Residual stress measurements and analysis by destructive and non-destructive techniques

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University of Nevada, Las Vegas

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RESIDUAL STRESS MEASUREMENTS AND ANALYSIS BY DESTRUCTIVE AND
NON DESTRUCTIVE TECHNIQUES

by

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Bachelor of Technology in Mechanical Engineering
University of Madras, Chennai, India
April 2001

A thesis submitted in partial fulfillment
of the requirements for the

Master of Science Degree in Mechanical Engineering
Department of Mechanical Engineering
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Graduate College
University of Nevada, Las Vegas
August 2004
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is approved in partial fulfillment of the requirements for the degree of

Master of Science in Mechanical Engineering

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ABSTRACT

Residual Stress Measurements and Analysis by Destructive and Non Destructive Techniques

Satish Dronavalli

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

This investigation is focused on the evaluation of residual stresses resulting from cold deformation and welding in candidate target structural materials such as martensitic Alloys EP-823 and HT-9, and austenitic Type 304L stainless steel (SS). Measurements of residual stresses were performed by both destructive and nondestructive techniques including ring-core (RC), neutron diffraction (ND) and positron annihilation spectroscopy (PAS). The results obtained by the PAS method indicate that the residual stresses in the cold-worked specimens were significantly enhanced with increased level of cold reduction. Residual stress measurements by the RC method on welded specimens consisting of similar materials revealed tensile stresses on both sides. The welded specimens consisting of similar and dissimilar materials showed compressive residual stresses on one side (Alloy EP-823) and tensile stresses on the other side (Type 304L SS). In general, a good agreement in the measured residual stresses was observed based on their comparisons on different configurations of testing specimens using state-of-the-art experimental techniques.
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CHAPTER 1

INTRODUCTION

With increased demand of energy, nuclear power has become a potential source of energy worldwide. Currently 20% of electric power in the United States is generated from roughly 100 nuclear power plants operating in this country. It is obvious that significant amounts of spent nuclear fuel (SNF) and high-level radioactive waste (HLW) will be generated from these nuclear plants, research reactors and government nuclear facilities. Significant efforts are in progress in the United States to dispose of SNF/HLW for 10,000 years.\(^1\) In view of this long disposable period and the necessity to accommodate future radioactive materials in the same repository the United States Department of Energy (US DOE) had been exploring the possibility of alternative approaches in addition to the direct disposal of SNF/HLW.

One such approach is transmutation of minor actinides (MA) and Fission Products (FP) from SNF/HLW to reduce their half-lives.\(^2\) The process of transmutation involves bombardment of target material with protons generated either by an accelerator or a thermal reactor. Thereby, producing fast moving neutrons, then these neutrons are impinged on SNF/HLW at a very high speed, thus ejecting MA/FP.\(^3\) This process results in a significant reduction in half-lives of radioactive materials contained in SNF/HLW. This reduction in half-lives would thus enable the disposal of this radioactive materials for shorter period in the proposed geological repository. This concept shown in

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Figure 1.1 would enable isolation of radioactive materials from SNF/HLW would facilitate in disposing a large volume at a relatively low cost.

In essence, during the transmutation process, the protons are impinged upon a target material such as molten lead-bismuth-eutectic (LBE), which can also act as a coolant. However, this molten LBE will be contained in a structural configuration made of a suitable metallic material, commonly known as target structural material.

![Diagram of Nuclear Waste Management Methods]

Figure 1.1 Different Methods for Nuclear Waste Management

Since these protons are impinged at a very high speed, the molten LBE will be subjected to the development of substantial amount of stresses, which can also be experienced by the structural material surrounding it. Further, significant amount of heat ranging from 400 to 600°C will be generated during the transmutation process. Thus, the target structural material will be subjected not only to high stresses but also to elevated
temperatures during transmutation. During the fabrication of the containment vessel for the molten LBE, the structural material may undergo operations such as cold working, mechanical forming and welding of similar and dissimilar materials, which can induce appreciable amount of residual or internal stresses inside these materials. (4) Further, during the welding operations involving either similar or dissimilar materials, enough residual or internal stresses can be generated due to the rapid rate of solidification, differences in coefficient of expansion/contraction, and dissimilar metallurgical microstructure developed at the weld, fusion line, heat-affected-zone (HAZ), and the base material. Combination of residual stresses and elevated operating temperatures during the transmutation process can, thus, significantly influence the performance of the target structural material. (5) Unless these internal stresses are relieved by proper thermal-treatment, their presence can impair the performance of the target structural materials, while the transmutation process is in progress.

Residual stresses can be defined as the stresses embedded inside a machined or welded component even after the forces causing the stresses are removed. The internal state of the stresses can be either compressive or tensile depending upon the nature of the operation performed on the component. Both the magnitude and distribution of the residual stresses are critical for performance, and should be considered while designing a component. (6) In any free standing body, stress equilibrium should be maintained, which means that the presence of a tensile residual stress in a component will be balanced by a compressive stress elsewhere in the component. Tensile residual stresses on the surface of a component are generally undesirable since they can aggravate the failure of a component, and often may cause fatigue failure, quench cracking and stress-corrosion.
cracking. Residual stresses can however be reduced or eliminated by inducing compressive stresses generated by short-peening operation.

