Residual stress characterization and defects analyses by microscopy

Subhra Bandyopadhyay

University of Nevada, Las Vegas

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RESIDUAL STRESS CHARACTERIZATION AND DEFECTS ANALYSES BY MICROSCOPY

by

Subhra Bandyopadhyay

Bachelor of Engineering in Mechanical Engineering
University of Mysore, India
November 2001

A thesis submitted in partial fulfillment of the requirements for the

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Graduate College
University of Nevada Las Vegas
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The Thesis prepared by

Subhra Bandyopadhyay

Entitled

Residual Stress Characterization and Defects Analyses by Microscopy

is approved in partial fulfillment of the requirements for the degree of

Master of Science in Mechanical Engineering

C. K. Joy
Examination Committee Chair

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Examination Committee Member

Examination Committee Member

Graduate College Faculty Representative
ABSTRACT

Residual Stress Characterization and Defects Analyses
by Microscopy

by

Subhra Bandyopadhyay

Dr. Ajit K. Roy, Examination Committee Chair
Associate Professor of Mechanical Engineering,
University of Nevada, Las Vegas

The structural material to contain the spallation target during transmutation of spent nuclear fuel (SNF) may develop internal stresses resulting from forming operations including plastic deformation and welding. Techniques such as pair-production, activation and life-time analysis based on classical positron annihilation spectroscopy (PAS) have been used to characterize residual stresses in candidate structural materials subjected to cold-reduction, plastic deformation and welding. Since the PAS techniques can only provide qualitative evaluation of residual stress, calibration curves were developed to estimate the resultant residual stress as a function of PAS line-shape parameters. Since plastic deformation can lead to the generation of defects in structural materials, an extensive effort has also been made to characterize the resultant defects such as dislocations and their densities as a function of the cold-reduction level through utilization of transmission electron microscopy (TEM). An excellent relationship has been established between the dislocation density and a PAS line shape parameter as a function of the cold-reduction level. Further, the metallurgical microstructures and the
morphology of failure in the tested specimens has been determined by optical microscopy and scanning electron microscopy.
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CHAPTER 1

INTRODUCTION

Energy derived from coal, fossil fuels and gas have been extensively used throughout the world during the past 100 years. However, fossil fuels can lead to the generation of green house effect producing global warming. Further, the price of energy developed from fossil fuels is also escalating at an alarming rate. Thus, energy derived from nuclear power plants appears to be the most viable option to circumvent the power crisis and unusually high prices associated with the fossil fuels. It should, however, be realized that the disposal of nuclear waste is a major challenge to all power generating nations utilizing the nuclear source. While different countries are considering nuclear waste disposal using different approaches\textsuperscript{[1]}, the United States Department of Energy (USDOE) has been considering disposal of spent nuclear fuel (SNF) and defense high level waste (HLW) in a geologic repository located 100 miles North West of Las Vegas, Nevada.\textsuperscript{[2, 3]} This repository, located at the Yucca Mountain site is designed to contain approximately 77,000 metric tones of SNF/HLW for 10,000 years. However, additional waste is gradually being generated by the current nuclear power plants, which may require their disposal in the near future in repositories yet to be built.\textsuperscript{[3]}

A process known as transmutation is currently being considered by the USDOE to dispose of SNF/HLW for shorter durations inside the proposed Yucca Mountain repository. This process can transform long-lived isotopes to species with relatively short
half lives and reduced radioactivity through capture and decay of minor actinides and fission products. \textsuperscript{[4,5]} This type of transformations of nuclear waste can occur when the nucleus of an atom changes due to the natural radioactive decay, nuclear fission, neutron-capture, or other related processes. While the concept of transmutation is being emphasized by the USDOE, other nuclear power-generating nations including France, United Kingdom, Japan and Russia are considering reprocessing of SNF. The reprocessing of SNF could enable the separation of uranium and plutonium from nuclear waste by chopping off the fuel rods and dissolving them in acids to separate the radioactive species. The recovered uranium could then be returned to a conversion plant for subsequent re-enrichment. The reactor grade plutonium would then be blended with enriched uranium to produce a mixed-oxide fuel which is known as MOX fuel. \textsuperscript{[1]}

The process of transmutation involves the bombardment of SNF by neutrons generated by directing protons from an accelerator or a reactor onto a target material. The molten lead-bismuth-eutectic (LBE) has been proposed to be a spallation target producing source neutrons from the incident proton beam, and simultaneously acting as a blanket coolant. \textsuperscript{[6]} The molten LBE will be contained in a structural vessel made of a suitable metallic material, often referred to as the target structural material. Fabrication of this containment vessel will involve normal manufacturing processes such as cold deformation, mechanical forming, and welding of similar and dissimilar materials. These types of fabrication processes can induce lattice defects such as voids or dislocations that can interact with the crystal lattice producing a higher state of internal energy, known as residual stress. These internal stresses can cause premature failures in target structural materials unless they are relieved by thermal treatments, which are commonly known as
stress relief operations. Figures 1.1 and 1.2 shows the SNF management approach and the transmutation process, respectively.

![Diagram of Spent Nuclear Fuel Management Approach](image)

**Figure 1.1 Spent Nuclear Fuel Management Approach**

The temperature generated during the transmutation process is expected to lie in vicinity of 400-550°C. Therefore, the structural material to contain the molten LBE must possess significant high-temperature metallurgical and structural stability as well as superior resistance to degradation in the presence of the molten LBE. In view of these rationales, both austenitic and martensitic stainless steels have been considered as candidate structural materials for transmutation applications. Martensitic Alloys EP-823, HT-9 and 422, and austenitic Type 304L stainless steel (SS) have been tested for characterization of residual stresses by previous investigators at UNLV using both
destructive and nondestructive techniques. However, the present investigation involving all four alloys is being conducted through utilization of the nondestructive testing methods only.

Figure 1.2 Transmutation Concept

Residual stress can be defined as a stress that can be contained inside the matrix of a structural component, in collaboration with its surroundings without application of any external load such as applied force or temperature fluctuations. Depending on the dimensions and metallurgical characteristics of a part to be analyzed, three different types of stresses can be categorized. The first type (type 1) of residual stress called macroscopic can vary continuously over large distances extending at least several grains of a material of interest. This type of stress is different from residual stresses which can
develop over a particular grain size, categorized as type II resulting from intergranular stresses. Type II stresses can exist in polycrystalline materials such as martensitic stainless steels due to their elastic and thermal properties among grains oriented in different directions. Thus, these types of stress can be generated in materials with different phrases and physical characteristics including the presence of inclusions. The type III stress can occur in submicroscopic areas, several atomic distances within a grain and can manifest due to the pileup of dislocations in the vicinity of the grain boundaries (Frank-Reid Mechanism) thus enhancing the retained stresses. The second and the third type of stresses are commonly referred to as microstresses. All three types of stresses are illustrated in Figure 1.3.

Figure 1.3 Different Types of Residual Macro and Micro Stresses

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The nature and the magnitude of residual stress generated in materials can be influenced by many factors including their physical characteristics and thermo-mechanical parameters. Physical properties such as thermal conductivity, elastic modulus, poisson’s ratio, coefficient of expansion and contraction and thermodynamic parameters can play significant roles in developing and enhancing the extent of residual stresses. Further, different manufacturing processes including melting, casting, forging, drawing, extruding, rolling and bending etc or machining of parts can induce internal stresses. In addition, welding, joining, and thermo-mechanical treatments including shot peening, laser peening, quenching, carburizing, nitriding, carbonitriding, case hardening, ionplanting and electrodeposition can influence the nature and the extent of residual stresses in structural materials.

The residual stresses may be either beneficial or detrimental depending on the magnitude, sigh and distribution of internal stresses with respect to the design stress. In general, the tensile residual stresses are undesirable since they can contribute to the enhancement in the susceptibility of a component to fatigue failure, creep deformation, quench cracking and environment-assisted embrittlement such as stress-corrosion cracking and hydrogen damage. On the contrary, the generation of compressive residual stresses is beneficial to counter balance the damaging effect of tensile stress. In view of this rationale, many components are subjected to induction of compressive residual stresses by processes such as shot-peening, and laser-peening.

Numerous quantitative and qualitative techniques have been developed over the past few decades to characterize residual stresses in structures and components. These include both destructive and nondestructive methods. A destructive method known as ring core
technique has been utilized by a previous investigator at UNLV, which involves milling of annular groove on the surface on the surface of the measurement object around a special strain-gauge rosette bonded to the surface. The strain relieved during the coring operation is used to calculate the residual stress using elastic-plastic relationships. The measured residual stresses were then compared to three nondestructive techniques such as X-Ray diffraction (XRD), neutron diffraction (ND) and positron annihilation spectroscopy (PAS). Both XRD and ND have been extensively used by numerous investigators to characterize residual stresses in metallic materials. However, the XRD technique cannot be used in materials with larger grain size. Therefore, the current investigation is focused on the utilization of ND and PAS techniques to characterize residual stresses in both austenite and martensitic materials.

The USDOE has been emphasizing the development of a non destructive technique based on the positron annihilation spectroscopy to characterize residual stresses in target structural materials having residual stress in target structural materials having residual stresses generated by plastic deformation, cold reduction in thickness and welding operations. Even though the PAS technique has been used in several investigations performed at UNLV,\textsuperscript{[13-15]} this technique still needs to be perfected for its utilization as a standardized tool in residual stress measurements. Further, the resultant data need to be compared to those obtained by other nondestructive techniques such as ND. It should, however, be noted that the residual stresses characterized by the PAS techniques in terms of the resultant line shape parameters is not a quantitative phenomenon. Thus, the calibration of the resultant parameters is needed to estimate the residual stress in materials subjected to the different manufacturing processes. The PAS technique is
capable of residual stress measurements by virtue of two different concepts known as pair production and activation. Both approaches have been used in this investigation along with the neutron-diffraction method.

Different configurations of specimens have been tested for characterization of residual stresses. Efforts have been made to develop calibration curves involving Alloys EP-823, HT-9, 422, and Type 304L SS. The PAS measurements have been performed at the Idaho Accelerator Center (IAC) of the Idaho State University. The residual stress measurements based on performed at the Atomic Energy of Canada, Limited, Chalk river laboratory.

Microscopic evaluations using optical microscopy, scanning electron microscopy, (SEM), and transmission electron microscope (TEM) were performed at UNLV. The optical microscopy was used to evaluate the metallurgical microstructures of the tested materials. SEM was used to analyze the morphology of failure in tensile specimens. Since significant amount of defects such as dislocations and voids can be generated in cold-worked material, TEM was used to develop micrographs for identification and characterization of defects by state of the art technique. This technique was based on the determination of dislocation density that has been compared to the PAS line shape parameter as a function of the degree of cold reduction in a martensitic alloy. The compressive test results including residual stress measurements, calibration data and microscopic evaluations has been presented in this thesis to develop a basic understanding on residual stress in candidate target structural materials.
CHAPTER 2

TEST MATERIALS AND SPECIMENS PREPARATION

2.1 Test Materials

The materials tested in this investigation include austenitic Type 304L stainless steel (SS), and martensitic Alloys EP-823, HT-9 and 422. These materials have been extensively used in Europe and Russia as containment materials in the transmutation system primarily due to their desirable properties including excellent corrosion resistance, optimum strength and the ease of manufacturing. Stainless steels are high alloy steels that possess excellent corrosion resistance compared to other steels due to the presence of high chromium (Cr) content. Stainless steels can be divided into three types based on their crystalline structure, namely austenitic, martensitic and ferritic.

