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FUSION MATERIALS SEMIANNUAL PROGRESS REPORT FOR THE PERIOD ENDING

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FOREWORD

This is the sixty-sixth in a series of semiannual technical progress reports on fusion materials science activity supported by the Fusion Energy Sciences Program of the U.S. Department of Energy. It covers the period ending June 30, 2019. This report focuses on research addressing the effects on materials properties and performance of exposure to the neutronic, thermal and chemical environments anticipated in the chambers of fusion experiments and energy systems. This research is a major element of the national effort to establish the materials knowledge base for an economically and environmentally attractive fusion energy source. Research activities on issues related to the interaction of materials with plasmas are reported separately.

The results reported are the products of a national effort involving a number of national laboratories and universities. A large fraction of this work, particularly in relation to fission reactor irradiations, is carried out collaboratively with partners in Japan, Russia, and the European Union. The purpose of this series of reports is to provide a working technical record for the use of program participants, and to provide a means of communicating the efforts of fusion materials scientists to the broader fusion community, both nationally and worldwide.

This report has been compiled under the guidance of F. W. (Bill) Wiffen and Stephanie Melton, Oak Ridge National Laboratory. Their efforts, and the efforts of the many persons who made technical contributions, are gratefully acknowledged.

Daniel Clark Research Division Office of Fusion Energy Sciences

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1. FERRITIC/MARTENSITIC STEEL DEVELOPMENT

1.1 MICROSTRUCTURAL STABILITY OF NEUTRON-IRRADIATED CNA-0 – A NITROGEN-CONTAINING RAFM STEEL—L. Tan and W. Zhong (Oak Ridge National Laboratory)

OBJECTIVE

Castable nanostructured alloys (CNAs) are being developed at Oak Ridge National Laboratory (ORNL) as a United States (U.S.) reduced-activation ferritic-martensitic (RAFM) steel. The alloys are engineered to contain an increased amount of stable nanoprecipitates using conventional, affordable steelmaking methods. An earl-developed CNA was neutron irradiated and is being examined to evaluate the irradiation response.

SUMMARY

A commercial heat CNA-0 of the first-generation CNAs was used to produce type SS-J2 miniature specimens that were irradiated in the High Flux Isotope Reactor (HFIR) of ORNL. Irradiation conditions were 0.02-7.44 displacements per atom (dpa) at ~390-750°C. Room-temperature tensile tests indicated nearly 26% softening after irradiation to 7.44 dpa at ~490°C. Microstructural characterization using transmission electron microscopy (TEM) revealed slight microstructural changes in the irradiated sample, such as lath structure recovery, dislocation loops, cavities, coarsening of M₂₃C₆, and dissolution of MX. Transmutation of Ta to W in MX was shown by energy dispersive x-ray spectroscopy analysis. Detailed analysis and discussion are to be published.

PROGRESS AND STATUS

Introduction

Unlike later heats of the first-generation CNAs having only carbides as MX nanoprecipitates, CNA-0 is a heat containing primarily MX carbonitrides. This heat exhibited superior tensile and creep properties and comparable Charpy impact toughness when compared with the general RAFM steels. Neutron irradiation experiments were conducted on CNA-0 to evaluate the microstructural stability of the alloy and for correlation with mechanical properties.

Experimental Procedure

A commercial heat (~25 kg) of CNA-0, cast by vacuum induction melting, has a measured composition of Fe-9.01Cr-1.1W-0.46Mn-0.23Si-0.32V-0.1Ta-0.10C-0.05N-0.008O-0.001S-<0.01P in weight percentage (wt%). The ingot was solution annealed at 1150°C for 4 h, followed by hot forging and rolling to a 2.5-cm thick plate, and then normalization at 1050°C for 1 h and tempering at 750°C for 1 h with air cooling. Type SS-J2 miniature specimens were machined from the plate with the specimen length along the rolling direction. Some of the specimens were irradiated to 0.02–7.44 displacements per atom (dpa) at ~390–750°C in the HFIR at ORNL. The irradiation temperatures were determined by characterizing the accompanied SiC samples. So far only the specimen irradiated to 7.44 dpa at ~490°C has been characterized in detail and is presented here.