The US Department of Energy is seriously interested to develop non destructive Positron Annihilation Spectroscopy (PAS) as a standardized tool for measuring residual stresses in materials subjected to plastic deformation, cold working and welding involving similar and dissimilar materials. While the PAS technique has been utilized for other applications, very little or no data currently exist as to the applicability of this technique for residual or internal stress measurements. Thus, it is a major challenge to the scientific community to apply this technique for such applications.

It should suffice to state that while standardizing the PAS technique as a useful method for stress measurement, other non destructive methods such as neutron diffraction (ND), x-ray diffraction (XRD), and destructive ring-core method also need to be explored in characterizing and comparing the resultant data. In light of this rationale, this project has been focused on using on all four techniques for measuring residual stresses in candidate target structural materials of interest.

However, this thesis is focused on the utilization of the PAS technique for such applications. Positron annihilation spectroscopies are based on detailed measurements, and evaluation of properties of annihilation gamma rays, and can provide electron density or momentum at the annihilation site. The data generated by the other three techniques have also been included in this thesis for valid comparison of the measured stresses as a function of different metallurgical variables. The metallographic data using optical microscopy have also been presented. In essence, an in-depth understanding of the
measured residual stresses has been developed through analysis and interpretation of experimental data generated by both destructive and non-destructive techniques.
2.1 Materials

Martensitic Alloys EP-823 and HT-9, and austenitic Type 304L Stainless Steel (SS) were used in this investigation as the candidate structural materials to contain the transmutation target material. While the austenitic Type 304L SS comes under the category of iron-chromium-nickel-molybdenum (Fe-Cr-Ni-Mo) steel, the martensitic alloys falls under the Cr-Mo steels. Experimental heats of these materials were melted by a vacuum-induction-melting (VIM) practice. They were subsequently forged, and rolled into plate materials of desired dimensions. These materials were then thermally-treated prior to the machining of the test specimens. Type 304L SS plates were austenitized at 1850°F for 1 hour followed by an air cooling, thus producing a fully austenitic microstructure. Alloys EP-823 and HT-9 were austenitized at a similar temperature followed by an oil quench. The quenched plates were subsequently tempered at 1150°F followed by air-cooling. This type of thermal treatment produced fully tempered martensitic microstructure without the formation of any retained austenite. The chemical compositions of all these tested material are given in Table 2.1.
Table 2.1 Chemical Composition of Materials Tested (wt %)

<table>
<thead>
<tr>
<th>Material/Heat No</th>
<th>Elements (wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C</td>
</tr>
<tr>
<td>EP-823/2154</td>
<td>0.17</td>
</tr>
<tr>
<td>HT-9/2239</td>
<td>0.20</td>
</tr>
<tr>
<td>Type 304L SS/2155</td>
<td>0.02</td>
</tr>
</tbody>
</table>

Alloy EP-823 is a martensitic stainless steel (SS) and has been extensively used as a structural material to contain the molten lead-bismuth-eutectic (LBE), which is used as a target and coolant during the transmutation process. Martensitic stainless steels are known for their high tensile strength, moderate creep, and fatigue properties, in combination with appreciable corrosion and heat resistance. These materials are essentially alloys of Cr and Mo that can possess a body-centered cubic (bcc) crystal structure in the hardened (martensitic) condition. The medium carbon content in the martensitic stainless steels enables the development of the desired metallurgical microstructures and properties resulting from hardening and tempering. The Cr and carbon (C) contents are balanced to ensure a fully-tempered martensitic microstructure following hardening and subsequent tempering. The presence of Mo can enhance the mechanical properties and the localized corrosion resistance. Alloy EP-823, containing a relatively high silicon level, possesses a superior corrosion resistance in oxygen-containing LBE coolant due to the formation of protective silicon oxide film on its surface. This material has been successfully used in Russia as a structural material to
contain molten LBE for transmutation applications. This alloy shows significant resistance to swelling during high neutron exposure at temperatures up to 420°C and can exhibit high creep resistance during irradiation.

Alloy HT-9 is a Cr containing martensitic stainless steel, also known as 12Cr-1MoVW. It contains 12 weight percent of Cr. Cr provides significant resistance to atmospheric corrosion, while Mo enhances the resistance of this alloy to seawater corrosion. A small Ni content in this alloy offset the ferritizing effect of low carbon content. Alloy HT-9 has a body centered cubic (BCC) lattice structure. Martensitic stainless steels are considered to be one of the most suitable candidate materials for the blanket and first-wall structures of a fusion reactor. These alloys are also finding increased application in fast breeder reactor systems. Alloy HT-9 possesses better corrosion and swelling resistance and can provide appreciable resistance to irradiation-embrittlement at 60°C. This alloy can also exhibit high creep strength. High strength and resistance to corrosion and swelling in the fast-neutron environment have made Alloy HT-9 a primary candidate material for use as cladding in the current U.S. fast reactor designs.

