

# **Distribution Limited to Sponsor**

# MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE POWER SYSTEMS

**Final Report** 

DE-FG07-05ID14642

May 12, 2005 to September 30, 2008

## **Submitted to:**

Mr. Dirk Cairns-Gallimore

U.S. Department of Energy NE-34 19901 Germantown Road Germantown, MD 20874-1290

## **Submitted by:**

Daniel P. Kramer and Chadwick D. Barklay

University of Dayton Research Institute 300 College Park Dayton OH 45469

Date of Report: October 7, 2008

# **Table of Contents**

Table of Contents	3
Summary	. 4
Selected Technical Highlights	4
Appendix A- Publications, Thesis, and Presentations	7
Appendix B- Quarterly progress Reports	. 8

## **Summary**

The University of Dayton Research Institute (UDRI), a division of the University of Dayton, with technical guidance from DOE/NE-34 personnel investigated materials related technical topics associated with supporting the efforts of DOE's Office of Nuclear Energy's Radioisotope Power Systems Program. Over the length of this sponsored program, UDRI performed a number of materials related tasks that helped to facilitate increased understanding of the properties and applications of a number of candidate program related materials. Some technical highlights of the project are listed below. Appendix A is a compilation of the technical papers/presentations that resulted from this work. The breath of the research and development performed during this project is described in detail in Appendix B which includes the Technical Quarterlies submitted during the course of this project.

In addition to the technical work discussed in the Quarterlies in Appendix B, this project also partially supported the Ph.D. research for a graduate student at the University of Dayton. Dr. Chadwick Barklay completed his Ph.D. thesis entitled, "Investigation of Low Level Neutron Radiation on Tantalum Alloys for Radioisotope Power System Applications," University of Dayton, Dayton OH, May 2007, 191 pages. This thesis helps to elucidate the effects of neutron irradiation at heat source levels on various refractory tantalum alloys.

## **Selected Technical Highlights**

- ➤ Technical support was provided to support Idaho National Laboratory in their evaluation of a potential fiber replacement for use in Fine Weave Pierced Fabric (FWPF) billets.
- A continuing relationship was developed with The Ohio State Nuclear Test Laboratory that is located in Columbus, OH
- ➤ The University of Dayton's radioisotope license was amended that permits the storage, handling, and testing of radioactive materials of interest on campus. This provides UDRI a relatively unique capability that allows the mechanical testing of irradiated metals.
- ➤ Identified potential protective coatings that could decrease the oxidation/ablation rate of FWPF graphitic material. Thin coatings of an alumina based high temperature refractory ceramic were applied to various graphitic specimens and these were subjected to the thermal pulse from the output of small rocket motor engines in a static rocket motor test stand.
- A software package has been identified that is be able to calculate based on thermodynamic constants, material phases in a weld or weld pool as it cools from a molten state down towards room temperature. The software is capable of

"predicting" the formation and/or resolution etc. of materials phases and polymorphs as a function of temperature, glove box atmosphere, chemical impurities, etc. Initial work employing the software has been on the T-111 and Ta-10W systems with an emphasis on determining the thermodynamically favorable nitrides, oxides, and carbides. The temperature region of interest is from  $\sim 4000\,^{\circ}$ C which is significantly above the expected weld pool temperature down through solidification of the HAZ to  $\sim 1000\,^{\circ}$ C.

- A total of twenty-four (24) Ta-10%W and twenty-four (24) T-111 metallurgical and test specimens were irradiated in two separate reactor runs at the Ohio State University Research Reactor (OSURR). The reactor runs were conducted at full power, approximately 0.5 MW, for a total of four (4) hours, which yielded a total flux of ~1.2E15 nvt.
- ➤ Various standard analytical techniques were applied to the steady state creep data of both irradiated and non-irradiated T-111 and Ta-10%W specimens obtained in this research. This allowed for better insight into the creep mechanisms, and the development of a predictive model for determining the secondary creep rates for these alloys that incorporates the effects of neutron displacement damage (dpa).
- ➤ Irradiated and non-irradiated rupture tested samples of Ta-10%W and T-111 specimens were analyzed using Transmission Electron Microscopy (TEM) at Oak Ridge National Laboratory's (ORNL) High Temperature Materials Laboratory (HTML) Materials Analysis User Center (MAUC). Preliminary conclusions are that the irradiation induced defect structures in these alloys are responsible for a pronounced microstructural evolution by further impeding dislocation motion thus increasing rupture times and negatively affecting strain to failure.
- The fracture surfaces of neutron irradiated and non-irradiated rupture tested specimens of Ta-10%W and T-111 were analyzed using Scanning Electron Microscopy (SEM) at UDRI. In general, the SEM images showed that the fracture surfaces of the stress rupture tested specimens of irradiated and non-irradiated Ta-10%W and T-111 exhibit a ductile failure mode based on the dimples observed throughout the fracture surfaces. All of the fracture surfaces are considerably rough and exhibit elongated dimples of various sizes with relatively flat regions in between. Such features are indicative of ductile failure mechanisms associated with uniaxial tests.
- A graduate student in materials science at the University of Dayton completed a Ph.D. based on a research project that increased our understanding of the effects of heat source level neutron irradiation on T-111 and Ta-10W.
- ➤ Elemental surface analysis and surface imagery of irradiated and mechanically tested Ta-10%W and T-111 samples was submitted to and accepted by the Oak Ridge National Laboratory (ORNL) Materials Analysis User Center (MAUC), which supports the High Temperature Materials Laboratory (HTML).

- At NE-34's request technical information was exchanged with representatives of the Heat Source program at INL focused on the potential surface contamination of parts that were machined with Moly-Dee cutting fluid. UDRI obtained information on the potential contamination and shared observations during several telecons. UDRI prepared a "Quick Reaction Force" memo that was submitted to NE-34.
- ➤ A possible replacement material for the FWPF presently used in the fabrication of GPHS aeroshells has been identified which appears to be intriguing from a functional point of view. POCOGRAPHITE, Inc. (Decatur, TX) has developed a family of silicon carbide products that are initially machined from a very pure form of graphite and which can be subsequently converted into silicon carbide. In general, silicon carbide is a ceramic material that has a high melting temperature of ~2540°C (~4600°F) and which is relatively chemically inert making it attractive as a possible FWPF replacement material.

The authors gratefully acknowledge the technical and financial support made possible by the DOE/NE-34 sponsor (Mr. Dirk Cairns-Gallimore) and look forward to assisting DOE's Office of Nuclear Energy's Radioisotope Power Systems Program in the future.

Dr. Daniel Kramer Division Head/Metals & Ceramics UDRI Dayton, OH Dr. Chadwick Barklay Group Leader/Adv. Ceramics & Glasses UDRI Dayton, OH

## **Appendix A- Publications, Thesis, and Presentations**

- 1. C. D. Barklay (UDRI), D.P. Kramer (UDRI), and R.G. Miller (ORNL), "The Effect of the Presence of 2 wt% Hafnium in T-111," presented at the Thirty-First Annual Dayton-Cincinnati Aerospace Science Symposium, Dayton, OH, March 2006.
- 2. C.D. Barklay, D.P. Kramer, and J. Talnagi (The Ohio State University), "Investigation of Effects of Neutron Radiation on Tantalum Alloys for Radioisotope Power System Applications," poster at the Materials Science & Technology 2006 Conference and Exhibition, Cincinnati, OH, October 2006.
- 3. W.E. Moddeman (Pantex), C.D. Barklay, J.C. Birkbeck (Pantex), R.G. Miller (ORNL), L.F. Allard (ORNL), and D.P. Kramer, "Thermodynamic Prediction and Transmission Electron Microscopy Results of Compositional Phases in Tantalum-based Alloys Welds for Power Source Application," poster at the Futures Technologies Conference II sponsored by the National Nuclear Security Agency (NNSA), Washington, D.C., October 2006.
- 4. C.D. Barklay, D.P. Kramer, and J. Talnagi, "Investigation of Effects of Neutron Irradiation on Tantalum Alloys for Radioisotope Power System Applications," published in the 25<sup>th</sup> Proceeding of Space Technology and Applications International Forum (STAIF-2007), American Institute of Physics Conference Proceedings 880, Albuquerque, NM, February 2007, pp. 224-228.
- 5. W.E. Moddeman, C.D. Barklay, J.C. Birkbeck, R.G. Miller, L.F. Allard, and D.P. Kramer, "Thermodynamic Prediction of Compositional Phases by Transmission Electron Microscopy on Tantalum-Based Alloy Weldments," published in the 25<sup>th</sup> Proceedings of Space Technology and Applications International Forum (STAIF-2007), American Institute of Physics Conference Proceedings 880, Albuquerque, NM, February 2007, pp. 229-233.
- 6. C. D. Barklay, "Investigation of Low Level Neutron Radiation on Tantalum Alloys for Radioisotope Power System Applications," Ph.D. Thesis, University of Dayton, Dayton OH, May 2007, 191 pages.
- 7. D.P. Kramer, C.D. Barklay, and J. Talnagi, "Neutron Irradiation of Refractory Tantalum Alloys (T-111 and Ta/10W) for Radioisotope Space Power System Applications," published in the Proceedings of the 2007 National Space & Missile Materials Symposium, Keystone, Colorado, June, 2007.
- 8. C.D. Barklay, J.Y. Howe, and D.P. Kramer, "Investigation of Stress Rupture Tested Neutron Irradiated Tantalum Alloys," published in the 26<sup>th</sup> Proceeding of Space Technology and Applications International Forum (STAIF-2008), American Institute of Physics Conference Proceedings 969, Albuquerque, NM, February 2008, pp. 439-445.

6

## **Appendix B- Quarterly progress Reports**

## **QUARTERLY PROGRESS REPORT**

**Project Title:** MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

**Covering Period:** May 12, 2005 through June 30, 2005

**Date of Report:** July 18, 2005

**Recipient:** University of Dayton Research Institute

300 College Park Dayton, OH 45469

**Award Number:** DE-FG07-05ID14642

**Subcontractors:** none

**Other Partners:** none

Contact(s): Dr. Daniel P. Kramer/daniel.kramer@udri.udayton.edu

937-229-1038

**DOE Project** NE-50 contacts – Dirk Cairns-Gallimore/Tim Frazier

Contacts: ID contract specialist – Suzette Olson

**Project Objective:** This project addresses several of DOE-NE-50's near-term and future

materials science and engineering related programmatic needs/goals.

#### **Background:**

The Office of Space and Defense Power Systems (NE-50) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions including the three large RTG's (Radioisotope Thermoelectric Generators) that are presently powering the Cassini spacecraft at Saturn. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific knowhow, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-50 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

#### **Status:**

During the reporting quarter technical work was initiated in a number of the assigned technical areas including:

- 1) Potential protective coatings are being identified that would decrease the oxidation/ablation rate of FWPF graphitic material. This would reduce the potential need and substantial cost of identifying and qualifying a completely new material. Discussions held with a number of suppliers around the country have identified two different techniques that could be employed. The first is a CVD based process that may be able to convert the top ~0.050" of the surfaces of a FWPF module into SiC. SiC is a high temperature ceramic material that exhibits very good high temperature properties including excellent oxidation resistance especially compared to unprotected graphite. SiC is also attractive since carbon (in the FWPF) and silicon (in the SiGe unicouples) have previously been utilized in space RTG's. One contacted company is currently converting very complex machined graphite shapes (with up to ~0.25" cross-sections) completely into SiC with minimal dimensional changes. Discussions have been held with the company centered on the possibility of converting the near surface of graphitic test specimens into SiC. If this is possible, specimens will be obtained and subjected to high temperature The second technique being investigated is the oxidation and/or ablative environments. application of different types of coatings. Various manufacturers produce ceramic paintable/sprayable materials that are SiC based that could be used to protect the graphitic modulus. We are also looking at the possibility of employing polymeric materials that can be converted into SiC via a thermal cycle.
- 2) A surplus glove box has been identified and it will be cleaned and set up to provide an inert atmosphere environment for relatively long term compatibility tests at elevated temperatures on various material couples such as T-111, Ta-10W, Haynes 188, Haynes 230, Hastelloys, etc.
- 3) A Ph.D. student in materials science has been identified and is preparing a research project possibly based on furthering our understanding of the material property differences between T-111 and Ta-10W.
- 4) At the request of Robert Wiley (DOE/NE-50) technical support was provided to Teledyne Energy Systems (TES). A telecon was held with various TES personnel and follow-up contact included providing TES technical information on techniques for measuring the emissivity of various materials at elevated temperatures. References to several of our written publications on this subject were also provided to TES.
- 5) We have identified an analytical technique that may be able to distinguish between Hf, Ta, and W in <T-111 insert/Ta-10W> welds that are being used in a program. Hf, Ta, and W are elements 72, 73, and 74 in the Periodic Table making their resolution in standard EDS systems nearly impossible. A new WDS analysis system may be able to resolve their various respective elemental spectrums. This would allow us to determine and understand the role of Hf in these welds and materials.

**Program Recommendations:** To become more familiar with the various program's materials related issues, it is suggested that our attendance at appropriate program meetings be encouraged. In addition, reinstating clearances could also greatly facilitate future program related discussions.

**Plans for Next Quarter:** During the next quarter R&D materials efforts will continue on determining techniques for protecting GPHS materials from oxidation, the initiation of compatibility tests, and on understanding the properties of Ta-10W and T-111.

Patents: none

Publications/Presentations: none

#### **Milestone Status Table:**

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
		-		
1	FWPF protection/replacement			
2	Role of Hf on the property differences between Ta-10W and T-111			
3	Potential materials replacements			
4	Compatibility of typical and future radioisotope materials			
5	Support Stirling based power systems development			
6	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05	
7	2 <sup>nd</sup> Quarterly Report	10/30/05		
8	3 <sup>rd</sup> Quarterly Report	01/30/06		
9	Final Report	08/11/06		
10	SF-269A	08/11/06		

# **Budget Data** (as of June 30, 2005):

			Approved Spending Plan		Actual S <sub>1</sub>	pent to Date
Budget Period: May 12, 2005 to May 11, 2006		DOE Amount	Total	DOE Amount	Total	
	From	То				
	05/11/05	06/30/05			\$3907	\$3907
		Totals	\$100,000	\$100,000	\$3907	\$3907

## **QUARTERLY PROGRESS REPORT**

**Project Title:** MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

**Covering Period:** May 12, 2005 through June 30, 2005

**Date of Report:** July 18, 2005

**Recipient:** University of Dayton Research Institute

300 College Park Dayton, OH 45469

**Award Number:** DE-FG07-05ID14642

**Subcontractors:** none

**Other Partners:** none

**Contact(s):** Dr. Daniel P. Kramer

937-229-1038

daniel.kramer@udri.udayton.edu

**DOE Project** NE-50 contacts – Dirk Cairns-Gallimore/Tim Frazier

Contacts: ID contract specialist – Suzette Olson

**Project Objective:** This project addresses several of DOE-NE-50's near-term and future

materials science and engineering related programmatic needs/goals.

## **Background:**

The Office of Space and Defense Power Systems (NE-50) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions including the three large RTG's (Radioisotope Thermoelectric Generators) that are presently powering the Cassini spacecraft at Saturn. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific knowhow, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-50 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

#### **Status:**

During the reporting quarter technical work was initiated in a number of the assigned technical areas including:

- 1) Potential protective coatings are being identified that would decrease the oxidation/ablation rate of FWPF graphitic material. This would reduce the potential need and substantial cost of identifying and qualifying a completely new material. Discussions held with a number of suppliers around the country have identified two different techniques that could be employed. The first is a CVD based process that may be able to convert the top ~0.050" of the surfaces of a FWPF module into SiC. SiC is a high temperature ceramic material that exhibits very good high temperature properties including excellent oxidation resistance especially compared to unprotected graphite. SiC is also attractive since carbon (in the FWPF) and silicon (in the SiGe unicouples) have previously been utilized in space RTG's. One contacted company is currently converting very complex machined graphite shapes (with up to ~0.25" cross-sections) completely into SiC with minimal dimensional changes. Discussions have been held with the company centered on the possibility of converting the near surface of graphitic test specimens into SiC. If this is possible, specimens will be obtained and subjected to high temperature oxidation and/or ablative environments. The second technique being investigated is the application of different types of coatings. Various manufacturers produce ceramic paintable/sprayable materials that are SiC based that could be used to protect the graphitic modulus. We are also looking at the possibility of employing polymeric materials that can be converted into SiC via a thermal cycle.
- 2) A surplus glove box has been identified and it will be cleaned and set up to provide an inert atmosphere environment for relatively long term compatibility tests at elevated temperatures on various material couples such as T-111, Ta-10W, Haynes 188, Haynes 230, Hastelloys, etc.
- 3) A Ph.D. student in materials science has been identified and is preparing a research project possibly based on furthering our understanding of the material property differences between T-111 and Ta-10W.
- 4) At the request of Robert Wiley (DOE/NE-50) technical support was provided to Teledyne Energy Systems (TES). A telecon was held with various TES personnel and follow-up contact included providing TES technical information on techniques for measuring the emissivity of various materials at elevated temperatures. References to several of our written publications on this subject were also provided to TES.
- 5) We have identified an analytical technique that may be able to distinguish between Hf, Ta, and W in <T-111 insert/Ta-10W> welds that are being used in a program. Hf, Ta, and W are elements 72, 73, and 74 in the Periodic Table making their resolution in standard EDS systems nearly impossible. A new WDS analysis system may be able to resolve their various respective elemental spectrums. This would allow us to determine and understand the role of Hf in these welds and materials.