Results

Figure 1 shows the room temperature tensile curves of the irradiated and control samples, illustrating the reduced yield (565 MPa) and ultimate (697 MPa) strength and increased uniform (7.22%) and total (20.16%) plastic elongation of the irradiated sample compared to the control sample with 773 and 875 MPa yield and ultimate strength and 4.53% and 14.04% uniform and total plastic elongation. The irradiation to 7.44 dpa at ~490°C resulted in nearly 26% softening with ~43% increase in total plastic elongation.



Figure 1. Tensile curves of the irradiated and control samples of CNA-0 tested at room temperature.

The irradiation resulted in slight recovery to the lath structure with a larger lath width, some dislocation loops with sizes of ~15 nm and density on the order of 10^{21} m⁻³, a few cavities with sizes <3.7 nm and density <9×10²⁰ m⁻³, slight coarsening of M₂₃C₆ precipitates to ~141 nm and negligible reduced size of MX precipitates at ~53 nm. Figures 2a-c show examples of matrix (a-b) MX interacting with dislocations and (c) a special core-shell type MX with W/V-rich core and V-rich shell. The core-shell type MX was also observed in the control sample, which has a Ta/V-rich core and V-rich shell, indicating a near "complete" transmutation of Ta to W as a result of the irradiation. Additionally, a few M₂₃C₆ nanoprecipitates were observed in the matrix as shown in Figure 2d. The high-resolution TEM in Figure 2e taken from the center of the matrix M₂₃C₆ indicates partial amorphization of this type of M₂₃C₆. Detailed results analysis, transmutation effects, and microstructure-property correlation are to be included in a journal paper that is being prepared.



Figure 2. Bright-field (a-d) STEM and (e) TEM images of the irradiated sample of CNA-0 showing (a-c) MX and (d-e) $M_{23}C_6$ precipitates in the matrix.

1.2 ALPHA-PRIME (α') **PRECIPITATE FORMATION IN ION IRRADIATED Fe14Cr AND Fe18Cr ALLOYS**—Y. Zhao and S. Zinkle (University of Tennessee), A. Bhattacharya (Oak Ridge National Laboratory)

OBJECTIVE

This work investigates the phase separation process to form α ' precipitates in high purity FeCr alloys under heavy ion irradiation conditions. The formation of α ' precipitates is the result of two competitive processes: radiation enhanced diffusion (mainly controlled by temperature, with some influence of dose rate) and ballistic dissolution (mainly controlled by dose rate and bombarding ion details). We aim to quantify these competing processes by tuning the temperature and dose rate used to irradiate Fe18Cr and Fe14Cr specimens and checking the distribution of Cr atoms after irradiation with atom probe tomography (APT) and irradiation-induced damage with transmission electron microscopy (TEM).

SUMMARY

Ultra-high purity Fe-Cr alloys with 12-18 wt.% Cr (in either solid solution or thermally aged to form preexisting α 'precipitates) were irradiated with 8 MeV Fe ions to a midrange (~1 um) dose of 0.35-3.5 displacements per atom (dpa) between 300-450 °C at 10⁻³, 10⁻⁴ and 10⁻⁵ dpa/s. Following irradiation, APT was employed to characterize the number density, radius and Cr concentration of Cr-rich clusters. Homogeneously distributed α ' precipitates were revealed when radiation-enhanced diffusion dominates over ballistic dissolution (>350°C).

INTRODUCTION

Ferritic-martensitic (FM) steels with 9-14% Cr concentration are promising structural materials for Gen. IV fission and fusion reactors. However, FM steels with this Cr concentration are known to undergo pronounced hardening and embrittlement when irradiated at low temperatures (~300-480 °C), due to the formation of dislocation loops and Cr-rich α ' precipitates.