Austenitic, or nonmagnetic stainless steels (SS), are classified as Type 200 and 300 series, with 16 to 30% Cr and 2 to 20% Ni for enhanced surface quality, formability and increased corrosion and wear resistance. They are nonhardenable by heat-treatment. All austenitic stainless steels are nonmagnetic in the annealed condition. Depending on their compositions, mainly the Ni content, austenitic SS can become slightly magnetic in the cold-worked condition. Ideally, austenitic stainless steels exhibit a single-phase, face-centered cubic (fcc) structure that can be maintained over a wide range of temperatures.

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This structure results from a balance of alloy content, primarily Ni, which can stabilize the austenitic phase at cryogenic to elevated temperatures. These single phase alloys can only be strengthened by solid-solution alloying or by work-hardening.\(^{(7)}\) The austenitic stainless steels were primarily designed for applications in both mild and severe corrosive environments. These materials can exhibit high toughness at different temperatures, in addition to enhanced oxidation resistance. Since the austenitic materials are nonmagnetic, they are sometimes used in applications where magnetic materials are not acceptable. The austenitic class of stainless steels is generally considered to be weldable by commonly used techniques. The physical and mechanical properties of all three tested materials are shown in Table 2.2.

<table>
<thead>
<tr>
<th>Property</th>
<th>304L SS</th>
<th>Alloy EP-823</th>
<th>Alloy HT-9</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal Conductivity, W/m*K</td>
<td>16.2</td>
<td>NA</td>
<td>28</td>
</tr>
<tr>
<td>Modulus of Elasticity, Gpa (10^6 psi)</td>
<td>193</td>
<td>207</td>
<td>160</td>
</tr>
<tr>
<td>Poisson’s Ratio</td>
<td>0.25</td>
<td>0.29</td>
<td>0.33</td>
</tr>
<tr>
<td>Coefficient of Thermal Expansion per °C * 10^-6</td>
<td>9.4</td>
<td>NA</td>
<td>12.5</td>
</tr>
</tbody>
</table>

2.2 Test Specimens

Heat-treated plates of all these alloys were plastically deformed by cold rolling to reduce the plate thickness by approximately 3, 7 and 11 percent. Some of the heat-treated rectangular machined beams were deformed in three-point bending to produce a gradual residual stress gradient along their lengths. Prior to the deformation process, all beams were electropolished to remove the surface cold-working resulting from the machining operation. The center of the beam was displaced 1.5 inches during the bending process. The outer supports were separated by 10 inches. Welded specimens
consisting of similar and dissimilar metals (Alloy EP-823, Type 304L SS and Alloy HT-9) were prepared using gas tungsten arc welding (GTAW) method. The configurations of all the test specimens are shown in the Figure 2.1.

Figure 2.1 Specimen Configurations
CHAPTER 3

EXPERIMENTAL TECHNIQUES

Four different techniques were used for the evaluation of residual stresses in target structural materials. Both nondestructive and destructive methods were utilized. Nondestructive techniques were Positron Annihilation Spectroscopy (PAS), Neutron Diffraction (ND) and X-ray Diffraction (XRD). On the other hand, the ring-core technique falls under the destructive category. A brief description of each method is presented in this section. However, an emphasis will be given to the PAS technique in this thesis.

3.1 X-Ray Diffraction Technique

The x-ray diffraction (XRD) technique exploits the fact that when a metal is under stress (applied or residual), the resulting elastic strains can cause the atomic planes in the metallic crystal structure to change their spacing. Since metals are composed of atoms arranged in a regular three-dimensional array to form a crystal, most metal components of practical interest consist of many tiny crystallites or grains that are randomly oriented with respect to their crystalline arrangement, and are fused together to make a bulk solid. When such a polycrystalline metal is subjected to stresses, elastic strains can be produced in the crystal lattice of the individual crystallite.\textsuperscript{(12)} XRD method can measure the
interatomic spacings, which are indicative of the elastic strain in the specimen. Changes in the interatomic spacing can, therefore, be related to the elastic strain in the material and hence, to the stress. This technique uses repeated scanning of a selected peak with the specimen orientated at an increasing angle to the incident beam shown in Figure 3.1. The x-ray beam is directed onto the sample surface at the location of interest. The diffracted beam is detected by a position-sensitive proportional counter. The angular position (2θ) of the diffracted beam is used to calculate the distance (d-spacing) between parallel planes of atoms using Bragg's law. A series of measurements made at different x-ray beam approach angles (ψ) are used to fully characterize the d-spacing. The slope of the least squares fit on a graph of the d-spacing versus sin² ψ is used to calculate the stress.

