Material Properties Measurement

Residual Stress Measurement in Alloy 182
J.J. Wall, K.J. Krzywosz, EPRI, USA; C.R. Hubbard, Oak Ridge National Laboratory, USA

ABSTRACT
Weld residual stresses in components in nuclear power plant piping systems can facilitate stress corrosion cracking and, ultimately, lead to failure. Nondestructive measurement of residual stresses in such components would allow identification of susceptible areas and allow preemptive mitigation or empirical welding process improvement. This study explores portable methods to measure residual stresses in Ni-based alloy 182 metallurgical specimens subjected to ex-situ thermal and mechanical loading. The “baseline” residual stresses were measured using neutron diffraction. The data from the neutron diffraction measurements was used to conduct a feasibility study of portable nondestructive residual stress measurement techniques based on Eddy current testing and portable x-ray diffraction. The experimental results of the feasibility study are presented.

INTRODUCTION
Residual stresses in welded Alloy 182 and austenitic stainless steel components contribute directly to primary water stress corrosion cracking (PWSCC) and fatigue cracking during service. Residual stresses in welded components develop during cooling due to expansion/contraction hysteresis across the weld and heat-affected zone. Material in a tensile stress field exists in a thermodynamic energy landscape that favors chemical reaction—for example, chloride attack—as compared to the unstressed material. Conversely, if one were to put the same material in a compressive stress field, corrosion would become energetically difficult. As a general practice, tensile stress of repaired welds, weld overlay, and/or preemptive overlay is minimized, and compressive surface stresses can be introduced by a mechanical heat stress improvement process (MSIP) and/or by induction heat stress improvement (IHSI).

Successful repair and mitigation of pipe welds requires knowledge of the residual stress state in the component. Nondestructive measurement of residual stresses is typically done using X-ray diffraction (XRD) and/or neutron diffraction (ND). However, these methods are usually not portable and X-ray diffraction is limited to near-surface measurements due to the short penetration depth of laboratory x-rays in metals (~ 101 µm). Thus, there is a strong interest in exploiting alternative technologies for residual stress characterization in engineering components.

EXPERIMENTAL
Sample Fabrication
A total of 20 test pieces of weld overlaid Alloy 182 plates were prepared by laying a plate of weld material on a ferritic steel substrate and cutting out plates of dimensions 10 mm x 50 mm x 150 mm. 4 mm diameter Alloy 182 welding rods were used. Note that the plates were hand ground to remove weld bead peaks prior to sectioning. After sectioning, the test pieces were subjected to heat treatment, simulated stress corrosion cracking, and/or crack repair. Three different heat treating schedules were employed:

1) Light: 650°C under Ar for 24 hours.
2) Medium: 750°C under Ar for 24 hours.
3) Heavy: 1050°C under vacuum for 1 hour then gas quenched.
An additional heat treatment stage of 600°C under Ar for 20 hours was done for samples subject to stress corrosion cracking. Three levels of corrosion were used for the samples subject to stress corrosion cracking.

1) Light: HCl, HNO₃, and CH₃COOH solution exposure for 10 seconds.
2) Medium: HCl, HNO₃, and CH₃COOH solution exposure for 120 seconds.
3) Heavy: HCl, HNO₃, and CH₃COOH solution exposure for 25 minutes.

Three stress levels were applied in a 3-point bending configuration to simulate stress corrosion cracking. The stress levels used were:

1) Light: 90% of the yield stress.
2) Medium: 110% of the yield stress.
3) Heavy: 120% of the yield stress.

Weld repair was done by physically grinding out cracks and TIG welding Inconel 82 filler material into the ground out region. Table 1 shows the sample preparation details for the Alloy 182 samples. In the interest of brevity, only the results from Samples NDT 897 and 906 will be presented in this paper.
Table 1 Alloy 182 Sample List

