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Use of Positron Annihilation Spectroscopy for Stress-Strain Measurements

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Use of Positron Annihilation Spectroscopy for Stress-Strain Measurements
(Year II Renewal)

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TRP Research Area: Transmutation Sciences

Budget Request: $ 107,073

Abstract

The purpose of this collaborative research project involving the University of Nevada, Las Vegas (UNLV) and the Idaho State University (ISU) is to evaluate the feasibility of determining residual stresses of welded, bent (three-point-bend), and cold-worked engineering materials using a new nondestructive technique based on positron annihilation spectroscopy. The proposed technique is to use x-rays from a small MeV electron 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 that could not be accomplished by conventional positron technique or other nondestructive methods. The data generated will be compared to those obtained by other nondestructive methods such as neutron diffraction and x-ray diffraction, and a destructive method known as ring-core technique. Materials to be tested in the initial phase will be unirradiated austenitic (Type 304L) and martensitic (EP-823) stainless steels that will be welded, bent and cold-worked prior to the evaluation of their residual stresses. Metallurgical microstructures will also be evaluated. In addition, deformation characteristics in terms of dislocations and their movements resulting from welding and plastic deformation will be analyzed by transmission electron microscope (TEM). Later, irradiated Alloy EP-823, HT-9, and austenitic materials (Type 316L stainless steel and Alloy 718) will be included in this program.

Work Proposed for Year 2 (Summer 2003 –Spring 2004), Goals, and Expected Results:

Two experimental heats of Alloy EP-823 and Type 304L stainless steel (heat numbers 2154 and 2155, respectively) were melted during the year 2002 at the Timken Research Laboratory (TRL) using vacuum induction melting process. They were subsequently forged, and rolled into plates of desired dimensions. Alloy EP-823 plates were subsequently austenitized and oil quenched, followed by tempering to achieve a fully tempered martensitic microstructure. Type 304L stainless steel plates were solution-annealed. Three types of specimens namely, cold-worked, bent (three-point-bending), and welded specimens were prepared from the heat-treated plates of both materials. The cold-worked specimens were prepared by TRL by cold-reduction of the heat-treated plates by 7 and 11 percent. The bent specimens were fabricated by three-point-bending at the Lambda Research Laboratory (LRL). The welded specimens were prepared by LANL by welding plates of similar and dissimilar materials (304L/304L, 304L/EP-823, and EP-823/EP-823).

All three types of specimens are currently being evaluated at the Idaho Accelerator Center (IAC), LANL, and LRL for determination of residual stresses using four different measurement techniques. Positron annihilation spectroscopy (PAS) is being used at IAC. Neutron diffraction technique is being applied at LANL. Both x-ray diffraction and ring-core methods are being used at LRL. Measurement of residual stresses using all four techniques will be carried out throughout the second year of this task, followed by the analyses of the resultant data. Some of these testing and analyses will be continued during the third year. In addition, the welded specimens will be subjected to post-weld thermal treatment to eliminate or minimize the residual stresses generated during welding, followed by stress measurements by different techniques. Microstructural analyses of the welded specimens before and after thermal treatments will be performed by using optical microscopy. Dislocation characteristics will be evaluated by TEM. Further, efforts will be made to irradiate the test specimens by low-energy electron beam at IAC/ISU, and subsequently measure the enhanced residual stresses due to radiation hardening using PAS.

Background and Rationale

Plastic deformation of metals and alloys produces an increase in the number of lattice imperfection 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 operations are usually carried out in several steps, with intermediate annealing operations to soften the cold-worked metal and restore the ductility. This sequence of repeated cold-working and annealing is frequently called the cold-work-anneal cycle. By suitably adjusting this anneal cycle, the 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 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 transmutation 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 will be aimed at evaluating the residual stresses of cold worked and welded austenitic Type 304L and martensitic EP-823 stainless steels by three different techniques. Alloy EP-823 is a leading structural material to contain lead-bismuth-eutectic (LBE) nuclear coolant needed for fast spectrum operations of transmutation systems. Type 304L stainless steel is a universally known corrosion resistant iron-nickel-chrome alloy having optimum formability and weldability.