Austenitic stainless steels exhibit a single-phase, face-centered-cubic (fcc) structure that can be maintained over a wide range of temperatures, and are categorized as 200 and 300 series, containing 16 to 30 weight percent (wt %) Cr and 2 to 20 wt% nickel (Ni). The presence of Cr and Ni can provide enhanced surface quality, formability, increased corrosion and wear resistance. Austenite is formed due to the presence of austenitizing elements such as Ni, manganese, and nitrogen. Since these alloys are predominantly single phase, they can be strengthened only by solid-solution alloying or by work-hardening. Austenitic stainless steels are effectively nonmagnetic in the annealed condition, and can be hardened by cold-working. Due to these characteristics,
they are sometimes used in applications where magnetic materials are not acceptable. Some ferromagnetism may be noticed due to cold-working or welding. These types of materials typically have reasonable cryogenic and high temperature tensile properties.

Austenitic stainless steels have also been reported to possess superior oxidation and corrosion resistance in molten LBE due to their relatively high chromium content\[^{[16]}\]. The austenitic classes of SS are those that are weldable by common fusion and resistance techniques.\[^{[17]}\] They are susceptible to cracking during solidification. Cracks can occur in various regions of the weld with different orientations, such as centerline cracks, transverse cracks, and microcracks in the underlying weld metal or adjacent heat-affected-zone (HAZ). These cracks are primarily due to low-melting liquid phases, which allow boundaries to separate under the thermal and shrinkage stresses during welding. The ratio of Cr and Ni can be modified to improve the formability of austenitic SS. The carbon (C) content can be reduced to improve their intergranular corrosion resistance. Similarly, the addition of molybdenum (Mo) can improve the localized corrosion (pitting/crevice) resistance in these materials.

Type 304 SS is an austenitic alloy possessing a minimum of 18% Cr and 8% Ni combined to a maximum C content of 0.08%. It is a universally-known corrosion-resistant alloy having iron-nickel-chromium (Fe-Ni-Cr) possessing optimum formability and weldability. Type 304L SS contains a maximum C content of 0.03% that can assist in prevention of carbide precipitation during thermal treatments. This material is known to provide excellent corrosion resistance for applications in the chemical, textile, petroleum, dairy and food industries. The maximum temperature to which Type 304L SS can be
continuously exposed without appreciable scaling is about 900°C. Since this material can work-harden rapidly, annealing may be necessary to reduce its hardness and, thus enhancing its ductility. The combination of relatively lower yield strength (Ys) and optimum ductility makes this material formable and drawable.\textsuperscript{[18]}

Martensitic stainless steels are currently finding extensive applications in nuclear reactors as substitutes for austenitic steels. They are basically Fe-Cr alloys with relatively higher C content having body-centered cubic (BCC) or body-centered tetragonal (BCT) martensitic crystal structures in the hardened condition. They are ferromagnetic, and hardenable by heat-treatments. Martensitic stainless steels are usually preferred for their relatively high strength, moderate corrosion resistance and optimum fatigue properties resulting from suitable thermal treatments. Their resistance to general corrosion is adequate in some corrosive environments, but not as good as other types of stainless steels.

The Cr content of the martensitic SS can range from 9 to 18 wt%. The Cr and C contents are balanced to ensure a martensitic microstructure after hardening. The presence of Mo and Ni in these alloys can also improve their mechanical properties and the corrosion resistance.\textsuperscript{[19]} The presence of Ni can also assist in maintaining the desired microstructure and preventing the formation of delta ferrite when high concentration of Cr is present. The brittle martensitic microstructures resulting from the austenitizing and quenching operations can be modified to enhance the ductility of the matrix by tempering at a lower temperature.\textsuperscript{[17]}

Alloy EP-823 is a Russian nuclear grade martensitic Fe-Ni-Cr-Mo stainless steel with high silicon (Si) content (1.0-1.3 wt %). It is a leading structural material to contain a
spallation target such as molten LBE that can also act as a coolant in the Accelerator-Driven-Spallation (ADS) system.\textsuperscript{[20]} This material has also been used in the United States as internal components in experimental liquid metal fast breeder reactors (LMFBR) due to its moderate corrosion resistance, optimum strength, ease of manufacturing and relatively lower cost. This alloy possesses significant resistance to swelling during high neutron exposure at temperatures up to 420°C and a low rate of irradiation creep. However, the strength of this steel may drop below 500°C.\textsuperscript{[21]} This steel can retain its high post-irradiation ductility at test temperatures ranging between 20 and 700°C,\textsuperscript{[22]} even in an irradiated condition.

Alloy HT-9 is a Swedish nuclear grade martensitic stainless steel containing Fe-Ni-Cr-Mo. Even though these alloys have excellent swelling resistance at doses up to 200 displacements per atom (dpa), their creep resistance decreases drastically above 550°C.\textsuperscript{[23]} Alloy HT-9 is also an excellent material for cladding and duct applications in liquid metal reactors. It has good swelling resistance and is also resistant to irradiation embrittlement, particularly at 60°C.\textsuperscript{[23]}

Type 422 stainless steels falls under the category of 9-12% chromium steel such as Types 410 and 420. This alloy can be heat treated to various strength levels, up to a maximum of 250,000 psi. It has higher mechanical strength, lower thermal expansion, higher swelling resistance, lower work-hardening index, higher thermal conductivity, higher resistance to irradiation creep and helium-embrittlement, and better liquid-metal compatibility than the austenitic stainless steels. Type 422 SS can retain high strength and toughness at temperatures up to 650°C.\textsuperscript{[24,25]} Besides nuclear applications, it can also be used for highly stressed components such as the turbine blades and high-strength
fasteners for applications in corrosive environments due to its ability to be heat-treated in large sections. Further, it has high damping capacity and good resistance to fatigue, thermal shock and hydrogen damage. The typical mechanical and physical properties of materials tested in this investigation are given in Table 2.1

Table 2.1 Mechanical and Physical Properties of Materials Tested[26, 27]

<table>
<thead>
<tr>
<th>Property</th>
<th>Material</th>
</tr>
</thead>
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<tr>
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<td>Type 304L SS</td>
</tr>
<tr>
<td>Thermal Conductivity (W/m*K)</td>
<td>16.2</td>
</tr>
<tr>
<td>Poisson’s Ratio at Ambient Temperature</td>
<td>0.23</td>
</tr>
<tr>
<td>Coefficient of Thermal Expansion °C * 10^6</td>
<td>9.4</td>
</tr>
<tr>
<td>Yield Strength Ksi (MPa)</td>
<td>55 (379.3)</td>
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<tr>
<td>Tensile Strength Ksi (MPa)</td>
<td>72 (496.5)</td>
</tr>
<tr>
<td>Modulus of Elasticity ( *10^3 ) Ksi</td>
<td>193</td>
</tr>
</tbody>
</table>

Experimental heats of all four materials were melted by a vacuum-induction-melting practice at the Timken Research Laboratory (TRL), Canton, Ohio. They were subsequently forged, and hot-rolled into plate materials of desired dimensions. These materials were then heat-treated prior to the machining of the test specimens. Type 304L SS plates were austenitized at 1010°C (1850°F) for 1 hour followed by air cooling, thus producing a fully-austenitic microstructure. Alloys EP-823, HT-9 and 422 were austenitized at a similar temperature followed by an oil-quench. The quenched plates
were subsequently tempered at 621°C (1150°F) followed by air-cooling. This type of thermal-treatment produced fully-tempered martensitic microstructure without any retained austenite. The chemical compositions of all four materials tested are given in Table 2.2

<table>
<thead>
<tr>
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<td>C</td>
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<td>12.83</td>
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<td>Ni</td>
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<td>0.53</td>
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<td>Mo</td>
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<tr>
<td>V</td>
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<td>--</td>
<td>0.32</td>
<td>--</td>
<td>0.29</td>
<td>0.22</td>
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<tr>
<td>W</td>
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<tr>
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<td>--</td>
<td>--</td>
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</tr>
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<td>0.033</td>
<td>0.0029</td>
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<td>--</td>
</tr>
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<td>Bal</td>
<td>Bal</td>
<td>Bal</td>
<td>Bal</td>
<td>Bal</td>
</tr>
</tbody>
</table>

2.2 Specimens Preparation

2.2.1 Tensile, Cold-Worked, and Welded Specimens

Three different types of specimens were fabricated from the experimental heats. They include cylindrical tensile, cold-worked and welded specimens. Smooth cylindrical specimens were machined from the heat treated plates in the longitudinal rolling direction. The configuration of the cylindrical specimens having 4-inch (101.6mm) overall length, 1-inch (25.4mm) gage length and 0.25-inch (6.35mm) gage diameter is
shown in Figure 2.1. A part of the heat-treated plates was plastically-deformed by cold-rolling to reduce the plate thickness by approximately 3, 5, 7, 11 and 15 percent, respectively. The configuration of the cold-worked specimens is shown in Figure 2.2. Welded specimens consisting of similar (Type 304L SS, Alloy EP-823 and Alloy HT-9) and dissimilar metals (Alloy EP-823 and Type 304L stainless steel) were prepared using the gas-tungsten-arc-welding (GTAW) method. For welded specimens consisting of similar materials (austenitic and martensitic SS) on both sides, Type 308L SS and Type 2283L SS, respectively were used as filler material. For the welded specimens consisting of dissimilar materials an (austenitic and martensitic SS) on either side, IN 82 was used as the weld metal. Figure 2.3 illustrates the welded specimens with detailed dimensions.
Figure 2.2 Configuration of the Cold Worked Specimens
2.2.2 TEM Specimens

Transmission Electron Microscopy (TEM) was used to develop micrographs for analysis of defects in Alloy EP-823 subjected to cold deformation at different levels (0, 7 and 11 percent). These micrographs were eventually used to estimate the dislocation density ($\rho$) as a function of the cold reduction level. The magnitude of $\rho$ was then compared to the line shape parameters determined by pair-production and lifetime analysis.

The preparation of the TEM samples is a tedious process. These samples have to be very thin in order for the electrons to penetrate into them. The sample thickness may vary
from 100-200 microns. The specimen preparation requires a sequence of processes, described below.

