

6-18-2003

## Use of Positron Annihilation Spectroscopy for Stress-Strain Measurements: Annual Progress Report (May 2002 – May 2003)

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Roy, A. K. (2003). Use of Positron Annihilation Spectroscopy for Stress-Strain Measurements: Annual Progress Report (May 2002 – May 2003). 1-11.

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**Annual Progress Report  
(May 2002 – May 2003)**

**Use of Positron Annihilation Spectroscopy for Stress-Strain  
Measurements  
TRP Task-14**

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**June 18, 2003**

# Use of Positron Annihilation Spectroscopy for Stress-Strain Measurements

## Introduction

The purpose of this collaborative research project involving the University of Nevada Las Vegas (UNLV) and Idaho State University (ISU) to evaluate the feasibility of determining residual stresses of welded, bent (three-point-bend), and cold-worked engineering materials using a new non-destructive technique based on positron annihilation spectroscopy. The proposed technique is the use  $\gamma$ -rays from a small MeV electron Linear accelerator (LINAC) to generate positrons inside the sample via pair production. This method can be used for materials characterization and investigation of defects in thick samples, which could not be accomplished by conventional positron technique or other non-destructive methods. The data generated will be compared to those obtained by other non-destructive methods such as neutron diffraction and X-ray diffraction (XRD), and a destructive method known as the ring-core technique. Materials that are currently being tested in the experimental program are Austenitic Type 304L stainless steel (SS), Martensitic Alloys EP-823 and HT-9.

## Personnel

The current project participants are listed below.

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## **Background and Rationale**

Plastic deformation of metals and alloys produces an increase in the number of lattice imperfections known as dislocations, which by virtue of their interaction results in higher state of internal stress and reduces ductility. This type of deformation which is carried out in a temperature region and over a time interval such that the strain hardening is not relieved is called cold-work. When cold-working is excessive (greater than the uniform elongation), the metal will fracture before reaching the desired size and shape. Thus, in order to avoid this drawback, cold-working and annealing is frequently done, a process known as the cold-work-anneal cycle. By suitable adjustment of this anneal cycle, this part can be produced with any desired degree of strain hardening. However, in order to remove the undesirable internal stresses, a stress relief thermal treatment needs to be done.

During welding of engineering components, thermal cycles can cause changes in physical state, metallurgical phase transformation, and transient thermal stress. The welded part may contain physical discontinuities that arise due to excessively rapid solidification, or adverse microstructure that are due to inappropriate cooling, or tensile residual stresses and distortion resulting from the existence of incompatible plastic strains. Presence of high tensile residual stresses in and around (such as the heat-affected-zone) the welded region can cause premature failure under certain conditions.

Materials used in accelerator-driven waste transmutation (ATW) systems, such as target and other structural parts, are likely to be influenced by adverse residual stresses resulting from cold work and welding. In view of this rationale, this research project is aimed at evaluating residual stresses in cold worked and welded Austenitic Type 304L stainless steel, and Martensitic Alloys EP-823 and HT-9 by three different techniques.

## **Experimental Procedure**

Three different techniques, namely  $\gamma$ -ray induced positron annihilation spectroscopy, neutron diffraction and ring-core method were used to determine the residual stresses present in the test materials.

### **Positron Annihilation Spectroscopy:**

The Positron Annihilation Spectroscopy (PAS) is well established non-destructive tool to characterize materials and defects. However the conventional positron annihilation spectroscopy use slow positron beams or wide energy spectrum beams from radioactive sources.

The technique proposed here exploits the high penetrability  $\gamma$ -rays and the high sensitivity of the positron to extend positron annihilation into thick samples and enable measurement of stresses, strains and defects in engineering materials (Figure 1).

The photon beam from the LINAC is incident on the material for which residual stresses have to be evaluated. On entering the material, this photon beam undergoes pair production and hence splits up into a positively charged positron and a negatively charged electron. Since the positron is a very sensitive particle, it has a tendency to settle down in a crystal defect which is caused due to plastic deformation. This positively charged positron annihilates with the valence electron of the neighboring atoms and hence produces two  $\gamma$ -rays which possess energy of 511 KeV. These  $\gamma$ -rays are detected by the HPGe detector. This information is plotted as shown in Figure 1. The PAS technique can be used for measuring different line shape parameters (S, W and T).

