EFFECTS OF ANNEALING ON SHOT PEELED COPPER

by

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ABSTRACT

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Shot peening has been used for many years to strengthen metal for industry. However, these metals that have gone through the shot peening process can become weaker if they are heated up, whether through process or in their application, to the point where the crystal lattice structure of the metal begins to change. This paper will attempt to look at the relationship of annealing temperature and the effect it has on metals that have been strengthened through a shot peening process. Using the technique of positron annihilation spectroscopy, copper metal coupons that have been shot peened and annealed at various temperatures will be measured according to their S-Parameter. This technique will show graphically that as the metal is annealed at higher temperatures, due to everyday applications, the amount of defects will begin to diminish significantly.
Chapter 1

Introduction

1.1 Purpose and Intent

Shot peening was invented by General Motors in the late 1920’s to improve the fatigue strength of a metal. [4] Shot peening is the process where small ball bearings are shot at high velocities, using air pressure, into the metal. This produces minute divots which leaves the metal in a “compressive state, which prevents the formation and propagation of cracks.” [3] However, the shot peening process can produce point defects in the metal. [2] Point defects are atoms that are missing from a crystal structure. Once these point defects are in the metal they are not easily removed. In fact, to remove point defects in metals, the metal has to be annealed.

The annealing process is where metal is heated up to the point where the crystal undergoes three stages. The first is called the recovery phase. This is when the crystal atoms gain enough energy that it will slowly move to fill in the point defects. The second phase is called re-crystallization. This is when new crystals will grow in the metal, which will also help remove any defects that were placed in the metal due to any internal stresses. The last and final stage is called grain growth. This is when the grain sizes will actually grow and get larger. These three stages of the annealing process will remove the point defects in the metal [3].

The removing of the point defects is not always a good thing. As mentioned above, shot peening prevents the formation and propagation of cracks, which helps prolong the fatigue life. But sometimes metal undergoes the annealing process when such a process is not wanted. For an
example, a jet engine is shot peened to help lengthen its life span; however when the engine heats up, it could possibly undergo annealing and in fact, undo the process that increases its lifespan.

Although annealing shot peened metal can be predicted, through that process the point defects will be taken out making the metal weaker. Through positron annihilation spectroscopy (PAS), or also known as Doppler broadening, it will be shown exactly what annealing does to shot peened copper. If we can get a better picture and understanding of the process of annealing shot peened metal, then we can increase the life of mechanical parts.
Chapter 2

Background

2.1 Positron Annihilation

Since the discovery of a positron (anti-electron), by Carl D Anderson in 1932, and the detection of two gamma-ray photons by S. DeBenedetti in 1949, positrons have been a means to probe solid-state material for many different applications. [5] In this case, positrons will be used to probe solid-state material for point defects in copper.

Searching for defects in copper can be complicated; however, using positron annihilation spectroscopy can help in that process. PAS is the process of shooting a positron into a material. When a positron enters a material it is moving with too much velocity to interact with the material. The forces acting on the particle once it enters the substance will cause it to slow down and to come to the same speed as the majority of the particles in the substance; this process of coming to equilibrium, takes only a few picoseconds. [3]

![Figure 2.1: Shows a positron and an electron annihilating forming two 511 keV gamma rays.](image)
Once the positron is at equilibrium it will travel around the material until it collides with and annihilates an electron, causing two gamma rays to appear. Using Einstein’s famous equation $E = mc^2$, “the direct conversion of mass into energy” will give us “two, and sometimes three, gamma-ray photons,” with energies of 511keV, when “an electron and a positron combine.” This equation will also show that it is impossible for one photon to appear from annihilation because “one photon alone cannot conserve both energy and momentum.” [6]

2.2 Doppler Broadening

Once these two gamma rays appear from the annihilated electron/positron, they carry sufficient information that can be extracted. If the material has little to no defects, then the positron will enter the material and most likely collide and annihilate with a core electron, which carries with it a great deal of momentum. A positron entering into a material that has many defects will most likely annihilate with a valance electron, which has substantially less momentum. (Figure 2.2)

![Figure 2.2](image_url)

