Project Title:
Development of a Mechanistic Understanding of High-Temperature Deformation of Alloy EP-823 for Transmutation Applications
(For Renewal)

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AFCI Research Area: Transmutation Sciences

Requested Funds: $92,645 (Year Three)

Abstract:
The purpose of this project is to evaluate the elevated temperature tensile properties of Alloy EP-823, a leading target material for accelerator-driven waste transmutation applications. This alloy has been proven to be an excellent structural material to contain the lead-bismuth-eutectic (LBE) nuclear coolant needed for fast spectrum operations. However, very little data exist in the open literature on the tensile properties of this alloy. The selection of Alloy EP-823 as the test material in the proposed task is based on the recommendation of our collaborator at the Los Alamos National Laboratory (LANL). The test material will be thermally treated prior to the evaluation of its tensile properties at temperatures relevant to the transmutation applications. The deformation characteristics of tensile specimens, upon completion of testing, will be evaluated by surface analytical techniques using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The overall results will lead to the development of a mechanistic understanding of the elevated-temperature deformation processes in this alloy as a function of thermal treatment. Understanding deformation mechanisms of Alloy EP-823 may also help the development of suitable target materials possessing enhanced LBE corrosion resistance and acceptable radiation damage.

Work Proposed for Year 3 (Fall 2003 - Summer 2004), Goals, and Expected Results:
The Materials Testing System (MTS) machine to perform tensile testing, located in room TBE B-150 at UNLV, was modified during the spring of 2003 to accommodate the desired high temperature tensile testing of Alloy EP-823 that was custom melted by vacuum induction melting, and subsequently processed by the Timken Company of Ohio. A high temperature chamber capable of testing tensile specimens in the temperature range of 200 to 1000^{o}C in the presence of an inert gas (nitrogen) was added to this MTS machine. Temperature during testing using this chamber can be monitored by two K-type thermocouples. A laser extensometer having a scan rate of 100 scans/second has also been added to measure the elongation in the gage section of the test specimen during the plastic deformation under the tensile loading at the desired strain rate. Further, custom-built water-cooled specimen grips made of maraging steel (M250) have been attached to this unit. This water-cooled

assembly was designed to prevent the grips from being heated from the temperature inside the chamber by conduction. In addition, a nitrogen line has been connected to this heating chamber.

Tensile specimens, machined from the heat-treated round bars of Alloy EP-823, have been tested at ambient temperature and 100^{o}C in the presence of nitrogen. The preliminary data indicate that both the yield strength (YS) and the ultimate tensile strength (UTS) of this material were slightly reduced at the elevated temperature. No significant reduction in ductility in terms of percent elongation and percent reduction in area was observed in these tests. Temperature profiles have also been established to determine the times needed to achieve the desired test temperature, as a part of the calibration process. Additional tests are in progress. Future testing will be performed at 300, 400, 500 and 600^{o}C. The tensile properties at all testing temperatures will be evaluated as a function of thermal treatments (austenitized, quenched and tempered), in particular the tempering times (1.25, 1.75 and 2.25 hours) following austenitizing. Some of these tensile specimens will be subjected to certain amount of plastic deformation beyond the elastic limit at elevated temperatures without causing failure. Metallurgical microstructures will be evaluated by high-resolution optical microscopy at the Materials Performance Laboratory (MPL) prior to the high temperature testing. The deformation characteristics of tensile specimens, with and without failure, will be analyzed by surface analytical techniques, such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM), both at UNLV and Los Alamos National Laboratory (LANL). Periodic contacts will be made with the LANL collaborator. Brief quarterly reports will be prepared, and semi-annual and final reports will be submitted.

Funding Profile:

Academic Year:	**2001-2002**	**2002-2003**	**2003-2004**
Total (K$)	$98,735	$98,736	$92,645

Note: LANL employees do not require funding from UNLV to participate in this project.