Experimental Procedure

Type 304L and EP-823 stainless steels will be received from the vendor in the form of plates of desired dimensions. The dimensions of these plate materials will be decided based on the type of specimens to be tested. They will be procured in properly heat-treated conditions. Austenitic Type 304L stainless steel is commonly used in a solution-annealed condition. On the other hand, Alloy EP-823 is austenitized and quenched, followed by tempering to achieve a fully tempered martensitic metallurgical microstructure. Subsequently, these materials will be subjected to two levels (7 and 11%) of cold reduction, bending, and welding, respectively. Welding of similar and dissimilar plate materials will be performed using the Gas-tungsten arc welding (GTAW) technique at LANL. Metallurgical microstructures of both materials will be evaluated in the as received, cold-worked and welded conditions using optical microscopy at UNLV and LANL.

Three different techniques, namely x-ray induced positron annihilation spectroscopy; neutron diffraction and ring-core method will be used to determine the residual stresses present in the test materials. The positron annihilation spectroscopy is a 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 thickness of the samples under investigation is severely limited by the range of the impinging positrons inside the sample, which is less than 1 mg/cm^2. The technique proposed here exploits the high penetrability x-rays to extend positron annihilation spectroscopy into thick samples and enable measurement of stress, strain and defects in engineering materials. The collimated bremsstrahlung beam from a small electron accelerator (6 MeV pulsed Linac) is used to generate positrons inside the test specimen via pair production. No photon-induced activation is involved in this process. Each generated positron thermalizes and annihilates with one of the sample electrons emitting two annihilation photons (511 keV) back to back. The annihilation photons are recorded by a high-energy resolution HPGe detector, and the data will be analyzed in terms of line shape parameters of 511 keV annihilation peak. The character and concentration of defects can be investigated from these measurements. The portability, reliability and relatively low cost of small-pulsed electron accelerator can create significant interest in commercial or industrial applications. Stress measurements using this technique will be performed at ISU/IAC.

Neutron diffraction is also a non-destructive method, which is based on measuring the spacing, *d*, 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*. 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 stress, as determined by this technique, provide knowledge of the possible location of fracture, 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 μm. In view of these deficiencies, a limited number of measurements will be performed on thin samples using LANSCE at LANL.

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 materials. 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. 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. X-ray diffraction method can also be used to measure residual stresses. Lambda Research Laboratory of Cincinnati, Ohio will perform these types of measurements.

Expected Data

The proposed research program will develop the following scientific/technical information, elucidating the applicability of positron annihilation spectroscopy.

- Metallurgical microstructures of as-received, cold-worked, welded and stress-relieved material(s)
- Residual strains resulting from cold work, bending, and welding operations
- Estimation of residual stresses corresponding to the measured strain values

Research Capabilities at UNLV

The following pieces of equipment are currently available in the Materials Performance Laboratory (Room TBE B-129) and the Materials Testing Laboratory (Room TBE B-150).

- Twelve Cortest Constant Load Testing Fixtures (Proof Rings – 7,500 lb Load Capacity)
- Four Cortest SSR Test Frames (Constant Extension Rate Test Fixture - 7,500 lb Load Capacity)
- Twelve High-Temperature (120^{o}C) Corrosion-Resistant Test Vessels (Hasteloy C-276)
- One High-Temperature (500^{o}C) Corrosion-Resistant Autoclave (Hasteloy C-276) with Lid having Electrochemical Connections
- Two EG&G Model 273A Potentiostats, and one EG&G eight-channel multiple potentiostat
- One Blue-M 1200^{o}C Heat Treatment Furnace
- High – Temperature Water Bath and Mettler Electronic Balance, one each
- Twelve Custom Luggin Probes for Polarization under Controlled Electrochemical Potential
- One 1000X Resolution Leica Optical Microscope with Digital Image Capture
- Buehler Sample Preparation Accessories – Isomet 4000 Linear Precision Saw, Abrasimet 2 Abrasive Cutter, Ecomet 6 Variable Speed Grinder/Polisher with Automet 2 Power Head
- One High-Temperature (1000^{o}C) Furnace with Inert Gas Purge for Tensile Properties Evaluation in Association with an MTS Axial/Torsional Test System (50,000 lb Load Capacity)

Additional Heat Treatment Facilities
Two high temperature furnaces are available:

1) Lindberg Furnace
The maximum temperature is 1200 ^{o}C (2200 ^{o}F). The working dimensions are 15” x 7.5” x 5.5”.