Previously, it was believed that the decomposition of α and α' phases cannot happen after ion irradiation due to intense ballistic dissolution at the high dose rates typical for heavy ion irradiation. In Pareige's [1] work, the same materials (Fe12Cr) were separately irradiated by both neutrons and self-ions at 573 K and the microstructural results are very different. Many Cr-rich clusters formed after neutron irradiation, while there were no such precipitates after ion irradiation. This absence of α' precipitates may be due to the high dose rate ($10^{-4} - 10^{-3}$ dpa/s) related to the ion irradiation, which makes ballistic dissolution more effective than radiation enhanced diffusion at this temperature. The recent work by Tissot et al. [2] confirmed that low dose rate ion irradiation can indeed give rise to the formation of Cr-rich clusters in FeCr alloys (2 MeV Fe ions, 300°C). The dose rate used in their work ranged from 3 to 6.1×10⁻⁵ dpa/s between the surface and peak damage position, which is much lower than the typical dose rates in ion irradiation experiments.

To identify the critical conditions for precipitate formation or dissolution, we have conducted a series of heavy ion irradiations on Fe-Cr alloys at varying temperatures and dose rates.

PROGRESS AND STATUS

The as-received high purity Fe(12-25)Cr specimens from The French Alternative Energies and Atomic Energy Commission (CEA), Saclay, France were cut with electronic discharge machining (EDM) into disks with 3mm in diameter and 1 mm in thickness. Part of the disk-shaped Fe18Cr and Fe25Cr samples were thermal aged at 500 °C for 100, 300 and 900 hours and the oxidation layer was removed prior to aging to remove possible artifacts from the EDM fabrication. The heat treatment was performed under the guidance of Novy [3] aiming at forming α ' precipitates with different size and number density.

Both the heat-treated and pristine samples were mirror polished finishing with 0.05 µm colloidal silica. The chemical composition and grain size information of the as-received samples are summarized in Table 1.

The as-polished samples were irradiated by 8 MeV Fe ions at conditions listed in Table 2 in the Michigan Ion Beam Laboratory (MIBL). The displacement damage and injected Fe ions concentration profiles are calculated with the Stopping and Range of Ions in Matter (SRIM) 2008 in the simple Kinchin-Pease (KP) ("quick calculation of damage") mode as shown in Figure 1 [4]. The flux and fluence of injected ions are calculated based on achieving 0.35 or 3.5 dpa damage at mid-range (~1µm) with the displacement energy of the target set to be 40 eV.

specimen	Cr (wt. %)	Cr (at. %)	C (ppm)	S (ppm)	O (ppm)	N (ppm)	P (ppm)	grain size(µm)	
Fe12Cr	11.63	12.38	5	2	4			500	
Fe14Cr	14.25	15.15	5	7	4	5	<10	141	
Fe18Cr	17.97	19.05	7	2	6	5		650	
Fe25Cr	24.97	26.33	4	1	3	3		570	

Table 1. Chemical composition and grain size of as-received FeCr specimens

lon Species	Mid-range / Peak dose (dpa)	Fluence (ions.cm ⁻²)	Flux/mid-range dose rate (ions.cm ⁻² .s ^{.1} / dpa.s ^{.1})	Temper- atures (ºC)	Estimated time (h)
8 MeV Fe ions	0.35/ 0.84	8.83x10 ¹⁴	2.52x10 ¹² /10 ⁻³	300 350 450	0.097 (5min50s)
8 MeV Fe ions	0.35/ 0.84	8.83x10 ¹⁴	2.52x10 ¹¹ /10 ⁻⁴	300 350 450	0.97
8 MeV Fe ions	0.35/ 0.84	8.83x10 ¹⁴	2.52x10 ¹⁰ /10 ⁻⁵	450	9.72
8 MeV Fe ions	3.5/ 8.4	8.83x10 ¹⁵	2.52x10 ¹¹ /10 ⁻⁴	350 450	9.72

Table 2. Preliminary irradiation matrix. All materials will be irradiated in a single sample holder for each condition



Figure 1. SRIM based estimates of the depth profile of displacement damage in dpa and implanted Fe ions concentration in pure Fe with 0.35 dpa at mid-range (~1µm depth).