- Abrasive cutting
- Diamond saw cutting
- Mounting of specimens
- Mechanical polishing
- Punching
- Electropolishing

Abrasive Cutting, Diamond Saw Cutting and Mounting of Specimens

The test specimens were initially sectioned transversely followed by longitudinal cutting using an abrasive cutting tool. These operations are illustrated in Figure 2.4. The sectioned specimens were then subjected to fine cutting by diamond saw at a low speed as shown in Figure 2.5. Some weights were placed on the cutting tool to apply a gravitational force on the specimen. The time to cut was controlled by the speed of cutting. The thickness of fine cut specimen varied between 500-700 microns. These specimens were then mounted on specimen holders heated to a desired temperature followed by the placement of a wax coating on this holder until the wax got melted and finally became attached to the specimen. The coated specimen was then subjected to mechanical polishing, as illustrated in Figure 2.6.
Figure 2.4 Cold Work Specimens used in this Study

Figure 2.5 Diamond Cutter with the specimens cut

Figure 2.6 Specimen Holder, Heater and Cold Plate
Mechanical Polishing, Punching and Electropolishing

The sample holder was then placed on a planar grinder, which assisted in the removal of large amount of material at a controlled rate. Mechanical Polishing was done using a rotary grinding/polishing wheel through utilization of a 600 grit abrasive paper\textsuperscript{[28]} as shown in Figure 2.7. The mechanically polished specimens were then punched to a point 0.3 mm diameter using a punching tool, as shown in Figure 2.8. Electropolishing of the punched specimens were then performed to remove material for preparing the final specimen for TEM analysis. The electropolishing is based on electrochemical reactions resulting from current distribution in flowing electrolyte to achieve the desired thickness and surface finish prior to the examination of the test specimen by TEM. Figure 2.9 shows an electropolishing cell and a separate power control.

Fig 2.7 Mechanical Polishing
Care was taken to control the flow of electrolyte to prevent the formation of anodic film that could etch the specimen rather than polishing. The composition of electrolyte used in TEM sample was a solution of 5% perchloric acid in ethanol using 40 volts and at a flow rate of 12 and at a temperature of -7°C (19.4°F). Due to current distribution within the specimen, electropolishing results in an overall smoothing of the specimen when compared to the valleys. A light source and photo-electric detector are utilized to
determine the precise time when a perforation occurs. Light is directed at the specimen by a fiber optic bundle. As the electrolyte action produces a perforation in the specimen, a second fiber optic bundle directs the light transmitted through the specimen to a photo detector. Once the hole is produced, electropolishing stopped automatically. The specimen is then cleaned in acetone and finally with ethyl alcohol. The final specimen is stored in air tight desicador before observation in TEM.
CHAPTER 3

EXPERIMENTAL TECHNIQUES

Residual stress measurements were performed on different specimens by two nondestructive techniques. They are positron annihilation spectroscopy (PAS) and neutron diffraction (ND). Even though, a major emphasis has been placed on the applicability of the PAS technique for characterization of residual stresses in this thesis, efforts have also been made to compare the results obtained from the ND technique. Two different methods based on the PAS technique including pair-production and activation has been used to characterize residual stresses in target structural materials. Further, positron lifetime studies have also been performed to evaluate the types of defects present in the cold-reduced specimens. Optical microscopy has been used to characterize metallurgical microstructures of the tested alloys. Morphology of failure in tensile specimens has been determined by scanning electron microscopy. Transmission electron microscopy has been used to characterize defects such as dislocations and their densities in the cold-worked specimens, and correlate them to the PAS line-shape-parameters as well as the positron lifetimes of the specimens subjected to different levels of cold-reduction.
3.1 Positron Annihilation Spectroscopy

3.1.1 Positrons

The positron is the anti-particle of an electron. It has a single positive charge, which has a same mass as an electron, but is quite unstable in the presence of electrons. A positron and an electron will annihilate each other, using their total mass to produce two high-energy photons (γ-rays) each having an energy of 511 KeV, that can be emitted in diametrically opposite directions if the original electron and the positron were both stationary. [30-31] This symmetrical emission of two γ-rays is to conserve zero initial momentum, and can be Doppler shifted due to the center of mass velocity of the electron-positron pair. The lifetime of a positron is infinite in the absence of electrons but its life is reduced significantly if it is introduced to an electron-rich environment. The positrons were discovered by Dirac in 1928. [32] The history of positron development is given in Table 3.1.

Table 3.1 History of Positron Development [33]

<table>
<thead>
<tr>
<th>Year</th>
<th>Development</th>
</tr>
</thead>
<tbody>
<tr>
<td>1928</td>
<td>Positrons were discovered by Dirac</td>
</tr>
<tr>
<td>1932</td>
<td>Positron found in cosmic radiation by Anderson</td>
</tr>
<tr>
<td>1940’s</td>
<td>Interaction of Positron with matter</td>
</tr>
<tr>
<td>1940-50</td>
<td>First studies of electronic structure of solid by angular co-relation</td>
</tr>
<tr>
<td>1950-60</td>
<td>Establishment of Doppler-Broadening and lifetime techniques</td>
</tr>
<tr>
<td>End of 60’s</td>
<td>Establishment of annihilation parameters sensitive to lattice imperfections.</td>
</tr>
<tr>
<td>1968</td>
<td>First LINAC based positron generation</td>
</tr>
<tr>
<td>1982</td>
<td>Positron moderation and slow-positron beams</td>
</tr>
<tr>
<td>1998</td>
<td>First microprobe under operation</td>
</tr>
</tbody>
</table>
3.1.2 Sources of Positrons

In conventional positron annihilation experiments, positrons can be readily obtained from radioactive isotopes such as $^{22}$Na, $^{58}$Co, $^{64}$Co, $^{68}$Ge, which can be directly injected into a specimen. The implantation depth of positrons in this case corresponds to 0.1-1 mm. Amongst these radioactive isotopes, the most popular positron source is $^{22}$Na since its half-life period is 2.6 years. In addition, the efficiency of the $\beta^+$ i.e. the positron branching ratio $\left(\frac{\text{number of Positron emissions}}{\text{total number of decay emissions}}\right)$ is 90%. $^{[33]}$ Further, $^{22}$Na is convenient for lifetime spectroscopy since it can emit a high energy $\gamma$-ray of the order of 1.28 MeV within 10 picoseconds. The simultaneous emission of $\gamma$-rays can be used in lifetime spectroscopy to identify the birth of positrons. The $\beta^+$ can be represented by the equation given below. A major limitation of using $^{22}$Na is its shallow penetration depth ranging between 0.1 and 1 mm.

$$^{22}\text{Na} \rightarrow ^{22}\text{Ne} + e^+ + \gamma\text{-rays (1.28 MeV)}$$

Alternatively, the production of low-energy positrons can be accomplished either by pair-production using a high-energy accelerator or use of an activation technique based on radioactive decay of a nucleus. When energetic electrons are decelerated in matter, photons can be emitted through Bremsstrahlung process. When the photon energy is more than twice the electron rest mass energy, positrons can be produced along with electrons by the pair conversion of these photons. This phenomenon can develop discontinuous positron beams at rate of $10^9$ positrons ($e^+$) per seconds. This technique had been efficiently utilized in many research facilities including the IAC at the Idaho State University. $^{[34]}$ Three different methods of residual stress measurements based on the
classical positron annihilation spectroscopic (PAS) method are described in the following sub-sections.

3.1.3 Measurements by Pair-Production

Different experimental techniques based on positron annihilation have been developed in the past. [31] Broadly, they can be classified into two categories. The first method is distinguished by the sensitivity of positron annihilation to the electron density, and to the electron momentum distribution which is known as positron annihilation spectroscopy. The second method, known as positron lifetime spectroscopy is based on the fact that positron annihilation rates are sensitive to the types of defects present in the metal lattice.

The electron momentum techniques are based on the fact that the positron rapidly thermalizes in matter before annihilating, and thus, the positron does not contribute significantly to the momentum whereas the electron, in contrast, may have significant momentum. Thereby, the electron contributes significantly to the center of mass of the annihilation pair. [31] From the conservation of momentum, the two photons of the annihilation process can be emitted in a nearly collinear position, but the presence of significant electron momentum may cause them to be emitted at a slight angle relative to each other. This is the basis of the angular correlation annihilation radiation and Doppler broadening techniques.

PAS has been successfully utilized in non-destructive evaluation of residual stresses in structural materials. PAS is highly sensitive for lattice disorders in crystalline materials. The underlying principle of the PAS technique is illustrated in Figure 3.1. The positron looses its high energy within a few seconds due to thermalization following its
production. The positron is sensitive to the lattice defects such as dislocations and vacancies existing in metals and alloys. At the defect site, the electron density is relatively lower compared to the perfect lattice because of the missing atoms. The positrons generated inside the test sample will be diffused into the metal lattice and subsequently be annihilated with an electron from the surrounding lattice. While the positrons will be localized in a defect-free lattice, it could be repelled by the nucleus, thus transforming them into delocalized condition primarily in the interstitial regions. In the presence of defects, the positron may be trapped in the vicinity of defects due to the absence of repulsive potential of the positively charged atomic nucleus. The momentum of the positron can eventually be neglected due to the thermalization process. Therefore Doppler broadening of the annihilation radiation is primarily determined by the electron momentum.

![Figure 3.1 Positron Annihilation Principle](image)

Figure 3.1 Positron Annihilation Principle \cite{32}
Each positron generated by this technique can be thermalized and annihilated with one of the sample electrons emitting two photons having a 511 KeV energy spectrum. The 511 KeV spectrum, also known as characteristic curve is used in analyzing defects. Line shape parameters S, W and T are used to characterize the annihilation peak in Doppler broadening spectroscopy, as shown in Figure 3.2. [31] The S parameter is sensitive to the annihilation with valence electrons and is defined as the ratio of the counts in central region to the total counts in the peak. The W parameter is more sensitive to the annihilation with high momentum core electrons and is defined as the ratio of the counts in the wing regions of the peak to the total number of counts in the peak. The T parameter is simply the ratio of W to the S parameter. As evident, it can be seen from the figure, the S-parameter is directly proportional to the residual stresses, while the W and the T-parameter is inversely proportional to the internal stress developed inside the specimens used in the investigation. [35-38]

![Figure 3.2 Characteristics of 511 KeV Gamma-Ray Energy Spectrum](image)

