Positron Defect Mapping

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by

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To the Graduate Faculty:

The members of the committee appointed to examine the dissertation of Marcus A. Gagliardi find it satisfactory and recommend that it be accepted.

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Positron Defect Mapping

Abstract

Positron annihilation spectroscopy has been used in the detection of monovacancies as well as dislocations in materials for many years. Using the technique of Doppler broadening, relative defect density measurements were made on several copper coupons that had been annealed as well as shot peened. Due to its nature, shot peening introduces defects such as monovacancies and dislocations into the material. This reproducible technique was used to implant these defects into the copper in order to show that correlations exist between S-parameter and shot peening intensity. Relative defect density maps were produced that allow for verification to be made of different defect density regions. Also, these relative defect density maps can be used to determine if shot peening intensity was uniform over the measured area.

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for my mother and my father

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Contents

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List of Figures

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List of Tables

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Chapter 1

Introduction

1.1 Positrons and Positron Annihilation Spectroscopy

Since its prediction by Dirac in the 1920's and its subsequent discovery by Anderson in 1933, many uses have been predicted for the positron [2, 15, 16]. In 1949 S. DeBenedetti et al. discovered that that the two γ-rays arising from the annihilation of thermalized positrons in a solid material were not collinear. This phenomenon was attributed to the electron momentum of the annihilating pair. Thus, began a new era of using positrons to probe solid-state materials [12, 54].

When the emitted positron enters a material it will thermalize, typically, within the first few micrometers of the surface [24, 43, 47]. Once thermal equilibrium is reached the positron will annihilate with an electron from the surrounding medium. The dominant annihilation of the positron with the electron leads to two γ-rays. This entire annihilation process will, in general, take place within 100 to 500 picoseconds (ps). If the lattice structure of the material is free of any defects such as dislocations, or point defects as seen in figure 1.1 a, the positron has a larger probability of annihilating with a high momentum core electron. In contrast, if the positron enters a material where monovacancies exist as seen in figure 1.1 b the positron can become trapped in the defect, decreasing the probability of annihilating with high momentum core electrons and hence increasing the probability of annihilating with low momentum valence electrons. Doppler Broadening Spectroscopy, or as it will be

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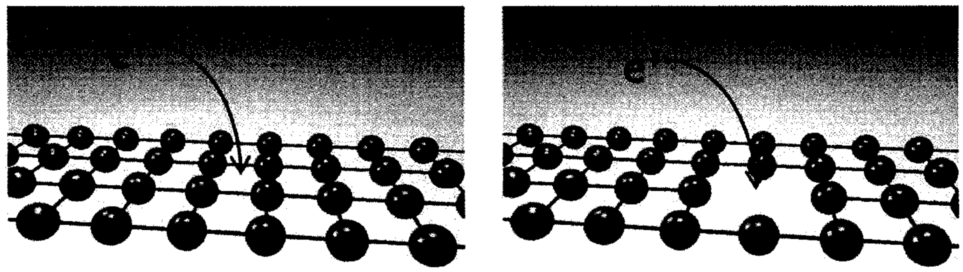

(a) (b)

Figure 1.1: Figure (a) shows the positron entering the lattice of the material being probed. The material here has no defects and therefore the positron has an equal probability of annihilating with any electron. Figure (b) shows the positron entering the lattice of a material with a point defect. The positron sees this defect as a potential well and in turn has a high probability of being trapped within the defect. Once trapped the positron then will annihilate with a valence electron from one of the surrounding atoms.

referred to in this text, positron annihilation spectroscopy (PAS), is commonly used for determining which of these two processes takes place.

The positron annihilation spectroscopy process in these experiments was conducted through the use of a high purity germanium (HPGe) detector [38]. The energies of the emitted γ-rays are detected as a peak with an energy of approximately 511 keV. Although more detail will be given later in this text as to the specifics of this measuring method, it is the change in width of this 511 keV peak that is used to distinguish the annihilation of positrons within defects. Figure 1.2 shows the resulting 511 keV annihilation lines as measured by the HPGe detector. The 511 keV line from the annealed copper is broader than that of the manufactured copper. This implies that the momentum of the annihilating electrons measured from the annealed copper is larger than that of the as manufactured copper. Due to the annealing process, defects like mono-vacancies are less prevalent. Therefore, the positrons have a higher probability of annihilating with core electrons than they would if they were in a material with a larger number of defects, such as mono-vacancies. In contrast, the manufactured copper has a large concentration of defects, due to the cold working