**Program Recommendations:** To become more familiar with the various program's materials related issues, it is suggested that our attendance at appropriate program meetings be encouraged. In addition, reinstating clearances could also greatly facilitate future program related discussions.

**Plans for Next Quarter:** During the next quarter R&D materials efforts will continue on determining techniques for protecting GPHS materials from oxidation, the initiation of compatibility tests, and on understanding the properties of Ta-10W and T-111.

Patents: none

Publications/Presentations: none

## **Milestone Status Table:**

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
Tuilloci		Completion	Completion	
1	FWPF protection/replacement			
2	Role of Hf on the property differences between Ta-10W and T-111			
3	Potential materials replacements			
4	Compatibility of typical and future radioisotope materials			
5	Support Stirling based power systems development			
6	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05	
7	2 <sup>nd</sup> Quarterly Report	10/30/05		
8	3 <sup>rd</sup> Quarterly Report	01/30/06		
9	Final Report	08/11/06		
10	SF-269A	08/11/06		

13

# **Budget Data** (as of June 30, 2005):

			Approved Spending Plan		Actual Sp	ent to Date
Budg	get Period:		DOE	Total	DOE	Total
May	12, 2005 to	May 11, 2006	Amount		Amount	
	From	То				
	05/11/05	06/30/05			\$3907	\$3907
		Totals	\$100,000	\$100,000	\$3907	\$3907

#### QUARTERLY PROGRESS REPORT

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

Covering Period: July 1, 2005 through September 30, 2005

Date of Report: October 18, 2005

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-50 contacts – Dirk Cairns-Gallimore/Tim Frazier

Contacts: ID contract specialist – Suzette Olson

Project Objective: This project addresses several of DOE-NE-50's near-term and future

materials science and engineering related programmatic needs/goals.

## Background:

The Office of Space and Defense Power Systems (NE-50) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions including the three large RTG's (Radioisotope Thermoelectric Generators) that are presently powering the Cassini spacecraft at Saturn. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific knowhow, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-50 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

#### Status:

During this reporting quarter investigations continued in the assigned technical areas and below are several focal points:

- a) Research on potential protective coatings that could decrease the oxidation/ablation rate of FWPF graphitic material continued during this quarter. In order to test potential coatings, a static rocket motor test stand was developed that allows the contained firing of small rocket motor engines. The test stand was fabricated using ceramic refractory materials and is designed to withstand the high temperatures of the rocket motors. Two types of small rocket motor engines are commercially available; 1) relatively low cost black powder based motors and 2) more expensive solid propellant based motors. The solid propellant used in these small rocket motors is very similar to the solid propellant that is used in the Space Shuttle's SRBs (Solid Rocket Boosters). The tentative plan is to initially employ the low cost black powder rocket motors in the screening experiments. When promising coatings are identified the more expensive solid propellant motors could be used. Initial experiments in the static test stand have been performed on coated graphite substrates using black power motors.
- b) A small box furnace has been placed within a glove box for long term compatibility tests on multiple material couples such as T-111, Ta-10W, Haynes 188, Haynes 230, Hastelloys, etc. The leak tightness of the surplus glove box is being enhanced to allow the long term tests to occur in an inert atmosphere environment.
- c) A relationship has been developed with The Ohio State Nuclear Test Laboratory that is located in Columbus, OH. Located within the laboratory is the Ohio State University Research Reactor (OSURR). The OSURR is a low-enriched uranium (LEU) fuel (19.5% enriched U<sub>3</sub>Si<sub>2</sub>) reactor, that has a licensed operating power of 500kW and a total neutron flux of approximately 1.5x 10<sup>13</sup> neutrons/(cm<sup>2</sup>-sec). The neutron flux from this reactor corresponds reasonably well with the magnitude of neutrons that are produced by GPHS units. This test reactor will be used by Chadwick Barklay (Ph.D. graduate student on this project) in his research regarding the investigation of possible changes in the mechanical properties of heat source related alloys as a function of irradiation. An initial reactor run on seventeen (17) test samples of various metal alloys of interest was completed. During the 1000 second irradiation activation test, metal samples were placed within cadmium capsules to reduce their exposure to thermal neutrons.

The irradiated test samples were analyzed using OSNTL's PC-based high-resolution gamma-ray spectroscopy system to determine their isotope and activity levels. The results of this experiment were decayed and extrapolated to values commensurate with material quantities and longer irradiation times. The results of this work will support future research efforts on this project. The extrapolated isotopic values were also provided to the University of Dayton's Office of Environmental Safety as input to the university's submission for their 2006 Radioactive Materials license from the State of Ohio. This will enable the transport and storage of specific radioactive materials for the conduct of future research efforts.

Both Dr. Dan Kramer and Chadwick Barklay have become authorized by the University of Dayton's Radiation Safety Committee as "Authorized Users" of radioactive material. Additionally, the protocol for the aforementioned research has been written and submitted for approval to the committee. The protocol outlines the use, storage, security, risks, hazards, exposure, and monitoring techniques associated with the handling of irradiated test specimens.

Various metal alloy sheet materials were requested from DOE/NE-50 in support of this research effort. UDRI has received informal confirmation that Idaho National Laboratory has been directed to provide the requested materials, but to date no material has been received.

- d) At the request of Robert Radzinski/Robert Wiley (DOE/NE-50) technical support was provided to support Idaho National Laboratory in their evaluation of a potential fiber replacement for use in Fine Weave Pierced Fabric (FWPF) billets. Discussions were held with Textron and other companies to determine the extent of the available background technical data on T-300 fiber. Several telecoms were held with NE-50 / INL during which we presented our opinion that the needed technical data on T-300 was not available, making it difficult to recommend it as a replacement fiber in FWPF at this time.
- e) Specimens of T-111 alloy and a {Ta-10W / T-111 insert / Ta-10W} weld were mounted in conductive mounts and polished. The specimens were sent to an outside laboratory that has developed a new WDS analytical instrument that may be able to differentiate the elemental lines of Hf, Ta, and W. If successful, this would allow us for the first time to possibly elucidate the role of Hf in these welds and materials.
- f) Discussions (e-mails) have been held with INL personnel on shipping the pressure burst furnace to UDRI. UDRI has requested more thorough details of the electrical power requirements needed to run the furnace and associated equipment; especially the furnace's <a href="mailto:amps">amps</a> and voltage requirements, and its exact dimensions. This information is required so that appropriate space within UDRI for the furnace can be identified.

<u>Program Recommendations:</u> To become more familiar with the various program's materials related issues, it is suggested that our attendance at appropriate program meetings be encouraged. In addition, reinstating clearances could also greatly facilitate future program related discussions.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including; determining if the state-of-the-art WDS equipment can identify the individual elements in Ta-10W and Ta-10W/T-111 insert weld specimens; the coating and "rocket" testing of carbon substrates; and preparing the experimental plan and experimental setup for the irradiation runs.

Patents: none

Publications/Presentations: none

# Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
		Composition	Стопросто	
1	FWPF protection/replacement			
2	Role of Hf on the property differences between Ta-10W and T-111			
3	Potential materials replacements			
4	Compatibility of typical and future radioisotope materials			
5	Support Stirling based power systems development			
6	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05	
7	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05	
8	3 <sup>rd</sup> Quarterly Report	01/30/06		
9	Final Report	08/11/06		
10	SF-269A	08/11/06		

# Budget Data (as of September 30, 2005):

			Approved Spending Plan		Actual Spent to Date	
Budget Period: May 12, 2005 to May 11, 2006		DOE Amount	Total	DOE Amount	Total	
	From	То				
	05/11/05	06/30/05			\$3,907	\$3,907
	05/11/05	09/30/05			\$25,958	\$25,958
		Totals	\$100,000	\$100,000	\$29,865	\$29,865

## **QUARTERLY PROGRESS REPORT**

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

Covering Period: October 1, 2005 through December 31, 2005

Date of Report: January 18, 2006

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-50 contacts – Dirk Cairns-Gallimore/Tim Frazier

Contacts: ID contract specialist – Suzette Olson

Project Objective: This project addresses several of DOE-NE-50's near-term and future

materials science and engineering related programmatic needs/goals.

## Background:

The Office of Space and Defense Power Systems (NE-50) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions including the three large RTG's (Radioisotope Thermoelectric Generators) that are presently powering the Cassini spacecraft at Saturn. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific knowhow, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-50 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

#### Status:

During this reporting quarter investigations continued in the assigned technical areas and below are several focal points:

a) Research on potential protective coatings that could decrease the oxidation/ablation rate of FWPF graphitic material continued during this quarter. High temperature refractory ceramic coatings were prepared which were loaded with SiC, Si<sub>3</sub>N<sub>4</sub>, Y<sub>2</sub>O<sub>3</sub>, or ZrO<sub>2</sub>. These ceramic materials exhibit very high melting temperatures. Samples of graphite were coated and subjected to the output of small rocket motor engines in a static rocket motor test stand that has been developed. Relatively low cost black powder rocket motors were used in these experiments and the rocket motor nozzles were positioned ~2.54cm from the surface of the test specimens. Figure 1 shows the results obtained on a coated graphite test specimen after being subjected to the output from one of the small black powder rocket motors. In general, tests on the various ceramic coated specimens were similar in that the output of the rocket tended to "erode" through the protective coatings. Figure 1 shows an "eroded hole" in the center of the picture and the radiating dark lines from the "eroded hole" is condensed reaction products from the rocket motor. Breakdown of the various coatings did not appear to be a function of the rocket motors' thermal spike or oxidation but due to the ablative characteristics of the reaction products.

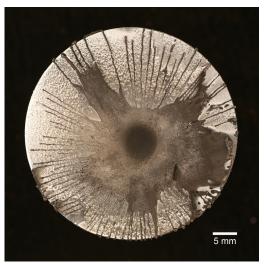


Figure 1. Surface of a coated graphite specimen after being subjected to the output of a small black powder rocket motor whose output nozzle was ~2.54 cm from the test specimen.

b) Technical discussions were held with Dr. C. Chen who has experience in determining the radiation tolerance and thermal stability of rare earth magnets. Some of her research was initiated by NASA as part of their research and development endeavors in support of Stirling generator development. Stirling generators utilized magnets that must be radiation tolerant over the duration of the assigned mission. During Dr. Chen's research both Samarium-Cobalt and Neodymium-Iron-Boron based magnet specimens were irradiated in a reactor to fluences of ~10<sup>18+</sup>n/cm<sup>2</sup>. In general, Sm-Co magnets are typically more radiation tolerant compared to Nd-Fe-B compositions. Boron containing magnets may undergo Boron Neutron Capture Transmutation (BNCT) as B<sup>10</sup> may transmute into Li<sup>7</sup>. It was also discussed, that after

irradiation some of the test magnets were determined to be fully demagnetized. The reported cause was that this was due to the samples being subjected to a "thermal spike" during the irradiation while in the reactor. However, from the discussions it is not clear if this hypothesis can be supported by the obtained data.

c) Various metal alloy sheet materials that were requested from DOE/NE-50 were received from Idaho National Laboratory. These materials are critical to future research efforts regarding the possible changes in the mechanical properties of heat source related alloys as a function of irradiation.

A purchase order was completed with The Ohio State Nuclear Test Laboratory, located in Columbus, Ohio, for use of the Ohio State University Research Reactor (OSURR). This test reactor will be used by Chadwick Barklay (Ph.D. graduate student on this project) in support of the aforementioned research. Additionally, hardware and expendable items were procured that will be used in conjunction with the OSURR reactor runs.

Dr. Dan Kramer and Chadwick Barklay met with Dr. Larry Allard of the Materials Analysis User Center (MAUC), which supports the High Temperature Materials Laboratory (HTML) at Oak Ridge National Laboratory (ORNL). The MAUC possesses unique capabilities and facilities that specialize in atomic structure, nano-scale composition, surface morphology, and surface chemistry. These unique capabilities will be essential in fully characterizing changes in mechanical properties of heat source related alloys as a function of irradiation.

While at ORNL, Dr. Dan Kramer and Chadwick Barklay also met with Dr. Steven Zinkle, leader of the Nuclear Materials Science and Technology Group in ORNL's Metals and Ceramics Division. Dr. Zinkle is considered an international authority in the study of radiation effects on materials. During this meeting Dr. Zinkle confirmed that research has not been conducted regarding changes in mechanical properties of heat source related alloys as a function of irradiation at the neutron fluence levels commensurate with HS/GPHS RTG environments.

- d) At the request of Dirk Cairns-Gallimore (DOE/NE-50), technical personnel and equipment located at UDRI were identified which could support Idaho National Laboratory in their evaluation of Haynes 25 capsules via eddy current NDE techniques. Dr. Ray Ko (UDRI) has a Ph.D. from Ohio State University and studied NDE techniques while in OSU's Welding Department. Dr. Ko is ASNT Level III certified, and eddy current test equipment is available at UDRI. Information on UDRI's capabilities in eddy current testing has been submitted to NE-50.
- e) The two T-111 alloy and a {Ta-10W / T-111 insert / Ta-10W} weld specimens that were sent to an outside laboratory have been analyzed using one of two state-of-the-art WDS analytical instruments. Unfortunately, the first spectrometer was not able to significantly differentiate the Ta, W, and Hf lines. A second more sensitive spectrometer is being shipped from Japan to the company's US location and it may be employed to attempt another analyses. If successful, this would allow the heat source program for the first time to possibly elucidate the role of Hf in these welds and materials.

- f) Discussions (e-mails) continue with INL personnel on shipping the pressure burst furnace to UDRI. UDRI has informed INL that more thorough details of the electrical power requirements needed to run the furnace and associated equipment; especially the furnace's amperage and voltage requirements. This information has been requested so that the appropriate space within UDRI for the furnace can be identified.
- g) Collaborations with Dr. William Moddeman (Pantex) have been initiated centered on the application of new software that may be able to calculate based on thermodynamic constants, material phases in a weld or weld pool as it cools from a molten state down towards room temperature. The software is capable of "predicting" the formation and/or resolution etc. of materials phases and polymorphs as a function of temperature, glove box atmosphere, chemical impurities, etc. Initial trials employing the software will be run on a "simple" system such as Ta-10W as this material is basically a binary alloy. In order to simulate glove box welds, low ppm levels of oxygen, nitrogen, carbon, water, etc. will be included in the thermodynamic calculations. Temperature regions that are to be investigated are from ~4000°C which is significantly above the expected weld pool temperature to ~1000°C.

<u>Program Recommendations:</u> To become more familiar with the various program's materials related issues, it is suggested that our attendance at appropriate program meetings be encouraged. In addition, reinstating clearances could also greatly facilitate future program related discussions.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including; determining if the state-of-the-art WDS equipment can identify the individual elements in Ta-10W and Ta-10W/T-111 insert weld specimens; the coating and "rocket" testing of carbon substrates; and preparing the experimental plan and experimental setup for the irradiation runs.

Patents: none

Publications/Presentations: none

# Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1	FWPF protection/replacement			
2	Role of Hf on the property differences between Ta-10W and T-111			
3	Potential materials replacements			
4	Compatibility of typical and future radioisotope materials			
5	Support Stirling based power systems development			
6	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05	
7	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05	
8	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06	
9	Final Report	08/11/06		
10	SF-269A	08/11/06		

# Budget Data (as of December 31, 2005):

			Approved Spending Plan		Actual Spent to Date	
Budg	et Period:		DOE	Total	DOE	Total
May	12, 2005 to	May 11, 2006	Amount		Amount	
	From	То				
	05/11/05	06/30/05			\$3,907	\$3,907
	05/11/05	09/30/05			\$25,958	\$25,958
	05/11/05	12/31/05			\$32,080	\$32,080
		Totals	\$100,000	\$100,000	\$61,945	\$61,945

## **QUARTERLY PROGRESS REPORT**

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

Covering Period: January 1, 2006 through March 31, 2006

Date of Report: April 18, 2006

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-50 contact – Dirk Cairns-Gallimore Contacts: ID contract specialist – Suzette Olson

Project Objective: This project addresses several of DOE-NE-50's near-term and future

materials science and engineering related programmatic needs/goals.

## Background:

The Office of Space and Defense Power Systems (NE-50) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions including the three large RTG's (Radioisotope Thermoelectric Generators) that are presently powering the Cassini spacecraft at Saturn. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific knowhow, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-50 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

Status: During this reporting quarter investigations continued in the assigned technical areas, and below are several focal points:

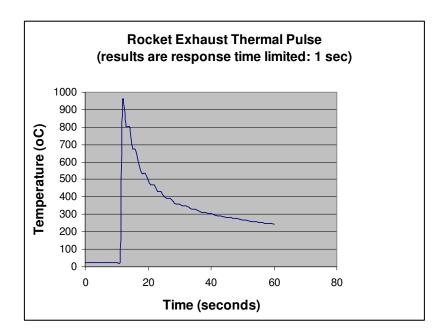
a) Work during this quarter on potential protective coatings that could decrease the oxidation/ablation rate of FWPF graphitic materials centered on an alumina based high temperature refractory ceramic coating. Thin coatings were applied to various graphitic specimens and these were subjected to the thermal pulse from the output of small rocket motor engines in a static rocket motor test stand. Relatively low cost black powder rocket motors were used in these experiments and the rocket motor nozzles were positioned ~2.54cm from the surface of the test specimens. The selected coating in initial testing has shown good adhesion, good thermal shock resistance, and most importantly, good erosion resistance to the reactants. A graphitic specimen was prepared that had one-half of its surface coated while the other half of its surface was left uncoated. This sample was positioned in the rocket test rig so that the output of the rocket motor was directed to the center of the circular specimen between the coated and uncoated sections of the specimen. The test specimen was placed ~2.54cm from the output of a rocket motor, and Figure 1 is a photograph taken post-test allowing a comparison between the coated and uncoated surfaces. Post-test analysis showed that the coating exhibited good integrity as it did not thermal shock, spall, or erode.