The precipitate microstructure of the irradiated Fe18Cr and Fe14Cr samples was characterized by APT located at Center for Nanophase Materials Sciences (CNMS) at the Oak Ridge National Laboratory (ORNL). The samples for APT analysis were prepared by focused ion beam (FIB). In order to ensure the mid-range irradiated region was analyzed, a depth of 800~900 nm from the sample surface was removed during the final polish with an ion beam probe of 2 KeV, 89 pA. The α ' precipitates are identified and quantified through the cluster analysis algorithm inherent to the Integrated Visualization and Analysis Software (IVAS). Each Cr-enriched precipitate is indexed by a different color. Figure 2 gives the result of Fe18Cr and Fe14Cr irradiated by 8 MeV Fe ions to a total dose of 0.35 dpa with different temperature and dose rate combinations. Figure 3 presents the distribution of α ' precipitates after irradiation at 350°C and 10⁻⁴ dpa/s to final doses of 0.35 dpa and 3.5 dpa. A high density of nanoscale Cr-rich precipitates are visible for all examined irradiation conditions (350-450°C, 10⁻⁵ to 10⁻⁴ dpa/s), with the lowest density and largest precipitates observed for the lowest dose rate and highest irradiation temperature.



Figure 2. α ' precipitates indexed by different colors in Fe14Cr and Fe18Cr specimens irradiated to 0.35 dpa at different temperature and dose rate.



Figure 3. α ' precipitates indexed by different colors in Fe14Cr and Fe18Cr specimens irradiated at 350 °C and 10⁻⁴ dpa/s to 0.35 dpa or 3.5 dpa.

The number density, radius, Cr concentration and volume fraction of clusters were calculated from the Cluster Analysis file generated by IVAS. The parameter d_{max} (maximum distance below which the atoms are considered from the same cluster) and N_{min} (the minimum number of atoms to define one statistically significant cluster) were determined from the cluster count distribution and cluster size distribution respectively [5]. The radius of precipitates is calculated from the number of atoms inside the cluster. All the numerical results are given in Figure 4.



Figure 4. numerical results for the ion irradiated Fe14Cr and Fe18Cr. (a) the radius of the clusters, (b) number density, (c) Cr concentration in the clusters, (d) volume fraction of the precipitates.

Precipitates formed in Fe18Cr at all the five irradiation conditions, which means that the effect of radiation enhanced diffusion is more effective than ballistic dissolution for all the investigated dose rate and temperature combinations.

The following trends can be summarized from these results:

- (a) Low dose rate (10⁻⁵ dpa/s compared to 10⁻⁴ dpa/s) irradiation gives relatively larger precipitates with higher Cr precipitates, lower number density and lower volume fraction.
- (b) Higher temperature (450 °C compare to 350 °C) results in precipitates with much higher Cr concentration and lower volume fraction. Its effect on radius and number density is within the error bar.

- (c) Higher final dose (3.5 dpa compared to 0.35 dpa) leads to precipitates with higher Cr concentration, lower number density and volume fraction. The total dose does not have a significant effect on the radius of precipitates.
- (d) For all investigated conditions, the precipitates formed in Fe14Cr have a lower number density and volume fraction than the Fe18Cr specimen. The effect of bulk Cr concentration on the radius and Cr concentration of the solute clusters is not very significant.

Both low dose rate and high temperature increase the impact of radiation enhanced diffusion for a constant dose irradiation, which accelerated the separation of matrix α and precipitate α ' phases. This can be understood based on the longer time for diffusion at low dose rates and the higher diffusivity at high temperatures, respectively. High final dose can lead to the growth of clusters if they form at a lower dose. High Cr concentration provides a larger driving force for the phase separation process. The lower volume fraction and number density in ion irradiated Fe14Cr or Fe18Cr are consistent with this trend. However, the Cr-rich precipitate radius seems to be constant over all of five irradiation conditions.

Future Work

- The heat-treated Fe18Cr and pristine Fe18Cr and Fe14Cr samples before and after irradiation at higher dose rate and/or lower temperatures will be characterized by APT.
- The collected APT data will be further analyzed to correct the trajectory aberration and to remove the effect of C, N or dislocations on the number density and cluster Cr concentration. Pearson coefficient analysis will be conducted in all the APT datasets.
- The dislocation microstructure in the irradiated Fe14Cr and Fe18Cr specimens will be characterized by TEM.

