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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**

**August 11, 2001**


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| **Investigators (UNLV):** | Martin Lewis, Graduate Student<br>Department of Mechanical Engineering, UNLV |
| **Collaborator (DOE):** | Dr. Stuart A. Maloy<br>Transmuter – Materials Contact Person<br>APT, MS-H809<br>LANL, Los Alamos, NM 87545<br>Phone: (505) 667-9784 Email: Maloy@lanl.gov |


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| **AAA Research Area:** | Transmuter / Target |
| **First Year Request:** | $98,735 (out years comparable) |

**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 1 (Fall 2001 – Summer 2002), Goals, and Expected Results**

Alloy EP-823 material will be ordered, heat-treated and machined into test samples. The graduate student will be trained on the mechanical testing equipment and surface analysis techniques. Approximately 50% of the experiments will be completed and about 25% of the surface analysis will be completed. Monthly contact will be maintained with the LANL collaborator. Brief quarterly reports will be prepared and detailed semi-annual reports will be submitted.

**Background and Rationale**

During his two presentations at the UNLV Advanced Accelerator Applications (AAA) Mini-Workshop held at the Harry Reid Center (HRC) on January 18, 2001, Dr. Stuart Maloy of 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 Ning Li to follow-up on related research topics. 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 UNLV Mechanical Engineering Department.

Target materials used in AAA systems will be subject to extreme temperature and corrosive environments. Issues related to environment-induced degradation of these materials are being investigated under a UNLV AAA research project that began summer term 2001. 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 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 AAA.

**Material and Experimental Procedure**

Martensitic EP-823 stainless steel will be tested to evaluate its tensile properties at temperatures ranging from ambient to 540 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 will be used with the MTS mechanical testing machine at 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.

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 and extent of failure (ductile versus brittle) 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 establishment of deformation mechanisms for the tested material.

## 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 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 1900 to 2000 $^{o}$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 1200 $^{o}$F for 1, 1.5, and 2 hours followed by air-cooling. All test specimens will be fabricated from these quenched and tempered materials.

Tensile properties will be evaluated in an inert (nitrogen/argon) environment primarily in the temperature range of 300 to 540 C (300, 400, 500 and 540 C). A few tests may also be performed at ambient temperature (25 C) to develop baseline data. At last three specimens will be tested under three (3) metallurgical conditions at these temperatures.

## Expected Data

The proposed research program will generate the following data:

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

## Research Capabilities at UNLV

<u>Heat Treatment Facilities</u>

Two high temperature furnaces are currently available:

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

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

A third furnace with larger working dimensions will be available within the next four months.

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 chambers are currently on order for a corrosion related research project sponsored by the UNLV Yucca Mountain Cooperative Agreement Program. Additionally, an order for a high temperature autoclave is being prepared 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. Equipment funding for improvements for a dedicated AAA materials preparation facility has been approved in the previous round of proposals.

Scanning Electron Microscopy

The UNLV Geosciences Department has a JEOL-5600 Scanning Electron Microscope http://www. unlv.edu/Colleges/Sciences/Geoscience/EMIL.htm. 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 is currently being developed.

### Transmission Electron Microscopy

A transmission electron microscopy facility is planned to be established as part of the UNLV AAA program. The UNLV AAA Infrastructure Committee is evaluating this possibility and a decision will be made in September or October of 2001. The anticipated date for the establishment of this facility is in the spring of 2002. TEM work will be arranged with Los Alamos National Laboratory if it is needed before this facility is established.

