seems to be substantially higher than those of texture and hardening. Finally, since the thermoelectric signal completely disappeared after the second heat treatment (i.e., after annealing) above the recrystallization temperature, the contribution of the rough surface topography to the measured signal is clearly negligible.
Figure 5.11  Comparison of the peak-to-peak magnetic signatures of shot-peened copper specimens with preexisting cold work in C11000 bars.
5.8.2 RESULTS ON SHOT-PEENED COPPER SPECIMENS WITHOUT PREEXISTING COLD WORK IN THE C11000 BARS

The results presented in the previous section were intended only to demonstrate the potential of the noncontacting thermoelectric technique to characterize the main effects produced by shot peening and to emphasize the need for a much more rigorous research procedure in order to separate the contributions of the residual stress and cold work in the measured magnetic signature. Recognizing that both the surface treatment and the thermal stress release processes are susceptible to random uncertainties and inevitable variations, five series of 12.7 mm × 38.1 mm × 500 mm C11000 copper specimens have been shot-peened to different degrees of intensity ranging from Almen 2 to Almen 16 in steps of 2 over areas of 25.4 mm × 25.4 mm by Metal Improvement Co. of Cincinnati. Again, the peening was done by commercially available Z-600 ceramic beads of 0.6-0.85 mm in diameter to a nominal coverage of 100%.

In order to further reduce the uncertainties caused by the presence of cold-work-induced hardening and texture and intrinsic microstructural variations, all specimens were annealed following machining for 30 minutes at 600 °C in a protective nitrogen atmosphere by Bodycote Thermal Processing of Cincinnati. In this case the main goal of the heat treatment that all specimens received before the surface treatment (shot-peening) was to remove near surface and bulk material variations caused by prior processing in the original plate from which the specimens were cut.

First, the shot-peened specimens underwent a detailed surface analysis to establish their rms roughness and correlation length. Figure 5.12 shows examples of the measured correlation functions (dashed lines) and best-fitting exponential distributions (solid lines) for shot-peened copper specimens using a WYKO NT-2000 scanning white light interference microscope. The goal of these measurements was to help obtain a better understanding of the relationship between the penetration depth of the compressive residual stress and the correlation length of
the rough surface topography and to verify that the inherent random variations of the surface treatment process are under control. The rms value of the surface roughness increased from \( \approx 2 \) \( \mu \)m to \( \approx 10 \) \( \mu \)m as the peening intensity increased from Almen 2 to Almen 16.

On the average, the correlation length was 15 times higher than the rms roughness, which is very similar to the ratio found in other ductile materials such as 7075 aluminum. In the next step, all specimens were inspected by the noncontacting thermoelectric technique described earlier in this chapter. Figure 5.13 shows the measured magnetic signatures on three series of C11000 copper specimens before and those of two series after stress release for 30 minutes at 315 °C for nine different shot peening intensities at 2-mm lift-off distance and 2.3 °C/cm temperature gradient. On the intact specimens (i.e., before stress release) the peak-to-peak value of the measured magnetic flux density increased from \( \approx 5 \) nT to \( \approx 20 \) nT as the peening intensity increased from Almen 2 to Almen 16 and the variation within the three series was found to be at an acceptable level. In comparison, on the stress-released specimens the magnetic flux density was approximately 75% lower.
Figure 5.12 Examples of the measured correlation functions (dashed lines) and best-fitting exponential distributions (solid lines) for shot-peened copper specimens.
before stress release  
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after stress release (30 min, 315 °C)

Figure 5.13  Magnetic signatures on three series of C11000 copper specimens before and those of two after stress release as functions of the shot peening intensity.
5.8.2.1 X-RAY DIFFRACTION RESIDUAL STRESS MEASUREMENTS

The first series of the shot-peened specimens were sent to Lambda Research of Cincinnati for X-ray diffraction analysis. X-ray diffraction residual stress measurements were made at the surface and at nominal depths of 1, 2, 3, 7, 10, and $15 \times 10^{-3}$ in. for specimens of Almen intensity 0 through 8. Measurements were performed at the surface and nominal depths of 2, 4, 7, 10, 15, and $20 \times 10^{-3}$ in. for specimens of Almen intensity 10 through 16, where the penetration depth was significantly higher. All measurements were made in the longitudinal direction of the bar at the corner of the shot-peened zone (incident beam divergence 1.0°, psi rotation 10° and 50°, irradiated area $0.2 \times 0.2$ in.). The samples were rocked through an angular range of $\pm 1.5°$ around the mean psi angles during measurement to integrate the diffracted intensity over more grains in order to minimize the influence of grain size. X-ray diffraction residual stress measurements were performed using a two-angle sine-squared-psi technique, in accordance with SAE J784a, employing the diffraction of Mn K-alpha radiation from the (311) planes of the FCC structure of the C11000 series copper.