2) Thermodyne Furnace
The maximum temperature is 1200 ^{o}C (2200 ^{o}F). The working dimensions are 6.5” x 4.5” x 4.5”.

Machine Shop
The UNLV College of Engineering has a machine shop with two vertical mills, two lathes, a welding station, and a variety of band saws, shear breaks, and drill presses. Because this equipment is not automated, some machining needs are contracted to local machine shops. There are several good local shops with CNC, EDM, water jet, and laser cutting capabilities that can be contracted at reasonable rates.

Mechanical Testing
The UNLV College of Engineering has a 55 kip Axial/Torsional Servo hydraulic MTS Materials Testing System. The machine has a hydraulically controlled actuator with 5.5” of stroke and approximately 55^O of angular rotation. It also has a hydraulic grip supply and two different hydraulic grips: a set of 55 kip axial/torsional collet grips and a set of 27 kip wedge grips. The axial motion can be controlled by force, displacement, or an external signal such as a strain gage. The torsional motion can be controlled by torque, angular position, or an external signal. The machine is equipped with an 8-channel signal-conditioning box from the Measurements Corporation for monitoring strain gages, extensometers, and other sensors. Signals from this box are processed directly by the MTS control software programs TestStar and TestWare SX. Other accessories for this machine include: digital longitudinal and transverse extensometers and a digital deflectometer. This machine has been used for tensile, torsion, flexure, and compression testing of metals, polymer composites, and polymer foams.

Microstructural Analysis
The UNLV Mechanical Engineering Department has a photomicroscopy lab with two 3-wheel sample polishing stations along with a sample potting machine and sanding wheels. The lab has a Unimet Unitron 8644 Inverted Metallurgical Microscope with 800X magnification equipped with a digital

camera and computer for recording micrographs. The lab also has a Leco M-400A microhardness tester, several Wilson and Clark Rockwell hardness testers, and a Beuler sample mounting press.

However, the metallographic laboratory needs to be upgraded with a high-resolution optical microscope and accessories to perform microstructural characterization planned in this project. Analyses of hydrogen content by SIMS can be performed at LANL.

Scanning Electron Microscopy (http://www.unlv.edu/Colleges/Sciences/Geoscience/EMIL.htm)
The UNLV Geosciences Department has a JEOL-5600 Scanning Electron Microscope (SEM). It is optimized for imaging micron to millimeter scale topographic detail of solid materials. Resolution of up to 50 nm at 100,000 times magnification is possible. The SEM is equipped with a BSE detector and an Oxford ISIS EDS system, capable of semi-quantitative analysis (± 10%). The topographic and compositional images can be processed directly on the screen to show pseudo-color and critical point measurement of features. The images can also be combined, allowing for easy comparison of samples or different magnifications. The manual stage can accommodate four 1-cm diameter samples or one sample up to 3.2-cm diameter. The SEM and EDS are controlled by two networked Windows 95 operating systems allowing for intuitive, simple operation.

The UNLV Geosciences Department also has the JEOL-8900 Electron Probe Microanalyzer (EPMA). It is optimized for quantitative, non-destructive chemical analysis of solid materials on a micron scale. Four fully automated wavelength dispersive spectrometers (WDS) are equipped with 2 crystals each and are capable of quantifying elements ranging from boron to uranium. Concentrations of at least 0.10 wt % can be measured to within ±1% of the measured abundance. In addition, elements present in smaller concentrations can be measured with somewhat less precision. The energy dispersive spectrometer (EDS) collects a full spectrum of x-rays at once and is capable of rapidly qualifying up to 8 elements at one time. Both EDS and WDS can also be used to obtain high-precision x-ray maps and line scans of spatial variation in chemical composition. The instrument is also equipped with backscattered electron, secondary electron, and cathodoluminescence detectors capable of producing "real time" images, or automated images in tandem with x-ray mapping to further characterize the area of interest. A fully automated stage, capable of holding up to nine one-inch round samples (or six petrographic sections) has reproducibility of less than one micron. Unmounted samples up to 15 cm in diameter can also be accommodated. The EPMA is controlled by a graphical user interface on a HP-UX UNIX workstation. These two instruments are available as a user facility. A fee structure has currently being developed.