**Figure 2.2:** Figure (a) shows a positron entering the crystal lattice of a material. This lattice does not have any point defects thereby giving the positron an equal chance of annihilating with a valance or core electron. Figure (b) shows a positron entering the crystal lattice of a material with a point defect. The forces acting on the positron will trap it in this defect. The positron will then annihilate only with a valance electron.
Since gamma rays are indeed electromagnetic waves, they exhibit wavelike properties, which include Doppler broadening. [6] The method of PAS also known as Doppler broadening spectroscopy, measures a gamma ray's width of a positron/electron annihilation to 511 keV. Since one gamma ray cannot tell us much about the material, a spectrum of gamma rays are taken and analyzed. The final spectrum consists of the sum of each individual different shifts from all different annihilations that occur. [3] This 511 keV peak can be interpreted into two separate parts; the first part is called the central part of the peak (figure 2.3), which is the positrons that annihilated with the low momentum valance electrons. The second part is known as the wings of the peak. This is the positrons that annihilated with the high momentum core electrons. Because of the high momentum, from the core electron, the positron/electron energy does not match the 511 keV but instead, will be slightly higher or lower depending on the momentum of the electrons. Because the positron and electron pair will gain energy from the electron’s orbital speed, the resulting peak will gain a significant amount of width, thus will gain wings on the original 511 keV peak. [3] The broadening effect can then be used to determine the momentum of the sum of each positron/electron pairs and therefore determine if the material has many defects, few defects or none at all.

If an atom is missing from the lattice crystals, this will attract the positron and trap it in a potential well. This will cause the probability of colliding with a valance electron to increase, which will interpret the 511 keV peak as being sharper. The opposite is for a material that does not have any defects. Since there are no point defects, or missing atoms, then the probability of a positron to annihilate with a core electron is increased, therefore causing the wings of the 511 keV peak to grow causing the peak to be wider. (Figure 2.3)
Figure 2.3: Figure (a) shows a positron annihilating with a valance electron giving off a pure 511 keV peak. Figure (b) shows a positron annihilating with a core electron giving off slightly different energies depending on the Doppler shifts.

2.3 S-Parameter

The sharpness parameter also known as the S-parameter can be measured by the width of these peaks. The S-parameter is calculated by dividing the central region (red), also known as the S-region, by the total area under the peak (blue), as shown in equation 2.1 and in figure 2.4.

\[
S = \frac{R_b}{I_b}
\]  

(2.1)

Figure 2.4 Example of a 511 keV Peak, red area represents that S-Region and the blue area represents the total peak region.
The full derivation of the S-parameter can be found on Dr. Marcus Gagliardi’s thesis and is shown in equation 2.2.

\[
\Delta S = \frac{1}{T_b} \left[ \sum_{j=p_0}^{r_o} S^2 \Delta y_j^2 + \sum_{j=r_o}^{r} (1-s)^2 \Delta y_j^2 + \sum_{j=r_f+1}^{p} S^2 \Delta y_j^2 + \sum_{i=r_o}^{r} \left( \frac{(i-P_o)}{P_f-P_o} - 1 \right)^2 \Delta b_i^2 + \sum_{i=r_o}^{r} \left( \frac{(i-P_o)}{P_f-P_o} - s \sum_{i=p_0}^{p} \left( \frac{(i-P_o)}{P_f-P_o} \right)^2 \Delta b_i^2 \right)^{1/2} \right]
\]

Dr. Gagliardi has taken these formulas and has created a computer program that can calculate the S-Parameter, with the information of the central and wings given.

For a side note the measurement of the S-parameter was very tricky. Before starting the official experiment a couple of sub experiments were preformed to guarantee the measurements of the S-parameter.

The first sub experiment was done by putting a sample in front of the detector and then without moving it at all taking 95 different measurements of the S-parameter. This will determine if the value of the S-parameter will change over time, or if it is indeed a means of measuring defects. As can be seen on figure 2.5 although the values of the S-parameter change, the values stay within 0.007 of each other signifying that the S-parameter doesn’t vary in time.
Figure 2.5 Shows the measurements of the S-parameter of the same sample 95 times.

The next sub experiment that was preformed was to determine if the S-parameters is affected by the number of counts detected. To do this a plot of the number of counts that was detected was compared to the error that was measured, using the same sample as from above. The graph of these results is found on figure 2.6.

Figure 2.6 Shows the measurement of the error due to the number of counts detected
As can be seen on figure 2.6 as the amount of counts increase the error will then decrease exponentially. Using this data it was determined that the measurements of the S-parameter that will be used will have a count of 100,000. This will give the S-parameter a range of ± 0.002 for its error.
Chapter 3

Experimental

The basic setup consisted of a data acquisition system, high purity germanium (HPGe) detector, copper coupons, and a 50 μCi $^{22}$Na radioisotope source.

3.1 Copper Coupons

The square copper coupons sides measured at 2.54 cm and had a thickness of 0.635 cm. Each of these coupons consisted of a pair in which they were sandwiched together with the $^{22}$Na source in the middle. [3] The copper coupons were first annealed at a temperature of 700° C, to make them all similar in S-parameter. The copper coupons were measured both before and after annealing as seen in figure 3.1.

After they were all annealed, point defects were introduced into the copper through the shot peening process.

![Graph showing S-Parameter vs Copper Pieces](image)

Figure 3.1: Unannealed vs Annealed copper coupons. The change in S-Parameter indicates that the annealing process removes defects from the original metal.