S = $A_o / A_0$

$W = A_w / A_0$

$T = W / S$

$A_o$=Total Area
3.1.4 Measurements by Activation

The activation technique was based on the utilization of a photo-nuclear (\(\nu, n\)) reaction involving a 20 MeV bremsstrahlung beam. This principle is based on the radioactive decay of a nucleus. \(^{[39]}\) In this case a bremsstrahlung beam having an approximate energy of 20 MeV induces photo-nuclear \((\gamma, n)\) reactions in the sample of interest and renders the sample radioactive. Some of the resulting neutron-poor (proton-rich) nuclei in turn can emit positrons through a beta decay process. This technique, thereby, creates positrons inside the matrix of the test material as a part of the photo-nuclear reaction. It is important to note that reactions other than photo-nuclear can also be employed for the activation technique. Any nuclear reaction that leaves nuclei in a proton-rich state can be used. The gamma-dose delivered to samples by this method is typically of the order of \(10^4 \text{ Gy}\) or more. The stainless steels, studied in this investigation, contained a substantial amount of iron that generated positrons according to the photo-nuclear reactions shown below. Since, the half life of radioactive Iron is at around 8.5 minutes, the evaluation of defects by this technique can be performed within a short time. The positron generated by the activation technique can also thermalize and annihilate with the sample electrons emitting 2 photons having a 511 KeV energy spectrum. It is still unknown if this activation technique can induce radiation hardening of Iron within such a short time period. \(^{[40]}\)

\[
^{54}\text{Fe}_{26} + \nu \rightarrow ^1\text{n}_9 + ^{53}\text{Fe}_{26}
\]

\[
^{53}\text{Fe}_{26} \rightarrow e^+ + ^{53}\text{Mn}_{25}
\]

Where, \(\nu\) and \(n\) represent the gamma ray and neutron, and \(e^+\) signifies positron.
3.1.5 Measurement by Positron Lifetime Spectroscopy (PLS)

Positron Lifetime spectroscopy is a well established tool in studying the properties of vacancy type defects in metals and semi conductors.\footnote{\cite{41}} Annihilation is a random process, thus the positron lifetime, i.e. the time interval between the injection of a positron into a sample and the instant of annihilation, has a statistical distribution. This is called the positron lifetime spectrum. The fact that open volumes defects can trap positron makes it possible to distinguish between defect-free sample and a sample containing vacancies.\footnote{\cite{42}} In defect-free homogeneous metals, the life time spectrum is a simple decaying exponential. In the presence of trapping at defects, the lifetime spectrum becomes a superposition of decaying exponentials. That means the average electron density at the vacancy is lower than in the bulk and therefore the lifetime of the trapped positron is increased compared to the value in the perfect bulk lattice.

In general, the lifetime of a component is a function of the number and the types of defects in it. The lifetime of positrons in metallic materials can range from 100 to 500 picoseconds (ps). For Iron without any imperfections, this lifetime is in the vicinity of 106 ps, which can be enhanced in presence of monovacancies and dislocations (ranging between 120 and 180 ps). However, the lifetime due to the presence of voids (40-50) can be increased upto 500 ps. Therefore, the estimation of lifetimes values of the positrons can shed a significant light on the nature of deformation and resultant defects at the materials. Figure 3.3 gives an example of such illustration.\footnote{\cite{43, 44}}
Figure 3.3 Lifetimes of Different types of Defects Associated in Single Fe Crystal

Lifetime analysis have been performed in this investigation on Alloy EP-823 (heat no 2154) subjected to cold deformation at two different levels (7 and 11 %). For evaluation of residual stresses in these cold-worked specimens, they were placed closed to two Boron-Fluoride detectors for recording the birth and death of positrons. Software named Program LT v9 was used to calculate the average lifetimes of the cold worked specimen using the best fit model.

3.1.6 Experimental Facility

The PAS techniques used in this investigation employed high penetrability gamma rays to extend PAS into thick samples and to enable measurements of residual stress, and defects in various materials of interest. These high penetrable gamma rays were produced by two linear accelerators (LINAC), a 30 MeV (for pair-production) and a 20 MeV for (Activation). The collimated bremsstrahlung beam from the 30 MeV LINAC was used to generate positrons inside the test specimen via pair production as seen in Figure 3.4.
The frequency of the accelerator, which is often referred as repletion rate is maintained at 500 Hz throughout for reliability of data. The resulting bremsstrahlung beams are doubly collimated with a 20 cm thick, 0.6 cm diameter stainless steel primary collimator, followed by a 15 cm thick, 1.8 cm diameter lead secondary collimator. As discussed earlier, each positron generated by this technique thermalized and annihilated with one of these electrons emitting two photons having 511 KeV, back to back. These were then recorded by a high purity germanium (HPGe) detector. The 30 MeV LINAC used for pair-production is shown in Figure 3.5.
The activation process involves using a 20 MeV accelerator. This accelerator was used to render the material radioactive. Very high energy gamma rays were required for this purpose. The materials used in this investigation were treated for 15 minutes. The 20 MeV accelerator is shown in Figure 3.6. After, this process, the materials were checked for required dose rates. Efforts were made to check for a consistency of the dose rates each sample received. This process created positrons as a result of radioactive decay or activation. These samples were then recorded using a high-energy germanium (HPGe) detector. These detectors are highly sensitive to any vibrations and other forms of distractions; hence this detector was shielded with 8" lead bricks all around. Two different sources Ba and Cs were used in this study for calibration. Ba gives two continuous energy photons in order of 81 KeV and 356 KeV respectively, Cs gives photon rays in order of 662 KeV. These were used for determining 511 KeV
characteristic curve. MPANT software was used to determine the energy curve which was then analyzed for calculation of (S, W and T) parameters by using a FORTRAN program. A typical MPANT showing 81, 356, 511 and 662 KeV lines are shown in Figure 3.7.

Figure 3.6 20 MeV LINAC

Figure 3.7 MPANT Software Showing Different Energy Peaks
3.2 Neutron Diffraction

The ND method relies on elastic deformations within a polycrystalline material that cause changes in the spacing of the lattice planes (d-spacing) from their stress-free value. During this experiment, a collimated neutron beam of known wave-length (0.156 nm) was diffracted by the test specimen, followed by its passage through a second collimator finally reaching the detector, as shown in Figure 3.8.

![Figure 3.8 ND Test Setup](image)

The interplanar distance ‘d’ can be evaluated using the Bragg’s law, and the corresponding lattice strain can be evaluated using the following equation.

\[
\varepsilon_{hkl} = \frac{(d_{hkl} - d_0)}{d_0}
\]

(Equation 1)
Where, $d_{hlkl}$ is the interplanar distance in the stressed material, $d_0$ is the interplanar distance in a stress-free material, and $\varepsilon_{hlkl}$ is the lattice strain.

The stress values were subsequently determined from these calculated lattice strains using appropriate mathematical equations. When the diffraction data are taken from many grains of randomly oriented polycrystals, the strains measured by the neutrons correspond to macro-strains and are related to the macro-stresses by the equation of isotropic elasticity. In an elastically-isotropic model, the principal stresses $\sigma_{xx}$, $\sigma_{yy}$, $\sigma_{zz}$ are related to the strains by the following equations.

\[
\sigma_{xx} = \frac{E}{(1 + \nu)(1 - 2\nu)} \left[ (1 - \nu)\varepsilon_{xx} + \nu(\varepsilon_{yy} + \varepsilon_{zz}) \right] \quad (Equation 2)
\]

\[
\sigma_{yy} = \frac{E}{(1 + \nu)(1 - 2\nu)} \left[ (1 - \nu)\varepsilon_{yy} + \nu(\varepsilon_{xx} + \varepsilon_{zz}) \right] \quad (Equation 3)
\]

\[
\sigma_{zz} = \frac{E}{(1 + \nu)(1 - 2\nu)} \left[ (1 - \nu)\varepsilon_{zz} + \nu(\varepsilon_{xx} + \varepsilon_{yy}) \right] \quad (Equation 4)
\]

where, $E$ is Young's modulus, $\nu$ is the Poisson's ratio, $\varepsilon_{xx}$ is the principal strain in the X-direction, $\varepsilon_{yy}$ is the principal strain in the Y-direction, and $\varepsilon_{zz}$ is the principal strain in the Z-direction.

Due to the anisotropy of elastic properties in crystalline materials, the values of $E$ and $\nu$ at a microscopic level depend on the lattice plains $(hkl)$ considered. Thus, the neutron elastic constants must be known or can be determined experimentally. The nature of residual stress (tensile versus compressive) was also evaluated by analyzing the magnitude of the $d$ parameter.
3.3 PAS Calibration Curve

The residual stresses characterized by the line shape parameters (S, W and T) can provide only the quantitative information. Therefore an effort was made to relate the magnitude of S, W and T parameters to the tensile stresses imparted to cylindrical specimens by applying loads beyond the Y.S but within the U.T.S. The engineering stress vs. strain (s-e) diagram was determined by pulling smooth cylindrical specimens at the ASTM-specified strain rate of $10^{-3}$ sec$^{-1}$ (E8). The material testing system equipment used in tensile testing is shown in Figure 3.9. The magnitude of stress and strains (e) was recorded from this plot. Subsequently, similar types of cylindrical specimens were strained in tension up to specific loads ranging between YS and UTS. Once the desired load was achieved in this range, the straining of the specimen was stopped and the corresponding elongation was recorded. These specimens were then subjected to residual stress characterization in terms of (S, W and T) parameters, providing a calibration curve slowing these parameters individually as a function the applied stress. In essence, these calibration curves can enable the estimation of residual stresses in terms of any of these line shape parameters corresponding to applied plastic stress for a material of interest.
3.4 Stress Relief Operation in Welding

The structural materials to be used to contain the molten LBE will be subjected to different manufacturing processes including welding. Irrespective of the type of material used in the welded structure, residual stress will be generated on both sides of the weld. However, the nature of residual stress may be different depending on whether similar or dissimilar materials are used on either side of the weld. Attempts have been made in this investigation to analyze residual stresses in welded specimens consisting of austenitic and martensitic stainless steels. Since the metallurgical characteristics of austenitic and
martensitic stainless steel are quite different, the magnitude of residual stress may be
different due to different metallurgical microstructures, grain size and physical properties
such as coefficient of expansion and rate of solidification.

During welding, different regions may be developed including the fusion line (line of
contact between the weld and base metal), heat affected zone (HAZ), and the base
material as shown in Figure 3.10. Since the rate of solidification in the vicinity of the
fusion line will be substantially faster compared to the other regions, it is anticipated that
the extent of residual stress close to the fusion line will be maximum followed by gradual
reduction away from this region. Since the presence of residual stress in a welded
structure can be determined from the design point of view, it is customary to minimize or
eliminate these internal stresses by means of thermal treatments that are commonly
known as stress relief operations. Minimization or elimination of these stresses can also

Figure 3.10 Different Zones in a Welded Structure [47]
be beneficial for stress corrosion cracking resistance in the presence of a molten metal such as LBE, and reduction in radiation hardening during the transmutation process.