Background and Rationale:

During his two presentations at the UNLV workshop held at the Harry Reid Center (HRC) on January 18, 2001, Dr. Stuart Maloy of Los Alamos National Laboratory (LANL) identified several research areas in the fields of Material Science and Corrosion Engineering. More recently, Drs. Roy and O'Toole had further interactions with Drs. Maloy and Li to follow-up on the related research topics, and to invite Drs. Maloy and Li of LANL to participate in HRC-sponsored collaborative research at UNLV in areas of Materials Science and Corrosion Engineering, respectively. Drs. Maloy and Li had expressed their willingness to collaborate with UNLV researchers and to actively participate in collaborative materials and corrosion research activities to be pursued at the Mechanical Engineering department of UNLV.

Target materials used in the nuclear transmutation systems will be subject to extreme temperature and corrosive environments. Issues related to environment-induced degradation of these materials are being investigated under a recently approved Advanced Fuel Cycle Initiative (AFCI) research project. However, the optimum mechanical properties of target materials for use in these environments are yet to be developed. The focus of this work is to determine the effect of elevated temperature (300-600^{o}C) on the tensile properties of one candidate target material, namely Alloy EP-823, subjected to three different heat treatments. It is anticipated that the resultant data will enable development of deformation mechanisms for Alloy EP-823 under conditions relevant to transmutation applications.

Material and Experimental Procedure:

Martensitic EP-823 stainless steel will be tested to evaluate its tensile properties at temperatures ranging from ambient to 600^{o}C. Tensile properties will be evaluated at different temperatures of interest by using the ASTM Standard E8 in an inert (nitrogen) atmosphere. The tensile specimens will be machined in the longitudinal rolling direction. An elevated temperature chamber with an inert gas atmosphere will be used with the MTS mechanical testing machine currently available in UNLV. This testing will enable the evaluation of ductility parameters such as the uniform elongation and reduction in area as well as the yield strength and ultimate tensile strength as a function of the test temperature and thermal treatments.

Metallurgical microstructures of the tensile specimens, before and after testing, will be evaluated by using standard metallographic methods (polishing and etching) involving high-resolution optical microscopy. SEM will be used to determine the morphology (ductile versus brittle) and extent of failure in each specimen tested at different temperatures. TEM will be used to develop high-temperature deformation characteristics including the distribution and nature of dislocations and other imperfections that will aid in the development of deformation mechanisms for EP-823 as functions of thermal treatment and testing temperatures.

Test Conditions:

Alloy EP-823 will be received in the form of bar stock. This material is called martensitic because it is capable of changing its crystal structure to martensite upon heating and cooling. It can be quench hardened to fully martensitic structure without any retained austenite. The EP-823 bars will be austenitized in the temperature range of 1850 to 2000^{0}F for 1 hour followed by a water/oil quench. The martensitic microstructure resulting from austenitizing and quenching is very hard and brittle. Therefore, the quenched material will further be tempered at 1150-1200^{0}F for 1.25, 1.75 and 2.25 hours followed by air-cooling. All test specimens will be fabricated from these quenched and tempered materials. Some of the tensile specimens will be cathodically charged prior to the elevated temperature testing to study the effect of hydrogen on the plastic deformation of Alloy EP-823.

The tensile properties will be generated in the presence of nitrogen primarily in the temperature range of 300 to 600^{o}C (300, 400, 500 and 600^{o}C). A few tests will also be performed at ambient temperature (25^{0}C) and 100^{o}C to develop baseline data. At least three specimens will be tested under each of the three (3) metallurgical conditions at these temperatures.

Expected Data:

The proposed research program will generate the following data:

- Uniform elongation versus (vs) temperature (T)
- Reduction in area vs T
- Yield strength vs T
- Ultimate tensile strength vs T
- Metallurgical microstructure vs thermal treatment (TT)
- Failure mode (ductile vs brittle) vs TT
- Deformation modes (TEM)

Research Capabilities at UNLV

The following equipment are currently available in the Materials Performance Laboratory (MPL), 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) Inert Gas Chamber for Tensile Properties Evaluation in Association with an MTS unit

<u>Additional Heat Treatment Facilities</u>
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”.