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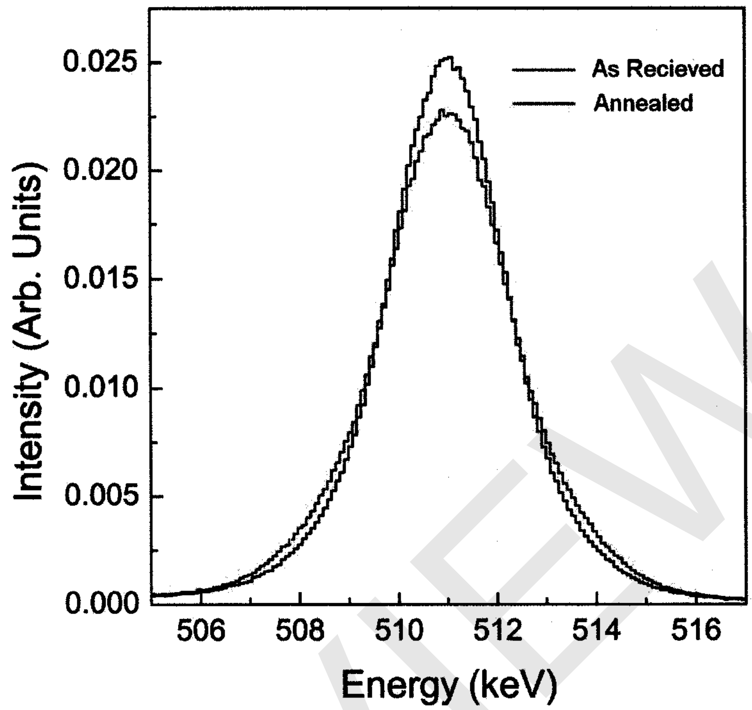

Figure 1.2: These are two 511 keV peaks taken from an annealed and unannealed copper sample. These spectra are normalized to the total number of counts in the peak. The difference in widths are used to illustrate the doppler shift experienced by the annihilating γ-rays due to a material with a relative low defect density (annealed) compared to a material with higher relative defect density (unannealed).

performed on the metal during the manufacturing process. As previously mentioned, positrons interacting with materials that have a larger number of defects will have a higher probability of annihilating with valence electrons, this in turn will cause a narrowing of the 511 keV annihilation line as seen in figure 1.2. Analysis of the 511 keV annihilation lines width allows a measurement of the relative defect densities of the material under test.

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1.2 Purpose and Intent

The goal of this doctoral research was to map relative defect densities from a two dimensional surface. Using positron annihilation spectroscopy, measurements of the 511 keV annihilation line width were taken from several points on a copper plate. Each of these data points were measured and reconstructed into a map of location versus relative defect density. The process of conducting these experiments began with the construction of the experimental apparatus. Although alterations were made for later experiments the following description of the system remained unchanged.

This research required a data acquisition and control system. The data acquisition system consisted of several nuclear instrumentation modules as well as data acquisition software a computer control program, and a HPGe. The control system consisted of a series of motion control tables. The details of these systems will be discussed in more detail in Chapter 3.

Two sets of experiments were conducted to test the experimental apparatus. The first set of experiments tested the two-dimensional mapping technique that would be used later for the construction of a relative defect density map. With these experiments the system resolution was calculated. The second set of experiments measured sensitivity to defects. An understanding of the system's sensitivity was paramount in distinguishing variations in the overall defects on the material surface. As discussed in Chapter 4.3 the bending of the copper plate induced a large amount of damage that extended throughout the plate. Hence, a more precise technique was required to create defects.

Shot peening was used to create and obtain well defined regions of relative defect density. Using copper coupons shot at different intensities, reproducible measurements were made of these relative defect densities. Comparison of those relative defect densities made by shot peening to those measurements made of annealed and unannealed pieces provided a scale of shot intensity versus relative defect density. A saturation point was reached in the measurements of the shot peened pieces, where no further variance could be observed between the relative defect densities. This saturation effect agrees with results from S. Saterlie et al. using PAS on shot peened

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aluminum [53].