The diffraction peak angular positions at each of the psi tilts employed for measurement were determined from the position of the K-alpha 1 diffraction peak separated from the superimposed K-alpha doublet assuming a Pearson VII function diffraction peak profile in the high back-reflection region. The diffracted intensity, peak breadth, and position of the K-alpha 1 diffraction peak were determined by fitting the Pearson VII function peak profile by least-squares regression after correction for the Lorentz polarization and absorption effects and for a linearly sloping background intensity. The macroscopic residual stress was calculated from the strain measured normal to the (311) planes of using tabulated values of Young's modulus and Poisson's ratio. Material was removed electrolytically for subsurface measurements in order to minimize possible alteration of the subsurface residual stress distribution. All data obtained as a function of depth were corrected for the effects of the penetration of the radiation employed for residual stress measurement into the subsurface stress gradient. The stress gradient correction
applied to the last depth measured is based upon an extrapolation to greater depths and may result in over correction at the last depth if the stress profile has been terminated in the presence of a steep gradient. Corrections for sectioning stress relaxation and for stress relaxation caused by layer removal were also applied as appropriate. The error shown for each residual stress measurement is ± 25 MPa, which is somewhat higher than one standard deviation resulting from random error in the determination of the diffraction peak angular positions. One reason for such additional error on the order of 15 MPa may result from sample positioning and instrument alignment errors. The fully corrected residual stress distributions measured as function of depth before stress release and after partial stress release at 315 °C are presented in Figure 5.14 for nine specimens (compressive stresses are shown as negative values). The residual stress data before stress release show that the maximum compressive layer was found close below the surface on all but the unpeened specimen (Almen 0). Generally, the results indicate increasing depth of compression with increasing intensity. The surface residual stress slightly decreases in magnitude as the peening intensity increases, which is typical of work-hardening materials. In comparison, the residual stress data after a 315-°C partial stress release show a substantial reduction in residual stress by approximately a factor of two at all Almen intensities (the weighed average reduces even more due to the almost complete relaxation at and directly below the surface).
5.8.2.2 X-RAY DIFFRACTION COLD WORK MEASUREMENTS

As a by-product of the above-described residual stress measurement, the obtained XRD data can also be used to quantitatively assess the degree of cold work below the shot-peened surface. Figure 5.15 shows the subsurface cold-work distribution for nine C11000 copper specimens of different shot peening intensity before and after the 315-°C partial stress release as measured by the (311) diffraction peak width, which is a sensitive function of the hardness and the degree to which the material has been cold worked. In work-hardening materials, the diffraction peak width significantly increases as a result of an increase in the average microstrain and the reduced crystallite size produced by cold working.[65] The (311) diffraction peak width can be indicative of how the material may have been processed and the degree to which it has been plastically deformed. The results before stress release indicate that maximum cold work occurs at the surface of the shot-peened samples. The maximum cold work does not differ significantly with peening intensity, but the depth of the plastically deformed layer appears to slightly increase with increased peening intensity. After the 315-°C partial stress release, the cold work is essentially gone at all Almen intensities.
Figure 5.14 Subsurface residual stress distribution for nine C11000 copper specimens of different shot peening intensity as measured by X-ray diffraction.
Figure 5.15  Subsurface cold work distribution for nine C11000 copper specimens of different shot peening intensity as measured by the (311) diffraction peak width.
5.8.2.3 X-RAY DIFFRACTION TEXTURE MEASUREMENTS

In a polycrystalline material each grain usually has a different crystallographic orientation with respect to its neighbors. The presence of a preferred crystallographic and/or morphologic grain orientation is called texture, which might lead to substantial macroscopic anisotropy in the material properties. The presence and degree of crystallographic texture can be illustrated by graphical representation of X-ray diffraction measurements, that are called pole figures. A pole figure is a stereographic projection that shows the probability density of a given crystallographic direction with respect to specimen orientation measured in spherical coordinates. In order to determine the preferred orientation of the grains, several pole figures are required for different crystallographic planes.