The temperature used during the post weld thermal treatment (PWTT) may vary depending on the type of material used in the welded structure. For welded specimens consisting of type 304L SS on both sides, PWTT consisted of rapid cooling from a temperature of 1850°F that prevented any possibility of sensitization of the grain boundaries. For martensitic stainless steel welds, the stress relief operation consisted of annealing at 1500°F followed by controlled cooling to 1100°F inside the furnace at a rate of 50°F per hour and then cooled in air. [47]

3.5 Optical Microscopy

The metallographic technique using an optical microscope enables the characterization of phases present, their distributions with in grains and their sizes which depend on the typical composition and thermal treatments performed on a material of interest. For example, a quenched and tempered martensitic stainless steel can develop a fine grained and fully tempered microstructure. On the other hand an austenitic microstructure can show large grain austenitic phase due to solution annealing. The principle of an optical microscope is based on the impingement of a light source perpendicular to the test specimen. The light rays pass through the system of condensing lenses and the shutters, up to the half-penetrating mirror. This brings the light rays through the objective to the surface of the specimen. Light rays reflected off the surface of the sample then return to the objective, where they are gathered and focused to form the primary image. This image is then projected to the magnifying system of the
eyepiece. The contrast observed under the microscope results from either an inherent difference in intensity or wavelength of the light absorption characteristics of the phases present. It may also be induced by preferential staining or attack of the surface by etching with a chemical reagent.

The test specimens were sectioned and mounted using the standard metallographic technique, followed by polishing and etching to reveal the microstructures including the grain boundaries. The polished and etched specimens were rinsed in deionized water, and dried with acetone and alcohol prior to their evaluation by a Leica optical microscope as shown in Figure 3.11. This microscope is capable of resolution of up to 1000X.

Figure 3.11 Optical Microscope
3.6 Scanning Electron Microscopy

In a scanning electron microscope (SEM), electrons from a metal filament are collected and focused, just like light waves, into a narrow beam. The beam scans across the subject, synchronized with a spot on a computer screen. Electrons scattered from the subject are detected and create a current, the strength of which makes the spot on the computer brighter or darker. This creates a photograph-like image with an exceptional depth of field. Normally, SEM provides black and white micrographs. A JEOL-5600 scanning electron microscope, capable of resolution of up to 50 nm at magnifications of up to 100,000 times, was used in this study as shown in Figure 3.12. The manual stage of this SEM can accommodate four 1 cm diameter samples or one sample with up to 3.2 cm diameter. The extent and morphology of failure in the tested specimens were determined by SEM. Analysis of failure in metals and alloys involve identification of the type of failure. The test specimens were sectioned into $\frac{1}{2}$ to $\frac{3}{4}$ of an inch in length to accommodate them in the vacuum chamber of the SEM. Usually, failure can occur by one or more of several mechanisms, including surface damage, such as corrosion or wear, elastic or plastic deformation and fracture. Failures can be classified as ductile or brittle. Dimpled microstructure and micro-void coalescence is a characteristic of ductile failure. Brittle failure can be of two types, intergranular and transgranular. An intergranular brittle failure is characterized by crack propagation along the grain boundaries while a transgranular failure is characterized by crack propagation across the grains. Energy Dispersive Spectroscopy (EDS), interfaced with this SEM, can also be used for elemental analysis in the vicinity of the resultant failures.
3.7 Transmission Electron Microscopy

The plastic deformation of a metal usually is accomplished by the development of imperfections or defects such as dislocations and voids. As indicated in the earlier part of this thesis, different specimen configurations including cold-reduced plastically deformed and welded specimens have been tested for characterization of internal or residual stresses resulting from these operations. Since dislocations and voids can be generated in these specimens, transmission electron microscopy was used to characterize these imperfections from the micrographs and relate them to the estimated residual stresses, as determined by non-destructive techniques described earlier. The use of TECNAI 20 transmission electron microscopy, shown in figure 3.13 was able to identify these defects in the micrographs. It should however be noted that the identification of the precise nature of these dislocations was beyond the scope of this investigation. Therefore, major
emphasis was placed on the estimation of dislocations and their densities from the TEM micrographs.

Figure 3.13 Transmission Electron Microscope

3.7.1 Calculation of Dislocation Density using TEM

The characterization of residual stresses due to plastic deformation was done by calculating dislocation density for the specimens cold reduced to different levels using transmission electron microscopy. Dislocation density was measured by using line
intersection method by superimposing a grid of horizontal and vertical test lines were superimposed on the TEM micrographs containing dislocations, as shown in Figure 3.14.\cite{48-50} The number of intersections of vertical (Σ n_v) and horizontal (Σ n_h) test lines with the dislocations presented on TEM were counted as well as the total length of horizontal (Σ L_h) and vertical (Σ L_v) test lines of the superimposed grids were measured. The dislocation density \( \rho \) was measured using the following equations.

\[
\rho = \frac{1}{t} \left( \frac{\sum n_v}{\sum L_v} + \frac{\sum n_h}{\sum L_h} \right)
\]

(Equation 5)

Where,

\( \Sigma n_v \) = No. of intersections of vertical test lines with dislocations
\( \Sigma n_h \) = No. of intersections of horizontal test lines with dislocations
\( \Sigma L_h \) = Total length of horizontal test lines, meter (m)
\( \Sigma L_v \) = Total length of vertical test lines (m)

Figure 3.14 Line Intersection Method for Calculation of Dislocation Density

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Dislocation density was measured by superimposing these grids on 10 different locations of the TEM micrographs and calculating the average of all these densities. The average thickness (t) of the specimen was measured using electron energy loss spectroscopy (EELS) technique available in the TEM. EELS was used to measure the local thickness of the specimens at different locations of the specimen using the following equation[51]

\[ t = \frac{\lambda}{\ln(I_t/I_0)} \]  

(Equation 6)

- \( t \) = average thickness of the sample
- \( I_t \) = total intensity reaching the spectrometer
- \( I_0 \) = zero-loss intensity reaching the spectrometer
- \( \lambda \) = mean free path

An example of local thickness calculation using EELS has been shown in Figure 3.15, where thickness calculations were done at 10 different locations of the specimen.

![Figure 3.15 EELS Measurements to Calculate Average Thickness](image)

Figure 3.15 EELS Measurements to Calculate Average Thickness
CHAPTER 4

RESULTS

This chapter presents the results of residual stress characterization on different types of specimens using pair production, activation and lifetime techniques. However, a major emphasis has been placed on the applicability of the pair production concept in estimating the residual stresses in cold-worked plates of Alloy EP-823 and HT-9. An extensive effort has also been made to develop calibration curves using cylindrical specimens of both alloys subjected to different levels of plastic deformation beyond the yield point. As mentioned earlier in this thesis, activation technique has also been used to develop calibration curves for plastically-deformed Alloys EP-823, HT-9 and 422, and Type 304L SS. With respect to the lifetime method, residual stresses have been characterized only in cold-worked Alloy EP-823. Preliminary results on welded specimens consisting of similar and dissimilar materials (Type 304L SS and Alloy EP-823) have also been presented in this chapter.

4.1 Metallurgical Characterization and Tensile Properties Evaluation

The metallurgical characterization of all four alloys was performed prior to the evaluation of internal stresses resulting from plastic deformation, cold-reduction and welding. The metallurgical microstructures of the austenitic and martensitic alloys,
determined by conventional metallographic techniques, are illustrated in Figures 4.1 through 4.4. Austenitic Type 304L SS was etched in solution containing 10 ml nitric acid (HNO₃), 10 ml acetic acid (CH₃COOH), 15 ml hydrochloric (HCl), and 2 drops of Glycerol. [52] All three martensitic alloys were etched in Fry’s reagent [53] [5g cupric chloride (CuCl₂), 40 ml HCl, 25 ml ethanol (C₂H₅OH)] and 30 ml H₂O. An examination of these micrographs revealed conventional austenitic grains and annealing twins for Type 304L SS. For martensitic stainless steels, fine-grained tempered martensitic microstructures having some delta ferrites were noted in the micrographs. The results of tensile testing at ambient temperature are given in Table 4.1 showing the magnitude of YS, UTS, percent elongation (% El), and percent reduction in area (% RA). As expected, the martensitic alloys exhibited higher strength resulting from the presence of tempered martensite compared to the austenitic Type 304L SS which was solution annealed. While the ductility of martensitic SS did not vary significantly, both the YS and UTS were relatively lower in the higher silicon-containing Alloy EP-823 due to the presence of lower carbon content.

Table 4.1 Room Temperature Tensile Properties of Test Materials

<table>
<thead>
<tr>
<th>Material / Heat No.</th>
<th>Thermal Treatments</th>
<th>YS ksi (MPa)</th>
<th>UTS ksi (MPa)</th>
<th>% El</th>
<th>% RA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy EP-823/2360</td>
<td>Quenched and Tempered (Q &amp; T)</td>
<td>97 (669)</td>
<td>121 (835)</td>
<td>25</td>
<td>61</td>
</tr>
<tr>
<td>Alloy HT-9/2361</td>
<td></td>
<td>105 (724)</td>
<td>138 (952)</td>
<td>25</td>
<td>61</td>
</tr>
<tr>
<td>Alloy 422/2051</td>
<td></td>
<td>122 (841)</td>
<td>145 (1000)</td>
<td>20</td>
<td>60</td>
</tr>
<tr>
<td>Type 304L SS/2155</td>
<td>Solution Annealed</td>
<td>47 (324)</td>
<td>72 (496)</td>
<td>66.6</td>
<td>51</td>
</tr>
</tbody>
</table>

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YS: Yield Strength  
UTS: Ultimate Tensile Strength  
% El: Percent Elongation  
% RA: Percent reduction in Area

Figure 4.1 Optical Micrograph of Solution Annealed Type 304L SS, Etched, 100X

Figure 4.2 Optical Micrograph of Q&T Alloy EP-823, Fry’s Reagent, 100X

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Figure 4.3 Optical Micrograph of Q&T Alloy HT-9, Fry's Reagent, 100X

Figure 4.4 Optical Micrograph of Q&T Alloy 422, Fry's Reagent, 100X
4.2 Residual Stress Measurements by Pair-Production

4.2.1 Cold-Worked Plate Specimens

The results of residual stress measurements on Alloys EP-823 and HT-9, subjected to reduction in thickness by different amount, are illustrated in Figures 4.5 and 4.6, respectively in terms of three line shape parameters (S, W, and T). These data indicate that the extent of residual stress in terms of the S-parameter was enhanced at intermediate level (7.2%) of cold-reduction of Alloy EP-823. A similar behavior was also noted for Alloy HT-9, which showed enhanced residual stress in terms of the S-parameter due to cold reduction by 3.2%. As indicated earlier, the S-parameter is directly proportional to the residual stress, while the W and T-parameters are inversely proportional to the internal stresses generated due to the cold reduction at different levels. The variations of W and T-parameters with % cold-work, shown in Figures 4.5 and 4.6, clearly demonstrate a similar phenomenon. It is interesting to note that the magnitude of all three parameters was not significantly changed at the highest level of cold reduction for either alloy. These results may suggest that the extent of residual stress induced at the intermediate level of cold reduction may play a more significant role compared to that at somewhat relatively higher cold-reduction levels. This phenomenon may be related to the higher driving force needed to move dislocations beyond the grain boundary at some intermediate stress level, beyond which the movement of dislocation may become relatively easier.
Figure 4.5 S, W, and T-Parameter vs. % Cold Reduction for Alloy EP-823 (Pair-Production)
Figure 4.6 S, W, and T-Parameter vs. % Cold Reduction for Alloy HT-9 (Pair-Production)
4.2.2 Plastically-Deformed Cylindrical Specimens