Figure 1. Post-rocket test photograph of a graphitic test specimen that had a protective coating applied to one-half of its surface (right side of the picture).



An initial attempt was also made to measure the thermal pulse from one of the rocket motors. A type K thermocouple was placed ~2.54cm from the motor and its output was captured using a data acquisition system. Figure 2 shows the results of one these thermal pulse tests. The rate of temperature rise was determined to be faster than the ability of the acquisition system to capture the data. The employed system had a 1 second measuring gate. Even with this limitation, the results in Figure 2 show a temperature rise in excess of >900°C/sec. A second acquisition system with a much faster measuring gate is being considered for further tests.

Figure 2. Thermal pulse obtained from a type K thermocouple placed ~2.54cm from the nozzle of a black powder rocket motor. Results are response time limited as data acquisition had a 1 second gate.



b) A literature search was performed using the Space Nuclear Technology Documentation Center Database maintained by Orbital Science Corporation, and it was conducted using the following search criteria:

Keyword	Documents in
	Database
Tantalum	56
T-111	20
Ta-10W	3

Approximately 7 of the 79 documents queried may be relevant to current research efforts and copies will be requested.

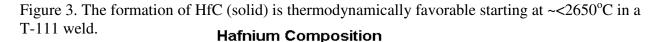
c) Ta-10%W and T-111 metallurgical and test specimens intended for use in planned irradiation experiments were fabricated via Wire Electrical Discharge Machining (WEDM). These test specimens meet the requirements of ASTM E8 (Standard Test Methods for Tension Testing of Metallic Materials) and are generally suitable for tests at elevated temperature and for creep tests. Once fabrication of the metallurgical and test specimens was completed, the specimens were immersed in a heated concentrated nitric acid solution (about 70% HNO<sub>3</sub>) to remove any residual brass material from the sample edges resulting from the WEDM process. Subsequently,

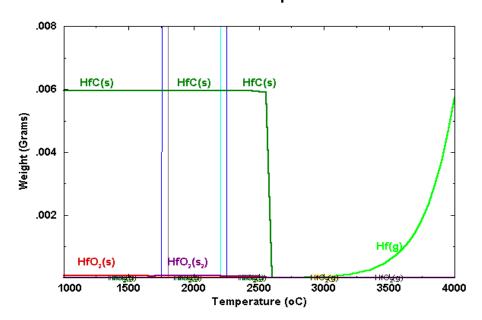
all specimens were cleaned in accordance with the tantalum alloy components cleaning procedure provided by Oak Ridge National Laboratory (ORNL).

Concurrently, all of the hardware and expendable items procured for use in conjunction with the Ohio State University Research Reactor (OSURR) reactor runs was configured and tested in the laboratory in order to characterize the response of the equipment. Additionally, mock-up and reactor runs were scheduled with OSURR facility. These reactor runs will be performed by Chadwick Barklay (Ph.D. candidate on this project).

A proposal for conducting elemental surface analysis and surface imagery of irradiated and mechanically tested Ta-10%W and T-111 samples was submitted to and accepted by the Oak Ridge National Laboratory (ORNL) Materials Analysis User Center (MAUC), which supports the High Temperature Materials Laboratory (HTML). The MAUC possesses unique capabilities and facilities that specialize in atomic structure, nano-scale composition, surface morphology, and surface chemistry. These unique capabilities will be essential in elucidating the mechanism(s) of long-term thermal and neutron irradiation stability of the precipitates responsible for high creep strength of Ta-10%W and T-111, and clarifying the mechanisms associated with the presence of hafnium in T-111.

- d) Discussions continued with INL personnel on shipping the pressure burst furnace. An appropriate laboratory space for the furnace has been identified.
- e) Collaborations with Dr. William Moddeman (Pantex) continued centered on the application of software capable of calculating, based on thermodynamic constants, the favorable material phases in a weld and/or weld pool as it cools from a molten state towards room temperature. The software is "predicting" the formation of possible materials phases and polymorphs as a function of temperature, glove box atmosphere, chemical impurities, etc. Initial work employing the software has been on the T-111 and Ta-10W systems with an emphasis on determining the thermodynamically favorable nitrides, oxides, and carbides. The temperature region of interest is from ~4000°C which is significantly above the expected weld pool temperature down through solidification of the HAZ to ~1000°C. Figure 3 shows an example of one application of the software that determined that HfC in solid form becomes thermodynamically stable in a T-111 weld below ~2650°C. It also shows that the expected quantity of HfC in a T-111 weld is greater than 10 times the expected quantity of HfO<sub>2</sub>. This work could be significant as it "identifies" which carbide, nitride, and/or oxide may form to help "pin" the grain boundaries in either T-111 and/or Ta-10W welds. This could provide a technical avenue for the direct welding of Ta-10W to Ta-10W without T-111 inserts, thus allowing a "complete" replacement of Ta-10W for T-111.





<u>Program Recommendations:</u> To become more familiar with the various program's materials related issues, it is suggested that our attendance at appropriate program meetings be encouraged. In addition, reinstating clearances could also greatly facilitate future program related discussions.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including; initiation of irradiation runs on tantalum alloys; the coating and "rocket" testing of FWPF substrates; and the running of additional software calculations for identifying thermodynamically stable species in T-111 and Ta-10W welds.

Patents: none

#### Publications/Presentations:

An abstract entitled ""Investigation of Effects of Neutron Radiation Exposure on Tantalum Alloys for Radioisotope Power System Applications" was submitted for consideration for presentation at the Materials Science & Technology 2006 Conference and Exposition scheduled for October, 2006.

An oral presentation of a paper entitled "The Effect of the Presence of 2 wt% Hafnium in T-111" was presented at the Thirty-First Annual Dayton-Cincinnati Aerospace Science Symposium, Dayton, OH, March 2006.

# Milestone Status Table:

ID	Task / Milestone Description	Planned	Actual	Comments
Number		Completion	Completion	
1	FWPF protection/replacement			
2	Role of Hf on the property differences between Ta-10W and T- 111			
3	Potential materials replacements			
4	Compatibility of typical and future radioisotope materials			
5	Support Stirling based power systems development			
6	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05	
7	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05	
8	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06	
9	4 <sup>th</sup> Quarterly Report	04/30/06		
	Final Report	12/30/07		
	SF-269A	12/30/07		

# Budget Data (as of March 31, 2006):

		Approved Spending Plan		Actual Spent to Date	
Budget Period:		DOE	Total	DOE	Total
May 12, 2005 to	May 11, 2006	Amount		Amount	
From	То				
05/11/05	06/30/05			\$3,907	\$3,907
05/11/05	09/30/05			\$25,958	\$25,958
05/11/05	12/31/05			\$32,080	\$32,080
05/11/05	03/31/06			\$30,145	\$30,145
	Totals	\$100,000	\$100,000	\$92,090	\$92,090

## **QUARTERLY PROGRESS REPORT**

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

Covering Period: April 1, 2006 through June 30, 2006

Date of Report: July 21, 2006

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-50 contact – Dirk Cairns-Gallimore Contacts: ID contract specialist – Suzette Olson

Project Objective: This project addresses several of DOE-NE-50's near-term and future

materials science and engineering related programmatic needs/goals.

## Background:

The Office of Space and Defense Power Systems (NE-50) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions including the three large RTG's (Radioisotope Thermoelectric Generators) that are presently powering the Cassini spacecraft at Saturn. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific knowhow, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-50 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

Status: During this reporting quarter investigations continued in the assigned technical areas, and below are several focal points:

a) The Ta-10%W metallurgical and test specimens were recrystallized under vacuum by INL prior to conducting the irradiation experiments at the Ohio State University Research Reactor (OSURR). Recrystallization of a metal produces properties that are comparable to those of the original, unstrained metal and results in a decrease in stored energy as well as a complete elimination of residual stresses in the recrystallized metal.

At total of twenty-four (24) Ta-10%W and twenty-four (24) T-111 metallurgical and test specimens were subsequently irradiated in two separate reactor runs at the Ohio State University Research Reactor (OSURR) reactor (Figure 1). These reactor runs were performed by Chadwick Barklay (Ph.D. candidate on this project). Both reactor runs were conducted at full power, approximately 0.5 MW, for a total of four (4) hours, which yielded a total flux of ~1.2E15 nvt. In accordance with the design of experiments, half of the samples were irradiated at ambient temperature and the remaining half were irradiated at 350°C, both in an atmosphere of less than 0.1 ppm oxygen. UDRI experimentation has demonstrated that in this temperature regime there is no oxidation of these materials. To further ensure oxidation protection of the metallurgical and test specimens during irradiation, the specimens were wrapped in tantalum foil.

Figure 1. (Left) Ta-10W and T-111 test specimens were placed within cadmium wrapped tube furnace prior to loading within reactor test pipe. (Right) Reactor core at bottom of water pool (middle of photograph) at Ohio State University.





Additionally, the heater system used during specimen irradiation incorporated a cadmium shield used to suppress the thermal neutron flux. Approximately, 95% of the flux had energy above the cadmium cutoff energy of approximately 1 eV, thus the neutron flux was primarily epithermal flux as expected. The first shipment of irradiated samples was received from OSURR and efforts have begun to initiate creep rupture testing in accordance with the design of experiments.

Concurrently, stress rupture tests were conducted on all of the non-irradiated test specimens in accordance with the design of experiments at static loads of 90 and 100 ksi and temperatures of 25°C and 300°C. The results of these experiments are being evaluated to determine the

activation energies for creep, the effects of load and temperature on the creep and rupture behavior of the materials, and to determine the degree of interaction of the experimental variables.

Preliminary results indicate that both Ta-10W and T-111 appear to be strain rate insensitive whether they are incrementally loaded up to failure or tested with a static load (per the design of experiments) and the corresponding elongations for each material are comparable. When loaded incrementally, both materials undergo instantaneous extension until the flow stress is equal to the applied stress, then creep ceases. This behavior seems to be more pronounced at 300°C than at 25°C and with Ta-10%W more so than with T-111. Efforts are ongoing to gain a better understanding of the mechanisms at play through fractography to determine if any microstructural instabilities are contributing to this behavior.

Preparations are underway for providing TEM samples of non-irradiated rupture tested Ta-10%W and T-111 specimens to the Oak Ridge National Laboratory (ORNL) Materials Analysis User Center (MAUC), which supports the High Temperature Materials Laboratory (HTML). The MAUC possesses unique capabilities and facilities that specialize in atomic structure, nano-scale composition, surface morphology, and surface chemistry. These unique capabilities will be essential in elucidating the mechanism(s) of long-term thermal and neutron irradiation stability of the precipitates responsible for high creep strength of Ta-10%W and T-111, and clarifying the mechanisms associated with the presence of hafnium in T-111.

- b) Collaborations with Dr. William Moddeman (Pantex) continued centered on the application of FactSage software capable of calculating, based on thermodynamic constants, the favorable material phases in a weld and/or weld pool as it cools from a molten state towards room temperature. Emphasis is on the possible thermodynamically favorable phases in T-111 weld pools. Support dollars have been received from Pantex to prepare T-111 and Ta-10W weld specimens that can be used in Transmission Electron Microscopy evaluations. Specimens were welded during this Quarter at ORNL. This work is very closely related to our present work in this area.
- c) Collaboration with Dr. Jan Birkbeck (Pantex) has been developed with emphasis on the effects of irradiation on T-111 and Ta-10W welds. This work is being funded by Pantex and also closely matches our present work in this area.
- d) A high temperature oxidation resistant coating that has successfully passed high temperature arc-jet tests performed by NASA has been identified for possible application as a FWPF oxidation protective coating. The NASA test was performed on coated carbon-carbon composite specimens at temperatures in excess of 3000°F without failure. The material when cured and pyrolyzed is silicon carbide based which is highly advantageous compared to other high temperature systems. Silicon and carbon have been successfully employed in long duration RTG space missions without detrimental compatibility issues. In addition, silicon carbide based systems for oxidation protection of carbon materials have the added advantage in that they can form a "glass" phase at high temperatures that can help fill any cracks that may form in the

coating. Test materials have been obtained and an atmosphere controlled pyrolyzing furnace is being designed.

<u>Program Recommendations:</u> To become more familiar with the various program's materials related issues, it is suggested that our attendance at appropriate program meetings be encouraged. In addition, reinstating clearances could also greatly facilitate future program related discussions.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including; mechanical testing of Ta-10W and T-111 specimens that have been irradiated; TEM work will be initiated on Ta-10W and T-111 specimens; design and fabrication of an atmosphere controlled furnace for the coating and "rocket" testing of FWPF substrates; and the running of additional software calculations for identifying thermodynamically stable species in T-111 and Ta-10W welds.

Patents: none

## **Publications/Presentations:**

An abstract entitled, "Application of Thermodynamic Based Software in the Prediction of Compositional Phases in Selected Tantalum Alloys Welds," has been submitted to STAIF'07 for consideration at the February 2007 meeting.

An abstract entitled, "Investigation of Effects of Neutron Radiation on Tantalum Alloys for Radioisotope Power System Applications," has been submitted to STAIF'07 for consideration at the February 2007 meeting.

Milestone Status Table:

ID Number	Task / Milestone Description	Planned Completion	Actual Completion	Comments
1	FWPF protection/replacement			
2	Role of Hf on the property differences between Ta-10W and T- 111			
3	Potential materials replacements			
4	Compatibility of typical and future radioisotope materials			
5	Support Stirling based power systems development			
6	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05	
7	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05	
8	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06	
9	4 <sup>th</sup> Quarterly Report	04/30/06	04/18/06	
10	5 <sup>th</sup> Quarterly Report	07/30/06	07/25/06	
	Final Report	12/30/07		
	SF-269A	12/30/07		

Budget Data (as of June 30, 2006):

_		Approved Spending Plan		Actual Spent to Date	
Budget Period:		DOE Amount	Total	Quarterly DOE Amount	Project Total
From	То				
05/12/05		\$100,000			
05/12/05	06/30/05			\$3,907	\$3,907
07/01/05	09/30/05			\$25,958	\$29,865
10/01/05	12/31/05			\$32,080	\$61,945
01/01/06	03/31/06			\$30,145	\$92,090
05/12/06		\$100,000			
04/01/06	06/30/06			\$16,842	\$108,932
	Totals	\$200,000			\$108,932

## **QUARTERLY PROGRESS REPORT**

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

Covering Period: July 1, 2006 through September 30, 2006

Date of Report: October 21, 2006

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-50 contact – Dirk Cairns-Gallimore Contacts: ID contract specialist – Suzette Olson

Project Objective: This project addresses several of DOE-NE-50's near-term and future

materials science and engineering related programmatic needs/goals.

#### Background:

The Office of Space and Defense Power Systems (NE-50) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions including the three large RTG's (Radioisotope Thermoelectric Generators) that are presently powering the Cassini spacecraft at Saturn. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific knowhow, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-50 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

Status: During this reporting quarter investigations continued in the assigned technical areas, and below are several focal points:

a) Statistical analysis of the tensile test data associated with irradiated and non-irradiated T-111 and Ta-10%W suggests that the mechanical properties of tensile, yield, UTS, and modulus for T-111 at 25 °C and 300 °C are unaffected when irradiated with a neutron fluence of  $\sim 1.2 \times 10^{15}$  nvt at both 25 °C and 350 °C. (Calculations have shown that a hypothetical RTG design that contains 2500 g of 238-Plutonium will have a total neutron emission rate of 1.5 x  $10^7$  neutrons/sec. Extrapolated to the expected 30-year lifespan of a RTG, the total neutron emission is approximately 1.5 x  $10^{16}$  neutrons. Assuming the material of interest is 1.0 cm from the fuel surface, the total neutron fluence is the approximately  $1.2 \times 10^{15}$  nvt).

However, the analysis of the percent elongation responses of T-111 tensile test data reveals that the high level of (A) irradiation level and the high level of (B) irradiation temperature are the "driving force" in the significance of the interaction of (A\*B). Thus the mechanical property of percent elongation of T-111 is negatively affected when irradiated with a neutron fluence of ~1.2 x  $10^{15}$  nvt at 350 °C. This work is significant because it shows that there are significant differences in some of the mechanical properties of Ta-10%W compared to T-111 when exposed to neutron fluences of ~1.2 x  $10^{15}$  nvt at increasing temperatures from 25 - 350 °C. Since the only major constituent difference between these two materials is the 2% hafnium addition in the T-111, it is reasonable to suggest that this neutron irradiation fluence represents a possible "threshold level" at which either hafnium or hafnium containing metalloid impurities are affected by the neutron irradiation, such that they begin to negatively influence the mechanical properties of T-111.