It is well known that cold work during shot peening will lead to the development of a localized sub-surface texture, which can be studied by X-ray diffraction and graphically represented by pole figures. The data necessary to construct the (200) pole figures were obtained using copper K-alpha radiation and a Schulz back-reflection pole figure device mounted on an automated Bragg-Brentano X-ray diffractometer. The diffracted intensity data were collected for one second at each orientation spaced at approximately 4° in all directions on a nominally equal area spherical net throughout the central 72°-range of the polar angle. During data collection, the sample was vibrated ±0.2" in a plane parallel to the sample surface in order to integrate the diffracted intensity. Each sample was oriented so that reference orientation marks on the sample were aligned in a direction parallel to a known reference azimuth direction for the pole figure. The background intensity measured on both sides of the diffraction peak for each angle of tilt was interpolated to determine the background intensity component of the diffraction peak.

After subtraction of the background, the corrected pole figure was constructed in spherical coordinates by linear interpolation of the data collected on the equal area spherical net. An analytical solution was used to correct for defocusing intensity losses, which occur as
the specimen is tilted in the X-ray beam. Figure 5.16 shows the plane (200) back-reflection pole figures for four C11000 copper specimens of different shot peening intensities (light and dark gray indicate areas of 50-100 % and 100-200 % intensity relative to the average, respectively). Generally, the pole figures of all samples appear to have a nearly random azimuth orientation. The unpeened specimen exhibits a “spotty” appearance, which decreases with increasing peening intensity. The lack of preferred orientation in the azimuth direction indicates that the initial annealing successfully removed the original texture of the bar stock as intended. After shot peening, the plane of the surface still remains essentially isotropic, but the emergence of a perceivable polar texture clearly indicates the effect of substantial plastic deformation at the surface. These results show that a slight localized texture is created below the surface by shot peening, which is revealed by the preferred orientation of the (200) planes parallel to the surface. It was found that, like the subsurface cold work, this texture was also completely eliminated by annealing at 315°C for 30 minutes.

Figure 5.17 shows the normalized intensity of the (200) back-reflection pole figures versus polar angle for nine C11000 copper specimens of different shot peening intensity (again, normalization is relative to the average intensity). These profiles were obtained at each polar angle simply by averaging the measured back scattered intensity for all azimuth angles. The results indicate that a slight localized texture is created below the surface by shot peening, that is revealed by the preferred orientation of the (200) planes parallel to the surface. In polycrystalline materials, texture can be either morphologic or crystallographic in origin. Although shot peening can lead to both kinds of texture, the crystallographic type, which is actually measured by XRD, is not expected to cause a detectable thermoelectric signature in cubic materials such as copper, since cubic crystals are completely isotropic from both thermal and electrical points of view.\textsuperscript{[30]} Therefore, the thermoelectric effects of texture are caused solely by morphological features, such as preferentially oriented dislocations, slip bands, and grain boundary imperfections and are generally very weak with the notable exception of titanium alloys.\textsuperscript{[17,18]}

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Figure 5.16  Plane (200) back-reflection pole figures for four C11000 copper specimens of different shot peening intensity (light areas 50-100 % relative to average, dark areas 100-200 % relative to average).
Figure 5.17 Normalized intensity of the (200) back-reflection pole figures versus polar angle for nine C11000 copper specimens of different shot peening intensity (normalization is relative to the average intensity).
5.8.2.4 OVERVIEW OF THE GEOMETRICAL AND PHYSICAL EFFECTS OF SHOT PEENING

Figure 5.18 summarizes the main quantitative results of our investigation on the shot-peened specimens before stress release. The different geometrical and physical parameters are shown as functions of the shot peening intensity in copper. It should be mentioned that the cold-work data were converted into true plastic strain by calibration on a series of compressed coupons. The most important observations we can make from these results are that (i) the peak value of the residual stress is rather insensitive to the peening intensity and that (ii) directly after shot peening the more variable parameter is not the peak value, but rather the penetration depth of the compressive residual stress, which is approximately three times deeper than the correlation length of the surface roughness. The penetration depth of the cold-worked region is roughly half of that of the residual stress, and the degree of cold work is also relatively insensitive to the peening intensity.