Figure 3.1 Conventional XRD Testing Technique
3.2 Neutron Diffraction Technique

The Neutron Diffraction (ND) method relies on elastic deformations within a polycrystalline material that cause changes in the spacing of the lattice planes from their stress-free value. Although stress measurements by XRD method are well established, they are practically limited to the near-surface stresses. Measurements by ND are carried out in much the same way as with XRD, with a detector moving around the sample, locating the positions of high intensity diffracted beams. A major advantage that neutrons have over x-rays is their capability to penetrate into greater depths that can make them suitable for measurements at depths ranging from around 0.2 mm to about 1 cm. With high spatial resolution, ND can provide complete three-dimensional strain maps of many engineered components. This is achieved through translational and rotational movements of the component. A collimated neutron beam of wavelength $\gamma$ is diffracted at an angle of $2\theta$ by the polycrystalline sample. The collimated beam then passes through a second collimator and finally reaches the detector as shown in Figure 3.2. The slits of the two collimators define the ‘gauge’ volume, the cross section of which can be as small as 1 mm x 1 mm and, in special cases, even smaller. The interplanar distance ($d$) can be evaluated using Bragg’s law, and the corresponding lattice strain can be evaluated. The stress values can, therefore, be determined from these strain readings using appropriate mathematical formulae. This method of stress evaluation, with a capability of collecting large quantities of data over the whole surface and depth has made ND a particularly useful technique for the validation of theoretical and numerical models.
3.3 Ring-Core Technique

The ring-core (RC) method is a mechanical/strain gage technique used to determine the principal residual stress as a function of depth in polycrystalline and/or amorphous materials. This method involves the localized removal of stressed material using an incremental ring coring or a mechanical dissection device, and measurement of strain-relief in the adjacent material. Strain gage rosettes (SGR) are usually used to measure the relieved strains. The method used in this study consisted of dissecting the desired location by a nominally 0.25 inch diameter plug containing the strain gages, as shown in Figure 3.3. A total nominal depth of \(70 \times 10^{-3}\) inch was cut by this plug at increments of \(2 \times 10^{-3}\) inch. The relieved strain measurements were made on the surface of the material remaining inside the ring. The residual stresses existing in the material before ring coring were calculated from the measured relieved strains.
3.4 Positron Annihilation Spectroscopic Technique

Positron annihilation results from the interaction of a positron with an electron. Positron is a positively charged particle and its annihilation with an electron produces two gamma rays, back to back. The energy and momentum conversation during annihilation can be used to study the properties of materials of interest. Numerous experimental techniques based on positron annihilation have been developed in the past. Broadly, they can be classified into two categories, which are distinguished by the sensitivity of positron annihilation to the electron density, and the to the electron momentum distribution. Positron lifetime measurements are based on the fact that positron annihilation rates are sensitive to the electron density, while two other techniques named “Doppler Broadening” (DB) and “Angular Correlation and Annihilation Radiation” (ACAR), are based on the electron momentum.

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. Thus the electron significantly contributes to the center of mass of the
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 will 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. For the spectroscopic studies here, Doppler broadening technique was used.

PAS has been successfully used as a method for non-destructive technique in material science. PAS is highly sensitive for lattice disorders in crystalline materials. The underlying principle of the PAS technique is illustrated in Figure 3.4. After the positron is produced in the sample by means of pair production, the positron loses its high energy within a few seconds by means of thermalization. In metals the positron is sensitive to lattice defects such as dislocations and vacancies. At the defect site the
electron density is relatively lower compared to the perfect lattice because of missing atoms.\(^{(26)}\) When positrons are produced in the test sample, it diffuses in to the lattice. At the end the positron annihilates with an electron from the surrounding lattice. In defect free lattice the positron is localized.\(^{(22)}\) Due to the positive charge, the atomic nucleus repels the positron and hence, it is found in the delocalized state mainly in interstitial regions. In the case of defects (missing of an atom) in the lattice, the positron may be trapped there due to the absence of the repulsive potential of the positively charged atomic nucleus. The measurements based on the momentum distribution of the annihilating positron electron pair, the momentum of the positron can be neglected because of its thermalization. Therefore, Doppler broadening of the annihilation radiation is mainly determined by the electron momentum.

The trapping of positrons at the defect site can lead to characteristic changes in the measured parameters of PAS. When the positron wave function is localized at defect site, its overlap with the core electrons will be decreased compared to its overlap with less tightly bound valence electrons and so, it will annihilate more with valence electrons. This is reflected in the Doppler broadening measurements, where a noticeable reduction of the annihilation peak is seen. 511 keV curve, also known as characteristic curve is used in characterizing the defects. Line shape parameters S, W and T are often used in characterizing the annihilation peak in Doppler broadening spectroscopy as shown in Figure 3.5.\(^{(24)}\) 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, which is most often used, is the ratio of W to the S parameter. As evident it can be seen from the figure, the S parameter is relatively high for a material having voids than the defect free material. In contrast the W and T parameter are high in magnitude for a defect free than the material containing vacancies.