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1.3 CAVITY DENUDED ZONE IN DUAL BEAM IRRADIATED Fe AND Fe-Cr ALLOYS—Yan-Ru Lin and Steven J. Zinkle (University of Tennessee at Knoxville), Arunodaya Bhattacharya (Oak Ridge National Lab)

OBJECTIVE

The objective of this task is to systematically study the void and bubble denuded zones adjacent to planar sinks such as grain boundaries and surfaces in neutron and ion irradiated Fe and Fe-Cr alloys at different doses, dose rates, and temperatures. The denuded zone width was dependent on temperature, damage rate, material and defect type (void or bubble). Many previous studies have not documented the He bubble denuded zone, which has a typical width 2-6 times smaller than void denuded zones.

SUMMARY

The denuded zone width of cavities in body centered cubic (BCC) pure Fe and high-purity binary Fe-Cr alloys was examined after dual ion irradiation (~0.1 appm He/dpa) to ~35 dpa at temperatures between 470° C and 550° C. The temperature dependence of the width of void denuded zone is proportional to exp(-Em/4KT), where Em is the vacancy migration energy.

PROGRESS AND STATUS

In our previous progress report [1], we quantified the cavity denuded zone of neutron irradiated Cu at 220-350°C. Furthermore, we confirmed that bubble denuded zone, which has a width 2-6 times smaller than void denuded zones were observed adjacent to surface and grain boundaries in irradiated materials.

In this study, we applied dual 8 MeV Ni ions and 3.55MeV He ions (with an energy degrader to produce a range of He ion energies) to irradiate a series of ultra-high purity bcc Fe and Fe-Cr alloys with Cr content ranging from 3-14% Cr (Fe, Fe-3Cr, Fe-10Cr, Fe-14Cr, Fe-10Cr-780appm C and Eurofer 97) at 470°C, 500°C and 550°C. In Figure 1, according to Stopping and Range of Ions in Matter (SRIM) calculations (quick calculation method, displacement energy of 40 eV), the total heavy ion fluence of 9.68×10²⁰ ions/m² produced a damage level of ~35 dpa at the mid-irradiation range. The concentration of He produced by degraded energy He ion beam was roughly constant at 0.1appm He/dpa at depths of 0.5µm to 1.5µm.





To minimize C contamination during ion-irradiations, plasma cleaning was conducted for two hours immediately before loading into the specimen holder. The 2 mm×10 mm specimens were attached with a thin silver paste layer onto a Cu substrate. The Cu substrate with samples were secured with a mask cover on the top (as shown in Figure 2). The irradiated area was 2 mm× 2 mm for each for the specimens. Thermocouple wires were attached outside the area of interest of each of the samples to accurately monitor the temperature during irradiation.



Figure 2. Materials and sample arrangement.

Generally, for ion beam irradiation experiments, surface-denuded zones and effect of implanted ions on void suppression were observed. In this study, near the irradiation surface and grain boundaries, void denuded zones were observed for the Fe and Fe-Cr specimens irradiated at 470°C, 500°C and 550°C, as shown in Figures 3- 5, respectively. Cavities were not observed in the peak implanted Ni ion region (~1.7-2.5µm). Furthermore, from 470°C to 550°C, the general trend showed that cavity size increased, and the density decreased, as expected based on simple cavity coarsening expectations. The average width of the surface denuded zones at various temperatures is given in Figure 5, which indicates that the denuded zone width gradually increased with elevating temperature. In addition, the transmission electron microscopy (TEM) images clearly showed that the void denuded zone width decreased with increasing Cr level, for Fe, Fe-3Cr and Fe-10Cr. However, the void denuded zone width went up for Fe-14Cr. From a comparison of the results in Fe-10Cr with and without C, the addition of carbon increased the denuded zone width by a factor of 4-6 times larger at 470-550°C. Lastly, only a low density of cavities was found in the Eurofer 97 material at 470°C. No cavities were observed in the Eurofer 97 material at 500°C and 550°C.



Figure 3. Near surface void denuded zone in dual beam irradiated Fe and Fe-Cr alloys at 470°C.



500°C - 8MeV Ni³⁺ / 3.55MeV He²⁺

Figure 4. Near surface void denuded zone in dual beam irradiated Fe and Fe-Cr alloys at 500°C.