Picture and caption were created by Dr. Marcus Gallardi [3]
For this experiment, there are certain specifications required for the size and material of the ball bearings. Each of the copper coupons were shot peened with Society of Automotive Engineers (SAE) 70 stainless steel shot. The SAE shots have a measured diameter of 0.1778 mm. [3]

The peening intensity is measured by a strip known as an Almen strip. Each Almen strip is exact in shape, measuring in length of 7.62 cm and in width of 1.905 cm each with specific thicknesses. The only two thicknesses used in this experiment are measured to be 0.7874 mm, which is referred to as the "N" scale, and 1.2954 mm which is referred to as the "A" scale. [2] Each strip is then shot by these SAE ball bearings until the saturation curve in the Almen strip is reached. Knowing the time and the arc height of the Almen strip, the intensity of the peening can be determined. In other words, the longer you peen the material, the greater the intensity.

3.2 HPGe Detector

The HPGe detector sat inside a box that was made to eliminate vibration or at best, dampen the vibration, so that it would not produce enough noise to cause any problems with the measurements. To do this the HPGe detector was positioned in a box that had a layer of fine sand on the bottom of the box. The box was 25.4 cm tall and was aligned with frequency absorbers, which were about .635 cm thick to further reduce any possible signal noise. To further cut down the vibration of the system from heat, the Dewar was filled with liquid nitrogen. The HPGe crystal protruded out the box into a 5.08 cm thick lead house, with a 1 cm collimator at the front of the detector, as seen in figure 3.2. [3]
Figure 3.2: The Schematic illustrates the internal setup of the HPGe detector inside of the shielded house. Picture and caption were created by Dr. Marcus Galiardi [3]

3.3 Calibration

Before each experiment, the HPGe detector was calibrated by using two, 1 μCi radioactive sources, $^{137}$Cs and $^{133}$Ba, which are known to have energies of 661.657 keV and 356.0 keV respectively. More significant than the energies, however, is the full width at half max (FWHM), which has resolutions as 1.4 keV and 1.1 keV respectively.
3.4 Setup

The copper samples under test were placed roughly 2.0 cm from the HPGe detector, using an aluminum holder as shown in figure 3.3, for 1000 seconds. This distance allotted the dead time to be under 20%. Dead time occurs when the detector has to process too many positron electron annihilations and is unable to record additional annihilations for a certain amount of time. Using this time is to assure accurate results, which needs to be below 25% dead time.

Figure 3.3: The holder for the copper coupon setup. It is positioned via a series of connecting rods and placed as close to the opening of the lead collimator as possible.

Picture and caption were created by Dr. Marcus Galiardi [3]
Chapter 4

Result

4.1 Copper coupons

As mentioned before the copper coupons were peened using two different scales; The A-scale and the N-scale. In the A-scale, the copper coupons were peened with different intensities, which measured 1A, 3A, 5A, and 10A. After these coupons were peened, they were viewed under a microscope (as seen in figure 4.1). As can be observed, the copper coupons that have been shot peened with lower intensities have far less defects than those that had been shot peened with higher intensities. Not only were the defects in the copper coupons able to be seen under the microscope, but their S-parameters, when measured, were very distinct as well. [3] This can be seen in figure 4.2.

Figure 4.1: Picture of the four initial shot peened copper coupons. A) is a copper coupon shot peened at 1A. B) is a copper coupon shot peened at 3A. C) is a copper coupon shot peened at 5A. D) is a copper coupon shot peened at 10A. s the intensity of the shot increases so does the amount of damage to the surface of the copper coupons.

Picture and caption were created by Dr. Marcus Galiardi [3]
Figure 4.2: S-Parameter versus shot peened intensity for the "A" scale. As the intensity increases so does the S-parameter. The annealed and unannealed samples are included to show a comparison.

To analyze the S-Parameter measurements, the graph in figure 4.2 shows that the lower intensities have considerably less surface defects than those that were peened with higher intensities. To compare the A-series with what is known, the annealed copper and unannealed copper are included in the graph. As predicted, the annealed copper showed a considerable drop in its S-parameter compared to the A-series, which signifies that it has significantly less amount of defects.

It can be determined that the pieces that were peened at intensity of 3A and 5A are within 1 standard deviation of each other, "indicat[ing] that a saturation point was reached for those two sets of [copper] coupons." [3] Even though the saturation point was reached, it cannot be
determined at this point if it was caused by the overload of positrons, or because of the overall amount of defects caused by the shot peening process.

Due to this saturation point that was reached in the A-series, another group of copper coupons were peened and measure using the N-scale. The copper coupons for the N-series were peened as follows: 2N, 4N, 6N, 8N, and 10N. As was mentioned above, the N-scale has a lower intensity than the A-scale, which hopefully will cause the defect density to be lower, causing that either “no saturation point would be reached or that the S-parameter measurements would be below those measured previously.”