<u>Machine Shop</u>
The UNLV College of Engineering has a machine shop with a Haas 3-axis CNC vertical mill, two vertical mills, two lathes, a welding station, and a variety of band saws, shear breaks, and drill presses. None of this equipment is automated so we have developed good working relationships with several 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.

<u>Microstructural Analysis</u>
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.

More recently, one 1000X resolution Leica optical microscope with digital image capturing capability has been installed in the MPL. The necessary sample preparation accessories including Buehler

Isomet-4000 linear precision saw, Abrasimet-2 abrasive cutter, Ecomet-6 variable speed grinder/polisher with Automet-2 power head have also been added to this facility.

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 (HRC) at UNLV. The anticipated date for the establishment of the TEM facility is in the fall of 2003.

Project Timeline

Timeline Narrative
This research project was initiated during the fall of 2001. Since the inception of this project, Mr. Martin Lewis has been working as a part-time graduate student to pursue his M.S. degree in mechanical engineering. Subsequently, Mr. John Motaka, an undergraduate student in mechanical engineering joined this project to assist Martin Lewis in related experimental work. Mr. Motaka will be completing his B.S. degree in mechanical engineering during the summer of 2003, and will subsequently join the graduate program (M.S.) in the same discipline. In addition, Mr. Mark Jones has also been working in this task as a part-time graduate student

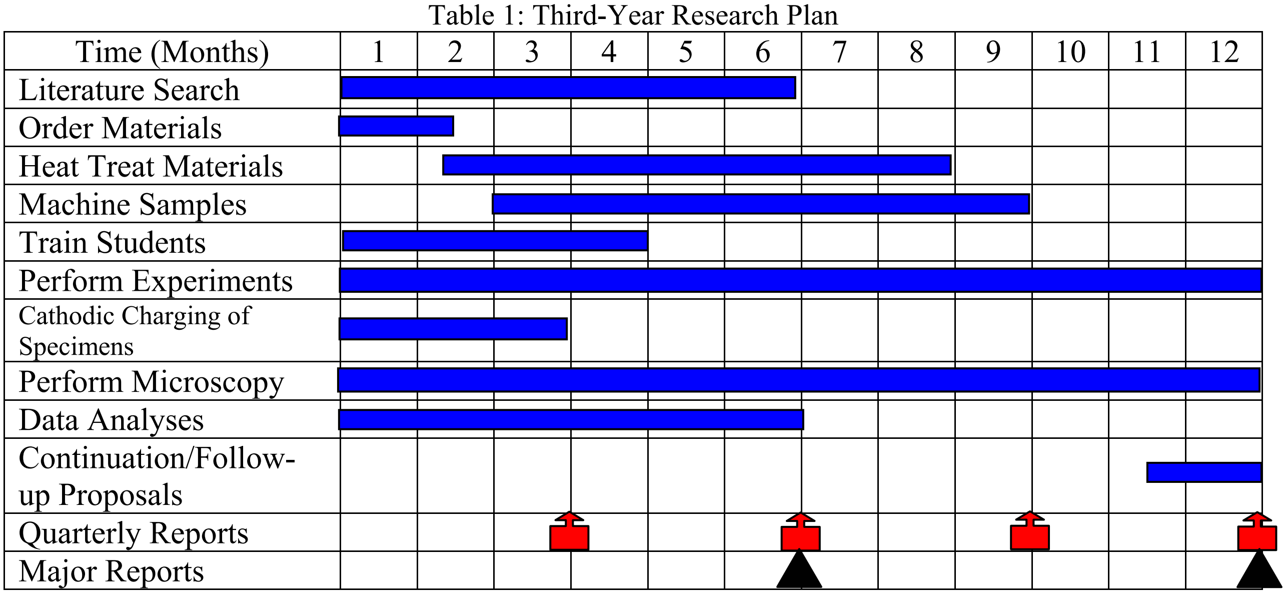
Development of infrastructures to pursue the desired experimental program is complete. Testing at ambient temperature and 100°C has just been completed. Future testing will be performed at 300, 400, 500 and 600°C using the modified MTS equipment. The tested specimens will be examined by optical microscopy, SEM and TEM. Upon review of the current and future tensile data, additional heat treatment of the processed bars of EP-823 will be done at the MPL to study the effect of tempering temperatures and times on the deformation characteristics. A trip is being planned to visit the LANL during the summer of 2003 to perform the metallurgical characterization of the tested specimens using TEM.