# Current research efforts are focusing on:

- Transmission Electron Microscopy (TEM) of Ta-10%W and T-111 irradiated and non-irradiated mechanically tests specimens in order to confirm differences in relative levels of lattice defects between materials.
- Conduct of additional mechanical testing such as creep (see Figure 1) and stress rupture for Ta-10%W and T-111 in order to determine if any statistically significant test differences result from irradiation.
- Determination of the activation energies for dislocation movement of irradiated and non-irradiated Ta-10%W and T-111 for given temperatures and irradiation levels.

These efforts will provide a level of understanding of irradiation effects in Ta-10%W and T-111 that will enable more confident extrapolation of relevant mechanical-property data to predict end-of-service behavior of components made from these alloys.

Figure 1. Test specimen (within black lined box below) irradiated during one of the reactor runs at Ohio State University is shown undergoing tensile creep testing. Computer software is used to capture the test data via a developed video camera system. Tests are being conducted at both room temperature and at elevated temperatures. Next to the tensile creep frame below is Chadwick Barklay (Ph.D. candidate) who is conducting the tests following approved radiological procedures.



b) Additional "matching" research funding was received during this quarter via technical collaborations with Dr. William Moddeman (Pantex Plant). This funding has allowed the project to leverage additional work that is of interest to both DOE/NE and Pantex. As shown in the Project's Budget Data at the end of this report, this collaboration has significantly reduced DOE/NE's expenditures during the quarter.

The collaborative research with Dr. Moddeman is centered on the application of FactSage software capable of calculating, based on thermodynamic constants, the favorable material phases in a weld and/or weld pool as it cools from a molten state towards room temperature. Emphasis is on the possible thermodynamically favorable phases in the weld pools. These support dollars have been used to prepare T-111 and Ta-10W test welds and this work is very closely related to our present work in this area. Specimens will undergo various mechanical property tests and samples have been prepared for Transmission Electron Microscopy evaluations.

c) A high temperature oxidation resistant coating that has successfully passed high temperature arc-jet tests performed by NASA has been purchased for testing as a FWPF oxidation protective coating. The NASA test was performed on coated carbon-carbon composite specimens at temperatures in excess of 3000°F without failure. An atmosphere controlled pyrolyzing furnace has been designed and fabricated. The furnace is presently undergoing thermal gradient testing.

<u>Program Recommendations:</u> To become more familiar with the various program's materials related issues, it is suggested that our attendance at appropriate program meetings be encouraged. In addition, reinstating clearances could also greatly facilitate future program related discussions.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including; mechanical testing of Ta-10W and T-111 specimens that have been irradiated; TEM analyses on Ta-10W and T-111 specimens at Oak Ridge National Laboratory; trial runs using the fabrication atmosphere controlled furnace for the coating and "rocket" testing of FWPF substrates; and the running of additional software calculations for identifying thermodynamically stable species in T-111 and Ta-10W welds.

Patents: none

## Publications/Presentations:

A poster was prepared for presentation at the Materials Science & Technology 2006 Conference and Exhibition entitled, "Investigation of Effects of Neutron Radiation on Tantalum Alloys for Radioisotope Power System Applications" by Chadwick Barklay (University of Dayton), Daniel Kramer (University of Dayton), and Joseph Talnagi (The Ohio State University), Cincinnati, OH, October 2006.

# **Task and Milestone Table:**

Task and	Milestone Table:		
ID Number	Task / Milestone Description		
1	FWPF protection/replacement		
2	Role of Hf on the property differences between Ta-10W and T-111		
3	Potential materials replacements		
4	Compatibility of typical and future radioisotope materials		
5	Support Stirling based power systems development		
		Planned Completion	Actual Completion
6	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05
7	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05
8	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06
9	4 <sup>th</sup> Quarterly Report	04/30/06	04/18/06
10	5 <sup>th</sup> Quarterly Report	07/30/06	07/25/06
11	6 <sup>th</sup> Quarterly Report	10/30/06	07/21/06
	Final Report	12/30/07	
	SF-269A	12/30/07	

# **Budget Data (as of September 30, 2006):**

Dudget Duta (as o		Approved Sp	Actual Sp	pent to Date	
Budget Period:		DOE Amount	Total	Quarterly DOE Amount	Project Total
From	То				
05/12/05		\$100,000			
05/12/05	06/30/05			\$3,907	\$3,907
07/01/05	09/30/05			\$25,958	\$29,865
10/01/05	12/31/05			\$32,080	\$61,945
01/01/06	03/31/06			\$30,145	\$92,090
05/12/06		\$100,000			
04/01/06	06/30/06			\$16,842	\$108,932
07/01/06 09/30/06				\$5,508	\$114,440
	Totals	\$200,000			\$114,440

#### **QUARTERLY PROGRESS REPORT**

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

POWER SYSTEMS

Covering Period: January 1, 2007 through March 31, 2007

Date of Report: April 17, 2007

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-34 contact – Dirk Cairns-Gallimore

Contacts: ID Contract Specialist – Patricia Alexander-Johnson

Project Objective: This project addresses several of DOE-NE-34's near-term and future

materials science and engineering related programmatic needs/goals.

#### Background:

The Office of Space and Defense Power Systems (NE-34) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific know-how, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-34 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

Status: During this reporting quarter investigations continued and below are several focal points;

# a) <u>Statistical Analysis of Low-Temperature Creep/Stress Rupture Data for Irradiated and Non-Irradiated Ta-10%W</u> and T-111

The low-temperature creep/stress rupture experiments of irradiated and non-irradiated Ta-10%W and T-111 associated with this research employed a fractional factorial design of experiments of resolution five. This type of design of experiments allowed for the simultaneous variation of irradiation level, irradiation temperature, creep load, and creep temperature. As a result, over 20,000 hours of low-temperature creep/stress rupture data was obtained on non-irradiated Ta-10%W and T-111 and specimens irradiated with a neutron fluence of approximately 1.2 x 10<sup>15</sup> neutrons/cm² (nvt). Exposure of Ta-10%W and T-111 to neutron fluence of 1.2 x 10<sup>15</sup> nvt results in an approximate neutron damage of 3.0 x 10<sup>-7</sup> displacements per atom (dpa). The general trend in the low-temperature creep/stress rupture data reveals that neutron damage in the 10<sup>-7</sup> dpa regime results in an approximate two-order of magnitude increase in the rupture time, and a corresponding two-order magnitude decrease in the steady state creep rate.

Statistical Analysis System (SAS) software was used to conduct an analysis of variance (ANOVA) of all of the low-temperature creep/stress rupture data. The responses of rupture time, elongation, and steady state creep rate were analyzed based on the dependent variables of irradiation level, irradiation temperature, creep load, and test temperature. The results of the ANOVA indicate the following:

Statistical Effect	Significance Level
	(p value)
Alloy vs. Rupture Time	0.0460
Interaction of Irradiation Fluence/Temperature vs. Rupture Time	0.0472
Alloy vs. Steady State Creep Rate	0.0567
Interaction of Irradiation Fluence/Temperature vs. Steady State Creep Rate	0.0572

These experimental results show that the rupture time and steady state creep rate of both Ta-10%W and T-111 tested at 25°C are affected when irradiated with a neutron fluence of ~ $1.2 \times 10^{15}$  nvt at both 25°C and 350°C. Additionally, the results demonstrate that the response of rupture time and steady state creep rate for T-111 are more pronounced than for Ta-10%W. This is significant because it demonstrates that neutron fluence levels that are equivalent to those of a  $^{238}$ PuO<sub>2</sub> fueled RPS have a positive influence on the steady state creep rate and rupture time of T-111 and Ta-10%W.

It should be noted that a 6-10% reduction of elongation was observed for irradiated T-111 and Ta-10%W when exposed to a creep load of 496-620 MPa. Additional testing is ongoing to verify if a statistical significance exists for these observations.

## b) Predictive of Steady-State Creep Model

Various standard analytical techniques were applied to the steady state creep data of both irradiated and non-irradiated T-111 and Ta-10%W specimens obtained in this research. This allowed for better insight into the creep mechanisms, and the development of a predictive model for determining the secondary creep rates for these alloys that incorporates the effects of neutron displacement damage (dpa). After significant analysis it was determined that the creep data associated with this research for both T-111 and Ta-10%W follows a hyperbolic sine relationship since it approximates the power law at low stresses and the exponential law at higher stresses. Thus, the minimum creep rate (m/s) for both irradiated and non-irradiated T-111 can be described by the following developed expressions:

## T-111 (non-irradiated)

$$\dot{\varepsilon} = 4.33 \times 10^{-10} \left[ Sinh(9.6 \times 10^{-3} \sigma) \right]^{5.14} e^{-57280/RT}$$

#### T-111 (Irradiated)

$$\dot{\varepsilon} = 2.16 \times 10^{-16} \left[ Sinh(9.6 \times 10^{-3} \sigma) \right]^{7.02} e^{-57280/RT}$$

Where,

R - Ideal gas constant (8.3145 J/mole-K)

T - Test temperature in Kelvin.

Correlating the neutron displacement damage (dpa) with the minimum strain rate equations for T-111 the minimum creep rate (m/s) for irradiated T-111 can be described by the expression:

$$\dot{\varepsilon} = (dpa)4.33 \times 10^{-10} \left[ Sinh(9.6 \times 10^{-3} \sigma) \right]^{1.8910 e^{1+dpa(1.04 \times 10^6)}} e^{-57280/RT}$$

This equation is valid for temperatures from 25 to 300°C, stresses between 496 to 690 MPa, and for a neutron damage levels between 10<sup>-7</sup> to 10<sup>-6</sup> dpa for T-111. Since the effects of neutron displacement damage are more pronounced for T-111 than for Ta-10%W the following expression describes minimum creep rate (m/s) for both irradiated and non-irradiated Ta-10%W:

$$\dot{\varepsilon} = 7.30 \times 10^{-12} \left[ Sinh(9.6 \times 10^{-3} \sigma) \right]^{5.25} e^{-57280/RT}$$

Which is valid for temperatures from 25 to 300°C, stresses between 496 to 690.

At the temperature and stress levels used in this research, the data reveals that the low temperature creep resistance of T-111 is sensitive to levels of neutron irradiation exposure that result in irradiation damage levels  $>10^{-7}$  dpa. The values of the stress exponents in the above equations suggest that the rate controlling mechanism for steady state creep of these materials is dislocation glide at temperatures between 25 °C and 300°C. The increase in the stress exponent

from 5.14 to 7.02 for the irradiated T-111 specimens further reinforces the conclusion that low levels of neutron irradiation have a pronounced effect on non-conservative dislocation motion. Additionally, the lack of change in the stress exponent between the irradiated and non-irradiated Ta-10%W specimens confirm that within the parameters of this research that neutron irradiation has a negligible effect on the creep properties of Ta-10%W.

These results provide a basic understanding of the effects of the creep properties of Ta-10%W and T-111 resulting from neutron radiation exposure at temperatures below 350°C. This enhances the level of understanding of potential irradiation hardening mechanisms in both these alloys. Additionally, mechanical-property trends of T-111 and Ta-10%W can more confidently predicted for these materials when exposed to neutron irradiation doses (irradiation damage levels) and internal pressure levels that correspond to actual RPS service lifetimes.

- b) Discussions were held with NE-34 during the reporting quarter on FY'08 project endeavors including continuing work on the effect of irradiation on various materials of interest to the Heat Source program. In anticipation of continuing sponsorship, negotiations are being held with representatives at The Ohio State University Research Reactor laboratory. These discussions are working towards obtaining commitments for FY'08 reactor time, and on maintaining the already operational UDRI developed experimental test equipment intact at the reactor laboratory until needed again early next fiscal year.
- c) Additional "matching" research funding was again received during this quarter via technical collaborations with representatives from Pantex. This funding has continued to allow the project to greatly leverage additional work that is of interest to both DOE/NE and Pantex. As shown in the Project's Budget Data at the end of this report, this collaboration has significantly reduced DOE/NE's expenditures again during the reporting quarter.

Discussions are presently underway on potential topic areas for FY'08 collaborative research with Pantex. Several of the topic areas again parallel work of potential interest to DOE/NE such as research on the effects of neutron irradiation on the mechanical properties of welded tantalum alloys (i.e. T-111).

#### Program Recommendations:

a) It is anticipated that Mr. Chad Barklay (Ph.D. graduate student working on this project) will complete his Ph.D. thesis requirements sometime during the next calendar quarter. Mr. Barklay's contribution to this program has been scientifically outstanding, and as a graduate student the cost benefit ratio to the program has been exceptionally advantageous. In order to assure continuation of the present line of research, it would be especially valuable to the project to take on another graduate student. A commitment to a graduate student typically entails the confidence of continued funding into at least the next fiscal year or two. It is requested that discussions be held between NE-34 and the University of Dayton to determine future anticipated funding levels for FY'08 and FY'09.

b) To become more familiar with the various NE-34 program's materials related issues, it is suggested that our attendance at appropriate program meetings be encouraged. In addition, reinstating clearances could also greatly facilitate future program related discussions.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including; mechanical testing of Ta-10%W and T-111 specimens that have been irradiated; concluding sample preparation and TEM analyses on the unirradiated and irradiated Ta-10%W and T-111 specimens at Oak Ridge National Laboratory; and continuing discussions with personnel from both Pantex and The Ohio State University Research Reactor laboratory.

Patents: none

#### Publications/Presentations:

- 1) C.D. Barklay, D.P. Kramer, and J. Talnagi, "Investigation of Effects of Neutron Irradiation on Tantalum Alloys for Radioisotope Power System Applications," published in the 25<sup>th</sup> Proceeding of Space Technology and Applications International Forum (STAIF-2007), American Institute of Physics Conference Proceedings 880, Albuquerque, NM, February 2007, pp. 224-228.
- 2p) C.D. Barklay, D.P. Kramer, and J. Talnagi, "Investigation of Effects of Neutron Radiation on Tantalum Alloys for Radioisotope Power System Applications," presented at Space Technology and Applications International Forum (STAIF-2007), American Institute of Physics Conference Proceedings 880, Albuquerque, NM, February 2007.
- 3) W.E. Moddeman, C.D. Barklay, J.C. Birkbeck, R.G. Miller, L.F. Allard, and D.P. Kramer, "Thermodynamic Prediction of Compositional Phases by Transmission Electron Microscopy on Tantalum-Based Alloy Weldments," published in the 25<sup>th</sup> Proceedings of Space Technology and Applications International Forum (STAIF-2007), American Institute of Physics Conference Proceedings 880, Albuquerque, NM, February 2007, pp. 229-233.
- 4p) W.E. Moddeman, C.D. Barklay, J.C. Birkbeck, R.G. Miller, L.F. Allard, and D.P. Kramer, "Application of Thermodynamic Based Software in the Prediction of Compositional Phases in Ta-10W and T-111 Weldments," presented at Space Technology and Applications International Forum (STAIF-2007), American Institute of Physics Conference Proceedings 880, Albuquerque, NM, February 2007.

**Deliverable Reports:** 

Number	Report	Planned Completion	Actual Completion
1	1st Quarterly Report	07/30/05	07/18/05
2	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05
3	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06
4	4 <sup>th</sup> Quarterly Report	04/30/06	04/18/06
5	5 <sup>th</sup> Quarterly Report	07/30/06	07/25/06
6	6 <sup>th</sup> Quarterly Report	10/30/06	10/21/06
7	7 <sup>th</sup> Quarterly Report	01/30/07	01/23/07
8	8 <sup>th</sup> Quarterly Report	04/30/07	04/17/07
	Final Report	12/30/07	
	SF-269A	12/30/07	

**Budget Data (as of March 31, 2007):** 

			Actual S	pent to Date	
Budget Period:		DOE Budgeted Amount	Quarterly Amount Spent	Project Total Spent	
From	То				
05/12/05		\$100,000			
05/12/05	06/30/05		\$3,907	\$3,907	
07/01/05	09/30/05		\$25,958	\$29,865	
10/01/05	12/31/05		\$32,080	\$61,945	
01/01/06	03/31/06		\$30,145	\$92,090	
05/12/06		\$100,000			
04/01/06	06/30/06		\$16,842	\$108,932	
07/01/06	09/30/06		\$5,508*	\$114,440	
10/01/06	12/31/06		\$7,938*	\$122,378	
01/01/07	03/31/07		\$12,068*	\$134,446	
Total: \$200,000 Total Budget Left: \$65					
	* Significant "matching" dollars obtained from				
	Pantex helped	to support this effort d	luring the Quar	ter	

#### **QUARTERLY PROGRESS REPORT**

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

Covering Period: September 30, 2006 through December 31, 2006

Date of Report: January 23, 2007

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-34 contact – Dirk Cairns-Gallimore Contacts: ID contract specialist – Suzette Olson

Project Objective: This project addresses several of DOE-NE-34's near-term and future

materials science and engineering related programmatic needs/goals.