The quantitative evaluation of residual stress by pair-production in a structural material is difficult to achieve since the resultant stress can be measured only in terms of line shape parameters (S, W and T) determined from the annihilation peaks. In view of the shortcoming associated with the pair-production method, an innovative approach was attempted to develop calibration curves based on plastic tensile stress/strain determined from engineering stress versus strain (s-e) diagram and to relate them to different line-shape parameters resulting from the pair-production method. As indicated in the previous chapter, the smooth cylindrical specimens were plastically loaded at stresses ranging between the YS and UTS. The magnitudes of S, W, and T-parameters for both alloys, determined by the pair-production technique, were plotted as a function of applied plastic stress, as illustrated in Figures 4.7 and 4.8 respectively for Alloys EP-823 and HT-9. An evaluation of these plots clearly indicates a gradual enhancement of the S parameter with increased applied plastic stress, suggesting enhanced residual stresses in the cylindrical specimens due to applied stresses. Conversely, the magnitude of W and T-parameters was gradually reduced with increasing applied stress, once again indicating a gradual increase in internal stresses resulting from the plastic deformations of different magnitude.
Figure 4.7 S, W, and T-Parameter vs. Applied Stress for Alloy EP-823 (Pair-Production)
Figure 4.8 S, W, and T-Parameter vs. Applied Stress for Alloy HT-9 (Pair-Production)
4.2.3 Welded Specimen

The results of residual stress evaluation involving welded specimens of similar and dissimilar materials by pair-production are illustrated in Figures 4.9 and 4.10, respectively. It is well known that the magnitude of the internal stresses developed in welded specimens will be highest at the fusion line (FL) followed by a gradual decline beyond this region. This phenomenon is associated with the differential rate of solidification between the weld and the base material, and the resultant microstructural variation with distance away from the welded region. A similar observation was made in Figure 4.9 for a welded specimen of Type 304L SS on both sides, showing reduced internal stresses at locations away from the fusion line in terms of both S and T parameters. As to the internal stresses generated in a welded specimen consisting of dissimilar materials (Type 304L SS/ Alloy EP-823), compressive stresses were generated in martensitic Alloy EP-823, which was characterized by enhanced S parameter value with distance indicating less tensile stresses, as seen in Figure 4.10. Simultaneously, the T parameter was also gradually reduced at locations away from fusion line due to the increased compressive internal stresses, as illustrated in the same figure.
Figure 4.9 S and T-Parameter vs. Distance from FL for (Pair-production)
Figure 4.10 S and T-Parameter vs. Distance from FL (Pair-Production)
4.3 Residual Stress Measurements by Activation

The results of residual stress evaluation by the activation technique involving specimens of both martensitic and austenitic stainless steels are shown in Figures 4.11 through 4.14. The variations of S, W and T parameters vs. the applied stress (YS through UTS) for Alloys EP-823, HT-9 and 422 are illustrated in Figures 4.11 through 4.13. As expected, the magnitude of S parameter was enhanced with applied plastic stress for all three alloys. Simultaneously, the residual stresses in terms of the W and T parameter was gradually increased with the reduction of both parameters, suggesting increased internal stresses at higher applied stresses. For austenitic Type 304L SS, the magnitude of S, W and T parameters of a controlled specimen without any deformation was determined by the activation technique to compare the resultant internal stresses to those at higher applied plastic loads. A similar trend was also noted, as anticipated.
Figure 4.11 S, W, and T-Parameter vs. Applied Stress for Alloy EP-823 (Activation)

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1 ksi = 6.895 MPa

(a)

(b)
Figure 4.12 S, W, and T-Parameter vs. Applied Stress for Alloy HT-9 (Activation)
Figure 4.13 S, W, and T-Parameter vs. Applied Stress for Alloy 422 (Activation)
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4.4 Residual Stress Measurements by Lifetime Analyses

As discussed in the previous chapter, the lifetime of a martensitic alloy such as Alloy EP-823 may be enhanced in the presence of linear defects such as dislocations. In order to estimate the lifetime of this alloy, subjected to different levels (7 and 11%) of cold-deformation, lifetime analyses based on the Positron Lifetime Spectroscopic (PLS) method was employed. The results, shown in Figure 4.15, indicate that the positron lifetime was enhanced with increased level of cold deformation from 0 to 7 to 11% indicating the presence more defects (dislocations) at higher cold deformation levels. The presence of higher number of dislocations signifies the generation of higher internal stresses. The positron lifetime corresponding to different levels of cold reductions are
also shown in Table 4.2. These data were subsequently utilized to relate the lifetimes to the dislocation density in a subsection presented later in this thesis.

Figure 4.15 Positron Lifetimes vs. % Cold Reduction for Alloy EP-823

Table 4.2 Positron Lifetime vs. % Cold-Reduction

<table>
<thead>
<tr>
<th>Material No / Heat No</th>
<th>% Cold Reduction</th>
<th>Positron Lifetimes (picoseconds)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy EP-823/2154</td>
<td>0</td>
<td>0.1949</td>
</tr>
<tr>
<td></td>
<td>7.2</td>
<td>0.226</td>
</tr>
<tr>
<td></td>
<td>11.6</td>
<td>0.232</td>
</tr>
</tbody>
</table>
4.5 Fractographic Evaluation by Scanning Electron Microscope

Scanning electron microscopy (SEM) was used to analyze the morphology of failure at the primary fracture surface of cylindrical specimens used in tensile testing at ambient temperature. The SEM micrographs of Alloys EP-823 and HT-9 are illustrated in Figures 4.16 and 4.17, respectively at two different magnifications. Classical dimple microstructures were observed in both cases, indicating ductile failure. Dimpled microstructures were also observed on the primary fracture surface of Type 304L SS, as shown in Figure 4.18.
Figure 4.16 Ductile Failure of Tensile Alloy EP-823 Specimen

(a) 35X
(b) 850X

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Figure 4.17 Ductile Failure of Tensile Alloy HT-9 Specimen

(b) 850X

(a) 35X

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4.6 Characterization of Dislocations by Transmission Electron Microscopy

It is well known that plastic deformation of structural material is accompanied by the formation of defects such as dislocations and voids, which can be characterized by transmission electron microscopy due to greater depth of penetration associated with this analytical tool. The TEM micrographs of plate materials of Alloy EP-823, with and without cold-reduction, are shown in Figures 4.19 through 4.21. As expected, Alloy EP-823 without any cold-deformation exhibited insignificant amount of defects, which could be the result of prior cold-work performed on this material. It is possible that the thermal treatment (tempering operation) performed to relieve the internal stresses may not be fully capable of eliminating some of the defects retained from prior cold deformation. An

Figure 4.18 Ductile Failure of Tensile Type 304L SS Specimen
examination of Figure 4.19 indicates the presence of tempered martensitic lathes and fine globular carbide precipitates possibly of types $\text{M}_7\text{C}_3$, $\text{M}_{23}\text{C}_6$, $\text{M}_3\text{C}$ and $\text{MC}$.\textsuperscript{[54-56]}

The TEM micrographs, shown in Figures 4.20 and 4.21, revealed the presence of enhanced dislocation clusters resulting from cold-reduction of the plate material by 7 and 11 percent, respectively. Both micrographs are characterized by clusters of dislocations crossing one another at various locations within the metal lattice. The TEM micrographs, shown in Figures 4.19 to 4.21 were used to calculate the dislocation density as a function of the cold-reduction level using the line intersection method, discussed in the previous chapter. The results, shown in Table 4.3 and Figure 4.21 indicate that the dislocation density ($\rho$) was gradually increased with an increase in the cold reduction level, as anticipated.

Figure 4.19 TEM Micrograph of Alloy EP-823 without any Cold-Reduction

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Figure 4.20 TEM Micrograph of Alloy EP-823 with 7% Cold-Reduction

Figure 4.21 TEM Micrograph of Alloy EP-823 with 11% Cold-Reduction
Table 4.3 Dislocation Density of Alloy EP-823 at Different Cold Reduction Levels

<table>
<thead>
<tr>
<th>Percent Cold Reduction</th>
<th>Dislocation Density, (\rho) (No./m(^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>(2.645 \times 10^{15})</td>
</tr>
<tr>
<td>7</td>
<td>(9.834 \times 10^{15})</td>
</tr>
<tr>
<td>11</td>
<td>(6.157 \times 10^{16})</td>
</tr>
</tbody>
</table>

Figure 4.22 Dislocation Density of Alloy EP-823 at Different Cold Reduction Levels

4.6 Comparison of Pair-Production Data to TEM results

An effort was made to compare the extent of residual stress determined by the pair-production technique to the dislocation density of cold-deformed Alloy EP-823 based on the analysis of the TEM micrographs. The variation of T-Parameter obtained by a
previous investigator\cite{13} on Alloy EP-823 subjected to 7 and 11 percent cold reduction is illustrated in Figure 4.23, showing reduced T-parameter values at higher cold-reduction level. Once again, the reduction of T-parameter signifies enhanced residual stress due to increased plastic deformation. The magnitude of the T-parameter shown in this figure was compared to the dislocation density given in Table 4.3 as a function of the cold reduction level. A comparative analysis, presented in Figure 4.24, provides a relationship between the estimated residual stress and dislocation density ($\rho$) at comparable cold-reduction levels.

![Figure 4.23 Effect of % CR on T-Parameter](image-url)
4.7 Comparison of Positron Lifetimes to the Dislocation Densities

The variations of both dislocation density and positron lifetimes with different levels of cold-reduction are shown in Figure 4.25. It is obvious from this figure that both the dislocation density and positron lifetimes were gradually enhanced with increasing cold deformation levels. Once again, enhanced internal stresses are indicative of the higher values of dislocation density and positron lifetimes, thus exhibiting a consistent pattern.
4.8 Comparison of Residual Stress by ND, Activation and Pair-Production

While this investigation is primarily focused on the characterization of residual stress by methods based on positron annihilation spectroscopy (PAS), the results of residual stress measurements by neutron diffraction (ND) are also presented here for comparative analysis. As indicated earlier, the ND technique is capable of providing quantitative residual stresses in structural materials subjected to plastic/cold deformation. The results of ND measurements in cylindrical specimens of Alloy EP-823, plastically deformed by tensile loading at stresses ranging between YS and UTS, are shown in Figures 4.26 (a). It is obvious from these data that the extent of internal stresses generated by plastic deformation was gradually enhanced with increasing applied stress beyond YS.
The estimated residual stresses in a similar material by pair-production and activation techniques already presented earlier are reproduced in this section to compare them to the residual stresses determined by the ND technique. An evaluation of the Figures 4.26 (b) and 4.26 (c) clearly suggests that the extent of residual stress in terms of the S-parameter determined by the pair-production and the activation technique was gradually increased with increased applied stresses, which is consistent with the basic understanding of the PAS technique. A comparison of this two figures to Figure 4.26 (a), in essence, demonstrate a similar trend in that both the S-parameter, and the residual stress determined by the ND technique are directly proportional to the magnitude of plastic stress applied to cause deformation using cylindrical specimens of Alloy EP-823. A comparison of the estimated residual stress in terms the S-parameter (activation/pair-production) and calculated residual based on the ND measurements are given in Table 4.4, once again showing a consistent pattern.