Transmission Electron Microscopy
A transmission electron microscope (TEM) has recently been procured from FEI, and is in the process of being installed at the Harry Reid Center. The anticipated date for the establishment of the TEM facility is in the fall of 2003.

Research Capabilities at ISU

The Idaho Accelerator Center (IAC) at Idaho State University provides opportunities for nuclear physics research and development to university, industrial, and governmental organizations. The center combines accelerators owned by the U.S. Department of Energy and the state of Idaho with the university's facilities and the technical expertise of university faculty and researchers. The IAC's mission is to conduct and to promote research and development in radiation science and accelerator applications. The laboratory creates partnerships with scientists and engineers in government, university and the private sector designed to lead to new advances and practical applications in nuclear and radiation science. The Center occupies some 16,000 square feet of laboratory space on the Idaho

State University campus, including the new Accelerator Center Building completed in October 1998, which houses Center operations and the major electron Linear Accelerator (LINAC). Center management is in the hands of a director and two part-time associate directors. Proposals for accelerator and facilities usage and advice on operations is given by a Users Advisory Group comprised of representatives from the university and national laboratories. Strategic direction and peer review is given by the Advisory Board, a group of nationally prominent scientists, public policy and business leaders.

The Center has available the following accelerators:

1. A 30 MeV electron linac which can produce 10 to 50 ps, 10nC pulses in the short pulse mode. The energy range in this mode is 0.5 to 28 MeV. Long pulses to 4 microseconds are also available. Repetition rates are to 360Hz. Beam energy spread can be as small as 0.5%. Three beam ports are available.
2. Two Van de Graaff type positive ion accelerators one is equipped for materials analysis studies (back-scattering, PIXE, channeling, etc.). The other Van de Graaff is principally for variable energy neutron production, using (p,n) and (d,n) reactions.
3. A mobile, variable energy (2-12 MeV) electron linac for photo-nuclear studies and applications.
4. A 6 MeV electron linac, equipped for radiation effects studies can deliver high radiation dose rates with precision dosimetry.
5. An 18 MeV electron linac for photo-nuclear studies.
6. A field portable 4MeV electron linac for radiography and neutron generation.
7. A large-scale industrial Scientific Measurements Systems x-ray tomography system, which can be equipped with 450keV or 4MeV x-ray source.

The Center has available a wide range of nuclear detector types, including neutron detectors, and supporting electronics. Custom electronics and mechanical design and fabrication are available.

Research & Applications

Current research activities include:

Radiography, tomography and nuclear techniques for NDE/NDA.
Industrial and agricultural applications of accelerator-produced radiation.
Ion and photon beam analysis for environmental and mineral extraction needs.
Radiation science in medicine; radioisotope production, accelerator based neutron sources
BNCT and other forms of neutron therapy.
Instrument and radiation detector testing for weapons surety studies and other applications.

Education

The Idaho Accelerator supports educational activities at all levels of ISU's academic areas, including:

- Physics
- Health Physics
- Engineering
- Waste Management
- Geology

- Biological Sciences
- Health Sciences

Facilities and services

Two other IAC facilities on campus house positive ion accelerators, scanners, imaging facilities, and support services. This arrangement provides a venue for convenient, inexpensive proof of principle testing, integrated demonstrations, and basic research. The broad range of ongoing scientific and engineering activities facilitates collaboration from a variety of private, university and governmental agencies, while providing an academic research environment with all the amenities of a university.

The Center can provide in house expertise in:

Neutron and photon transport calculations using various codes including MCNP, and ACCEPT.
Neutron spectroscopy using various detector types
A wide range of nuclear and radiation measurements
Precision dosimetry for gamma radiation and neutrons

Research Capabilities at LANL

Los Alamos National Laboratory (LANL) has numerous capabilities for manufacturing and characterizing materials. The facilities proposed to be used in this work include qualified gas-tungsten arc (GTA) welding by Felix Olivas of LANSCE-2, and neutron diffraction using the facilities at the Manuel Lujan Neutron Scattering Center by Mark Bourke. After a proposal is submitted and accepted, stress measurements by the neutron diffraction technique can be performed during the July-December 2002 run cycle.