![Figure 3.5. Characteristics of 511 keV γ-ray Energy Spectrum](image)

3.5 Experimental Facility

The PAS technique used in this investigation employed high penetrability gamma rays to extend PAS into thick samples and to enable measurement of stress, strain and defects in three materials of interest. These high penetrable gamma rays were produced by a linear accelerator (LINAC). The collimated bremsstrahlung beam from a LINAC was used to generate positrons inside the test specimen via pair production as shown in Figure. 3.6 and Figure 3.7. A 6 meV LINAC was used in this study for the production of
positrons by impinging high penetrable gamma rays onto the test specimen. The frequency of the accelerator, which was often referred as repetition rate is maintained at 500Hz throughout for the reliability of the data obtained. The resulting bremsstrahlung photons 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 the electrons emitting two photons having energy of 511 keV, back to back. These photons were recorded by a high-energy germanium (HPGe) detector, and the resultant data were analyzed in terms of three line-shape parameters, (S W and T) as discussed earlier. The energy resolution of HPGE detectors varied from 1.2 to 1.6 keV at the $^{133}$Ba $\gamma$-line of 356 keV. The relatively large error bars are due to low counting rates associated with pulsed beams because the count rate is limited by the low repetition rate of the electron linac. An improvement of these statistical errors can be achieved by increasing the repetition rate of the accelerators.

Figure 3.6 - PAS Test Setup

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3.6 PAS Calibration Curve

Efforts are ongoing at the Idaho Accelerator Center (IAC), to develop calibration curves for different alloys for residual stress measurements by the PAS technique. These calibration will be utilized to precisely determine the magnitude of residual stresses by knowing different line shape parameters such as S, W or T, as described earlier. Cylindrical tensile specimens made of different materials were tested according to the ASTM designation E 8 (25) to generate stress strain diagrams at ambient temperature. The magnitude of yield strength (YS) and ultimate tensile strength (UTS) were determined from these diagrams. Tensile specimens were subsequently loaded at stress values ranging between YS and UTS, and the corresponding strains were recorded. Line shape parameters including S and T were then determined by subjecting these loaded specimens

Figure 3.7 – PAS Test Setup Geometry
to PAS measurements. The magnitude of $S$ and $T$ parameters corresponding to each stress/strain value were then utilized to generate calibration curves showing $S$ or $T$ as a function of measured stress/strain for a particular material.

3.7 Optical Microscopy

Microstructural evaluations of test materials were performed by using a Leica optical microscope. An important aspect of optical microscopy is to characterize the metallurgical microstructures resulting from special thermal treatment imparted to test material, 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 test specimens were properly sectioned and mounted with epoxy material. The mounted specimen was then polished and etched by appropriate procedures for each material to reveal the microstructures. The polished and etched specimens were then rinsed in deionized water, and dried with acetone and alcohol prior to their evaluations of metallurgical microstructures.
CHAPTER 4

RESULTS

The results of residual stress measurements by the PAS, ND and RC techniques on all three test materials are presented in this section. While detailed data obtained by the PAS technique have been presented here, comparative analyses of these data have also been made to those obtained by the other two techniques. Further, approaches taken to establish the calibration curves for the PAS data analyses have been presented in this section.

4.1. Mechanical Properties

The results of tensile and hardness testing of Alloys EP-823 and HT-9, and Type 304L SS are shown in Table 4.1. These results indicate that the hardness values of two martensitic stainless steels in the quenched and tempered condition were much higher than that of austenitic Type 304L SS that was tested in a solution-annealed condition.
Table 4.1 Tensile Properties Including Hardness Data

<table>
<thead>
<tr>
<th>Material / Heat No.</th>
<th>Thermal Treatments</th>
<th>Yield Strength (Ksi)</th>
<th>Ultimate Tensile Strength (Ksi)</th>
<th>% El</th>
<th>% RA</th>
<th>Hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>EP-823/2154</td>
<td>Quenched and Tempered</td>
<td>103.4</td>
<td>124.4</td>
<td>24.9</td>
<td>61</td>
<td>25 Rc</td>
</tr>
<tr>
<td>HT-9/2155</td>
<td>Quenched and Tempered</td>
<td>115.7</td>
<td>139.4</td>
<td>21.4</td>
<td>61.4</td>
<td>31 Rc</td>
</tr>
<tr>
<td>304L SS/2239</td>
<td>Solution Annealed</td>
<td>47.6</td>
<td>72.4</td>
<td>66.6</td>
<td>51</td>
<td>64 Rb</td>
</tr>
</tbody>
</table>

4.2. PAS Measurements

4.2.1 Cold-Worked Specimens

The results of residual stress measurements on cold-worked (CW) Alloy EP-823 and Type 304L SS specimens by the PAS technique are shown in Figure 4.1 and 4.2, respectively. Both of these materials were subjected to three different levels of cold-reduction (CR) by rolling. The resultant data are plotted using the T-parameter values as a function of different levels of CR, as shown in these two figures. These results indicate that the magnitude of the reduction in T-parameter was higher at increased level of cold-work. Usually, a lower T-parameter is indicative of higher residual stress due to enhanced plastic deformation. Thus, both figures have shown a consistent trend in T-parameter versus %CW for Alloy EP-823 and Type 304L SS. In general, the internal/residual stresses developed inside the metal matrix due to plastic deformation are tensile in nature. But the PAS technique, as of now, is not capable of differentiating the nature (tensile vs compressive) of these residual stresses.