![Graph showing residual stress vs. applied stress for Alloy EP-823](image)

(a) Neutron Diffraction
(b) Pair-Production

(c) Activation

Figure 4.26 Comparison of Residual Stress by Different Methods

Table 4.4 Residual Stress Values vs. Different Techniques

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Applied Stress, ksi</th>
<th>Measurement Technique</th>
<th>Neutron Diffraction</th>
<th>Pair-Production</th>
<th>Activation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Residual Stress, ksi</td>
<td></td>
<td>Average S-Parameter</td>
<td>Average S-Parameter</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>96.3 (YS)</td>
<td>27</td>
<td>0.348</td>
<td>0.351</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>102.4</td>
<td>38</td>
<td>0.351</td>
<td>0.355</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>108.6</td>
<td>43</td>
<td>0.360</td>
<td>0.364</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>121 (UTS)</td>
<td>50</td>
<td>0.360</td>
<td>0.366</td>
<td></td>
</tr>
</tbody>
</table>
4.9 Comparison of Residual Stress by Pair-Production and Activation

Comparisons of S, W and T parameter determined on Alloys EP-823 and HT-9 by both pair-production and Activation techniques are given in Tables 4.5 and 4.6 respectively. A cursory evaluation of all three parameters indicates a consistent pattern in that their magnitudes are gradually enhanced with increased applied plastic stress ranging between YS and UTS.

Table 4.5 Comparative Results for P.P and Act in Alloy EP-823 (Plastically Deformed)

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Applied Stress, ksi</th>
<th>Average S-Parameter</th>
<th>Average W-Parameter</th>
<th>Average T-Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>P.P Act</td>
<td>P.P Act</td>
<td>P.P Act</td>
</tr>
<tr>
<td>1</td>
<td>96.3 (YS)</td>
<td>0.348 0.351</td>
<td>0.421 0.419</td>
<td>1.209 1.193</td>
</tr>
<tr>
<td>2</td>
<td>102.4</td>
<td>0.351 0.355</td>
<td>0.416 0.420</td>
<td>1.184 1.183</td>
</tr>
<tr>
<td>3</td>
<td>108.6</td>
<td>0.360 0.364</td>
<td>0.415 0.404</td>
<td>1.155 1.113</td>
</tr>
<tr>
<td>4</td>
<td>121 (UTS)</td>
<td>0.360 0.366</td>
<td>0.406 0.404</td>
<td>1.130 1.105</td>
</tr>
</tbody>
</table>

P.P: Pair-Production
Act: Activation
Table 4.6 Comparative Results for P.P and Act in Alloy HT-9 (Plastically Deformed)

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Applied Stress, ksi</th>
<th>Average S-Parameter</th>
<th>Average W-Parameter</th>
<th>Average T-Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>P.P</td>
<td>Act</td>
<td>P.P</td>
</tr>
<tr>
<td>1</td>
<td>104.5 (YS)</td>
<td>0.347</td>
<td>0.352</td>
<td>0.427</td>
</tr>
<tr>
<td>2</td>
<td>121.2</td>
<td>0.356</td>
<td>0.361</td>
<td>0.411</td>
</tr>
<tr>
<td>3</td>
<td>137.9 (UTS)</td>
<td>0.367</td>
<td>0.364</td>
<td>0.406</td>
</tr>
</tbody>
</table>
CHAPTER 5

DISCUSSION

This investigation was aimed at characterizing the residual stresses in candidate structural materials subjected to different types of deformation processes including cold-reduction, plastic deformation and welding consisting of similar and dissimilar materials. The materials tested include austenitic Type 304L SS and martensitic alloys EP-823, HT-9 and 422. An emphasis placed on the residual stress characterization of these alloys predominantly applying techniques based on the classical Positron Annihilation Spectroscopy (PAS) methods. Such techniques include pair-production, activation, and lifetime analysis. The PAS data were eventually compared to the neutron diffraction (ND) on identical materials and types of specimens.

Since the plastic deformation of engineering materials due to different forming processes is accompanied by the formation of lattice defects such as dislocations and voids, an extensive effort was focused on characterizing these defects and relating them to the residual stresses in a cold deformed structural material (Alloy EP-823). The characterization and extent of defects were determined by Transmission Electron Microscopy (TEM). The magnitudes of residual stresses evaluated by all three PAS techniques, are qualitative in nature. Therefore, calibration curves were generated based on the applied plastic stress imparted to the cylindrical specimens and the resultant line shape parameters (S, W and T) determined from the PAS measurements.
It is well known that the line shape parameters including S, W and T can be related to the extent of residual stress generated to the extent of residual stress generated due to different forming processes. The analyses of the PAS data indicate that the magnitude of residual stress characterized by the S-parameter was gradually increased with increasing applied stress ranging between the ambient temperature yield stress (YS) and ultimate tensile stress (UTS) of the test materials. The S-parameter simply characterizes the residual stress based on the energy spectrum of 511 KeV annihilation peak. It is well known that the S-parameter is directly proportional to the generated internal stresses. Thus, the results of the current investigation in terms of the S-parameter are significantly consistent with the established phenomenon on the characterization of residual stress by the line shape parameter. The residual stresses evaluated in terms of the other two parameters, namely W and T, are also consistent in that they were inversely proportional to the applied stresses, showing their reduced values at higher applied stresses. These observations are quite significant since the resultant data are valuable in developing calibration curves for estimation of internal stresses at different applied stress levels in the plastic region of the engineering stress vs. strain (s-e) diagrams of material of interest.

Compared to the pair production and activation techniques, residual stress characterization by the lifetime analysis are somewhat novel, which is based on enhanced lifetimes at higher levels of cold deformation, thus in turn, causing internal stresses in the form of dislocations. It should suffice to state that, based on the comparative analysis of the resultant data, all three techniques based on the positron annihilation and lifetime concepts were successful in characterizing the resultant residual stresses as a function of the magnitude of applied stress to the specimens of different types of materials.
Compared to the PAS technique, the ND provides quantitative evaluation of residual stresses in plastically deformed structural materials by measuring the interplanar distance \( d \) in a metal lattice. The results of residual stress measurements by the ND technique on plastically deformed Alloy EP-823 also revealed a similar trend showing enhanced internal stresses with increasing applied plastic stress imparted to a similar type of cylindrical specimen. The results of limited studies involving welded specimens consisting of similar materials (Type 304L SS) showed tensile residual stresses, the extent of which gradually reduced at locations away from the fusion line. Conversely, the residual stress generated on the martensitic (Alloy EP-823) side of the welded specimen was compressive in nature, which was gradually increased along the base material away from the fusion line. The enhanced compressive stress as a function of distance signifies the reduction in internal stress in this alloy. Thus, the overall data verifies a common conviction that the internal stresses generated in the vicinity of the fusion line (closed to the weld) will always be significantly higher compared to those at locations away from the weld.

The characterization of defects (dislocations) in cold-worked martensitic Alloy EP-823 based on the TEM micrograph showed an order magnitude higher density of defects at increased cold reduction levels compared to that in materials without any cold reduction. The dislocation density \( \rho \) calculated from the TEM micrograph showed a consistent pattern on the relationship of the T-parameter to \( \rho \) as a function of comparable cold-deformation level. A similar relationship has also been demonstrated involving positron lifetimes and dislocation density.
The microstructures determined by conventional metallographic techniques were consistent for the type of material incorporated in this investigation. As to the morphology of failure of the tensile specimens used in calibration studies, ductile failures characterized by dimpled structures were observed.
CHAPTER 6

SUMMARY AND CONCLUSION

Pair-production, activation and positron lifetime spectroscopy have been used to characterize residual stress in austenitic and martensitic stainless steels subjected to cold-reduction, plastic deformation and welding. Transmission electron microscopy has been used to characterize the nature and extent of defects in these in these materials. Metallography and fractography have been used to utilize to characterize microstructures and failure morphology of these alloys. The significant conclusions are summarized below.

- The martensitic alloys exhibited higher tensile strength resulting from the presence of tempered-martensitic microstructures compared to the austenitic structure of Type 304L SS resulting from solution-annealing, as observed in the optical micrographs.
- Maximum residual stress was generated in materials subjected to intermediate level of cold-reduction, as characterized by the S, W and T-parameters developed from the annihilation spectrum.
Both pair-production and activation data revealed a gradual enhancement in residual stresses with increasing applied plastic stress ranging between YS and UTS in terms of the S, W and T-parameters.

Welded specimen consisting of austenite stainless steels on both sides exhibited tensile residual stress, which was gradually reduced away from the fusion line. However, compressive residual stresses were observed on the martensitic stainless steel side of the weld for specimens having dissimilar materials.

Increased positron lifetimes were observed for cold-worked Alloy EP-823 at higher levels of cold-reduction, compared to specimens without any cold-reduction.

Residual stress determined by the ND technique was higher in specimens subjected to increased level of plastic deformation imparted by tensile loading.

The TEM micrographs revealed martensitic lathes and fine globular carbide precipitates along with dislocations irrespective of the level of cold-reduction in Alloy EP-823.

The dislocation density was gradually due to the reduction in thickness by 0, 7 and 11 percent, respectively indicating higher levels of internal stresses.

The dislocation density (\(\rho\)), calculated from the TEM micrographs, showed a consistent pattern on the relationship of the T-parameter to \(\rho\) as a function of the comparable cold-reduction level. A similar relationship was observed between the positron lifetime and dislocation density at similar cold-reduction levels.

Morphology of failure in tensile specimens used in calibration studies by SEM revealed ductile failures characterized by dimpled microstructures.
CHAPTER 7

SUGGESTED FUTURE WORK

Additional work, as indicated below, is suggested to develop a better understanding of residual stresses measured by the PAS technique.

• Characterization of residual stresses in welded specimens following Post Weld Thermal Treatment.

• Evaluation of radiation hardening effect on the residual stresses.

• Characterization of metallurgical microstructures of welded specimens.