Project Timeline

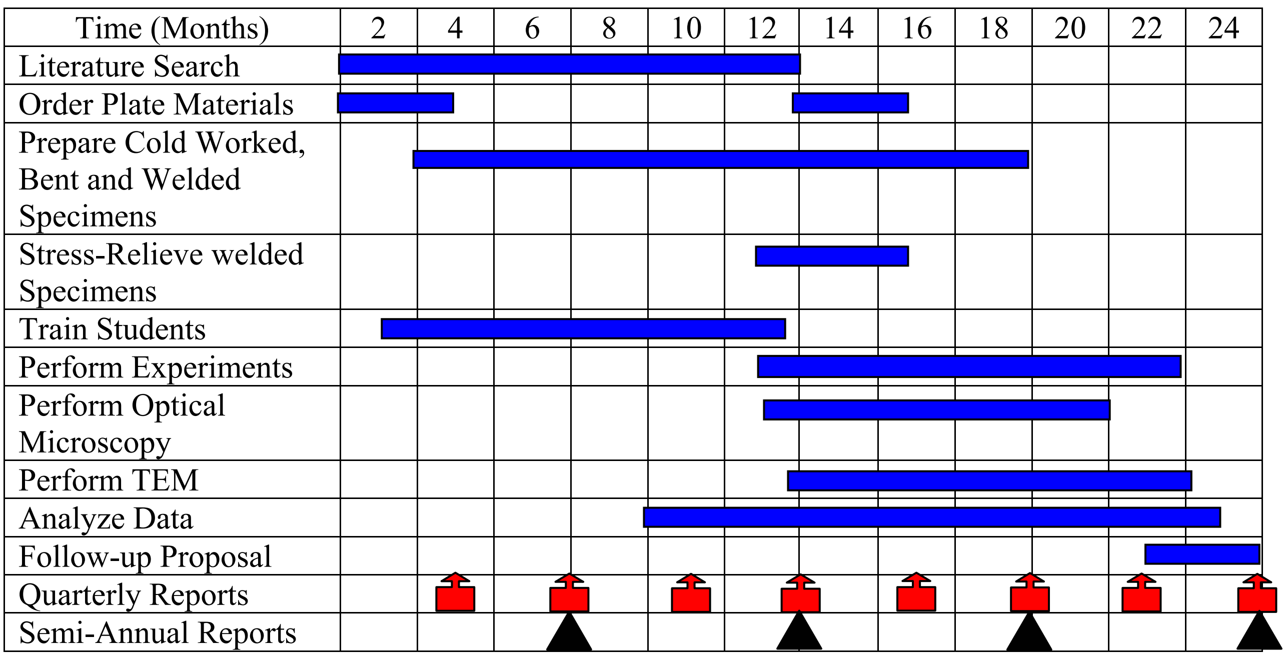
<u>Timeline Narrative</u>

Significant progress has been made during the first year of this research project. As proposed, the test materials (Type 304L stainless steel and Alloy EP-823) were melted and processed into plates within the specified time period. They were thermally treated, followed by cold reduction, bending and welding, as discussed earlier. The bent specimens were analyzed by X-ray diffraction technique at LRL for determination of residual stress due to three point bending. The measurements of residual stresses in the welded specimens are currently being performed at LRL using the ring-core method. The imperfections generated in the cold-worked (7 and 11%) specimens were characterized by PAS at IAC/ISU. Similar types of measurements are currently being performed on bent specimens at IAC/ISU. Subsequently, PAS measurements will be performed on both welded plates and tensile specimens deformed at various stress levels. Neutron diffraction technique will be used at LANL to determine the residual stresses in both welded and bent specimens. The welded specimens will eventually be stress relieved at UNLV's Materials Performance Laboratory. Microstructural analyses of welded specimens, before and after stress relief, will be performed by optical microscopy both at UNLV and LANL. The deformation characteristics (dislocations and their interactions) in bent and cold worked specimens will be analyzed by TEM both at LANL and UNLV.

Significant interactions took place during this past year among collaborators (IAC/ISU and LANL) and an independent research laboratory (LRL). The UNLV researchers (faculty and graduate students) have visited both LANL and IAC/ISU to familiarize themselves with the related experimental facilities, develop testing plans, and conduct experiments. A similar trip will be made to visit LRL in March 2003 to perform residual stress measurements on welded specimens by using ring-core method. These trips also provide the researchers with the special capabilities such as software codes and equations needed to analyze the resultant experimental data.