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Figure 4.1 Effect of %CW on T- Parameter

Figure 4.2 Effect of %CW on T- Parameter
4.2.2 Welded Specimens

Welded specimens consisting of both similar and dissimilar metallic materials were tested for residual stress evaluations by the PAS technique. A welded specimen consists of a fusion zone next to the weld, followed by a heat-affected-zone (HAZ) and the base metal. The maximum residual stresses are usually generated along the fusion line of a welded specimen. The distribution of S-parameter as a function of weld-distance is shown in Figure 4.3 for a welded specimen consisting of Type 304L SS only on both sides. A higher S-parameter indicates a higher amount of residual stress. An examination of this plot clearly indicates that the magnitude of the residual stress was highest at a location closest to the fusion line by virtue of the highest S-parameter. The value of the S-parameter was gradually reduced with increasing distance from the fusion line, indicating reduced residual stresses, as expected. A similar trend was also observed with a welded specimen consisting of two dissimilar materials (Type 304L SS / Alloy EP 823), as illustrated in Figure 4.4 for the Type 304L SS side of the welded specimen. Due to different metallurgical microstructures between the austenitic and martensitic stainless steel, the rate of solidification/contraction may be different in two alloys. This effect of different metallurgical properties was reflected in the plot of T-parameter versus weld-distance for the Alloy EP-823 side of the welded specimen (Figure 4.5), showing lower T-parameter values with increasing distance. For a welded specimen consisting of two dissimilar materials, the nature of residual stresses can generally be different showing tensile and compressive residual stresses, respectively in different alloys. Thus, the higher T-parameter with Alloy EP-823 at the fusion line may indicate a compressive
stress that may gradually become less negative or close to zero stress level at distances away from the fusion line.

![Graph](image)

**Figure 4.3 Effect of Weld-Distance on S-Parameter**

![Graph](image)

**Figure 4.4 Effect of Weld-Distance on T-Parameter**

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4.2.3 Tensile Specimens

PAS measurements were performed on tensile specimens of Alloy HT-9, subjected to different stress levels using a MTS testing machine. Initially, a cylindrical specimen was loaded in tension to complete failure according to the ASTM designation E 8\(^{25}\). The corresponding stress-strain diagram is shown in Figure 4.6. The magnitudes of the yield strength, ultimate tensile strength and the failure stress were recorded from this stress-strain diagram. Subsequently, one specimen each was loaded in tension up to certain load levels corresponding to the yield stress ($\sigma_y$), the maximum stress ($\sigma_m$) and a stress value close to the failure stress ($\sigma_f$), as shown in Figures 4.7, 4.8 and 4.9, respectively. Stress measurements by the PAS technique were performed on an unstressed (control) specimen, and specimens subjected to these three stress levels, and the magnitude of the...
T-parameter was subsequently determined for each stress level. A plot of T-parameter versus applied stress is shown in Figure 4.10. An examination of this plot reveals that the unstressed (control) specimen and the specimen loaded up to $\sigma_y$ showed the highest T-parameter values indicating the minimum internal/residual stress, as expected. The lowest value of the T-parameter was observed with the specimen loaded up to $\sigma_m$, indicating the maximum internal stresses developed due to plastic deformation in Alloy HT-9.

![Figure 4.6 Ambient-Temperature Stress-Strain Diagram](image-url)
Figure 4.7 Stress-Strain Diagram for Specimen Loaded up to $\sigma_y$

Figure 4.8 Stress-Strain Diagram for Specimen Loaded up to $\sigma_m$
Figure 4.9 Stress-Strain Diagram for Specimen Loaded Close to $\sigma_y$

Figure 4.10 T-Parameter versus Applied Stress

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4.3 Comparative Analyses of Welded Specimens

Comparisons of residual stresses in a welded specimen consisting of Alloy EP-823 and Type 304L SS, determined by the ND, RC and PAS techniques are shown in Figures 4.11, 4.12, 4.13 and 4.14, respectively. Since the PAS technique, so far, has been incapable of providing quantitative residual stresses in the welded specimen, attempts have been made to compare the residual stress values measured by the PAS technique in terms of a T-parameter. As indicated earlier, a larger T-parameter usually indicates a lower stress value. An examination of Figure 4.11 indicates that the value of T-parameter for the Alloy EP-823 side of the welded specimen gradually became smaller with increasing distance from the weld. As explained earlier, compressive residual stresses may be generated in the Alloy EP-823 side of the welded specimen consisting of dissimilar materials adjacent to the fusion line. At a location one inch away from the fusion line, which represents the base metal, these stresses became less compressive (approximately zero stress) in nature indicating a reduction in internal/residual stress at distances away from the fusion line.

