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Development of a Mechanistic Understanding of High-Temperature Deformation of Alloy EP-823 for Transmutation Applications

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

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AAA Research Area: **Target / Transmuter**

Funding Profile:

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

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

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 in the United States.

Work Proposed for Year 2 (Fall 2002 - Summer 2003), Goals, and Expected Results:
Three experimental heats (#2054, 2055 and 2056) of martensitic Alloy EP-823 have been melted and processed into round bars at the Timken Research Laboratory (TRL), Canton, Ohio. One heat (#2054) of this material has

been heat treated at TRL. The sectioned bars were austenitized at 1850^0F for one hour, followed by an oil-quench. These oil-quenched bars were subsequently tempered at 1150^0F for 1.25, 1.75 and 2.25 hours, respectively, followed by air-cooling. The measured hardness of these quenched and tempered (QT) materials ranged between R_c 29 and 30. Fabrication of round tensile specimens from these thermally treated alloys has just been completed. A high-temperature inert gas chamber with water-cooled jacket has been ordered for installation in the existing Materials Testing System (MTS) to perform tensile testing of Alloy EP-823 under controlled environmental conditions at ambient and elevated temperatures. Air-cooled extensometers will be used to monitor the displacement of these specimens while being pulled at the desired strain rate. The extensometer has just been received, and the test chamber is due in the research facility any time soon.

Construction of the "Materials Performance Laboratory (MPL)" in TBE #B129 is almost complete. This world-class research facility will have capabilities to perform numerous scientific and technical investigations in areas of materials and corrosion science and engineering. A state-of-the-art sample preparation unit has been procured to perform metallographic evaluations using mounted test specimens. A high-resolution (1000X) optical microscope has also been added to the MPL for evaluating the metallurgical microstructures and morphology of failures in tested specimens. The deformation characteristics of tensile specimens, upon failure, will be analyzed by surface analytical techniques, such as SEM and TEM, both in-house and off-site.

It is anticipated that approximately 75% of the tensile test matrix (using MTS) and 25% of the surface analyses (using optical microscopy, SEM and TEM) will be accomplished during the second year of this project. Periodic contacts will be made with the LANL collaborator. Brief quarterly reports will be prepared, and semi-annual reports will be submitted.

Background and Rationale:

During his two presentations at the AAA-UNLV Mini-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 AAA 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 (300-540 ^{0}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 540^{0}C. Tensile properties will be evaluated at different temperatures of interest by using the ASTM Standard E 8 in an inert 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 broken tensile specimens will be evaluated by using standard metallographic methods (polishing and etching). 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, 1.5 and 2 hour(s) followed by air-cooling. All test specimens will be fabricated from these quenched and tempered materials.

The tensile properties will be generated in an inert (nitrogen/argon) environment primarily in the temperature range of 300 to 540^{0}C (300, 400, 500 and 540^{0}C). A few tests may also be performed at ambient temperature (25^{0}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 UNLV AAA Infrastructure Committee has reviewed the need for some state-of-the-art equipment to accomplish the proposed metallurgical and corrosion research goals. The following equipment has recently been ordered using funds approved by this committee, some of which have already arrived at the campus and are currently being installed at MPL.

- Multi-channel potentiostat/Corrosion software for electrochemical studies (PerkinElmer Inc.)
- Proof ring assemblies for constant-load SCC testing (Cortest Inc.)
- Constant-extension-rate-testing (CERT) system for SSR testing (Cortest Inc.)
- Electrochemical cells/water bath/electrodes etc.
- High-resolution (1000X) inverted optical microscope for metallographic evaluation (Leica)
- Abrasive cutter for sample preparation (Buehler)
- Heat-treating furnace (1200^{0}C) with larger working dimensions (Lindberg)
- High-temperature inert gas chamber for mechanical testing (MTS)
- Other laboratory-related parts and equipment

<u>Heat Treatment Facilities</u>
Two high temperature furnaces are available:

1) Lindberg Furnace
The maximum temperature is 1200 ^{0}C (2200 ^{0}F). The working dimensions are 15" x 7.5" x 5.5".

2) Thermodyne Furnace
The maximum temperature is 1200 ^{0}C (2200 ^{0}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. 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.

Mechanical Testing

The UNLV College of Engineering has a 55 kip Axial/Torsional Servo hydraulic MTS Materials Testing System. The machine has 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.

Several elevated temperature vessels made of Alloy C-276 have recently been procured for corrosion related research projects sponsored by both the UNLV-AAA and the UNLV-Yucca Mountain Cooperative Agreement Research Programs. Additionally, a high temperature autoclave has been ordered for a recently funded AAA corrosion-related research project.

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.

A high-resolution (1000X) inverted optical microscope (Leica) has recently been added in MPL for metallographic evaluation including characterization of metallurgical microstructures and determination of grain sizes resulting from thermal treatments. Analyses of dissolved hydrogen content resulting from cathodic polarization can be performed at LANL by using secondary ion mass spectrometry (SIMS).

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 microscopy (TEM) is planned to be established as part of the UNLV AAA program. The UNLV AAA Infrastructure committee is currently evaluating the capabilities of three different TEM manufacturers, and a procurement order will be placed with the successful bidder. The anticipated date for the establishment of this facility is in the fall of 2002.