In figure 4.3 this graph shows that there was indeed a saturation point that was reached for the N-series, but it is interesting to note that the S-Parameter for the A-series and the saturation point for the N-series were within 1 standard deviation of each other. This would lean heavily on the possibility that the “saturation point was being reached through the method referred to as positron saturation. Although large variations can be observed on the surface of the varying shot peened pieces, it would appear that these defects do not extend deep into the coupons.”
Figure 4.3: S-Parameter versus shot peened intensity for the N-scale. As the intensity increases so does the S-parameter. The annealed and unannealed samples are included to show a comparison.

4.2 Quadrants

So now the A-series and the N-series have been determined that they have effectively been shot peened. Having its own unique S-parameter associated and compared to the annealed copper, the experiment can now proceed to the next phase. This part of the experiment will determine if the copper coupons can be dissected into different squares so that each can be annealed at different temperatures.

To perform this experiment the 6N copper was annealed at different temperatures ranging from 150° C, 400° C and 500° C. After these coupons were annealed at their respective temperatures, their S-parameter was measured. The results for this experiment are displayed in figure 4.3
Figure 4.4: S-Parameter versus temperature for the 6N series. As the temperature increases the measured S-Parameter decreases.

According to the graph in Figure 4.3, as the copper is annealed the defects are slowly removed. With 150° C, there isn’t much difference in that of the 20° C room temperature copper coupon. However, as the copper coupons are heated to 400° C and 500° C, there is a dramatic drop in the S-Parameter, signifying a significant drop in the amount of defects.

The problem with this data is that the information between the temperatures of 150° C to 400° C was not determined. The missing data is key in understanding the relationship of strength of metal vs. annealing temperature. It is imperative to know what is occurring between 150° C and 400° C to totally understand the annealing process in these copper coupons. Not
knowing if the graphical information of the slope of the missing data is more of a line, or a curve is significant to the outcome and conclusion of this experiment. It begs the question: Does the missing data demonstrate a significant drop of S-parameter at a specific temperature or does the annealing process have a gradual effect on S-parameter across the range of temperature? With only the information that is shown, the data cannot be extracted.

The need for this missing data has led to another set of experiments to determine the middle parts of the graph; however, obtaining this data is not so cut and dry. Due to the lack of copper coupons, the same coupons from the previous experiments will need to be cut into square quadrants each ranging in about (1.26 cm). The Series that will be tested is the 10N series.

Cutting the copper coupons might pose additional problems. When cutting copper, heat is introduced into the copper in the form of friction. This heat could possibly begin the annealing process, which might skew the results. Therefore, an experiment needed to be created that will help determine if, in fact, cutting the copper would indeed skew the results.

For this experiment, a copper coupon, which was annealed at 400 °C was measured and then cut into quadrants and then re-measured. The end results will determine if, when cutting the coupons, the heat due to friction will indeed skew the results. It is anticipated that the S-parameter will indeed be similar throughout the graph. When measuring the results, and to remain accurate with no computational error, the experiment is performed twice, once on June 30th 2009, and the second on July 9th 2009. Both results of this experiment came out to be roughly the same and the data for July 9th 2009 can be found in figure 4.4.
Figure 4.5: S-Parameter versus the four quadrants. All of the quadrants are pretty much aligned within 1 standard deviation of each other. The annealed piece is included for comparison.

As the graph shows from the July 9th experiment, all of the points are within 1 standard deviation of each other. The results show that cutting the copper coupons will not affect the S-parameter in a way that will skew the final outcome.

4.3 10N –Series

Knowing that the S-parameter is in no way affected by cutting the copper coupons, the final experiment was carried out to determine the middle ranges of the annealing process. The 10 N series copper coupons were cut up into four quadrants, making in total, 8 new coupons. Each was then annealed at specific temperatures. (200° C, 250° C, 300° C, 350° C, 400° C, 450°
C, and 500° C). Once these copper coupons were annealed at their specific ranges, they were each measured and their results are shown in figure 4.5.

![Graph](image)

**Figure 4.5**: S-Parameter versus temperature for the 10N series. As the temperature increases the measured S-Parameter decreases.

As can be seen in figure 4.5, as the temperature increases the S-parameter decreases gradually until 400°C. Once this temperature has been reached the slope of the curve begins to levels out asymptotically. The temperatures 400°C and 500°C are within the error of measurement, signifying that they could actually be the same, but there are distinct differences between all the other temperatures. This is a major breakthrough for annealed shot peened copper. The results signify that as temperatures increase the metal will become weaker, counteracting the shot peen process put in place to make it stronger.
Work Cited