A comparison of these PAS data to those obtained by the ND technique on the EP-823 side indicate that compressive residual stresses were developed near the fusion line. However, these residual stresses became almost neutralized (close to zero stress level) away from the fusion line to the base metal (Alloy EP-823). A similar trend was also observed when the PAS data was compared to the RC data, as shown in Figure 4.12. Comparison of residual stresses measured by the PAS and the ND techniques on the Type 304L SS side of the welded specimen are shown in Figure 4.13. Here the lowest-value of
the T-parameter measured by the PAS technique may indicate the highest tensile residual stresses adjacent to the fusion line, as expected.

A comparison of residual stresses measured by the PAS technique to those obtained by the RC method indicates that the maximum residual stresses were developed near the fusion line of the Type 304L SS side of the welded specimen, as shown in Figure 4.14. These data clearly show a consistent pattern as to the effect of weld-distance as the resultant residual stresses as measured by two different testing techniques. The ND data (Figure 4.13) indicate that the residual stresses generated on the Type 304L SS side near the fusion line gradually became less compressive. However, somewhat higher tensile residual stresses were observed at a distance one inch from the fusion line. No explanation based on the current understanding can be provided for this increased residual stresses in the base material (Type 304L SS).

Figure 4.11 Comparison of Residual Stress Profiles by ND and PAS Techniques
Figure 4.12 Comparison of Residual Stress Profiles by PAS and RC Techniques

Figure 4.13 Comparison of Residual Stress Profiles by PAS and ND Techniques
4.4 Metallography

Even though this investigation is primarily focused on the evaluation of residual stresses in austenitic and martensitic target structural materials using destructive and nondestructive techniques, it appears appropriate to characterize the metallurgical microstructures, especially in a welded specimen. In view of this rationale, metallographic evaluations were performed on welded specimens consisting of similar and dissimilar materials. The optical micrograph of Alloy HT-9 is illustrated in Figure 4.15, showing fine-grained martensitic microstructure with some delta-ferrite (white particles). The optical micrographs of a welded specimen consisting of Alloy EP-823 and Type 304L SS are shown in Figures 4.16 and 4.17, respectively. A similar martensitic microstructure was observed with Alloy EP-823, as shown in Figure 4.16.
Type 304L SS was characterized by equiaxed austenite grains (dark) and annealing twins, as illustrated in Figure 4.17.

Figure 4.15 Optical Micrograph of Alloy HT-9

Delta Ferrite

(a) EP-823, Base metal, Etched, 10X  (b) EP-823, Base and Weld Metal, Etched, 10X

Figure 4.16 Optical Micrographs of Alloy EP-823 Welded Specimen
Figure 4.17 Optical Micrographs of Type 304L SS Welded Specimen
CHAPTER 5

DISCUSSION

Both destructive and nondestructive techniques have been used to measure residual/internal stresses in candidate target structural materials that were subjected to cold deformation and welding operations. The materials tested include martensitic Alloys EP-823 and HT-9, and austenitic Type 304L SS. All materials were tested in properly heat-treated conditions. As presented in the previous section, detailed analyses of residual stress data based on the PAS technique have been performed. In addition, residual stress measurements obtained by the other two techniques namely neutron diffraction and ring-core methods have been included for the sake of comparison. This section is aimed at providing a critical discussion on the overall testing data. For simplicity, the data obtained on different types of specimens by each measurement technique have been discussed separately.

5.1 PAS Data – CW Specimens

The cold-worked specimens of Alloy EP-823 and Type 304L SS showed a consistent pattern in residual stresses in that a reduction in the T-parameter value was observed with increased level of cold-reduction (CR). As indicated in the previous section, a reduced T-parameter is indicative of higher residual/internal stresses embedded in the metal matrix resulting from plastic deformation due to cold reduction. Since a substantial
amount of residual stresses by rolling in the longitudinal direction can be generated in a metallic material due to plastic deformation, it is obvious that a lower T-parameter, as seen here, would signify, higher residual stresses in both Alloy EP-823 and Type 304L SS in the cold-worked condition. However, the magnitude of the T-parameter at each level of CR was different for different material, which can be attributed to the differences in metallurgical characteristics such as the chemical composition, microstructure and resultant mechanical properties due to thermal treatment. Thus, the resultant data suggest that the PAS technique is an effective tool in measuring residual stresses in plastically-deformed engineering materials, such as the three candidate target structural materials incorporated in this research program.

5.2. PAS Data – Welded Specimens

The welded specimens consisting of Type 304L SS on both sides showed a decrease in the value of S-parameter away from the fusion line. It is well-known that the residual stresses in the vicinity of heat affected zone would be higher and would tend to be reduced at distances away from the fusion line. This phenomenon was in fact substantiated by the highest S-parameter value observed at locations adjacent to the fusion line. On the other hand, the data generated on the welded specimen consisting of two dissimilar metals (Type 304L SS and Alloy EP-823) revealed a decrease in the T-parameter on the Type 304L SS side and a gradual increase in the T-parameter on the Alloy EP-823 of the welded specimen side away from the fusion line. These data may indicate the development of a tensile residual stresses in Type 304L SS while generating compressive residual stresses for Alloy EP-823. This difference in the nature of the
residual stress may be due to the differences in metallurgical properties of both alloys the filler material used to weld them.