#### Background:

The Office of Space and Defense Power Systems (NE-34) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions including the three large RTG's (Radioisotope Thermoelectric Generators) that are presently powering the Cassini spacecraft at Saturn. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific knowhow, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-34 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

Status: During this reporting quarter investigations continued in the assigned technical areas, and below are several focal points:

a) Irradiated and non-irradiated rupture tested samples of Ta-10%W and T-111 specimens were analyzed using Transmission Electron Microscopy (TEM) at Oak Ridge National Laboratory (ORNL) High Temperature Materials Laboratory (HTML) Materials Analysis User Center (MAUC). Preliminary TEM analysis of electropolished specimens of irradiated and non-irradiated Ta-10%W and T-111 revealed the basic microstructural evolution of these materials when subjected to low-dose (~10<sup>-7</sup> dpa) neutron irradiation both before and after being mechanically tested. Although the irradiation induced defect structures in both materials are not visible using conventional TEM techniques, their effects in mechanically tested microstructures are evident.

The primary and latent slip systems in tantalum are <111> $\{110\}$  and <111> $\{112\}$  respectively. Plastic flow of all bcc metals is controlled by  $a_0/2<111>$  screw dislocations that possess a high Peierls stress, which leads to a strong temperature dependence of the flow stress at low temperatures. For tantalum at low temperatures (<0.3  $T_m$ ), the motion of the screw dislocations is believed to be associated with the formation of mobile kinks on the dislocation line. When moving under the influence of an applied stress, screw dislocations will intersect thus leading to the formation of jogs. The dragging of these jogs by the moving dislocations results in the formation of intrinsic atomic defects, in particular of self-interstitials. At the temperatures regimes used in the creep/rupture testing for this research (<0.3  $T_m$ ), screw dislocations can cross-slip to the latent slip plane.

Preliminary conclusions are that the irradiation induced defect structures in these alloys are responsible for a pronounced microstructural evolution by further impeding dislocation motion thus increasing rupture times and negatively affecting strain to failure. Figure 1 is a TEM image of virgin recrystallized T-111. Figure 2 reveals that the dislocation activity in creep/tensile tested T-111 becomes apparent through the formation of characteristic cellular or sub-granular patterns where dislocations arrange in walls at the boundaries of cells/sub-grains with relatively low internal dislocation density.

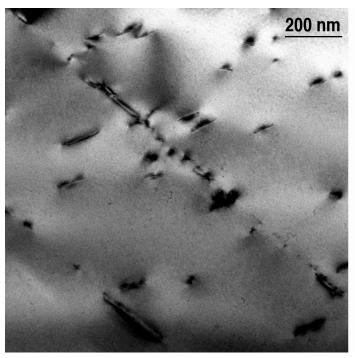
The driving force behind the creation of a mosaic cells/sub-grains has long been unclear. It is understood that dislocations of the same slip system with the same Burgers vector and slip plane interact weakly. During stage I of creep the work-hardening coefficient is relatively small. However, during stage II creep there is a high degree of work hardening as dislocations operate on mutually intersecting slip planes. As a result, they form junctions by recombination and have to intersect their "forest" dislocations during glide thus leading to sub-grain formation. The atomistic nature of the interaction between dislocations and small defect clusters, produced by low temperature neutron irradiation, makes these processes very difficult to study using conventional experimental techniques.

Figure 3 shows an increase in dislocation density in the irradiated T-111 rupture tested specimen accompanied by a pronounced mosaic of cells/sub-grains resulting from the interaction of gliding screw dislocations with irradiation induced defect structures.

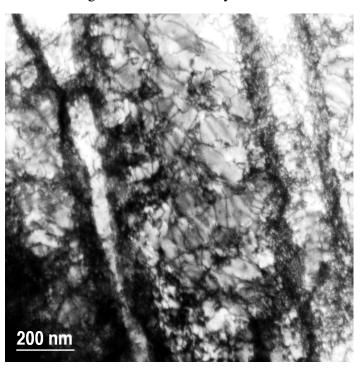
Due to the low level of radiation damage in the irradiated test specimens, approximately 3.0 x 10<sup>-7</sup> dpa, it is difficult to elucidate the increase in obstacle distribution that the dislocations have to surmount during deformation.

The TEM results for Ta10%W follow the same microstructural evolutionary trend as T-111, but effects of irradiation are much less pronounced. These observations are consistent with the preliminary creep/rupture test data.

The TEM analyses coupled with the preliminary statistical analysis of the creep/stress rupture test data associated with irradiated and nonirradiated T-111 and Ta-10%W confirm that the primary/secondary creep rates and rupture times for both materials at 25°C and 300°C are positively affected when irradiated with a neutron fluence of  $\sim 1.2 \times 10^{15}$ nvt at both 25°C and 350°C. However, the rupture strains for both materials are negatively affected, which is generally consistent with materials that have experienced irradiation damage.



**FIGURE 1.** TEM image of virgin recrystallized T-111 showing a dislocation density of  $\sim 10^{10}$  m<sup>-2</sup>.



**FIGURE 2.** TEM image of rupture tested non-irradiated T-111 showing a dislocation density of  $\sim 10^{16}$  m<sup>-2</sup>.

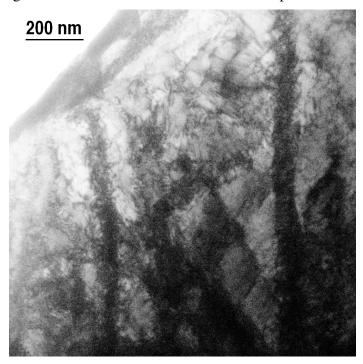
This work is significant because it begins to elucidate the difference in the responses of Ta-

10%W and T-111 to neutron fluences of  $\sim 1.2 \times 10^{15}$  nvt ( $\sim 10^{-7}$  dpa) when irradiated at increasing temperatures from 25 - 350°C.

Current research efforts are focusing on:

- Finalizing the Transmission Electron Microscopy (TEM) of irradiated Ta-10%W and T-111 mechanically tested specimens.
- Finalizing the mechanical testing of irradiated Ta-10%W and T-111.

These efforts will provide a level of understanding of the irradiation effects of Ta-10%W and T-111 that will enable more confident materials selection and extrapolation of relevant mechanical-property data to predict end-of-service behavior of components made from these alloys.



**FIGURE 3.** TEM image of rupture tested irradiated T-111 showing a dislocation density of  $>10^{16}$  m<sup>-2</sup> and more pronounced sub-grain formation.

b) Additional "matching" research funding was again received during this quarter via technical collaborations with Dr. William Moddeman (Pantex). This funding has continued to allow the project to greatly leverage additional work that is of interest to both DOE/NE and Pantex. As shown in the Project's Budget Data at the end of this report, this collaboration has significantly reduced DOE/NE's expenditures again during the reporting quarter.

Collaborative research with Pantex and the University of Dayton continues to be centered on radioisotope power sources via the application of FactSage software capable of calculating, based on thermodynamic constants, the favorable material phases in a weld and/or weld pool as it cools from a molten state towards room temperature. The collaborative support dollars have been used to prepare T-111 and Ta-10%W test welds and this work is very closely related to our present work in this area. Welded T-111 and Ta-10%W specimens have been neutron irradiated at the Ohio State University Research Reactor facility. The reactor run was conducted at full power, approximately 0.5 MW, for a total of one (1) hour, which yielded a total flux of ~ 3.0 x  $10^{14}$  nvt. The weld specimens were irradiated at  $400^{\circ}$ C under a purged argon atmosphere similar to what was used in our previous irradiations on non-welded Ta-10%W and T-111 specimens.

To ensure further oxidation protection of the metallurgical and test specimens during irradiation, the specimens were double wrapped in pure tantalum foil prior to the irradiation.

This parallel work on welded T-111 and Ta-10%W is primarily being funded by Pantex. It is not certain that Pantex will continue this work at the present funding level for the remainder of FY07 or into FY08.

#### **Program Recommendations:**

- a) It is anticipated that Mr. Chad Barklay (Ph.D. graduate student working on this project) will complete his Ph.D. thesis requirements sometime during the next two calendar quarters. Mr. Barklay's contribution to this program has been scientifically outstanding, and as a graduate student the cost benefit ratio to the program has been exceptionally advantageous. In order to assure continuation of the present line of research, it would be especially valuable to the project to take on another graduate student. A commitment to a graduate student typically entails the confidence of continued funding into at least the next fiscal year or two. It is requested that discussions be held between NE-34 and the University of Dayton to determine future anticipated funding levels for the continuation of this program.
- b) To become more familiar with the various NE-34 program's materials related issues, it is suggested that our attendance at appropriate program meetings be encouraged. In addition, reinstating clearances could also greatly facilitate future program related discussions.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including; mechanical creep testing of Ta-10%W and T-111 specimens that have been irradiated; sample preparation and TEM analyses on the unirradiated and irradiated Ta-10%W and T-111 specimens at Oak Ridge National Laboratory; and the running of additional software calculations for identifying thermodynamically stable species in T-111 and Ta-10%W welds.

#### Patents: none

#### Publications/Presentations:

- 1) A poster was presented at the Materials Science & Technology 2006 Conference and Exhibition entitled, "Investigation of Effects of Neutron Radiation on Tantalum Alloys for Radioisotope Power System Applications" by Chadwick Barklay (University of Dayton), Daniel Kramer (University of Dayton), and Joseph Talnagi (The Ohio State University), Cincinnati, OH, October 2006.
- 2) A poster was presented at the Futures Technologies Conference II sponsored by the National Nuclear Security Agency (NNSA) entitled, "Thermodynamic Prediction and Transmission Electron Microscopy Results of Compositional Phases in Tantalum-based Alloys Welds for Power Source Application," by W.E. Moddeman (Pantex), C.D. Barklay (University of Dayton), J.C. Birkbeck (Pantex), R.G. Miller (ORNL), L.F. Allard (ORNL), and D.P. Kramer (University of Dayton), Washington, D.C., October 2006.

**Deliverable Reports**:

Number	Report	Planned Completion	Actual Completion	
1	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05	
2	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05	
3	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06	
4	4 <sup>th</sup> Quarterly Report	04/30/06	04/18/06	
5	5 <sup>th</sup> Quarterly Report	07/30/06	07/25/06	
6	6 <sup>th</sup> Quarterly Report	10/30/06	10/21/06	
7	7 <sup>th</sup> Quarterly Report	01/30/07	01/23/07	
	Final Report	12/30/07		
	SF-269A	12/30/07		

**Budget Data (as of December 31, 2006):** 

			Actual S	pent to Date		
Budget Period:		DOE	Quarterly Amount	Project Total Spent		
		Budgeted Amount	Spent	Spent		
From	То					
05/12/05		\$100,000				
05/12/05	06/30/05		\$3,907	\$3,907		
07/01/05	09/30/05		\$25,958	\$29,865		
10/01/05	12/31/05		\$32,080	\$61,945		
01/01/06	03/31/06		\$30,145	\$92,090		
05/12/06		\$100,000				
04/01/06	06/30/06		\$16,842	\$108,932		
07/01/06	09/30/06		\$5,508*	\$114,440		
10/01/06	12/31/06		\$7,938*	\$122,378		
Total: \$200,000 Total Budget Left: \$77,62						
	* Significant "matching" dollars obtained from					
	Pantex helped	to support this effort d	uring the Quar	ter		

#### **QUARTERLY PROGRESS REPORT**

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

Covering Period: April 1, 2007 through June 30, 2007

Date of Report: July 16, 2007

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-34 contact – Dirk Cairns-Gallimore

Contacts: ID Contract Specialist – Patricia Alexander-Johnson

Project Objective: This project addresses several of DOE-NE-34's near-term and future

materials science and engineering related programmatic needs/goals.

#### Background:

The Office of Space and Defense Power Systems (NE-34) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific know-how, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-34 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

Status: During this reporting quarter investigations continued and below are several focal points;

a) Scanning Electron Fractography of Irradiated and Non-Irradiated Ta-10%W and T-111

The fracture surfaces of neutron irradiated and non-irradiated rupture tested specimens of Ta-10%W and T-111 were analyzed using Scanning Electron Microscopy (SEM) at the University of Dayton Research Institute (UDRI). In general, the SEM images, shown in Figures 1 through 4 taken of the fracture surfaces of the stress rupture tested specimens of irradiated and non-irradiated Ta-10%W and T-111 exhibit a ductile failure mode based on the dimples observed throughout the fracture surfaces. All of the fracture surfaces are considerably rough and exhibit elongated dimples of various sizes with relatively flat regions in between. Such features are indicative of ductile failure mechanisms associated with uniaxial tests.

When an alloy is subjected to mechanical overload, it may fail by a process called microvoid coalescence, which is the result of complex slip. Voids nucleate at regions of localized strain discontinuity, mostly associated with second phase particles, inclusions, grain boundaries and dislocation pile-ups. With increasing strain, the microvoids grow, coalesce and eventually form a continuous fracture surface, which is characterized by numerous cuplike depressions, also referred to as dimples. The failure mode of all creep specimens associated with this research can be classified as transgranular microvoid coalescence.

What is consistently observed between irradiated and non-irradiated specimens of both Ta-10%W and T-111 is the emergence of a bimodal distribution of the mean dimple size of both irradiated alloys when compared with their non-irradiated counterparts.

#### Fractography of Ta-10%W

As shown in Figure 1, the fracture surface of non-irradiated Ta-10%W has a fairly narrow dimple size distribution, 2µm maximum estimated diameter. This type of uniform fracture surface is characteristic of a ductile material. The photomicrograph in Figure 2, shows the fracture surface of a neutron irradiated Ta-10%W specimen with an approximate atomic displacement damage of 3.0 x 10<sup>-7</sup>dpa. The micrograph shows a bimodal distribution of small and large dimples on the fracture surface and evidence of poorly defined cleavage facets connected by deeply indented dimples. The estimated mean dimple diameters are 2µm and 7µm.

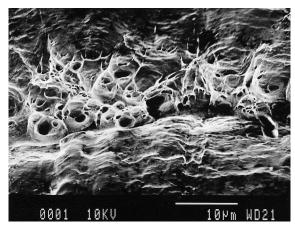


Figure 1. SEM photomicrograph of fracture surface of a rupture tested <u>non-irradiated</u> Ta-10%W sample (5C).

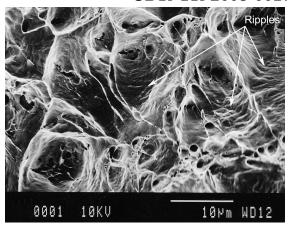


Figure 2. SEM photomicrograph of fracture surface of a rupture tested <u>irradiated</u> Ta-10%W sample (8C).

## Fractography of T-111

Similar to Ta-10%W the non-irradiated T-111 rupture tested sample shown in Figure 3 has a fairly narrow dimple size distribution,  $3\mu m$  estimated mean diameter, which is also characteristic of a ductile failure mode. The photomicrograph in Figure 4 shows the fracture surface of an irradiated T-111 specimen with an approximate atomic displacement damage of 3.0 x 10-7 dpa. The photomicrograph shows a bimodal distribution of small and large dimples on the fracture surface and evidence of poorly defined cleavage facets connected by deeply indented dimples. The estimated mean dimple diameters are <1 $\mu m$  and  $7\mu m$ .

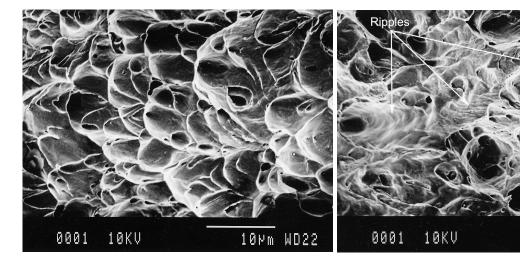


Figure 3. SEM photomicrograph of fracture surface of a rupture tested <u>non-irradiated</u> T-111 sample (T1C).

Figure 4. SEM photomicrograph of fracture surface of a rupture tested <u>irradiated</u> T-111 sample (T4C).

10 km MD23

#### Discussion

The fracture surfaces of the irradiated alloys, which exhibit a statistical difference in elongation in T-111 and dramatic increase in rupture time (Ref. Quarterly Progress Report for Materials Technology Support for Radioisotope Power Systems dated January 1, 2007 through March 31, 2007), clearly exhibit a more planar aspect with shallower dimples and poorly defined cleavage facets, when compared to their non-irradiated counterparts. This is indicative of materials that have experienced a loss of ductility.

It can also be seen that qualitative differences between the irradiated and non-irradiated fracture surfaces are more pronounced in T-111 than in Ta-10%W. Although the fracture surface of the irradiated and non-irradiated samples are different, the failure mode of all samples are essentially transgranular and ductile. The fracture surface of the irradiated creep test specimens shown in Figures 2 and 4 exhibit various dimple sizes. This variation in dimple size results from a non-uniform distribution of nucleating particles and the nucleation and growth of isolated microvoids early in the loading cycle, which is a function of the interaction between dislocations and small defect clusters, produced by low temperature neutron irradiation.

Another interesting observation regarding the irradiated fracture surfaces shown in Figures 2 and 4 is that deformation markings are visible on the dimple walls of both photomicrographs. These markings occur when slip-planes at the surface of the dimples are favorably oriented to the major stress direction and result in slip-plane displacement at the surface of the dimple as deformation proceeds. This suggests that transgranular fracture occurred by ductile rupture along coarse slip bands with ductile tearing between slip band fractures. This is another indication of a reduction of ductility in the irradiated samples due to the interaction between dislocations and small defect clusters. produced temperature neutron irradiation. Figure 5 schematically shows this phenomenon.