<table>
<thead>
<tr>
<th>Label</th>
<th>Weld Direction</th>
<th>Heat Treatment</th>
<th>Corrosion</th>
<th>Stress</th>
<th>Crack</th>
<th>Repair</th>
<th>Re-Heat Treatment</th>
</tr>
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<tbody>
<tr>
<td>NDT 896</td>
<td>Transverse</td>
<td>Light</td>
<td>None</td>
<td>Medium</td>
<td>7 mm complex</td>
<td>1.3 mm Excavation</td>
<td>None</td>
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<tr>
<td>NDT 897</td>
<td>Longitudinal</td>
<td>Light</td>
<td>None</td>
<td>Medium</td>
<td>9 mm, 7 mm &amp; 3 mm complex</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>NDT 898</td>
<td>Transverse</td>
<td>Light</td>
<td>None</td>
<td>Light</td>
<td>None</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
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<td>Light</td>
<td>Light</td>
<td>None</td>
<td>None</td>
<td>None</td>
<td>None</td>
</tr>
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<td>NDT 900</td>
<td>Transverse</td>
<td>Light</td>
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<td>None</td>
<td>None</td>
<td>None</td>
<td>None</td>
</tr>
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<td>NDT 901</td>
<td>Longitudinal</td>
<td>Sensitized</td>
<td>None</td>
<td>Medium</td>
<td>Multiple complex</td>
<td>7.7 mm Excavation Medium</td>
<td>Medium</td>
</tr>
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<td>Sensitized</td>
<td>None</td>
<td>Medium</td>
<td>9 mm complex</td>
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<td>Medium</td>
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<td>None</td>
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<td>None</td>
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</tr>
<tr>
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<td>None</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>NDT 906</td>
<td>Transverse</td>
<td>Sensitized</td>
<td>None</td>
<td>Heavy</td>
<td>14.6 mm &amp; 8.9 mm complex</td>
<td>2.4 mm Excavation Heavy</td>
<td>Heavy</td>
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<tr>
<td>NDT 907</td>
<td>Longitudinal</td>
<td>Sensitized</td>
<td>None</td>
<td>Heavy</td>
<td>21 mm &amp; 19.5 mm complex</td>
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<td>Heavy</td>
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<td>NDT 908</td>
<td>Transverse</td>
<td>Heavy</td>
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<td>Heavy</td>
<td>None</td>
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<td>None</td>
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<tr>
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<td>Sensitized</td>
<td>Heavy</td>
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<td>None</td>
<td>None</td>
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<tr>
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<td>None</td>
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<td>None</td>
<td>None</td>
<td>None</td>
</tr>
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<td>NDT 911</td>
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<td>None</td>
<td>None</td>
<td>None</td>
<td>None</td>
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</tr>
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</table>
Diffraction Residual Stress Measurement

Diffraction strain measurement is based on the geometrical solution of constructive interference when radiation interacts with a crystalline lattice. This is best described using Bragg’s Law, which states:

\[ \lambda = 2d \sin \theta \]

where \( \lambda \) is the wavelength of radiation, \( d \) is the spacing between diffracting planes of atoms, and \( \theta \) is the scattering vector angle. A simple illustration of this concept is shown in Fig. 1.

![Diagram showing the geometry of diffraction in a crystal lattice.](image)

Figure 1 - The geometry of diffraction in a crystal lattice. The conditions are satisfied for constructive interference, resulting in an intensity spike in the \( l(2\theta) \) XRD spectrum.

As such, diffraction can be used to determine the average interatomic strain in a volume of polycrystalline materials by irradiating a large number of grains and characterizing the nature of diffraction. The lattice strain is determined by relating the measured \( d \) to that of a known stress free standard \( (d_o) \).

\[ \varepsilon = \frac{d - d_o}{d_o} \]

Lattice strain can then be used to determine the residual stress using Hooke’s Law:
\[
\sigma_i = \frac{E}{(1+\nu)} \left[ \varepsilon_{ii} + \frac{\nu}{1-2\nu} \left( \varepsilon_{xx} + \varepsilon_{yy} + \varepsilon_{zz} \right) \right]
\]

Where \( i \) refers to the principal direction; \( x, y, \) or \( z \) (longitudinal, transverse, or normal, respectively). \( E \) is the Young's modulus and \( \nu \) is the Poisson's ratio. For the present material (Alloy 182) \( E \) and \( \nu \) are 22,775 ksi and 0.3, respectively [2].