Even though a quantitative assessment of the residual stresses measured by the PAS techniques is yet to be established, an extensive effort is in progress at UNLV in collaboration with the Idaho Accelerator Center to develop calibration curves for a specific material, which eventually would enable the estimation of residual stresses using the line-shape parameters obtained by the PAS technique. The PAS data measured on a tensile specimen loaded up to $\sigma_m$ yielded the lowest T-parameter value, indicating the highest residual stresses as expected. On the other hand, the specimens that were loaded up to $\sigma_y$ and a stress close to $\sigma_f$, the magnitudes of the T-parameter were higher, as anticipated. The generated PAS data on a control or unloaded specimen revealed the highest T-parameter indicating very little or no residual stresses. A similar behavior has been reported by other investigators.\(^{(23 \text{ and } 26)}\)
CHAPTER 6

SUMMARY AND CONCLUSIONS

Destructive and nondestructive techniques have been used to evaluate residual stresses in three candidate target structural materials including Alloys EP-823, HT-9 and Type 304L SS. The PAS, ND and RC techniques have been utilized to determine the residual stresses in cold-worked and welded specimens of all three alloys. This thesis is primarily focused on the use of the PAS technique, in an effort to standardize this method as a nondestructive tool to measure residual stresses in thick materials. A preliminary testing was carried out using tensile specimens of Alloy HT-9 to establish a calibration curve, which eventually would enable the estimation of residual stresses using the line-shape parameter (S, W, T) obtained from the PAS data. Although this investigation is primarily focused on the evaluation of residual stresses, an effort has also been made to characterize the metallurgical microstructures of welded specimens. The significant conclusions that can be drawn from this investigation are summarized below.

- The resultant residual stress data on the cold-worked specimens showed a reduction in the T-parameter value with increased plastic deformation. Specimens of Alloy EP-823 and Type 304L SS showed a consistent pattern on the effect of cold-reduction level showing a decrease in the T-parameter value with an increase in cold-reduction level. An increase in the magnitude of the residual stress is associated with a reduced T-parameter value.
• Welded specimens consisting of similar materials were tested by the PAS, RC and ND methods. The data generated on the welded specimens consisting of Type 304L SS on either side of the weld, showed a tensile residual stresses in the vicinity of the fusion line.

• The data generated on the welded specimen consisting of dissimilar materials (Alloy EP-823/Type 304L SS) by the PAS, ND and RC techniques revealed compressive residual stresses on the Alloy EP-823 side and tensile residual stresses on the Type 304L SS side at the fusion line. This difference in the nature of residual stress can be attributed to the difference in the metallurgical characteristics between these two materials.

• The residual stresses measured on the tensile specimens of Alloy HT-9, loaded to different stress levels, showed the maximum T-parameter value at an applied stress corresponding to the \( \sigma_m \) value. Very little or no stresses are observed with a specimen loaded up to \( \sigma_y \) value which was characterized by a higher T-parameter value, as anticipated.
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.

- Development of calibration curves showing T or S-parameter verses stress for all three alloys tested in this investigation.
- Residual stress measurements on three point bent specimens by the PAS technique for comparison of data obtained by the other methods.
- Evaluation of the effect of post-weld-thermal-treatment on residual stresses in welded specimens.
- Characterization of defects in plastically deformed and welded specimens by Transmission Electron Microscope.
- Evaluation of radiation hardening in target structural materials by activation through an accelerator.
BIBLIOGRAPHY


2. A.V. Locating, V.V. Olav, A.I. Filin, “Transmutation of Long-Lived Nuclides in The Fuel Cycle of Brest-Type Reactors”, (RDPE, Moscow, Russia)

3. FJ/OH Summer School, 2002-Beam Targets Design in Accelerator Driven Systems


9. Development of Martensitic steels for high neutron damage applications, D.S GELLES, BATTELLE PACIFIC NORTHWEST NATIONAL LAB


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15. The determination of the residual strains and stresses in ATIG welded sheet of IN718 superalloy using ND


18. Tanaka, Keisuke (Dept. of Mechanical Engineering, Nagoya University); Akiniwa, Yoshiaki; Hayashi, Makoto, *Materials Science Research International*, Vol. 8, No. 4 SPEC, December 2002, p. 165-174

19. Measurement of residual stresses in welded steel joints using the hole drilling method, Weng, C.C.; Lo, S.C


22. G.Dulbek, N.Meyendorf, "Positron Annihilation Spectroscopy"


27. Karsten Bennewitz, Matz Haaks, Torsten Staab, Stephan Eisenberg, Thomas Lampe, Karl Maier, “Positron annihilation spectroscopy- a non-destructive method for lifetime prediction in the field of dynamic material testing”


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