• Characterization of defects in welded specimens by Transmission Electron Microscope.
APPENDIX A

MTS DATA

A. Stress-Strain Diagrams using Smooth Specimens Tested at Room Temperature

(a) Alloy EP-823
1 ksi = 6.895 MPa

(b) HT-9

(c) Alloy 422
APPENDIX B

OPTICAL MICROGRAPHS

B1. Optical Micrograph of Solution Annealed Type 304L SS

(a) Etched, 50 X

(b) Etched, 200 X
B2. Optical Micrograph of Q&T Alloy EP-823, Fry's Reagent

(a) 50 X

(b) 200 X
B3. Optical Micrograph of Q&T Alloy HT-9, Fry's Reagent

(a) 50 X

(b) 200 X
B4. Optical Micrograph of Q&T Alloy 422, Fry's Reagent

(a) 50 X

(b) 200 X
APPENDIX C

SCANNING ELECTRON MICROGRAPHS

C1. Ductile Failure of Tensile Type 304L SS Specimen

(a) 35 X

(b) 500 X
C2. Ductile Failure of Tensile Alloy EP-823 Specimen

(a) 35 X

(b) 850 X
C3. Ductile Failure of Tensile Alloy HT-9 Specimen

(a) 35 X

(b) 850 X
C4. Ductile Failure of Tensile Alloy 422 Specimen

(a) 35X

(b) 500X
APPENDIX D

TRANSMISSION ELECTRON MICROGRAPHS

D1. TEM Micrograph of Alloy EP-823 without any Cold-Reduction

Sample 1

Sample 2
Sample 3

Sample 4

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D2. TEM Micrograph of Alloy EP-823 with 7 percent Cold-Reduction

Sample 1

Sample 2
Sample 3

Sample 4

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D3. TEM Micrograph of Alloy EP-823 with 11 percent Cold-Reduction

Sample 1

Sample 2
D4. STEM Micrograph of Alloy EP-823 Showing Carbide Precipitation

Energy Dispersive Spectroscopy (EDS)

Point 1 Showing Presence of Carbon

Point 2 Showing No Carbon Content
D5. Select Area Diffraction Pattern (SADP) for Alloy EP-823

SADP – 800 mm
APPENDIX E

POSITRON ANNihilation SPECTROSCOPY

El. Pair-production Data

Results for input file =
dec16_2004_223_hist.txt

passed 1st peak-find at Cs-peak = 7305.000

passed 2nd peak-find at Cs-peak = 7304.633
std. dev. of Cs peak = 6.009033
fwhm of Cs peak = 14.12123

total bkgd = 1044.370
channels = 50.00000
bk per chan = 20.88740

passed 3rd peak-find Cs-peak = 7304.647
std. dev. of Cs peak = 5.564457
fwhm of Cs peak = 13.07647
50%-width of Cs peak = 3.756008
BKGD of peak (per chan) = 20.88740

The fitting of the function yielded
passed 4th peak-find Cs-peak = 7304.649
std. dev. of Cs peak = 6.014511
fwhm of Cs peak = 14.13410
50%-width of Cs peak = 4.059795
BKGD of peak =
-2.308235 + 6.5590084E-02 *X + -8.6052096E-06 *X^2
Reduced Chi-Squared Value = 1.085224

passed 1st peak-find at Ba-peak = 3931.000

passed 2nd peak-find at Ba-peak = 3931.192
std. dev. of Ba peak = 6.159107
fwhm of Ba peak = 14.47390

passed 3rd peak-find Ba-peak = 3931.203
std. dev. of Ba peak = 4.447959
fwhm of Ba peak = 10.45270
50%-width of Ba peak = 3.002373
BKGD of peak (per chan) = 92.56676

The fitting of the function yielded
passed 4th peak-find Ba-peak = 3931.241
std. dev. of Ba peak = 4.833731
fwhm of Ba peak = 11.35927
50%-width of Ba peak = 3.262768
BKGD of peak =
-3220.842 + 1.854557 *X + -2.574413E-04 *X^2
Reduced Chi-Squared Value = 1.090910

passed 1st peak-find of ann-peak = 5641.000

passed 2nd peak-find of ann-peak = 5641.975
std. dev of 511 peak = 12.51595
fwhm of 511 peak = 29.41248

passed 3rd peak-find of ann-peak = 5642.162
std. dev. of 511 peak = 13.64159
fwhm of 511 peak = 32.05772
BKGD of peak (per chan) = 22.39765

The fitting of the function yielded
passed 4th peak-find 511-peak = 5642.202
std. dev. of 511 peak = 14.50684
fwhm of 511 KeV peak = 34.09108
50%-width of 511 peak = 9.792120
BKGD of peak =
-1434.672 + 0.5316734 *X + -4.8459529E-05 *X^2
Reduced Chi-Squared Value = 1.118598

cs_50pct_width = 3.756008
cs_50pct_width = 7.512017
cs_50pct_width = 5.634013
cs_50pct_width = 4.695011
cs_50pct_width = 4.225510
cs_50pct_width = 3.990759
cs_50pct_width = 4.108134
cs_50pct_width = 4.049447
cs_50pct_width = 4.020103
cs_50pct_width = 4.034775
cs_50pct_width = 4.042110
cs_50pct_width = 4.038443
cs_50pct_width = 4.040277
ba_50pct_width = 3.002373
ba_50pct_width = 6.004745
ba_50pct_width = 4.503559
ba_50pct_width = 3.752966
ba_50pct_width = 3.377669
ba_50pct_width = 3.190021
ba_50pct_width = 3.283845
ba_50pct_width = 3.236933
ba_50pct_width = 3.260389
ba_50pct_width = 3.272117
ba_50pct_width = 3.266253
ba_50pct_width = 3.263321
ba_50pct_width = 3.264787

total_50pct_width_fit = 4.597770

The 511 baseline sigma is = 14.55223

sum of center of 511 peak = 6333.844
sum of background of 511 center = 309.7174
sum of entire 511 peak counts = 17356.91
sum of entire background counts = 3074.047
sum of 511 wing counts = 6922.724
sum of 511 wing background = 1134.112

MAJOR RESULTS:

S-factor_fit = 0.3649178 +/- 5.5773119E-03
W-factor_fit = 0.3988455 +/- 6.1624437E-03
T-factor_fit = 1.092974 +/- 2.0096175E-02
E2. Activation Data

Results for input file = timing946_hist.txt

First the 137Cs peak parameters:

passed 1st peak-find: Peak = 7466.000

passed 2nd peak-find: Peak = 7466.676
std. dev. of peak = 4.883075
fwhm of peak = 11.47523

low_limit_cs = 7456
high_limit_cs = 7476
total bkgd = 531.0000
channels = 12.00000
bk per chan = 44.25000

passed 3rd peak-find Cs-peak = 7466.714
std. dev. of Cs peak = 4.636768
fwhm of Cs peak = 10.89641
50%-width of Cs peak = 3.129819
BKGD of peak (per chan) = 44.25000

low_limit_cs = 7456
high_limit_cs = 7476
low_limit_cs = 7456
high_limit_cs = 7476

The fitting of the function yielded
passed 4th peak-find Cs-peak = 7466.683
std. dev. of Cs peak = 6.041771
fwhm of Cs peak = 14.19816
50%-width of Cs peak = 4.078196
BKGD of peak = -3712.494 + 0.5046824 * X
Reduced Chi-Squared Value = 0.7496251

Second the 133Ba peak parameters:

passed 1st peak-find: Peak = 4247.000
passed 2nd peak-find: Peak = 4246.586
std. dev. of peak = 3.636060
fwhm of peak = 8.544741

low_limit.ba = 4239
high_limit.ba = 4253
passed 3rd peak-find  Ba-peak = 4246.612
std. dev. of Ba peak      = 3.459134
fwhm of Ba peak           = 8.128965
50%-width of Ba peak      = 2.334916
BKGD of peak (per chan)   = 379.8000

low_limit_ba = 4239
high_limit_ba = 4253
low_limit_ba = 4239
high_limit_ba = 4253

The fitting of the function yielded
passed 4th peak-find Ba-peak = 4246.298
std. dev. of Ba peak      = 5.580328
fwhm of Ba peak           = 13.11377
50%-width of Ba peak      = 3.766722
BKGD of peak = -86995.57 + 20.51595 *X

Reduced Chi-Squared Value = 0.4724198

Third the 511 peak parameters:

passed 1st peak-find: Peak = 5758.000

passed 2nd peak-find: Peak = 5757.478
std. dev. of peak         = 13.00992
fwhm of peak              = 30.57330

low_limit_511 = 5727
high_limit_511 = 5787
passed 3rd peak-find of ann-peak= 5757.601
std. dev. of 511 peak     = 11.90309
fwhm of 511 peak          = 27.97226
BKGD of peak (per chan)   = 180.9643

low_limit_511 = 5727
high_limit_511 = 5787
low_limit_511 = 5727
high_limit_511 = 5787

The fitting of the function yielded
passed 4th peak-find 511-peak= 5757.688
std. dev. of 511 peak      = 13.99304
fwhm of 511 KeV peak       = 32.88364
50%-width of 511 peak      = 9.445302
BKGD of peak               = 2125.193 + -0.3108103 *X

Reduced Chi-Squared Value = 0.8167945
cs_50pct_width = 3.129819
cs_50pct_width = 6.259637
cs_50pct_width = 4.694728
cs_50pct_width = 3.912273
cs_50pct_width = 3.521046
cs_50pct_width = 3.716660
cs_50pct_width = 3.618853
cs_50pct_width = 3.667757
cs_50pct_width = 3.643305
cs_50pct_width = 3.631079
cs_50pct_width = 3.637192
cs_50pct_width = 3.634135

ba_50pct_width = 2.334916
ba_50pct_width = 4.669831
ba_50pct_width = 3.502373
ba_50pct_width = 2.918644
ba_50pct_width = 2.626780
ba_50pct_width = 2.772712
ba_50pct_width = 2.845678
ba_50pct_width = 2.809195
ba_50pct_width = 2.827437
ba_50pct_width = 2.836558
ba_50pct_width = 2.831997
ba_50pct_width = 2.834278
ba_50pct_width = 2.833138

total_50pct_width_fit = 4.193104

The 511 baseline sigma is = 12.91638

sum of center of 511 peak = 56369.99
sum of background of 511 center = 4222.145
sum of entire 511 peak counts = 173505.3
sum of entire background counts = 20487.31
sum of 511 wing counts = 72996.20
sum of 511 wing background = 13450.37

MAJOR RESULTS:

S-factor_fit = 0.3248891 +/- 1.6444699E-03
W-factor_fit = 0.4207144 +/- 2.0286599E-03
T-factor_fit = 1.294948 +/- 7.7136341E-03
E3. Lifetime Data

Timing 996

Analysed between channels 310 and 600. Calibration=0.04028 ns/chann.
Total counts=4326 Bkgr./signal=6.70%

Fit's variance = 0.2219 Date: 8/23/2005 6:11:45 PM

SAMPLE:
intensities [%] lifetimes [ns] dispersions [ns]
100.0000 0.1949(0.0099) 0.0000

ZERO CHAN. 314.3301(0.0082) BACKGROUND 1.093(0.066)[Counts]

RESOLUTION CURVE:
ESG fract. (%) Shift (chnns) FWHM (ns) tau(left) (ns) tau(right) (ns)
100.0000 0.0000 0.4625 0.0000 0.0000
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