Two diffraction methods were used to characterize the residual stresses in the test samples and reference samples: x-ray diffraction (XRD) and neutron diffraction (ND). X-ray diffraction is a surface characterization method, limited to a depth of the order of \( 10^1 \) \( \mu \)m, meaning that in-plane stress components cannot be measured directly. As such, using this method requires an assumption that the surface normal strain, \( \varepsilon_{zz} \), is zero. The measurement of \( \varepsilon_{xx} \) and \( \varepsilon_{yy} \) is done by rotating angle of incidence of the x-ray beam, \( \psi \), toward the transverse and longitudinal directions, respectively. The strains are extrapolated based on the behavior of \( \varepsilon(\psi) \) as \( \psi \) is varied in +/- increments from zero (\( \psi = 0 \) corresponds to \( \varepsilon_{zz} = 0 \)). A simple illustration of this is shown in Fig. 2.

![Diagram of XRD measurement geometries](image)

Figure 2 - (a) shows the change in \( d \) with \( \psi \) as the incident beam is rotated relative to the normal (unstressed) geometry. (b) shows a normal component measurement geometry \((d_n)\). (c) shows an off-normal measurement geometry \((d_i)\). Taking multiple \( d \) measurements at different \( \psi \) rotations allows extrapolation to estimate the in-plane d-spacing, \( d_\psi \), corresponding to \( \psi = 90^\circ \).

Neutron diffraction allows the direct measurement of the in-plane components as well as the normal components with variable depth. Direct characterization of \( \varepsilon_{xx} \) and \( \varepsilon_{yy} \) is possible because neutrons have a much larger penetration depth in most engineering materials than laboratory x-rays. The depth and size of the gage volume may be defined by collimation of the incident and scattered neutron beam. Typical measurement geometries for the determination of \( \varepsilon_{xx}, \varepsilon_{yy}, \) and \( \varepsilon_{zz} \) are illustrated in Fig. 3. Note that to compute the strain in this manner, one must first measure \( d_o \) in an unstressed representative section of the material. For a more comprehensive treatment of x-ray and neutron diffraction residual stress measurements, see texts by Cullity and Stock [1] and Krawitz [2].
Figure 3 - (a) typical geometry for the measurement of normal diffraction plane spacing ($d_n$) in a plate sample using neutron diffraction. (b) that for longitudinal ($d_l$), and (c) shows that for transverse ($d_t$). Note that the transverse direction points out of the page in (b).

Eddy Current Residual Stress Measurement

Nondestructive eddy current residual stress measurement is based on the piezoresistivity effect, in which the electrical conductivity in a material changes with stress state. Under isotropic plane-state stress, the relative change in conductivity, $S$, can be determined by:

$$S = \frac{\Delta \sigma}{\sigma_0} = \kappa_{ip} \frac{\tau}{E}$$

where $\Delta \sigma$ is the change in electrical conductivity due to stress, $\sigma_0$ is the electrical conductivity of the stress-free material, $\kappa_{ip}$ is the electroelastic coefficient, which is the sum of the parallel and normal coefficient components, $\kappa_{11}$ and $\kappa_{12}$, respectively, and $\tau$ is the isotropic stress. The $\kappa_{11}$ and $\kappa_{12}$ coefficients must be obtained to characterize stresses using eddy currents. This is done by loading a test specimen in uniaxial tension and compression and measuring the conductivity using both nondirectional and directional eddy current probe coils as shown in Figure 3.
Once the coefficients are known, residual stress can be determined by measuring the local electrical conductivity in the sample.

RESULTS

The XRD measurements were performed using a Proto portable x-ray diffractometer housed at EPRI. 2-dimensional surface maps were collected in an area of 3” x 1” (76.2 x 25.4 mm) using lateral spacings between data points of ½” (12.7 mm) in the “x” and “y” directions. The measurement points on a typical sample are illustrated in Fig. 3.4. The experimental parameters used for the XRD measurements are listed below. It should be noted here than sample NDT 911 was sectioned for a separate study, hence only the centerline data was acquired (centerline along Y in Fig. 5).