Brief quarterly reports will be prepared and detailed semi-annual reports will be written. It is anticipated that Martin Lewis will finish his Master's thesis during the spring of 2004. It is also anticipated that a follow-up proposal investigating the fracture toughness of this material will be submitted towards the end of the third year. The third year milestones are shown below and a schedule is shown in Table 1.

Year 3 Milestones (Assuming a start date of September 1, 2003)

- (November 2003): Quarterly report to include the first batch of mechanical test data.
- (February 2004): Quarterly report to include the surface analysis data (microscopy, SEM, TEM).
- (May 2004): Submit 2nd batch of mechanical test data.
- (July 2004): Prepare follow-up proposal.
- (August 2004): Final report to include all data.

Table 1: Third-Year Research Plan



Deliverables

- **Trained Graduate Student:** The graduate student is trained to independently perform the mechanical testing, surface analyses, fractographic evaluations, data analyses, and to prepare an M.S. thesis of high academic standard in a timely manner. The undergraduate student is also given adequate training in numerous tasks related to this experimental program.

- **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 AAA 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.

Role of Principal Investigators:

Drs. Roy and O'Toole have been training Martin Lewis and John Motaka in numerous aspects of experimental work related to this research project. Dr. O'Toole has been training them in mechanical testing using the computer-controlled MTS equipment and the recently procured high-temperature inert gas chamber and extensometer. Dr. Roy has been providing them with in-depth metallurgical understanding of metallography and surface analyses including interpretation of microstructures, failure characteristics, dislocation patterns and their interactions as functions of thermal treatments and testing temperatures. In essence, these diversified training are providing both students with the aptitude and competence in carrying out the specified experimental tasks, analyzing data and preparing high-quality technical reports. Significant efforts will be made during this year to present and publish the generated data, elucidating a plausible mechanistic understanding of high temperature deformation of Alloy EP-823.

Role of Graduate Student:

Martin Lewis has been given a significant amount of responsibility in this project. The faculty investigators are providing him with guidance, training and assistance, but he will be conducting all related experiments including tensile testing, metallographic and fractographic evaluations, and data analyses. All these tasks will be geared towards the preparation of his M.S. thesis. He is also expected to present his research findings in national scientific and technical society meetings/conferences with eventual goals of publications in reputed professional journals. He has already participated in a few technical workshops, and has given a technical presentation at the ANS students' conference in Berkeley, CA, April 2003, and the ANS annual conference in San Diego, CA, June 2003. He has worked on several complex undergraduate projects, and has taken several courses towards his graduate degree. He has taken a few advanced level graduate courses in experimental mechanics, corrosion engineering, mechanical metallurgy and manufacturing processes thus, rendering him suitable for this research project. John Motaka will join this task during the fall of 2003, and will perform the desired experimental work and data analyses, as needed.

Los Alamos National Laboratory

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Date:May 13, 2003

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Subject: The continuation of research activities relating to Proposal entitled: "Development of a Mechanistic Understanding of High-Temperature Deformation of Alloy EP-823 for Transmutation Applications"

Dear Dr. Roy:

I am impressed with your progress and enthusiastically support continuation of your research on the "Development of a Mechanistic Understanding of High Temperature Deformation of Alloy EP-823 for Transmutation Applications."

Sincerely,
Stuart A. Maloy
AFCI Materials Task Leader

Stuart A. Maloy