The final set of experiments combined the sensitivity measurement with the mapping techniques. A new annealed copper plate was used and surface regions were shot peened at different intensities. A series of two-dimensional maps were produced in order to distinguish the shape and intensity of relative defect density at each location. It is shown that although defect density maps are relative to each material being used, defect densities of varying intensity can be imaged and distinguished using this technique.

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Chapter 2

Fundamentals of positron annihilation spectroscopy

2.1 Positron Thermalization

Upon entering a solid, energetic positrons rapidly loose energy by several processes until reaching thermal energies. For positrons in copper with energies above $\sim$ 20 MeV, this thermalization process is first dominated by the emission of bremsstrahlung radiation [11, 25]. Since $\beta+$ spectra typically have endpoint energies of $\sim$ 1 MeV, bremsstrahlung production is often negligible. At energies below $\sim$ 20 MeV positron energy loss is dominated by collisions with the electron gas until $\sim$ 20 eV. Below this ionization threshold, positrons loose energy by plasmon creation in metals and interband excitation in semiconductors [35, 37, 39]. Finally, positrons loose energy to phonon interactions below $\sim$ 0.1 eV until the positron reaches thermal energies [35, 37, 39].

At energies down to $\sim$ 20 MeV, energy loss due to bremsstrahlung is dominant and given by

$$-\frac{dE_o}{dx} = N \int_0^1 k\phi_k \, \mathrm{d}\left(\frac{k}{E_o - \mu}\right), \tag{2.1}$$

where E_o is the positrons total energy, N is the atomic number density, k is the photon energy, μ is the rest energy of the electron, and ϕ_k is the bremsstrahlung

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cross section. Performing the integration, equation 2.1 can be written as

$$-\left(\frac{dE_o}{dx}\right)_{rad} = NE_o\phi_{rad}, \tag{2.2}$$

where ϕ_{rad} is the cross section for the energy lost by radiation, is related to equation 2.1, and takes the form of,

$$\phi_{rad} = \frac{1}{E_o}\int_0^1 k\phi_k \,\mathrm{d}\left(\frac{k}{E_o - \mu}\right). \tag{2.3}$$

After performing the integration equation 2.3 takes the form

$$\begin{aligned}\phi_{rad} &= \bar{\phi}\left[\frac{12E_o^2 + 4\mu^2}{3E_o p_o}\, log\frac{E_o + p_o}{\mu} - \frac{(8E_o + 6p_o)\mu^2}{3E_o p_o^2}\left(log\frac{E_o + p_o}{\mu}\right)^2\right.\\ &\left. -\frac{4}{3} + \frac{2\mu^2}{E_o p_o}F\left(\frac{2p_o(E_o + p_o)}{\mu^2}\right)\right],\end{aligned} \tag{2.4}$$

where p_o is the momentum of the positron; $\bar{\phi} = \frac{Z^2 r_o^2}{137}$; and F is a function defined by the integral

$$F(x) = \int_0^x \frac{log(1+y)}{y}\,\mathrm{d}y \text{ [25]}.$$

Once the positron's energy has fallen below $\sim$ 20 MeV the process of ionization dominates as the main cause of energy loss. As the positron moves through the material its energy is transferred to the electrons, which can also cause ionization. Energy loss calculations were first conducted by Bohr using classical theory [6]. These calculations have also been performed using quantum mechanics. The formula for energy lost by the positron traveling with velocity $v = c\beta$ is

$$-\left(\frac{dE}{dx}\right)_{coll} = NZ\phi_o\mu\frac{3}{4}\frac{e^2}{\beta^2}\left[log\frac{2\mu\beta^2 W_m}{I^2 Z^2(1-\beta^2)} - 2\beta^2\right], \tag{2.5}$$

where ϕ_o is $\left(\frac{8\pi}{3}\right) r_o^2$, e is the charge of the positron, Z is the atomic number, and I is the average ionization energy, which varies for elements of different types. W_m is the maximum energy that can be transferred to a free electron by the positron. For the case of the positron W_m is $\frac{1}{2}(E - \mu)$.

The energy loss equation for positrons with non-relativistic energies ($E - \mu \equiv T \ll \mu$) is

$$-\left(\frac{dE}{dx}\right)_{coll} = NZ\phi_o\mu\frac{3}{4}\frac{\mu}{T}\left[log\frac{T}{IZ\sqrt{2}} + \frac{1}{2}\right], \tag{2.6}$$

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