Detectors: solid state scintillators (2)
Bragg peak: Ni 311
Radiation: MnKα (2.XXX Å)
Incident beam collimation: 3 mm x 1 mm
Tension: 18 kV
Current: 1.5 mA
ψ range: +/- 30°, 7 rotations.
ψ oscillation : +/- 3°
Count time: 20 seconds / rotation
Gain material: β-Ti
Standard: 304 stainless steel powder
Fitting function: Gaussian
Cutoff: 60%
Background subtraction: linear.
Figure 5 - 2-D surface map data points for x-ray diffraction strain measurement. Upper shows the top surface, lower shows the bottom surface (sample rotated 180° about the Y-axis.)
Figure 6 - Sample NDT 897 (Centerline is Y = 25.4 mm) characterized using XRD
Figure 7 - Sample NDT 906 (Centerline is Y = 25.4 mm) characterized using XRD
The ND measurements were performed using the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory on the Neutron Residual Stress Facility (NSRF) diffractometer. The longitudinal, transverse, and normal lattice parameters were measured. These were converted to principal strains using the lattice spacing of the center of volume of sample NDT 911 as a stress free reference. Principal stresses were computed using Hooke’s Law. The experimental parameters used for the ND measurements are listed below.

**General**

Peak: Ni 311  
Monochrometer: Si 422 (1.54 Å)  
Count time/point: Normal = 2 minutes, transverse/longitudinal = 6 minutes.  
Detectors: He-3 chamber array  
Fitting function: Pseudo-Voigt

**Sample Specific**

2-D maps of the near-surface residual stress using the measurement configuration shown if Figure 5 were acquired. The average depth of measurement was 1 mm from the surface. The scattering volume was approximately 10 mm x 10 mm x 2 mm. X and Y were defined by collimation in 1 dimension and tracking in second dimension, Z was defined by collimation. A rocking angle of +/- 4° was used to improve diffraction statistics.
Figure 8 - Sample NDT 897 (Centerline is $Y = 25.4$ mm) characterized using ND.
The eddy current residual stress measurement technique was found not to be feasible to characterize the Alloy 182 samples. 2-D images of the electric conductivity in a test piece were obtained to see whether eddy current measurements might be suitable for residual stress characterization in the Alloy 182 weld overlay plates. Initial imaging is necessary before high-precision conductivity measurements are conducted to see whether the microstructure is sufficiently homogeneous to get meaningful data. A conductivity image of the front surface of the measured specimen is shown in Fig. 10. The image shows only a 2.5"-by-2.5" center area of the specimen. The picture was taken at 2 MHz, so the effective penetration depth is around 200 microns.
The initial test indicated that the various sources of inhomogeneity in the Alloy 182 specimen appeared to be too severe to use the standard Eddy current residual stress characterization technique. As can be seen from the example raw data, Fig. 11, the stress-dependence of the electric conductivity exhibited a significant hysteresis that indicated the presence of a minute, but influential ferromagnetic phase. The average circular (directionless) electroelastic coefficient was \(-3.17 \times 10^{-5} \% \text{IACS/ksi}\) or \(-4.6 \times 10^{-6} \% \text{IACS/MPa}\), Fig. 3.

Typical values seen in nickel-base superalloys are between \(-2.3\) and \(-5.3 \times 10^5 \% \text{IACS/MPa}\). As such, the magnitude and sign of the electroelastic coefficient is fairly typical and would allow eddy current residual stress assessment. However, the hysteretic behavior clearly shows the presence of a ferromagnetic phase, which effectively excludes the application of the eddy current method. Unfortunately, it was determined that there was no realistic chance to use eddy current conductivity measurements for stress assessment in the weld deposited Alloy 182 material since the otherwise rather weak ferromagnetic phase causes an unacceptable measurement uncertainty.
CONCLUSIONS

1) The near surface residual stresses in Alloy 182 weld deposited plates were characterized using neutron diffraction and laboratory x-ray diffraction.

2) Surface maps of the residual stresses were characterized using x-ray diffraction. The measured data were dominated by surface effects and did not correspond directly to the ND measurements.

3) Eddy current residual stress was determined not to be feasible for the Alloy 182 samples due to heterogeneity of the conductivity as well as the presence of a ferromagnetic phase that probably nucleated during weld deposition.