5.8.2.5 THERMOELECTRIC RESIDUAL STRESS ASSESSMENT

The next task was to repeat all these measurements after different degrees of partial stress release to separate the individual effects of (i) residual stress, (ii) cold work induced texture and hardening, and (iii) surface topography on the measured thermoelectric signature. As a first tentative step, we had two series of shot-peened copper specimens stress released at 315 °C for 30 minutes. Figures 5.13-5.15 also showed the corresponding magnetic signatures, residual stress and cold work profiles measured in all nine specimens after this stress release. It should be mentioned that the X-ray pole-figure measurements were also repeated on the stress released specimens and all of them were found to be essentially random, i.e., all perceivable indications of texture were eliminated, which is not surprising in light of the fact that the cold work induced plastic strain has also completely disappeared (see Fig. 5.15).
Figure 5.18  Different geometrical and physical parameters as functions of the shot peening intensity in copper.
Figure 5.13 showed the magnetic signatures of three series of C11000 copper specimens before and those of two series after stress release. Clearly, the detected signals are very similar, but the peak-to-peak amplitudes significantly decreased as a result of the diminishing residual stress levels. On the average, this incomplete stress release reduced the amplitude of the magnetic signature by roughly a factor of 4. Two important observations should be made. First, the standard deviation of the relative drop for the different peening intensities was only 10%. This modest variation indicates that the decrease is essentially independent of the preexisting stress profile, which is an indication that the combined residual stress/cold work relaxation phenomenon is more or less proportional in all grades. It is also evident that the substantial near-surface cold work greatly accelerates thermal stress release and that cold work, as measured by the plastic strain, decays much faster than the residual stress at larger depths. In order to further investigate the parallel decay of residual stress and cold work and their combined effect on the measured thermoelectric signature, we conducted a number of additional experiments.

On one side, the remaining third partially stress released series, that was not destroyed by X-ray diffraction measurements, was further annealed first again at 315°C, and then at 460°C, and finally at 600°C (all heat treatments were done for 30 minutes) and the magnetic signatures were recorded after each step. In order to study lower levels of stress release, the last two series of intact shot-peened specimens were stress released at 235°C and 275°C, then tested by the thermoelectric technique, and finally also tested by X-ray diffraction measurements. The main goal of the heat treatment that all specimens received at different temperatures was to remove gradually the near surface material variations caused by the surface treatment. These variations are associated with subtle near surface effects such as the presence of residual stress, increased hardness, dislocation density and anisotropic texture. With the exception of surface roughness, annealing above the recrystallization temperature of the material effectively eliminated all other near surface variations, that contribute to the magnitude of the magnetic signature.
Figure 5.19 shows the peak-to-peak amplitudes of the magnetic signatures recorded on all of the C11000 copper specimens as functions of the shot peening intensity before and after partial stress release. The partial stress relaxations at 235°C and 275°C resulted in only modest 30% and 50% drops in the magnetic signature, respectively, while the repeated annealing at 315°C (second time) and 460°C further reduced the amplitude. Finally, there was no detectable magnetic signature left after full recrystallization at 600°C. Figure 5.19 also shows that the measured magnetic signature is essentially a linear function of the shot peening intensity and that this signature gradually decreases during relaxation to essentially zero in fully recrystallized specimens.

These trends are very promising for the feasibility of nondestructive monitoring of thermal relaxation in shot-peened copper specimens, but they do not provide unequivocal evidence whether the magnetic signature is caused mainly by the presence of residual stresses, the presence of cold work, or a certain combination of both. One thing however is already clear from our previously shown results in Fig. 5.15. Since the cold work appears to be completely gone after partial stress release for 30 minutes at 315°C while roughly half of the average residual stress is still retained, the 75% drop in the magnetic signature indicates that the residual stress contribution cannot be less than 50%.

In order to better separate these two effects, we had a second series of X-ray diffraction measurements conducted on the less relaxed 235-°C and 275-°C specimens. Figure 5.20 shows examples of the residual stress and cold-work profiles recorded on these specimens. The compressive residual stress gradually decreased as the annealing temperature increases at all Almen intensities. On the other hand, after the 315-°C annealing temperature, the cold work is essentially gone. This explains the fact that some physical and mechanical properties do not recover their initial values properties at the same time as others, which is an indication of the complicated nature of the recovering process. These figures give a relatively detailed picture of the initial relaxation process, though the accuracy of the very expensive and time-consuming destructive X-ray diffraction measurement is obviously less than sufficient to precisely map
Figure 5.19  Average peak-to-peak amplitudes of the magnetic signatures recorded on C11000 copper specimens as functions of the shot peening intensity before and after partial stress release.
these distributions and some of the ruggedness of the profiles was certainly caused by experimental errors. The accuracy of the residual stress measurement was estimated at approximately ±25 MPa, while the accuracy of the peak width measurement is approximately ±0.15°, which translates into approximately ±10% plastic strain.