Figure 5. Near surface void denuded zone in dual beam irradiated Fe and Fe-Cr alloys at 550°C.

Figure 6 summarizes the void denuded zone width of the ion irradiated Fe and Fe-Cr alloys at 470-550°C. Following the relationship that the width of the void denuded zone is proportional to exp(-Em/4KT), the slope of this line gives the vacancy migration energies (Em) of each materials [2]. It is worth to notice that this relationship is valid for recombination-dominant conditions. For sink-dominant conditions, the predicted behavior is independent of temperature. The value of the calculated migration energies from this experiment (assuming recombination-dominant conditions) are listed in Table 1. The vacancy migration energy of pure Fe agrees with the accepted value obtained from experimental data and atomistic modeling (~0.7 eV). In addition, for Fe-Cr alloys, with the increasing of Cr concentration, the variation of migration energy trend obtained by a recent density functional theory (DFT) study seems to qualitatively support our denuded zone width results.[3] The DFT results found that the vacancy migration energy decreased with increasing Cr concentration up to 5%, then the vacancy migration energy tended to slightly increase and stay steady between 7-14% of Cr. Lastly, although our samples were dual beam irradiated, a clear distinction between bubble and void denuded zone was not observed. Similarly, a bimodal size distribution of cavities was not found in all the specimens. This may be due to the relatively low ~0.1 appm He/dpa value investigated in this study.



Figure 6. Void denuded zone width vs. inverse temperature for irradiated Fe and Fe-Cr alloys.

	Vacancy migration energy, E _{m,v} (eV)					
Material	This study	DFT study [2]				
Fe	0.72	0.71				
Fe-3Cr	0.24	0.48				
Fe-10Cr	1.32	0.53				
Fe-14Cr	0.08-0.6	0.51				

Table 1. Vacancy migration energy of Fe and Fe-Cr alloys

In conclusion, void denuded zones were observed adjacent to the surface and grain boundaries in the irradiated Fe and Fe-Cr materials. The denuded zone width was dependent on temperature, Cr and C concentration. It is important to consider the denuded zone width to avoid artifacts in analysis of irradiated materials. In addition, quantification of the defect-free zone width can provide insight on mobility (activation energy) for controlling defect species.

Future Plans

We will complete the study on the same materials with the same irradiation condition (~0.1 appm He/dpa, ~35 dpa midrange dose) at 400 and 435°C. Furthermore, the depth-dependent void size and density in the materials will be quantified. Cavity information of similar dual beam irradiations at ~10 appm He/dpa, ~35 dpa midrange dose will be reported.

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1.4 EFFECTS OF Cr AND C ON DISLOCATION LOOPS IN HEAVY ION IRRADIATED ULTRA-HIGH PURITY FeCr ALLOYS—Yao Li and Steven J. Zinkle (University of Tennessee at Knoxville), Arunodaya Bhattacharya (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this task is to study the effects of Cr and C additions on dislocation loop size and type in high-purity Fe and Fe-Cr model alloys after heavy ion irradiation.

SUMMARY

Dislocation loops are examined in Ultra-high purity (UHP) iron and several Fe-Cr alloys containing 3, 5, 8, and 10 wt% of Cr irradiated by 8MeV Fe ions to midrange doses of 0.35 dpa and 3.5 dpa at 350°C and 450 °C, respectively. Conventional transmission electron microscope (CTEM) was used to characterize the size, density, and type of loops. By using detailed Burgers vector analysis $g \cdot b$ method, we will touch on the effect of temperature, Cr concentration, C concentration, dose rate and dose on the formation of dislocation loops. In addition, Cr segregation onto dislocation loops will be quantified by energy dispersive X-ray spectroscopy (EDX).