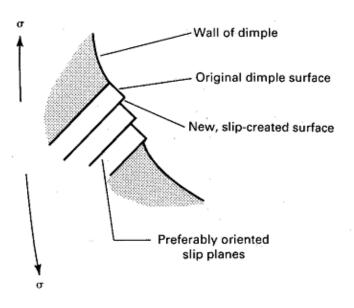


Figure 5. Slip step formation resulting in serpentine glide and ripples on a dimple wall.

b) Discussions are continuing with Pantex who may in FY'08 again help support various aspects of research of mutual interest to this project centered on investigating the effects of neutron irradiation on the mechanical and physical properties of welded tantalum alloys.

c) A technical poster based on aspects of this project's research entitled "Neutron Irradiation of Refractory Tantalum Alloys (T-111 and Ta/10W) for Radioisotope Space Power System Applications," won an Outstanding Poster award in the category of Space Exploration at the 2007 National Space and Missile Materials Symposium held June 2007.

## **Program Recommendations:**

- a) Dr. Chad Barklay (Ph.D. graduate student working on this project) completed his Ph.D. thesis entitled "Investigation of Low Level Neutron Radiation on Tantalum Alloys for Radioisotope Power System Applications" and completed all of his Ph.D. requirements during the calendar quarter. Dr. Barklay's contribution to this program has been scientifically outstanding and as a graduate student the cost benefit ratio to the program has been exceptionally advantageous. In order to assure continuation of the present line of research, it would be especially valuable to the project to take on another graduate student. A commitment to a graduate student typically entails the confidence of continued funding into at least the next fiscal year or two.
- b) To become more familiar with the various NE-34 program's materials related issues, it is suggested that our attendance at appropriate program meetings be encouraged. In addition, reinstating clearances could also greatly facilitate future program related discussions.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including; continued mechanical testing of Ta-10%W and T-111 specimens that have been irradiated. It is also anticipated that preliminary testing may be initiated on welded T-111 and welded Ta/10W specimens that have been neutron irradiated.

It is requested that discussions be held between NE-34 and the University of Dayton to determine future anticipated funding levels for FY'08 and FY'09 and future technical guidance.

Patents: none

#### Publications/Presentations:

- 1) C. D. Barklay, "Investigation of Low Level Neutron Radiation on Tantalum Alloys for Radioisotope Power System Applications," Ph.D. Thesis, University of Dayton, Dayton OH, May 2007, 191 pages.
- 2) C. D. Barklay, D.P. Kramer, and J. Talnagi, "Neutron Irradiation of Refractory Tantalum Alloys (T-111 and Ta/10W) for Radioisotope Space Power System Applications," 2007 National Space and Missile Materials Symposium, June 2007.

**Deliverable Reports:** 

Number	Report	Planned Completion	Actual Completion
1	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05
2	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05
3	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06
4	4 <sup>th</sup> Quarterly Report	04/30/06	04/18/06
5	5 <sup>th</sup> Quarterly Report	07/30/06	07/25/06
6	6 <sup>th</sup> Quarterly Report	10/30/06	10/21/06
7	7 <sup>th</sup> Quarterly Report	01/30/07	01/23/07
8	8 <sup>th</sup> Quarterly Report	04/30/07	04/17/07
9	9 <sup>th</sup> Quarterly Report	07/31/07	07/16/07
	Final Report	12/30/07	
	SF-269A	12/30/07	

**Budget Data (as of June 30, 2007):** 

			Actual S	pent to Date
Budget Period:		DOE Budgeted Amount	Quarterly Amount Spent	Project Total Spent
From	То			
05/12/05		\$100,000		
05/12/05	06/30/05		\$3,907	\$3,907
07/01/05	09/30/05		\$25,958	\$29,865
10/01/05	12/31/05		\$32,080	\$61,945
01/01/06	03/31/06		\$30,145	\$92,090
05/12/06		\$100,000		
04/01/06	06/30/06		\$16,842	\$108,932
07/01/06	09/30/06		\$5,508*	\$114,440
10/01/06	12/31/06		\$7,938*	\$122,378
01/01/07	03/31/07		\$12,068*	\$134,446
04/01/07	06/30/07		\$17,466*	\$151,912
		Total: \$200,000		et Left: \$48,088

#### QUARTERLY PROGRESS REPORT

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

POWER SYSTEMS

Covering Period: July 1, 2007 through September 30, 2007

Date of Report: October 17, 2007

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-34 contact – Dirk Cairns-Gallimore

Contacts: ID Contract Specialist – Patricia Alexander-Johnson

Project Objective: This project addresses several of DOE-NE-34's near-term and future

materials science and engineering related programmatic needs/goals.

#### Background:

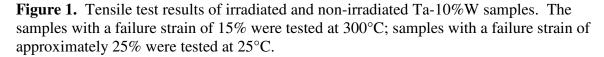
The Office of Space and Defense Power Systems (NE-34) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific know-how, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-34 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

Status: During this reporting quarter investigations continued and below are several focal points;

- a) Chad Barklay and Dan Kramer traveled early August 2007 to DOE/Germantown and briefed DOE/NE-34 project personnel on the technical status of the program. Two informal presentations were given entitled, "University of Dayton Research Institute Overview" and "Materials Technology Support for Radioisotope Power Systems (May 05 July 07)." Discussions centered on the direction of future program technical endeavors.
- b) At NE-34's request technical information was exchanged with representatives of the Heat Source program at INL focused on the potential surface contamination of parts that were machined with Moly-Dee cutting fluid. UDRI obtained information on the potential contamination and shared observations during several telecons.
- c) Pantex has submitted a request for FY08 internal development dollars that could help support various aspects of research of mutual interest to this project. If dollars are eventually appropriated by Pantex, research and development will be performed on investigating the effect of neutron irradiation on the mechanical and physical properties of welded tantalum alloys.
- d) Notification has been received from DOE/INL that the program has been extended through 09/30/08.
- e) Tensile Testing Results of Irradiated and Non-Irradiated Ta-10%W and T-111 Specimens;

Figure 1 graphically represents the tensile test results of irradiated and non-irradiated Ta-10%W samples. As can be observed from the data plots, the testing temperature has a significant impact on the yield strength, UTS, and elongation of the specimens regardless of the level of neutron radiation exposure. An increase in test temperature from 25°C to 300°C did not have an effect on the elastic modulus of Ta-10%W but the variation in test temperature did result in an approximate; 29% reduction in yield strength, a 20% reduction in UTS, and a 40% reduction in elongation.

Statistical Analysis System (SAS) software was used to conduct an analysis of variance (ANOVA) for the Ta-10%W tensile tests. The results infer that the mechanical properties of Ta-10%W tested at 25°C and 300°C are unaffected when irradiated with a neutron fluence of  $\sim 1.2 \text{ x}$   $10^{15}$  nvt at both 25°C and 350°C. Therefore, an atomic displacement damage of approximately 3.0 x  $10^{-7}$  dpa did not produce a statistically significant effect on the tensile properties of Ta-10%W when subjected to a strain rate of 0.0017 s<sup>-1</sup>.



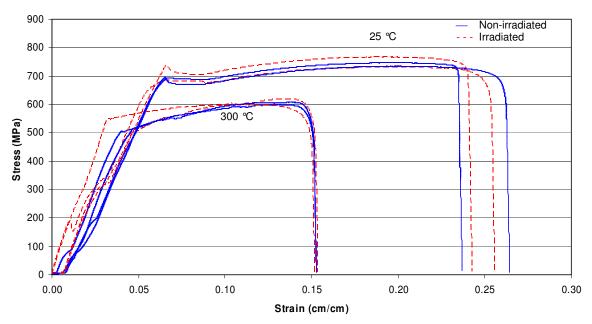
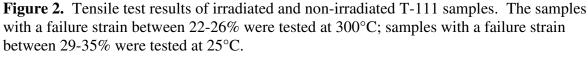
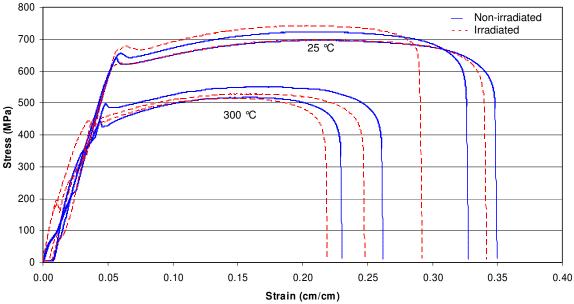


Figure 2 graphically represents the tensile test results of irradiated and non-irradiated T-111 samples. Similar to Ta-10%W, the results show that tensile testing temperature has a significant impact on the yield strength, UTS, and elongation of the specimens regardless of the level of neutron radiation exposure. An increase in tensile test temperature from 25°C to 300°C did not have an effect on the elastic modulus of T-111, but the variation in test temperature results in an approximate; 31% reduction in yield strength, a 28% reduction in UTS, and a 25% reduction in elongation.

Statistical Analysis System (SAS) software was also used to conduct an analysis of variance (ANOVA) for the T-111 tensile tests. These results infer that the tensile property of elongation of T-111 tested at 25°C and 300°C are affected when irradiated with a neutron fluence of ~1.2 x 10<sup>15</sup> nvt at both 25°C and 350°C. The yield strength, UTS, and elastic modulus of T-111 are unaffected by the research parameters for irradiation fluence and irradiation temperature. Therefore, an atomic displacement damage of approximately 3.0 x 10<sup>-7</sup> dpa produces an approximate 6.6% reduction in the ductility of T-111 at 25°C when subjected to a strain rate of 0.0017 s<sup>-1</sup>; the average reduction in elongation was 0.8039 cm (irradiated) compared to 0.8603 cm (non-irradiated). This was determined to be a statistically significant effect at the 0.01 significance level. Additionally, the effects of neutron irradiation in T-111 are more pronounced at an irradiation temperature of 350°C than at 108°C. This was determined to be a statistically significant effect at the 0.05 significance level.





## **Program Recommendations:**

- a) It is suggested that our attendance at appropriate program meetings be encouraged in order to gain additional technical familiarity with the various NE-34 program's materials related issues. This would help facilitate UDRI's utilization as a materials related "Quick Reaction Force (QRF)." Reinstating clearances would also greatly facilitate future program related discussions.
- b) In order to assure continuation of the present line of research, it would be valuable to the project to take on another graduate student. A commitment to a graduate student typically entails the confidence of continued funding into at least the next fiscal year or two. A graduate student has a cost benefit ratio that is exceptionally advantageous.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including;

- Continue mechanical testing of Ta-10%W and T-111 specimens that have been irradiated
- It is also anticipated that preliminary testing may be initiated on welded T-111 and/or welded Ta/10W specimens that have been neutron irradiated
- Investigations into the surface contaminants present on L-605 (Haynes 25) surfaces that are machined using a sulfur/chloride containing cutting fluid (Moly-Dee)

Patents: none

# Publications/Presentations:

1) A paper entitled, "Investigation of Stress Rupture Tested Neutron Irradiated Tantalum Alloys," C.D. Barklay, J.Y. Howe (ORNL), and D.P. Kramer has been submitted to the 2008 Space Technology Applications International Forum (STAIF-2008).

# **Deliverable Reports:**

Number	Report	Planned Completion	Actual Completion
1	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05
2	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05
3	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06
4	4 <sup>th</sup> Quarterly Report	04/30/06	04/18/06
5	5 <sup>th</sup> Quarterly Report	07/30/06	07/25/06
6	6 <sup>th</sup> Quarterly Report	10/30/06	10/21/06
7	7 <sup>th</sup> Quarterly Report	01/30/07	01/23/07
8	8 <sup>th</sup> Quarterly Report	04/30/07	04/17/07
9	9 <sup>th</sup> Quarterly Report	07/31/07	07/16/07
10	10 <sup>th</sup> Quarterly Report	10/30/07	10/17/07
	Final Report	12/30/08	
	SF-269A	12/30/08	_

# **Budget Data (as of September 30, 2007):**

			Actual S	pent to Date
Budget Period:		DOE Budgeted Amount	Quarterly Amount Spent	Project Total Spent
From	То			
05/12/05		\$100,000		
05/12/05	06/30/05		\$3,907	\$3,907
07/01/05	09/30/05		\$25,958	\$29,865
10/01/05	12/31/05		\$32,080	\$61,945
01/01/06	03/31/06		\$30,145	\$92,090
05/12/06		\$100,000		
04/01/06	06/30/06		\$16,842	\$108,932
07/01/06	09/30/06		\$5,508*	\$114,440
10/01/06	12/31/06		\$7,938*	\$122,378
01/01/07	03/31/07		\$12,068*	\$134,446
04/01/07	06/30/07		\$17,466*	\$151,912
07/01/07	09/30/07		\$18,747*	\$170,659
		Total: \$200,000	Total Budge	et Left: \$29,341

<sup>\*</sup> Significant "matching" dollars obtained from Pantex helped to support this effort during the Quarter

#### **QUARTERLY PROGRESS REPORT**

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

Covering Period: October 1, 2007 through December 31, 2007

Date of Report: January 23, 2008

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-34 contact – Dirk Cairns-Gallimore

Contacts: ID Contract Specialist – Patricia Alexander-Johnson

Project Objective: This project addresses several of DOE-NE-34's near-term and future

materials science and engineering related programmatic needs/goals.

#### Background:

The Office of Space and Defense Power Systems (NE-34) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific know-how, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-34 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

Status: During this reporting quarter investigations continued and below are several focal points;

a) At NE-34's request technical information was exchanged with representatives of the Heat Source program at INL focused on the potential surface contamination of parts that were machined with Moly-Dee cutting fluid. UDRI obtained information on the potential contamination and shared observations during several telecons. UDRI also prepared a "Quick Reaction Force" memo dated November 1, 2007 that was sent to NE34 which is discussed below.

#### Subject:

XPS surface scans on Haynes 25 coupons machined with Moly-Dee

#### Purpose:

To determine if machining with Moly-Dee results in Sulfur (S) and/or Chlorine (Cl) surface contamination

#### Experiment:

Haynes 25 sample discs (~0.125" thick by ~0.5" diameter) were machined out of ~0.5" rod stock using Moly-Dee as the coolant. Machined sample discs were prepared via; Procedure #1) first ultrasonic cleaning with acetone plus a second ultrasonic cleaning with isopropanol after which the disc was placed in the XPS instrument and; Procedure #2) the above sample disc was taken out of the XPS and hand surface "polished" using 600 grit SiC paper followed by an ultrasonic cleaning with acetone plus a second ultrasonic cleaning with isopropanol after which the disc was re-inserted into the XPS.

#### Results:

Figure 1 is an S 2p XPS scan that was obtained on a sample that was processed following Procedure #1. Figure 1 shows signs of S consistent with a metallic sulfide (~161 to 162 eV binding energy). There is evidence of a SO<sub>4</sub> (sulfate) peak ~168 eV, as well, although it is weaker (less sulfate than sulfide).

Figure 2 is an S 2p XPS scan that was obtained on a sample that was processed following Procedure #2. Figure 2 shows no obvious signs of S after the polishing and cleaning following Procedure #2. Table 1 presents approximate atom % surface compositions as determined from the XPS survey scans on the Haynes 25 samples that were processed following either Procedure #1 or Procedure #2.

#### **Summary:**

The XPS results show a significant increase (greater than two orders of magnitude) in the atom % of S on the surface of the Haynes 25 samples that were machined using Moly-Dee. The results also indicate that a "mild" mechanical process may be employed to remove the S surface contamination even though its affect on further component processing (i.e. welding, etc.) needs to be investigated.

Figure 1. S 2p scan obtained on Haynes 25 sample that was processed via Procedure #1

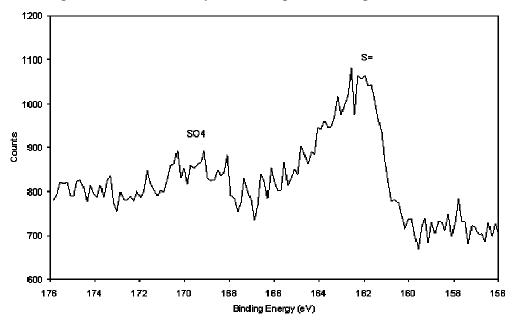
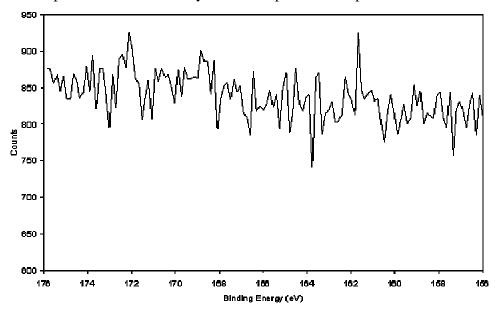


Figure 2. S 2p scan obtained on Haynes 25 sample that was processed via Procedure #2



69

Table 1. Approximate atom % surface compositions as determined from XPS survey scans on Haynes 25 sample discs that were processed via Procedure #1 or Procedure #2.