### **Ring Core Method:**

The ring-core method is a mechanical/strain gage technique employed to determine the principal residual stress field as a function of depth in polycrystalline or amorphous material. The method involves placing a strain gage rosette at the surface at the location of interest on a given component. An annular groove is machined around the strain gage rosette at predetermined depth increments (Figure 2). The strain relaxation that occurs as a function of machined depth is recorded. The final residual stress values are calculated using the measured change in strain with depth. The ring-core method works well on materials that are coarse grained, such as cast metals and weldments. XRD method was also used to measure residual stresses.

### **Neutron Diffraction Method:**

Neutron diffraction is also a non-destructive method, which is based on measuring,  $d$ -spacing between the atomic planes of a crystal lattice. When a neutron beam of known wavelength is impinged upon a crystalline specimen, neutrons are diffracted at an angle that depends on  $d$ -spacing. With accurate measurement of the diffraction angle, the  $d$ -spacing between the lattice planes can be calculated, to determine if the planes are being pushed together (compression), or pulled apart (tension). The measured patterns of residual stresses, as determined by this technique, provide knowledge of the possible location of defects, and the effectiveness of thermal treatments to relieve the internal stresses arising from welding and cold deformation. However, this technique is limited to very thin specimens with the neutron beam penetrating only a small depth (a few millimeters) below the specimen surface. Further, this method is not effective if the grain size is larger than 100  $\mu\text{m}$ . In view of these deficiencies, a limited number of measurements will be performed on these samples using the facilities at both the Atomic Energy of Canada Ltd (AECL) laboratory and Los Alamos National Laboratory (LANL).

### **Microstructural Evaluation and Metallurgical Characterization**

The following tasks will also be performed.

- Transmission Electron Microscopy to analyze the imperfections due to plastic deformation/welding.

- Microstructure evaluation of the welded specimens showing base metal/heat affected zone.

### **Accomplishments**

Experimental heats of Type 304L (SS), and Alloys EP-823 and HT-9 were recently melted at the Timken Research, Canton, OH using vacuum induction melting (VIM) process. They were degassed using argon. The chemical compositions of all three heats of materials are given in Table 1.

### **Specimen Configuration:**

Cold-worked, three-point-bent and welded specimens (Figure 3) of all three alloys were fabricated using heat treated plates.

### **Results and Discussion**

#### **Positron Annihilation Spectroscopy (PAS):**

PAS measurements were made on three-point-bent and cold-worked specimens of Alloy EP-823 and Type 304L SS.

- Data indicate that the magnitude of T-parameter ( $T = W/S$ ) was gradually reduced with the increase in the degree of cold working (Figure 4).
- The effect of increased cold working was more pronounced in 7% cold reduction compared to that at 11% cold reduction. It is possible that the extent of residual stresses due to cold reduction became stabilized at 7% cold reduction value.
- Discrepancies were observed in PAS measurements of three point bent beam specimens, which are currently being rectified using larger frequency.

#### **X-Ray Diffraction method (XRD):**

- Residual stress measurements using XRD technique was not possible on Type 304L SS due to its coarse grained structure.
- XRD measurements on three-point-bent specimens of Alloy EP-823 revealed compressive residual stresses on the convex side and tensile residual stresses on the concave side (Figure 5).
- XRD measurements were made at a nominal depth of 10 mils of the welded specimen configurations (EP-823/EP-823, 304L SS/EP-823) on the EP-823 side.
  - The residual stresses on the EP-823 side of EP-823/304L SS were compressive from the fusion line to a nominal distance of 1 inch away.
  - Residual stresses were tensile near the weld for the EP-823/EP-823 sample.

**Ring Core Method:**

- Destructive evaluation using ring-core method was performed on the welded specimens of similar and dissimilar metals. Configurations of EP-823/EP-823, EP-823/304L SS and 304L SS/304L SS were tested. Measurements were made at four locations on the specimens (adjacent, 0.25, 0.5 and 1 inch from the fusion line). Results of ring-core measurements are shown in Figure 6.

**Problems**

No problems are anticipated.

**Status of Funds**

Expenditures incurred during the first year are within the target amount allocated.

**Plans for Year 2**

- Residual stress measurements on welded specimens using PAS technique.
- Establishment of a calibration curve by applying PAS technique to unstressed tensile specimens (S/T parameter vs applied stress/strain).
- Residual stress measurement by neutron diffraction method at AECL/LANL.
- Use of Transmission Electron Microscope to analyze voids and dislocations due to plastic deformation/welding.
- Microstructural evaluation of welded specimens showing base metal/heat affected zone.
- Standardization of Positron Annihilation Spectroscopy for residual stress evaluation.