Figure 5.21 shows the thermal relaxation of the (a) integrated residual stress, (b) integrated cold work, and (c) peak-to-peak magnetic signature in copper C11000 for different Almen intensities. In order to avoid that the integrated profiles be dominated by the experimental errors at large depth where the true values are very low, we assumed that the residual stress and cold work values are zero wherever their magnitude was less than their respective errors, i. e., 25 MPa and 10%. A statistical comparison of these integrated residual stress and cold-work results to the magnetic signature revealed that in shot-peened C11000 copper, on the average, ≈64% of the thermoelectric signal is due to residual stresses. These experimental results verify the feasibility of nondestructive evaluation of thermal relaxation in shot-peened C11000 copper.
Figure 5.20  Thermal relaxation of residual stress and cold work in Copper C11000 for different Almen intensities (30 minutes in a vacuum furnace).
Figure 5.21  Thermal relaxation of the (a) integrated residual stress, (b) integrated cold work, and (c) peak-to-peak magnetic signature in Copper C11000 for different Almen intensities.
5.8.3 PRELIMINARY RESULTS ON IN100 NICKEL-BASE SUPERALLOY

Although the development of a quantitative residual stress measurement method might require additional more accurate and more detailed tests, the obvious next question to be addressed is whether the thermoelectric method is applicable to other engineering materials of special importance to the aerospace industry. In particular, it is very important to develop NDE techniques for high-strength, high-temperature engine materials such as nickel-base super alloys. Therefore, as a final experiment, we conducted additional thermoelectric measurements on two series of shot-peened IN100 specimens both before and after partial stress relaxation. Figure 5.22 shows the average peak-to-peak amplitude of the magnetic signatures recorded on IN100 nickel-base superalloy specimens as a function of the shot peening intensity before and after partial stress release ($\approx 2.5^\circ$C/mm temperature gradient). The most important difference between our thermoelectric results in IN100 and copper specimens is the presence of a significant background signature. In IN100, as well as in other engine materials such as Ti-6Al-4V, the magnetic signature is not entirely negligible in the unpeened specimens and it does not completely vanish in the shot-peened specimens after complete stress release. This background signature is caused by the anisotropic texture and inhomogeneity of the specimen and it was the subject of the previous two chapters. It should be mentioned that a similar, though somewhat weaker, signature was also observed in cold rolled copper specimens, so the material was annealed before shot peening to eliminate this adverse feature. In this respect, the IN100 measurements are more representative of a real application, in which the base metal is not necessarily homogeneous or texture-free even before surface treatment. Currently, efforts are underway to establish the most effective methods of baseline compensation based on the specific features of the baseline signature.
Figure 5.22  Average peak-to-peak amplitude of the magnetic signatures recorded on IN100 nickel-base superalloy specimens versus peening intensity before and after partial stress release (= 2.5 °C/mm temperature gradient).
Nondestructive evaluation of the existing residual stress in the shallow subsurface layer of shot-peened components could be very beneficial during manufacturing to monitor and minimize process variations. Even more importantly, NDE is absolutely necessary after extended service if residual stresses were to be taken credit for in fatigue life predictions of critical components because of the very significant and highly variable stress release that might occur at elevated operating temperatures. Currently, the only reliable NDE method for residual stress assessment is based on X-ray diffraction measurement which is limited to an extremely thin (≈1 mil) surface layer, that is approximately one order of magnitude less than the typical penetration depth of compressive residual stresses produced by shot peening. Conventional ultrasonic and eddy current NDT methods are simply too sensitive to surface roughness to quantitatively assess the subtle variation in material properties, such as ultrasonic velocity and attenuation or electrical conductivity, caused by material surface imperfections. On the other hand, thermoelectric techniques are very unique among all other methods used in nondestructive materials testing in that they are solely sensitive to intrinsic material variations regardless of the size, shape, and surface quality of the specimen to be tested. It is also well known that the thermoelectric power of most metals is fairly sensitive to elastic strains.