PROGRESS AND STATUS

It is known that in pure Fe and Fe-Cr alloys, a/2<111> and a<100> dislocation loops are formed after ion, neutron, and electron irradiations^[1-3]. At elevated temperatures, it is observed that there is a general evolution towards a<100> loops whereas a/2<111> loops are more prevalent near room temperature^[3]. Also, the addition of Cr favors the nucleation of a/2<111> loop and leads to higher loop density. The Fe-Cr specimens were received from Commissariat à l'énergie atomique et aux énergies alternatives (CEA), Saclay France, in the shape of cylindrical rods. The as-fabricated chemical composition, grain size and dislocation density were analyzed by CEA and summarized in Table 1. The samples were cut into 2.7 mm diameter and 1.5 mm thick disks with electronic discharge machining (EDM). Brass wire was applied in EDM work, resulting in a thin copper-bearing coating layer on specimen surface. Due to the high temperature (close to the melting temperature) in the cutting step, copper atoms may diffuse into specimen. Based on Anand and Agarwala's work ^[4] and diffusion calculations, we ground away ~0.5 mm surface layer after EDM work to totally remove the copper diffused-in region. This was followed by mechanical polishing to produce a mirror surface finish on the surface to be ion irradiated.

alloys	Cr wt%	C (ppm)	S (ppm)	O (ppm)	N (ppm)	P (ppm)	Grain size (µm)	Dislocation density (/cm²)
pure iron	<2 ppm	4	2	4	1	<5	183	1.2×10 ⁸
Fe3Cr	3.05	4	2	6	2		390	
Fe5Cr	5.4	4	3	6	2	<5	68	1.2×10 ⁸
Fe8Cr	7.88	5	2	7	2		320	
Fe10Cr	10.1	4	4	4	3	<5	82	< 10 ⁸
Fe10CrC	10.10	820	5	2	3	<5		

Table 1.	Elemental	information	and di	slocation	density	/ of a	s-received	samples
					,			

* The values lower than 5 ppm are given as an order of magnitude. The values between 5 and 10 ppm can be considered as +/- 3ppm (approximately).

Table 2 shows all the $g \cdot b$ combinations to identify dislocation loops. Numbers in Table 2 are the dot product of corresponding g and b. For example, the number in the second cell of [0 0 1] loop row is zero indicating this loop is invisible under [0 1 1] zone axis with g= [-2 0 0] two-beam condition.

	0 1 1 zone axis		0 0 1 zo	1 1 1 zone axis	
b g	0 1 -1	-2 0 0	1 -1 0	110	1 0 -1
001	-1	0	0	0	-1
010	1	0	-1	1	0
100	0	-2	1	1	1
111	0	-2	0	2	0
-1 1 1	0	2	-2	0	-2
1 -1 1	-2	-2	2	0	0
11-1	2	-2	0	2	2

Table 2. Detailed *g*·*b* analysis for dislocation loops

So far, the characterized samples were irradiated by 8MeV iron ion. The midrange dpa is 0.35. Irradiation temperature was 450°C. Dose rate was 10⁻⁴ dpa/s. Fluence was 8.83×10¹⁴ ion/cm². The result of stopping and range of ions in matter (SRIM) calculation is shown in Figure 1.



Figure 1. SRIM calculation result of 8MeV Fe in Fe-8Cr sample with Kinchin-Pease model. The yellow region is for (S)TEM analysis.

The TEM samples were prepared by Zeiss Auriga focused ion beam (FIB). The 30kV Ga⁺ probes were for thinning down to ~150nm in thickness and 10kV as well as 5kV probes were for fine polishing. Preliminary characterization has been performed on UHP iron, Fe-3Cr, Fe-5Cr, and Fe-8Cr samples. As an example, two sets of nearly edge-on loops in a Fe-5Cr specimen are shown in Figure 2 under dynamical two-beam bright field conditions with a beam direction of [011]. From the knowledge in Table 2, the dislocation loops in Figure 2a are [0 0 1] and/or [0 1 0] type. The loops in Figure 2b are [1 0 0], [1 1 1], and/or [-1 1 1]. The mottled contrast in background is probably attributed to FIB artifacts.



Figure 2. Dislocation loops in ion irradiated 5Cr specimen under $[0\ 1\ 1]$ zone axis with g = $[0\ 1\ -1]$ (Figure 2a) and g = $[-2\ 0\ 0]$ (Figure 2b) two-beam condition. Sample was irradiated at 450°C and midrange dpa is 0.35. The marked regions are shown in Figure 3a and Figure 3b, respectively.