Sample	Ni	Co	Cr	О	С	Cl	S	W
Procedure #1								
Haynes 25 - MolyDee								
- area 1	1.2	3.2	8.5	31.1	49.9	0.7	3.7	1.7
- area 2	0.9	2.2	3.7	27.8	54.6	0.7	8.8	1.3
Procedure #2								
Haynes 25 - MolyDee								
+ SiC polish								
- area 1	2.4	11.9	5.0	38.0	40.6	< 0.5	< 0.2	1.5
- area 2	2.0	10.8	4.7	37.9	42.5	< 0.4	< 0.3	1.5

- b) Pantex has received initial positive internal feedback to a request for GFY08 development dollars that would help support work of mutual interest to this project. If these dollars are eventually released by Pantex in the form of a purchase order to UDRI, research and development will be performed on investigating the effect of neutron irradiation on the mechanical and physical properties of welded tantalum alloys.
- c) Tensile Creep Testing on Irradiated and Non-Irradiated Ta-10%W and T-111 Specimens

A Ta/10W tensile creep specimen that was irradiated with a neutron fluence of ~1.2 x 10<sup>15</sup> nvt at room temperature (due to self heating actual temperature was 108°C) has exceeded over a total of 11,000 hours of continuous load at 90ksi and is ongoing.

#### Program Recommendations:

- a) It is suggested that our attendance at appropriate program meetings be encouraged in order to gain additional technical familiarity with the various NE-34 program's materials related issues. This would help facilitate UDRI's utilization as a materials related "Quick Reaction Force (QRF)." Reinstating clearances would also greatly facilitate future program related discussions.
- b) In order to assure continuation of the present line of research, it would be valuable to the project to take on another graduate student. A commitment to a graduate student typically entails the confidence of continued funding into at least the next fiscal year or two. A graduate student has a cost benefit ratio that is exceptionally advantageous.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including;

- Continue tensile creep testing of Ta-10%W and/or T-111 specimens that have been irradiated
- Initiate initial studies on DOE/NE supplied "aged" F8 aeroshell material and possible FWPF replacement materials
- Complete investigations into the surface contaminants present on L-605 (Haynes 25) surfaces that are machined using a sulfur/chloride containing cutting fluid (Moly-Dee)

Patents: none

Publications/Presentations: none

**Deliverable Reports:** 

Number	Report	Planned Completion	Actual Completion
1	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05
2	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05
3	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06
4	4 <sup>th</sup> Quarterly Report	04/30/06	04/18/06
5	5 <sup>th</sup> Quarterly Report	07/30/06	07/25/06
6	6 <sup>th</sup> Quarterly Report	10/30/06	10/21/06
7	7 <sup>th</sup> Quarterly Report	01/30/07	01/23/07
8	8 <sup>th</sup> Quarterly Report	04/30/07	04/17/07
9	9 <sup>th</sup> Quarterly Report	07/31/07	07/16/07
10	10 <sup>th</sup> Quarterly Report	10/30/07	10/17/07
11	11 <sup>th</sup> Quarterly Report	01/31/08	01/23/08
	Final Report	12/30/08	
	SF-269A	12/30/08	

**Budget Data (as of December 31, 2007):** 

			Actual Spent to Date	
Budget Period:		DOE Budgeted Amount	Quarterly Amount Spent	Project Total Spent
From	То			
05/12/05		\$100,000		
05/12/05	06/30/05		\$3,907	\$3,907
07/01/05	09/30/05		\$25,958	\$29,865
10/01/05	12/31/05		\$32,080	\$61,945
01/01/06	03/31/06		\$30,145	\$92,090
05/12/06		\$100,000		
04/01/06	06/30/06		\$16,842	\$108,932
07/01/06	09/30/06		\$5,508*	\$114,440
10/01/06	12/31/06		\$7,938*	\$122,378
01/01/07	03/31/07		\$12,068*	\$134,446
04/01/07	06/30/07		\$17,466*	\$151,912
07/01/07	09/30/07		\$18,747*	\$170,659
10/01/07	12/31/07		\$7,649*	\$178,308
Total: \$200,000 Total Budget Left: \$21				

<sup>\*</sup> Significant "matching" dollars obtained from Pantex helped to support this effort during the Quarter

#### **QUARTERLY PROGRESS REPORT**

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

POWER SYSTEMS

Covering Period: January 1, 2008 through March 31, 2008

Date of Report: April 10, 2008

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer / Dr. Chadwick D. Barklay

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-34 contact – Dirk Cairns-Gallimore

Contacts: ID Contract Specialist – Patricia Alexander-Johnson

Project Objective: This project addresses several of DOE-NE-34's near-term and future

materials science and engineering related programmatic needs/goals.

#### Background:

The Office of Space and Defense Power Systems (NE-34) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific know-how, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-34 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

Status: During this reporting quarter investigations continued and below are several focal points;

a) At NE-34's request a significant amount of the efforts during this reporting period were directed at investigating possible replacement materials for the FWPF presently used in the fabrication of the aeroshells. Technical information on a number of potential replacement materials was obtained, examined, and compared to various physical and chemical properties of FWPF as described in the *Acceptance Specification for FWPF Graphite* General Electric NS0060-01-20. It is our understanding that the principle function of the FWPF aeroshell is to protect the GISs during an emergency reentry or launch abort in terms of acting as an oxidation ablative layer. The aeroshell also has secondary functions such as providing some degree of mechanical protection during an impact, etc.

This effort has identified a potential material that at this point appears to be intriguing from a functional point of view. POCOGRAPHITE, Inc. (Decatur, TX) has been developing a family of silicon carbide products that are very attractive on a number of levels. In essence as shown in Figure 1, POCO can take a very pure form of graphite and convert it into silicon carbide. Parts can be readily machined out of the graphite to near net shape and then purified to less than 100's of ppm of impurities. (For comparison, the discussed *Acceptance Specification for FWPF Graphite* has a purity specification under 3.7.1 of a maximum ash content of 3000 ppm). After purification the graphite is converted into a high purity silicon carbide.

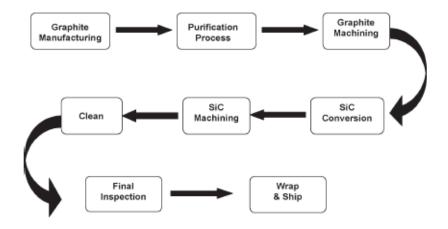


Figure 1. A flowchart of POCO's graphite conversion process.

UDRI has been able to obtain an example of complex components that have been fabricated using the POCO process. Below in Figure 2 on the left is a very intricate cube within a cube within a cube, etc. that was machined out of graphite. Graphite is very easy to machine and it holds tolerances well. On the right of Figure 2 is a "graphite cube within a cube" that was converted into a high purity "silicon carbide cube within a cube". It would be very difficult to

machine a silicon carbide cube as shown in the figure out of a solid block of silicon carbide. By first machining the cube out of graphite and then converting it into silicon carbide it is possible as demonstrated in the example to form very complex silicon carbide shapes.

Figure 2. (Left) Graphite cube within a cube and (Right) after conversion into silicon carbide.



In general, silicon carbide is a ceramic material which is attractive as a possible FWPF replacement for a number of reasons. It has a high melting temperature of ~2540°C (~4600°F), it is relatively chemically inert, the POCO material is mainly β-SiC, and it should exhibit very good oxidation resistance especially compared to the graphitic based FWPF. In addition, the elemental species in silicon carbide, Si and C, have previously been present within RTG's. The present aeroshells are essentially C, and Si is a chemical species in the previously employed SiGe unicouples. So at first glance, employing silicon carbide within a RTG would not add any "foreign" chemical species that have not been used before. Of course, the long term compatibility, vapor pressures, etc. would still need to be investigated.

Initial analysis has determined that the POCO converted graphite to silicon carbide material is very interesting as a possible replacement for FWPF;

- Initially machine an aeroshell, and perhaps aeroshell caps, lock screws, and lock members out of graphite
- Convert the graphite components into SiC
- Machine converted surfaces if needed as there is a reported 0.5% dimension change from the conversion process. The dimensional change is a known quantity that can easily be taken into account during the initial graphite machining thus minimizing or eliminating this processing step.
- Silicon carbide has many attractive physical and mechanical properties
- Keep the GIS material as-is since it is the actual impact component

It is understood that a lot more additional work comparing FWPF and the converted silicon carbide needs to be preformed. This includes the preparation and/or machining of test specimens out of both materials in order to attempt to obtain direct actual mechanical and physical properties comparisons. UDRI would need to obtain up-to-date FWPF component prints from the DOE to perform this work. Testing could include coefficient of thermal expansion, thermal diffusivity, thermal conductivity, density, oxidation testing, etc. Depending on the budget it is anticipated that we will be able to initiate testing of actual materials next calendar quarter.

b) Tensile Creep Testing on Irradiated and Non-Irradiated Ta-10%W and T-111 Specimens

A Ta/10W tensile creep specimen that was irradiated with a neutron fluence of  $\sim 1.2 \times 10^{15}$  nvt at room temperature (due to self heating actual temperature was  $108^{\circ}$ C) has exceeded over a total of 13,000 hours of continuous load at 90ksi and is ongoing.

### **Program Recommendations:**

- a) It is suggested that UDRI attendance at appropriate program meetings be encouraged in order to gain additional technical familiarity with the various NE-34 program's materials related issues. This would help facilitate UDRI's utilization as a material's related "Quick Reaction Force (QRF)." Reinstating clearances would also greatly facilitate future program related discussions.
- b) In order to assure continuation of the present line of research, it would be valuable to the project to take on another graduate student. A commitment to a graduate student typically entails the confidence of continued funding into at least the next fiscal year or two. A graduate student has a cost benefit ratio that is exceptionally advantageous.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including;

- Acquire additional property information on the POCO SiC material either from the literature or obtain some additional converted material to perform actual mechanical and/or physical property tests
- Meet with POCO personnel to learn more about the conversion process and to discuss the number of different converted materials that they presently produce. This may require some travel and would greatly help identify the actual POCO converted materials that could meet the requirements for a FWPF replacement.
- Obtain FWPF components prints
- Continue tensile creep testing of Ta-10%W and/or T-111 specimens that have been irradiated

Patents: none

### <u>Publications/Presentations:</u>

C.D. Barklay, J.Y. Howe, and D.P. Kramer, "Investigation of Stress Rupture Tested Neutron Irradiated Tantalum Alloys," 26<sup>th</sup> Proceeding of Space Technology and Applications International Forum (STAIF-2008), American Institute of Physics Conference Proceedings 969, Albuquerque, NM, February 2008, pp. 439-445.

**Deliverable Reports:** 

Number	Report	Planned Completion	Actual Completion
1	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05
2	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05
3	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06
4	4 <sup>th</sup> Quarterly Report	04/30/06	04/18/06
5	5 <sup>th</sup> Quarterly Report	07/30/06	07/25/06
6	6 <sup>th</sup> Quarterly Report	10/30/06	10/21/06
7	7 <sup>th</sup> Quarterly Report	01/30/07	01/23/07
8	8 <sup>th</sup> Quarterly Report	04/30/07	04/17/07
9	9 <sup>th</sup> Quarterly Report	07/31/07	07/16/07
10	10 <sup>th</sup> Quarterly Report	10/30/07	10/17/07
11	11 <sup>th</sup> Quarterly Report	01/31/08	01/23/08
12	12 <sup>th</sup> Quarterly Report	04/30/08	04/10/08
	Final Report	12/30/08	
	SF-269A	12/30/08	

## **Budget Data (as of March 31, 2008):**

Actual Spent to Da			pent to Date	
Budget Period:		DOE Budgeted Amount	Quarterly Amount Spent	Project Total Spent
From	То			
05/12/05		\$100,000		
05/12/05	06/30/05		\$3,907	\$3,907
07/01/05	09/30/05		\$25,958	\$29,865
10/01/05	12/31/05		\$32,080	\$61,945
01/01/06	03/31/06		\$30,145	\$92,090
05/12/06		\$100,000		
04/01/06	06/30/06		\$16,842	\$108,932
07/01/06	09/30/06		\$5,508*	\$114,440
10/01/06	12/31/06		\$7,938*	\$122,378
01/01/07	03/31/07		\$12,068*	\$134,446
04/01/07	06/30/07		\$17,466*	\$151,912
07/01/07	09/30/07		\$18,747*	\$170,659
10/01/07	12/31/07		\$7,649*	\$178,308
01/01/08	03/31/08		~\$9,116	~\$187,424
Total: \$200,000   Total Budget Left:~\$12,57			t Left:~\$12,576	

<sup>\*</sup> Significant "matching" dollars obtained from Pantex helped to support this effort during the Quarter

#### **QUARTERLY PROGRESS REPORT**

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

Covering Period: April 1, 2008 through June 30, 2008

Date of Report: July 10, 2008

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer / Dr. Chadwick D. Barklay

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-34 contact – Dirk Cairns-Gallimore

Contacts: ID Contract Specialist – Patricia Alexander-Johnson

Project Objective: This project addresses several of DOE-NE-34's near-term and future

materials science and engineering related programmatic needs/goals.

#### Background:

The Office of Space and Defense Power Systems (NE-34) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific know-how, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-34 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

Status: During this reporting quarter investigations continued and below are several focal points;

a) The effort during this reporting period was directed at investigating possible replacement materials for the FWPF presently used in the fabrication of the aeroshells. This work has identified a potential material that at this point appears to be intriguing from a functional point of view. As discussed in the last quarterly, POCOGRAPHITE, Inc. (Decatur, TX) has been developing a family of silicon carbide products that are very attractive on a number of levels. POCO can take a very pure form of graphite and convert it into silicon carbide. In general, silicon carbide is a ceramic material which is attractive as a possible FWPF replacement for a number of reasons. It has a high melting temperature of ~2540°C (~4600°F) and it is relatively chemically inert as the POCO material is mainly  $\beta$ -SiC.

During the present reporting period samples of FWPF and POCO SC-1 were obtained and machined into various test specimens (Figures 1 and 2). SC-1 is isotropic but FWPF is anisotropic requiring that during machining and testing the "directional axis" was maintained. Coefficient of Thermal Expansion (CTE), Thermal Diffusivity, and Specific Heat test specimens were prepared to various ASTM standards. The results of these experiments are described below.

Figure 1. (Left) Thermal diffusivity and (Right) CTE test samples fabricated out of POCO SC-1.

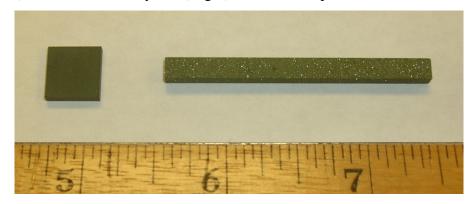
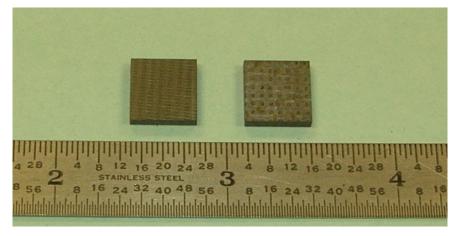


Figure 2. (Left) FWPF XY and (Right) FWPF Z thermal diffusivity test samples.



### **Coefficient of Thermal Expansion:**

Coefficients of thermal expansion (CTE) measurements were made as described in ASTM E 228, "Standard Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer." CTE was measured over a temperature range of 20-1000°C in a 95/5 argon/hydrogen atmosphere. This gas was employed to eliminate any oxidation of the FWPF specimens during the high temperature testing procedure. The equipment used for these tests is a Linseis L75 dual pushrod differential dilatometer. The test specimens were measured differentially using an aluminum oxide standard. Testing in this configuration calculates thermal expansion by comparing the specimen to the standard. A heating rate of 2°C/min was used for these tests. Figure 3 shows a specimen being loaded into the dilatometer and ready for testing within the dilatometer. The results obtained on the SC-1 and FWPF X-Y direction and FWPF Z direction specimens are presented in Figures 4, 5, and 6.

Figure 3. (Left) Specimen being loaded into the Dilatometer and (Right) specimen prior to testing.



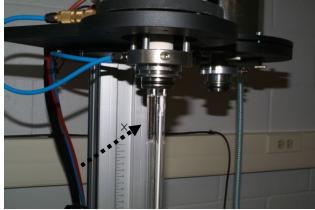
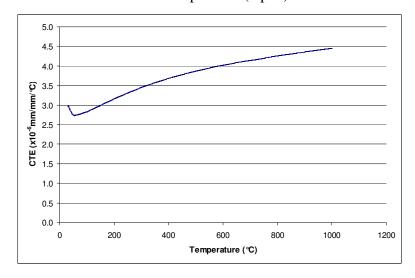


Figure 4. Coefficient of Thermal Expansion (alpha) of SC-1 which is isotropic.



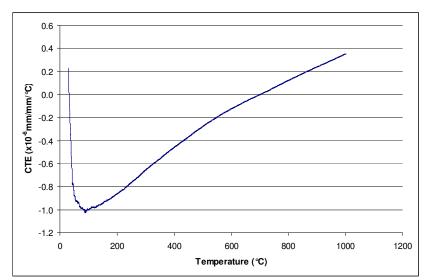
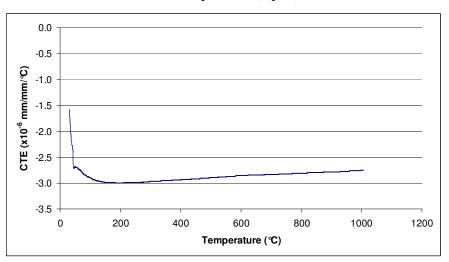


Figure 5. Coefficient of Thermal Expansion (alpha) of FWPF in the X-Y direction.