## Project Timeline

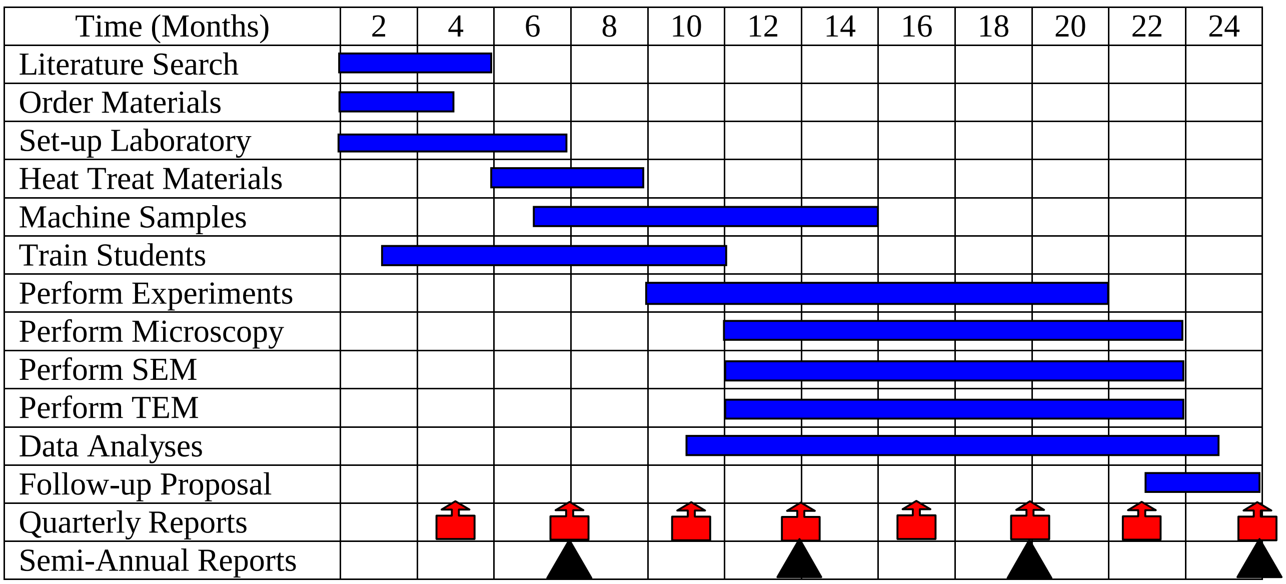
### Timeline Narrative

The proposed research is planned over two years, starting in fall 2001. Initial tasks will be ordering bar stock materials. The UNLV research team (faculty and students) will visit LANL during the first quarter to meet with our collaborators, refine our testing plan, and learn about related projects at LANL. An initial goal of this project will be to define a set of realistic objectives for the Master's Thesis of Martin Lewis.

The bar stock will be heat treated at UNLV and machined into test samples at UNLV or at a local machine shop. Mechanical testing will begin when the environmental chamber has been installed and the samples have been machined. All of the tested samples will be evaluated with photomicroscopy, SEM, and TEM. The effect of temperature on mechanical properties will be determined and correlated with changes in microstructure for the different heat treatments.

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 degree 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 during these first two years. A two-year schedule is shown in Table 2.

Table 2. Two-Year Research Plan




<u>Year 1 Milestones (Assuming a start date of September 1, 2001)</u>

- December 2001: All material and equipment has been obtained.
- February 2002: Heat Treatments have been completed and sample machining has started.
- March 2002: Mechanical testing has started.
- June 2002: Microscopy, SEM and TEM have started, data analysis has started
- August 2002: Year 1 report is submitted.

<u>Deliverables</u>

- **Trained Graduate Student:** The primary deliverable will be a graduate student trained in a field relevant to the national AAA 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 AAA 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 AAA 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**

Dr. Roy and Dr. O'Toole will train Martin Lewis and the undergraduate student (yet to be hired) in sample preparation and mechanical testing. They will also provide guidance to these students in carrying out the research project, documenting the progress, and analyzing the test results.

**Role of Graduate Student**

Martin Lewis will be given a significant amount of the responsibility for this project. The faculty investigators will provide guidance, training, and assistance but he will have to prepare samples, test the samples, perform the microstructural analyses, analyze the data, and write a M.S. thesis. He should attend a conference or workshop during the first year and plan to present his work at a conference during the second year. Martin is well suited for this project. He has worked on several complex undergraduate projects and has already taken several courses towards his graduate degree. He has taken an advanced experimental mechanics course and manufacturing courses so he will be a productive part of the team from the beginning.

# Los Alamos National Laboratory

***Advanced Accelerator Applications***
***Technology Project Office***
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Date:July 16, 2001

Brendan J. O'Toole
Associate Professor of Mechanical Engineering
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**Subject: Support for Proposal entitled: "Development of a Mechanistic Understanding of High Temperature Deformation of Alloy EP-823 for Transmutation Applications"**

Dear Dr. O'Toole

I enthusiastically support your proposal to understand the high temperature deformation mechanisms of Alloy EP823. I believe that this proposal will lead to important mechanical test results to understand the deformation mechanisms in this class of materials. Such results will be extremely important for the design of the targets for the Accelerator Driven Transmutation Facility as such research is not being performed at any other facilities that I am aware of.

Sincerely,
Stuart A. Maloy
Materials Team Leader