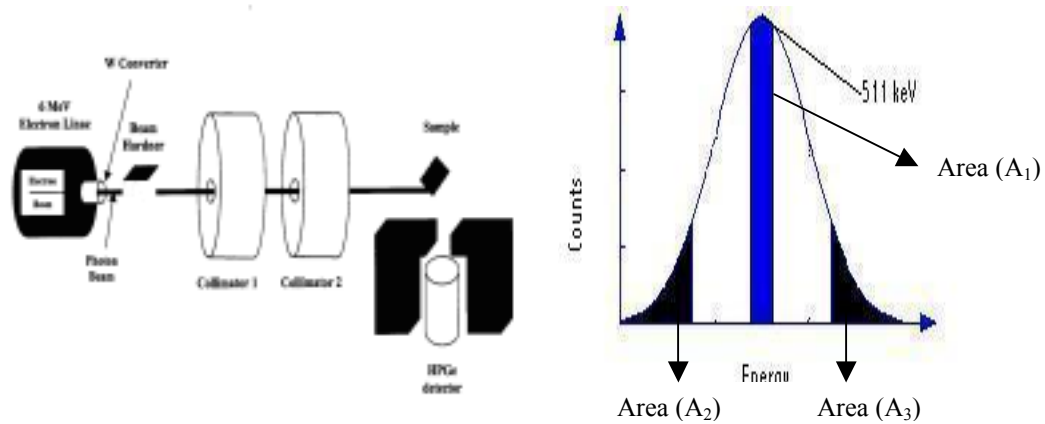
Table 1. Chemical composition of materials used\*.

Material/Heat Number	Elements (wt %)															
	C	Mn	P	S	Si	Cr	Ni	Mo	Cu	V	W	Cb	B	Ce	Al	Fe
EP-823/2154	0.17	0.54	0.005	0.004	1.11	11.69	0.65	0.73	0.01	0.34	0.63	0.26	0.0058	0.08	0.023	Balance
Type 304L SS/2155	0.02	1.63	0.003	0.005	0.40	18.20	9.55	0.03	0.03	--	--	--	--	--	0.011	Balance
HT-9/ 2239	0.20	0.40	0.011	0.003	0.19	12.50	0.53	0.99	0.01	0.29	0.46	--	--	--	0.029	--

\* Thermal Treatments Performed:

Alloys EP-823/HT-9: 1850 °F / 1 hr / Oil Quenched  
 1150 °F / 1 hr / Air Cooled

Type 304L SS : 1850 °F / 1hr / Air Cooled  
 1150 °F / 1 hr / Air Cooled



$$S\text{-Parameter} = \text{Area } A_1 / \text{Total Area (A)}$$

$$W\text{-Parameter} = (\text{Area } A_2 + \text{Area } A_3) / \text{Total Area (A)}$$

$$T\text{-Parameter} = W/S$$

Figure 1. Principle of Positron Annihilation Spectroscopy





Figure 2. Principle of Ring Core Method



Bent Specimen

Welded Specimen

Figure 3. Specimen Configurations

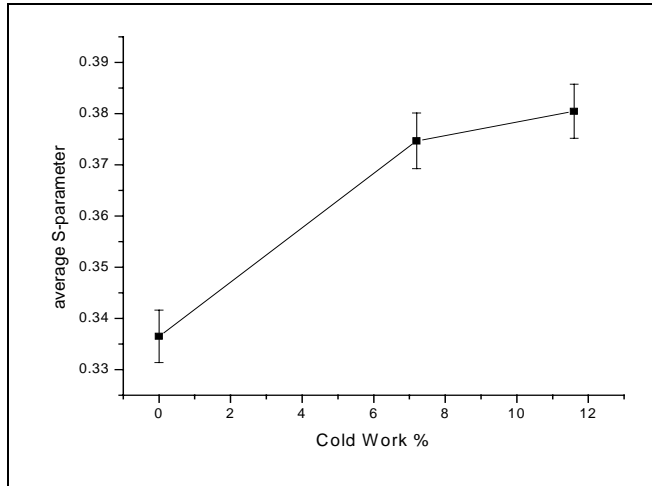
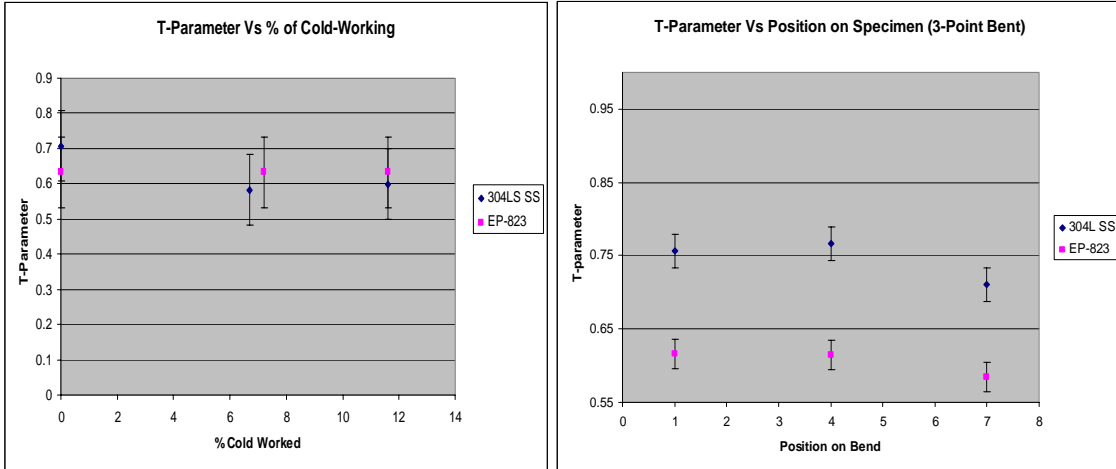