Figure 6. Coefficient of Thermal Expansion (alpha) of FWPF in the Z direction.



It is interesting to observe that the CTE of FWPF X-Y can be negative or positive as a function of temperature. Perhaps more significantly, FWPF Z is basically <u>negative</u> at a magnitude that should be taken into consideration during "pre-load" operations during the building of power units.

#### **Thermal Conductivity:**

Thermal diffusivity was measured as described in ASTM E1461 "Standard Test Method for Thermal Diffusivity of Solids by the Flash Method." The specimens were evaluated at 30, 100, 200, and 300°C. Measurements were made using a Netzsch LFA 447 flash diffusivity unit. The LFA 447 utilizes a xenon flash lamp to provide heat pulse which is measured using an IR detector. The specimen thickness was measured using micrometers in the center of the specimen. The specimens were then coated on both sides using spray graphite. The flat black surface ensures the heat pulse is evenly distributed over the entire surface. Thermal diffusivity is calculated using the software provided with the LFA 447. These tests employed the Cowen + pulse correction model for the thermal diffusivity calculation. The LFA 447 being loaded with test specimen is shown in Figure 7. The thermal diffusivity results are shown in Table 1.



Figure 7. LFA 447 Thermal Diffusivity unit being loaded.

Density measurements were made geometrically. A Sartorius analytical balance (Model CP124S, S/N 15650688) was used to weigh the specimens in air. The specimen dimensions were measured using micrometers (Mitutoya Model 293-721-10, S/N 2365678). The specimen volume was calculated and density was calculated. The densities of SC1, FWPF XY, and FWPF Z were 2.50, 2.00, and 1.99 g/cc respectively.

Specific heat was measured as described in ASTM E 1269, "Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry". Testing was completed using a TA Instruments Differential Scanning Calorimeter (Model Q1000, SN 1000 0138) over a temperature range of 20 to 500°F with data being collected every 10°C. Before beginning the tests, the DSC was calibrated using a sapphire standard. Tests were completed using

a heating rate of  $10^{\circ}$ C/min in a dry nitrogen atmosphere at a flow rate of 50 ml per minute. The specific heat results are presented in Table 1.

Thermal conductivity results are shown in Table 1 and Figure 8 and the reported values were calculated from the experimentally determined values of thermal diffusivity, specific heat, and density using the following equation:

$$k = \alpha \rho C_p$$
 (Equation 1)

where:

k =thermal conductivity,

 $\alpha$  = thermal diffusivity,

 $\rho$  = density and,

 $C_p$  = specific heat

Table 1. Density, Thermal Diffusivity, and Specific Heat test results with Thermal Conductivity values calculated using Equation 1 above.

0 1	Temperature	Material		
	(℃)	SC1	FWPF XY	FWPF Z
Density (g/cc)	21	2.50	2.00	1.99
Thermal Diffusivity (mm <sup>2</sup> /s)		64.2	92.1	51.7
Specific Heat (J/g/℃)	30	0.732	0.816	0.816
Thermal Conductivity (W/mK)		117	150	84
Thermal Diffusivity (mm <sup>2</sup> /s)		45.4	69.4	39.8
Specific Heat (J/g/℃)	100	0.878	1.030	1.030
Thermal Conductivity (W/mK)		100	143	82
Thermal Diffusivity (mm <sup>2</sup> /s)		31.8	51.7	30.3
Specific Heat (J/g/℃)	200	0.984	1.256	1.256
Thermal Conductivity (W/mK)		78	130	76
Thermal Diffusivity (mm <sup>2</sup> /s)		26.2	44.0	27.3
Specific Heat (J/g/℃)	300	1.082	1.469	1.469
Thermal Conductivity (W/mK)		71	129	80

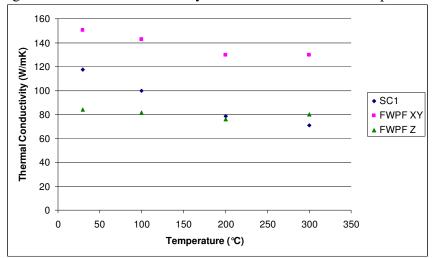


Figure 8. Thermal conductivity results as a function of temperature.

It is understood that additional work comparing FWPF and the converted silicon carbide needs to be preformed. This includes the preparation and/or machining of test specimens out of both materials in order to attempt to obtain other direct actual mechanical and physical properties comparisons. UDRI also needs to obtain up-to-date FWPF component prints from the DOE to perform this work. Depending on the budget it is anticipated that we will be able to perform additional testing on these materials next calendar quarter.

b) Tensile Creep Testing on Irradiated and Non-Irradiated Ta-10%W and T-111 Specimens

A Ta/10W tensile creep specimen that was irradiated with a neutron fluence of  $\sim 1.2 \times 10^{15}$  nvt at room temperature (due to self heating actual temperature was  $108^{\circ}$ C) has exceeded over a total of 15,000 hours of continuous load at 90ksi and is ongoing.

c) Discussions are underway with BWXT Pantex on possible additional funding for a project that would investigate the effects of neutron irradiation on the mechanical properties of welded T-111 and possibly Ta-10W test specimens.

#### <u>Program Recommendations:</u>

- a) It is suggested that UDRI attendance at appropriate program meetings be encouraged in order to gain additional technical familiarity with the various NE-34 program's materials related issues. This would help facilitate UDRI's utilization as a material's related "Quick Reaction Force (QRF)." Reinstating clearances would also greatly facilitate future program related discussions.
- b) In order to assure continuation of the present line of research, it would be valuable to the project to take on another graduate student. A commitment to a graduate student typically entails

the confidence of continued funding into at least the next fiscal year or two. A graduate student has a cost benefit ratio that is exceptionally advantageous.

<u>Plans for Next Quarter:</u> During the next quarter R&D efforts will continue in several areas including;

- Continue to acquire additional property information on the POCO SC-1 material either from the literature or obtain some additional converted material to perform actual mechanical and/or physical property tests
- Meet with POCO personnel to learn more about the conversion process and to discuss the number of different converted materials that they presently produce. This may require some travel and would greatly help identify the actual POCO converted materials that could meet the requirements for a FWPF replacement.
- Continue tensile creep testing of tantalum specimens that have been irradiated

Patents: none

Publications/Presentations: none

#### **Deliverable Reports:**

Number	Report	Planned Completion	Actual Completion
1	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05
2	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05
3	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06
4	4 <sup>th</sup> Quarterly Report	04/30/06	04/18/06
5	5 <sup>th</sup> Quarterly Report	07/30/06	07/25/06
6	6 <sup>th</sup> Quarterly Report	10/30/06	10/21/06
7	7 <sup>th</sup> Quarterly Report	01/30/07	01/23/07
8	8 <sup>th</sup> Quarterly Report	04/30/07	04/17/07
9	9 <sup>th</sup> Quarterly Report	07/31/07	07/16/07
10	10 <sup>th</sup> Quarterly Report	10/30/07	10/17/07
11	11 <sup>th</sup> Quarterly Report	01/31/08	01/23/08
12	12 <sup>th</sup> Quarterly Report	04/30/08	04/10/08
13	13 <sup>th</sup> Quarterly Report	07/30/08	07/10/08
	Final Report	12/30/08	
	SF-269A	12/30/08	

# Budget Data (as of June 30, 2008):

	Actual Spent to Da			pent to Date
Budget Period:		DOE Budgeted Amount	Quarterly Amount Spent	Project Total Spent
From	То			
05/12/05		\$100,000		
05/12/05	06/30/05		\$3,907	\$3,907
07/01/05	09/30/05		\$25,958	\$29,865
10/01/05	12/31/05		\$32,080	\$61,945
01/01/06	03/31/06		\$30,145	\$92,090
05/12/06		\$100,000		
04/01/06	06/30/06		\$16,842	\$108,932
07/01/06	09/30/06		\$5,508*	\$114,440
10/01/06	12/31/06		\$7,938*	\$122,378
01/01/07	03/31/07		\$12,068*	\$134,446
04/01/07	06/30/07		\$17,466*	\$151,912
07/01/07	09/30/07		\$18,747*	\$170,659
10/01/07	12/31/07		\$7,649*	\$178,308
01/01/08	03/31/08		\$9,116	\$187,424
04/01/08	06/30/08		\$5,307	\$192,731
		Total: \$200,000	Total Budge	t Left: ~\$7,269

<sup>\*</sup> Significant "matching" dollars obtained from Pantex helped to support this effort during the Quarter

#### **QUARTERLY PROGRESS REPORT**

Project Title: MATERIALS TECHNOLOGY SUPPORT FOR RADIOISOTOPE

**POWER SYSTEMS** 

Covering Period: July 1, 2008 through September 30, 2008

Date of Report: October 10, 2008

Recipient: University of Dayton Research Institute

300 College Park Dayton, OH 45469

Award Number: DE-FG07-05ID14642

Subcontractors: none

Other Partners: none

Contact(s): Dr. Daniel P. Kramer / Dr. Chadwick D. Barklay

937-229-1038

daniel.kramer@udri.udayton.edu

DOE Project NE-34 contact – Dirk Cairns-Gallimore

Contacts: ID Contract Specialist – Patricia Alexander-Johnson

Project Objective: This project addresses several of DOE-NE-34's near-term and future

materials science and engineering related programmatic needs/goals.

#### Background:

The Office of Space and Defense Power Systems (NE-34) is responsible for supplying radioisotope power systems (RPS's) in support of our country's space exploration and defense missions. Over the last forty years several different systems have been developed and employed on a number of missions. The design, development, evaluation, and testing of RPS's requires expertise in various areas of materials science and engineering. The University of Dayton Research Institute (UDRI), the research arm of the University of Dayton, provides a unique combination of materials based scientific know-how, technical capabilities and facilities, direct hands-on radioisotope power systems program experience, and historical RPS program knowledge that resides in several of the personnel at the University. As NE-34 plans to meet its programmatic and technical challenges over the next decade, UDRI provides broad technical and personnel support to help ensure the continued success of future radioisotope power system programs.

Status: During this reporting quarter investigations continued and below are several focal points;

a) Efforts continued during this reporting period investigating possible replacement materials for the FWPF presently used in the fabrication of the GPHSs. Previously reported work has identified a POCOGRAPHITE, Inc. (Decatur, TX) graphite product that can be converted into silicon carbide. Initial physical property experiments including Coefficient of Thermal Expansion (CTE), Thermal Diffusivity, and Specific Heat on both FWPF and POCO SC-1 have been obtained showing favorably results. A set of initial oxidation experiments was completed during this reporting period on both FWPF and POCO SC-1. The experiments were performed by first drying the test specimens in an oven at ~240°F for several hours after which they were weighed and placed directly within a preheated furnace at 2000°F. At various times the test specimens were removed, allowed to cool, and then reweighed. The results of the 2000°F oxidation tests are shown in Figure 1 and they show that the POCO SiC material showed no oxidation while FWPF oxidized significantly as a function of time. Figure 2 shows pictures of the test materials both before and after the oxidation tests.



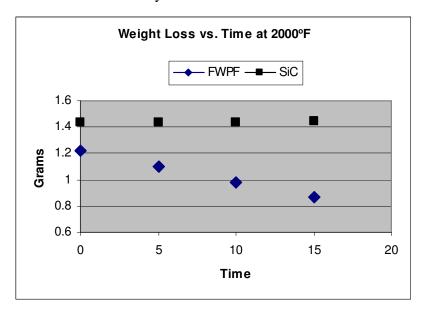
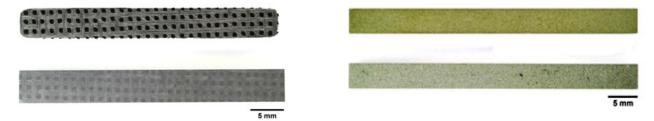


Figure 2. (Left-bottom) FWPF before the 2000°F oxidation tests and (Left-top) after the oxidation tests showing significant material removal. (Right-bottom) POCO SiC before the 2000°F oxidation tests and (Right-top) after the oxidation tests showing insignificant effects.



- b) It is readily understood that the above oxidation tests are only initial screening experiments and that they do not represent the time-temperature profile of what has been modeled to occur in a launch mishap. In order to simulate more closely a launch mishap, discussions were held with personnel from the Laser Hardened Materials Evaluation Laboratory (LHMEL) at Wright-Patterson Air Force Base. The LHMEL has two main CO2 lasers (~10kW and ~150kW) that can be programmed in terms of output power vs. time. From our discussions we have been able to obtain a commitment to employ at no cost their 10kW for a small series of additional oxidation tests. This is a significant benefit to the program as current daily cost for the 10kW CO2 laser is \$5,000 per day. Test specimens of both FWPF and POCO SiC based materials are being fabricated. These additional tests will enable us on a first order basis to better simulate the thermal-time pulse that a GPHS would see in a launch mishap. If these experiments prove useful we should consider employing their high power laser for future tests that may be able to more closely simulate a launch accident condition. Experimental data from these tests could also be used to help "benchmark" the safety models.
- c) Jay Doucette from Textron visited with Chad Barkley/Dan Kramer at UDRI to discuss a DOD project that is outside the scope of this work. During our initial discussions it was uncovered that Jay is one of the main people at Textron currently working on the production order for the FWPF billets. He is intimately familiar with FWPF production technology and appears to have been instrumental a number of years ago in the saving and storing of the piercing machines. This contact could be helpful in future endeavors.
- d) Tensile Creep Testing on Irradiated and Non-Irradiated Ta-10%W and T-111 Specimens

A Ta/10W tensile creep specimen that was irradiated with a neutron fluence of  $\sim 1.2 \times 10^{15}$  nvt at room temperature (due to self heating actual temperature was  $108^{\circ}$ C) has exceeded over a total of 17,000 hours of continuous load at 90ksi and is ongoing.

#### Program Recommendations:

- a) It is suggested that UDRI attendance at appropriate program meetings be encouraged in order to gain additional technical familiarity with the various NE-34 program's materials related issues. This would help facilitate UDRI's utilization as a material's related "Quick Reaction Force (QRF)." Reinstating clearances would also greatly facilitate future program related discussions.
- b) In order to assure continuation of the present line of research, it would be valuable to the project to take on another graduate student. A commitment to a graduate student typically entails the confidence of continued funding into at least the next fiscal year or two. A graduate student has a cost benefit ratio that is exceptionally advantageous.

<u>Plans for Next Quarter:</u> UDRI will submit the Final Project Report and other required documentation for DE-FG07-05ID14642 which is ending.

Patents: none

Publications/Presentations: none

### **Deliverable Reports:**

Number	Report	Planned Completion	Actual Completion
1	1 <sup>st</sup> Quarterly Report	07/30/05	07/18/05
2	2 <sup>nd</sup> Quarterly Report	10/30/05	10/18/05
3	3 <sup>rd</sup> Quarterly Report	01/30/06	01/18/06
4	4 <sup>th</sup> Quarterly Report	04/30/06	04/18/06
5	5 <sup>th</sup> Quarterly Report	07/30/06	07/25/06
6	6 <sup>th</sup> Quarterly Report	10/30/06	10/21/06
7	7 <sup>th</sup> Quarterly Report	01/30/07	01/23/07
8	8 <sup>th</sup> Quarterly Report	04/30/07	04/17/07
9	9 <sup>th</sup> Quarterly Report	07/31/07	07/16/07
10	10 <sup>th</sup> Quarterly Report	10/30/07	10/17/07
11	11 <sup>th</sup> Quarterly Report	01/31/08	01/23/08
12	12 <sup>th</sup> Quarterly Report	04/30/08	04/10/08
13	13 <sup>th</sup> Quarterly Report	07/30/08	07/10/08
14	14 <sup>th</sup> Quarterly Report	10/30/08	10/07/08
	Final Report	12/30/08	
	SF-269A	12/30/08	

## **Budget Data (as of September 30, 2008):**

Actual Spent			pent to Date	
Budget Period:		DOE Budgeted Amount	Quarterly Amount Spent	Project Total Spent
From	То			
05/12/05		\$100,000		
05/12/05	06/30/05		\$3,907	\$3,907
07/01/05	09/30/05		\$25,958	\$29,865
10/01/05	12/31/05		\$32,080	\$61,945
01/01/06	03/31/06		\$30,145	\$92,090
05/12/06		\$100,000		
04/01/06	06/30/06		\$16,842	\$108,932
07/01/06	09/30/06		\$5,508*	\$114,440
10/01/06	12/31/06		\$7,938*	\$122,378
01/01/07	03/31/07		\$12,068*	\$134,446
04/01/07	06/30/07		\$17,466*	\$151,912
07/01/07	09/30/07		\$18,747*	\$170,659
10/01/07	12/31/07		\$7,649*	\$178,308
01/01/08	03/31/08		\$9,116	\$187,424
04/01/08	06/30/08		\$5,307	\$192,731
07/01/08	09/30/08		\$7,269	\$200,000
	Total: \$200,000 Total Budget Left: \$0			t Left: \$0

<sup>\*</sup> Significant "matching" dollars obtained from Pantex helped to support this effort during the Quarter