This chapter presented experimental results that illustrated the potential for a new NDE technique to detect plastic deformation and the presence of residual stresses, which are very difficult to characterize by other more conventional NDE methods. It has been found that the noncontacting thermoelectric method can be used to characterize the relaxation of the prevailing residual stress in shot-peened metals. Besides the primary residual stress effect, the thermoelectric method is also sensitive to the secondary “material” effects of shot peening (local texture, increased dislocation density, hardening), but it is entirely insensitive to its “geometrical” by-product, i.e., the rough surface topography.
Our experimental results in copper indicate that the developed method is more sensitive to residual stress effects than to the secondary material effects, but unequivocal separation of residual stress relaxation from the parallel decay of secondary cold work effects is probably not feasible. Preliminary results on IN100 nickel-base superalloy were also presented to demonstrate that the proposed method might be applicable to a wide range of alloys including high-strength, high-temperature engine materials. Further development of this very promising new NDT method for other engineering materials of great interest, namely high-strength aluminum alloys, Ti-6Al-4V and other titanium alloys, and nickel-base superalloys, will be investigated.
6.1 SUMMARY AND CONCLUDING REMARKS

This dissertation presented experimental results on a novel NDE technique based on the magnetic field strength produced by thermoelectric currents due to a temperature difference established between the host medium and an imperfection. The flux density due to the magnetic field was sensed by a magnetometer in order to detect and quantitatively assess imperfections such as inclusions, texture, anisotropy, inhomogeneities, residual stresses, cold work, hardening and fretting damage. In the following, we summarize the most important findings of each chapter in the dissertation.

Chapter II presented experimental data for the magnetic field produced by thermoelectric currents around surface-breaking spherical tin inclusions in copper under external thermal excitation for different lift-off distances between the sensor tip and the specimen surface.[16] These results were found to be in good agreement with previously published theoretical predictions.[15] In addition, it was found that the copper specimens cut from a plates and bar stock both produced a substantial background signature that interfered with the accurate measurement of some of the smaller inclusions. This background signature is due to case hardening and axial texture caused by material manufacturing procedures such as rolling, forging, extrusion, pressing etc.

Chapter III reviewed a theoretical method[17] developed by Nayfeh and Nagy for calculating the magnetic field produced by thermoelectric currents in anisotropic materials under two-dimensional directional heating and cooling and presented an experimental verification of this analytical model. Experimental results on a textured Ti-6Al-4V titanium-alloy plate were shown to be in very good qualitative agreement with the predictions of this model. Since the detectability of small imperfections is ultimately limited by the intrinsic
thermoelectric anisotropy and inhomogeneity of the material to be inspected, the described analytical method can be used to optimize the thermoelectric inspection procedures and to evaluate the macroscopic texture of metals from their characteristic magnetic signatures.

Chapter IV investigated the spurious magnetic signature produced by the simplest type of macroscopic inhomogeneity when the material properties exhibit a linear spatial variation in the cross-section of a slender bar. An analytical method has been developed by Nayfeh and Faidi for calculating the normal and tangential magnetic fields produced by the resulting thermoelectric currents, which was subsequently compared to experimental measurements. Experimental results from a Ti-6Al-4V titanium-alloy bar were shown to be in very good qualitative agreement with the predictions of the analytical model. The results clearly indicate that the observed normal and tangential background signatures on the four sides of the specimen can be attributed to a linear thermoelectric current distribution in the cross-section of the slender bar. However, the larger than predicted magnitude of the observed signatures essentially excludes the possibility that the actual source of these thermoelectric currents is simply a minor inhomogeneity of the isotropic material. Further efforts are needed to better understand the relationship between the strength of the thermoelectric background signature and the microstructural and chemical features of the material.

Chapter V presented a noncontacting thermoelectric method that can be used to characterize the prevailing residual stress in shot-peened specimens. The experimental results in copper indicate that the developed method is more sensitive to residual stress effects than to the secondary material effects, but unequivocal separation of residual stress relaxation from the parallel decay of secondary cold work effects is generally not feasible. However, since the ratio of residual stress to cold work is primarily determined by the material and the specific surface treatment used, the thermoelectric method still offers the unique capability of nondestructively monitoring thermo-mechanical relaxation below the treated surface. Preliminary results on IN100 nickel-base superalloy were also presented to demonstrate that the
proposed method might be applicable to a wide range of alloys including high-strength, high-temperature engine materials.

Finally, although the detectability of these material imperfections has been experimentally verified in this dissertation, qualitative identification and, especially, quantitative assessment of these imperfections will require additional well coordinated theoretical and experimental efforts. The main purpose of this dissertation was to lay down the groundwork for a better understanding of the underlying physical phenomena, the development of new, more sensitive experimental procedures, and, ultimately, increased probability of detection (POD) for small inclusions and weak material imperfections.
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