Figure 4. Results of PAS Measurements

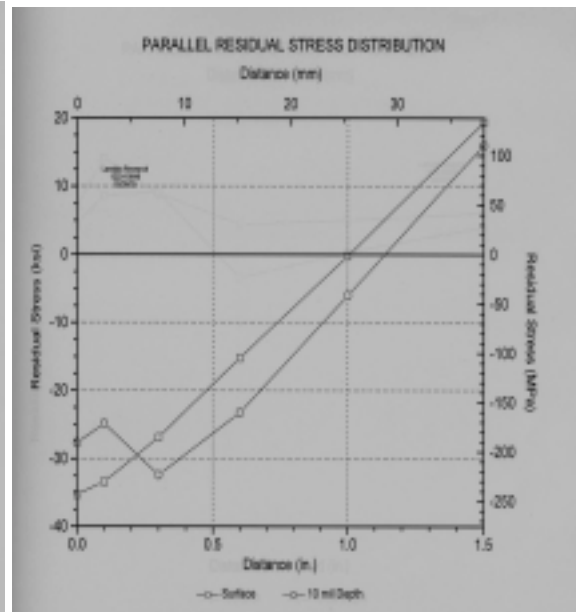
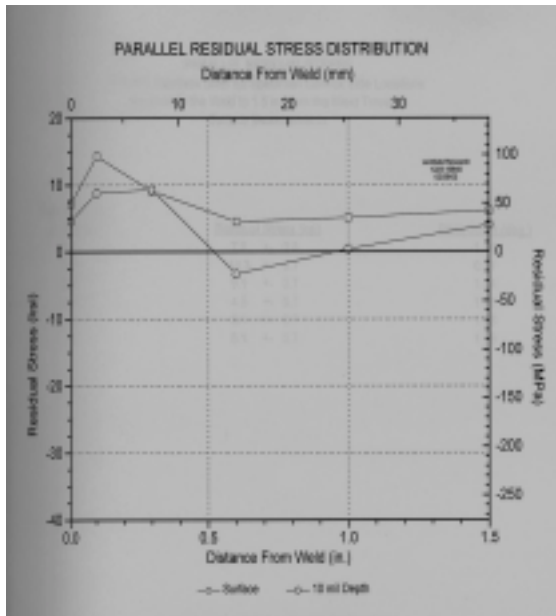
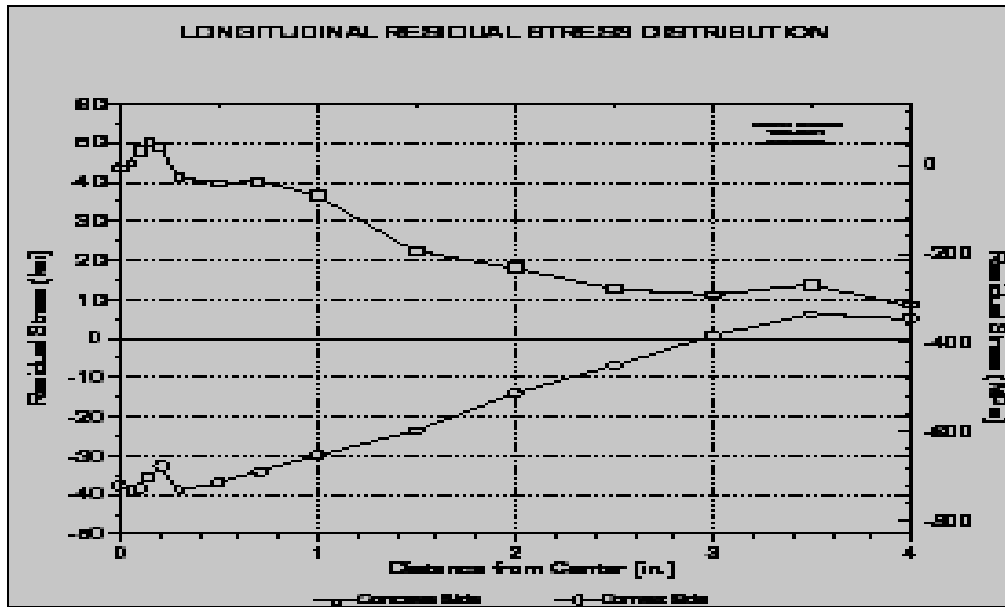