One technical paper based on the recent results has been accepted for presentation at the San Diego American Nuclear Society (ANS) conference in June 2003, and its subsequent publication in the symposium proceedings. Two more technical papers are currently being prepared for future conferences (MRS, ASM and SAMPE) to be held during 2003-2004. In addition, quarterly reports will be prepared as usual. A two-year schedule is shown in Table 1.

Table 1: Two-Year Research Plan



<u>Year 2 Milestones (Assuming a start date of May 20, 2002)</u>

- (April 2003): Initiate stress measurements on bent specimens.
- (July 2003) Complete stress measurements on cold worked specimens.
- (October 2003) Complete post-weld thermal treatment of welded specimens and complete additional specimen procurement.
- (December 2003) Complete Metallographic evaluation of welded specimens.
- (February 2004) Evaluate overall data and prepare follow up proposal.
- (April 2004) Prepare Year 2 report.

<u>Deliverables</u>

- **Train Graduate Students:** The primary deliverable will be two graduate student trained in a field relevant to the national AFCI program needs. An undergraduate student will also be added to train in relevant technical areas.

- **Collaboration with DOE project:** Monthly communications (by phone or in person) with National Project collaborator and/or technical lead to update on progress, discuss problems, and allow for re-focusing if necessary to address shifts in direction by the National Project.
- **Progress Reports:** Brief reports indicating progress will be provided every quarter (to support DOE AFCI quarterly meetings).
- **Bi-Annual Reports:** Written reports detailing experiments performed, data collected and results to date.
- **Final Report:** Written report detailing experiments performed, data collected, results, and conclusions to be submitted at the end of the project.
- **Project Samples:** For archival purposes, samples generated during the experimental campaigns will be turned over to the National Laboratory partner. For experiments where multiple samples were prepared, only one sample will be turned over. This sample archive will allow the Project researchers (either from the National Laboratories or UNLV or other academic partners) to re-examine samples as necessary, either in support of this work or for use in other research projects.

Roles and Responsibilities

UNLV Researchers

As Principal Investigator (PI), Dr. Roy will take the lead in the overall coordination of this project. He will assume responsibility for many different aspects of this project to ensure that all desired tasks are accomplished in a timely and cost-effective manner. He will train all three graduate students for performing the various research tasks, supervise the preparation of their thesis, and provide them with an interdisciplinary working environment so that they can make steady progress towards their research goals. As their supervisor, Dr. Roy himself will not perform the related research work but will ensure that high quality work is performed by these students through continuous guidance and consultation in areas of metallurgy, mechanics, thermal treatment, fractography and fabrication techniques. He will also be responsible for specifying and ordering test materials from prospective vendors. The graduate students will conduct and monitor the related experimental work, and will record all test data in the scientific notebooks under the supervision of the PI. Dr. Roy will analyze the resultant data, and will subsequently outline the follow-up experimental work to be performed by the graduate students. He will also prepare the interim and final reports.

ISU Collaborators

Doug Wells, Associate Professor and Farida Selim, Post Doctoral researcher at ISU/IAC will be responsible for the overall design and completion of the experiments of positron annihilation spectroscopy, which will be performed at ISU. They will perform positron measurements on the specimens supplied from UNLV to evaluate the residual stresses and microstructures and provide UNLV with the data. They will also provide guidance and daily hands on scientific expertise to the graduate students involved in the project.

Los Alamos National Laboratory

Advanced Accelerator Applications
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Date:February 12, 2002

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Subject: Support for Proposal entitled: "Use of Positron Annihilation Spectroscopy for Stress-Strain Measurements"

Dear Dr. Roy:

I enthusiastically support your proposal on the "Use of Positron Annihilation Spectroscopy for Stress-Strain Measurements" after welding of Alloy EP823 and 316L stainless steel. I believe that this proposal will lead to proof for using such a method for analyzing residual stresses. Such results will be extremely important for the design of the targets for the ATW project and such research is not being performed at any other facilities that I am aware of.

Sincerely,
Stuart A. Maloy
AAA Materials Team Leader

Stuart A. Maloy