Project Timeline

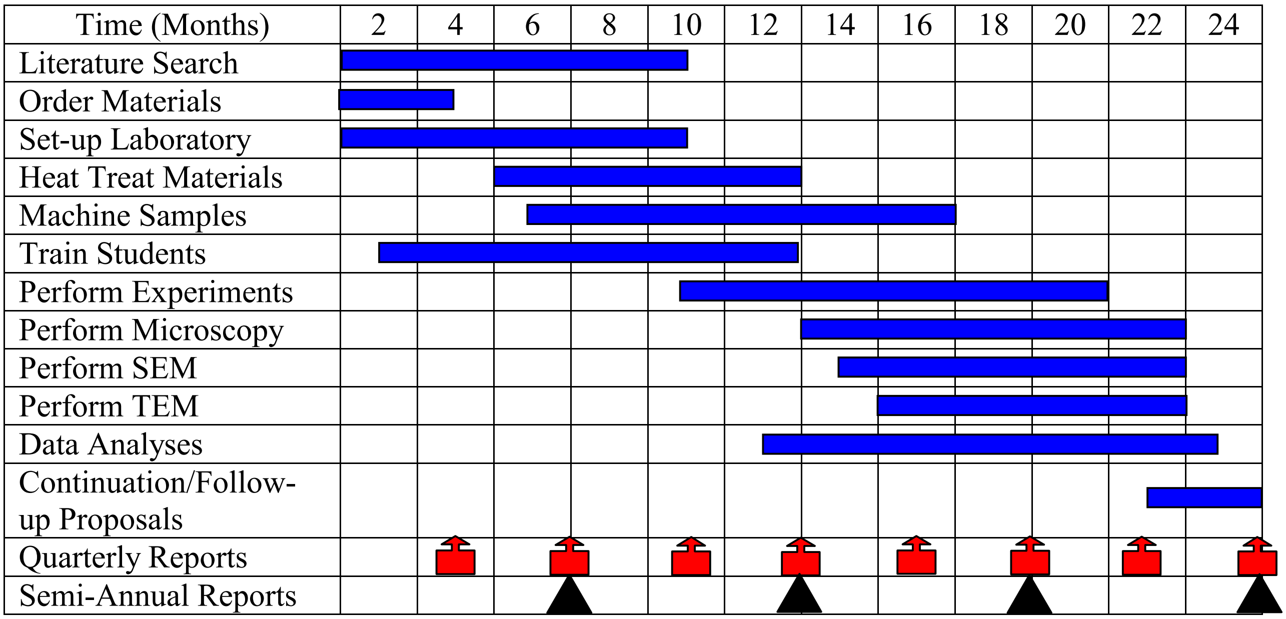
Timeline Narrative

This research project was initiated during the fall of 2001. Since the inception of this project, Mr. Martin Lewis has been working as a 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. In addition, Mr. Konstantin Zabotkin, a recent UNLV M.S. graduate in mechanical engineering was added to the overall AAA research projects to expedite the planned experimental program. Mr. Zabotkin also played a key role in developing the MPL in UNLV's engineering building. Recently, a group of UNLV researchers participating in AAA-funded projects visited LANL to become familiar with related research projects pursued by our collaborators at this national laboratory. This trip proved to be very useful in enhancing coordination and cooperation among researchers from both UNLV and LANL.

Machining of EP-823 tensile specimens has just been completed. Current efforts are being focused in generating ambient temperature tensile data in air using our existing MTS equipment and recently procured extensometer. Subsequently, specimens will be tested both at ambient and elevated temperatures in the presence of an inert environment, once this high-temperature inert gas chamber becomes available in the very near future. Upon review of the tensile data to be generated soon, additional heat treatment of the processed bars of EP-823 will be done at MPL to study the effect of tempering temperatures and times on the deformation characteristics. Upon completion of tensile testing, all samples will be analyzed using optical microscopy, SEM, and 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 towards the end of the second year of this project. It is also anticipated that a follow-up proposal investigating the fracture toughness of this material will be submitted towards the end of the second year. A two-year schedule is shown in Table 1.

Table 1: Two-Year Research Plan



Year 2 Milestones (Assuming a start date of September 1, 2002)

- (September 2002): Substantial ambient temperature mechanical tests completed, and elevated temperature tests initiated.
- (October 2002): Tensile data analyses initiated
- (November 2002): Microstructural analyses of tested specimens by optical microscopy initiated.
- (December 2002): Fractographic evaluations by SEM, and elemental analyses by EDS initiated.
- (February 2003): Deformation characteristics study by TEM initiated.
- (February 2003): Semi-annual progress report submitted.
- (May 2003): Preparation of the preliminary draft of the year 2 annual report encompassing the overall generated data initiated.
- (June 2003): Continuation of this project proposal, and a follow-up proposal to evaluate the fracture toughness of target materials submitted.
- (August 2003): Year 2 annual report submitted.

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 in an ANS conference in Reno, NV. 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. A second graduate student may be added next year depending on the progress of this project, if needed.

Role of Research Associate

Mr. Konstantin Zabotkin has been added to assist in the overall AAA-related research tasks currently being performed in the MPL and other laboratories in the UNLV engineering building. He has provided substantial technical assistance during the establishment of the MPL, and is responsible for the day-to-day operation of these research facilities. He has been keeping track of all existing and incoming equipment, calibrating them, performing dry runs and analyzing data. As a research associate, he ensures that all related testing equipment are working properly, and all necessary laboratory supplies are maintained. Further, he provides technical assistance to all graduate students in respective project-related tasks. The duration of his appointment as an AAA research associate will be six months during this year.

702-895-137

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Date:June 11, 2002

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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 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
AAA Materials Team Leader

Stuart A Maloy 6/11/02