Figure 5. Results Obtained by XRD Technique.

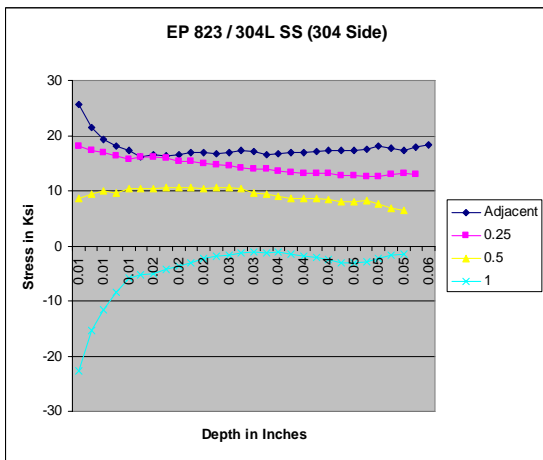
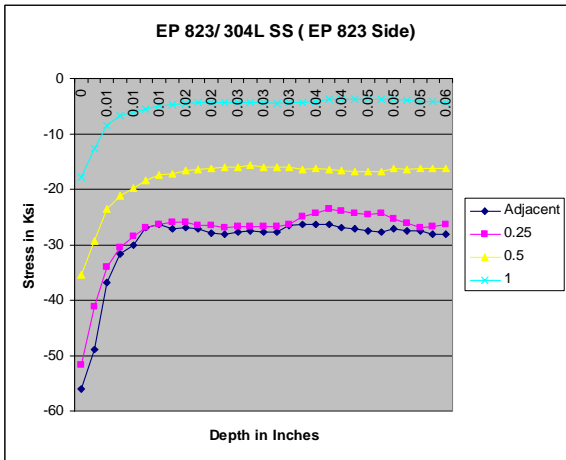
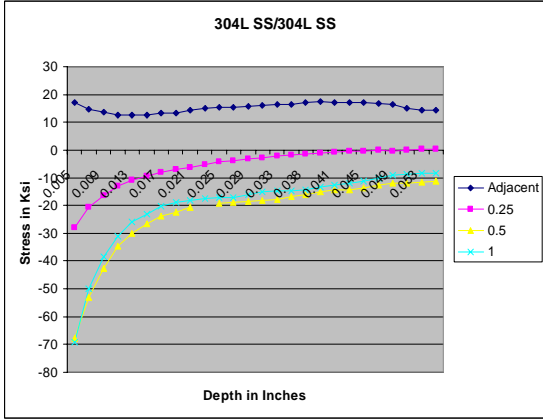
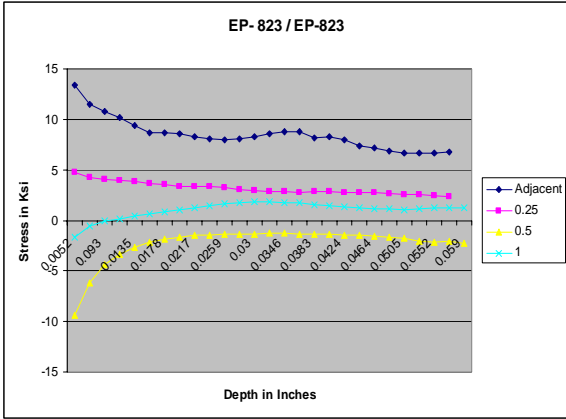


Figure 6. Results Obtained by Ring Core Method.