Images in Figure 3 were taken in ~1 μ m deep region from the surface in order to minimize influences from the specimen surface and implanted ion regions. They show more details regarding the loop features from Figure 2. The big "loop" (~200nm in diameter) in Figure 2b consists of several small dislocation loops. For loops in Figure 2a, they show "multi-layer" structure. They are clustered together while they are not on the same plane (Figure 3a). Meanwhile, it seems that the loops clustered as a big "loop" in Figure 2b sit on the same plane (Figure 3b).



Figure 3. Higher magnification images of the marked regions in Figure 2a and b under $[0\ 1\ 1]$ zone axis with g= $[0\ 1\ -1]$ (Figure 3a) and g = $[-2\ 0\ 0]$ (Figure 3b) two-beam condition, respectively.

Future Work

As shown in Figures 2 and 3, the damage induced by FIB is heavy. The contrast due to FIB surface ion milling damage under some imaging conditions almost mask all the details of dislocation loops. Figure 4 is taken from the same region as the one in Figure 2b and Figure 3b. The wavy contrast is due to FIB damage.



Figure 4. Dislocation loops in Fe-5Cr specimen near [0 0 1] zone axis with g= [1 -1 0] two-beam condition. This image was taken at the same region as the one shown in Figure 2b.

In order to remove artifacts associated with FIB ion milling, we are pursuing another TEM sample preparation method called flash electropolishing. Essentially, this method consists of brief electropolishing after FIB milling. Researchers prepare a FIB lamella by thinning down to 200-300 nm in thickness and then transfer it to a flash electropolishing system for ~0.08s. After cleaning artifacts, more characterizations are needed.

Two sets of irradiations are expected to be completed at the Michigan Ion Beam Laboratory soon. One irradiation set will compare the difference caused by raster beam and steady beam on the same samples and another will investigate carbon effects on dislocation loop formation and evolution.

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1.5 BUBBLE FORMATION BEHAVIOR FOR IN-SITU He-IMPLANTED 14YWT AND CNA ADVANCED NANOSTRUCTURED FERRITIC ALLOYS—Yan-Ru Lin, Steven J. Zinkle (University of Tennessee at Knoxville), David T. Hoelzer, Lizhen Tan (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this task is to study the cavity size and density in He implanted nanostructured ferritic alloys (14YWT and CNA3) with different starting dispersoid sink strengths. Helium effects in irradiated materials can degrade the mechanical performance of structural materials and impact the economics and safety of future fission power plants; they ultimately determine the upper operating temperature limit for materials. It has been proposed that these effects could be mitigated by increasing the number of He trapping sites to control the bubble size or to shield He from the grain boundaries. This concept has led to the development of high sink strength materials with nanoclusters, such as Oxide dispersion strengthened (ODS) alloys. However, there is no systematic irradiation data showing how the density of nanoclusters in advanced ferritic alloys such as 14TWT and CNA materials affect the helium bubble density and size at elevated temperatures.

SUMMARY

Cavity formation in Fe-10Cr, CNA3 and 14YWT materials with in-situ He implantation (to ~12000 appm at 600 and 900°C) was examined by transmission electron microscopy (TEM). From 600-900°C, the bubble size was observed to increase with increasing irradiation temperature for all three of the materials. At both 600°C and 900°C, the bubble density of 14YWT were measured to be ~2x10²³m⁻³ whereas the bubble density in Fe-10Cr and CNA3 decreased from ~6x10²²m⁻³ to ~8x10²¹m⁻³ and from 3x10²²m⁻³ to 1.5x10²²m⁻³ with increasing temperature, respectively. The observed bubble number densities for the two Nano clustered alloys (CNA3 and 14YWT) are comparable to the nanocluster density, suggesting that the nanoclusters in both alloys were effective at trapping He at elevated temperatures. The results indicate that the 14YWT alloy appears to sequester the helium into smaller bubbles more effectively than the CNA3 alloy. This can be attributed to the much higher sink strength associated with the nanoclusters in the 14YWT alloy.

PROGRESS AND STATUS

In our previous progress report [1], He bubbles were only found in the He implanted (~7000appm) Ni material, but not in the 14YWT and CNA3 alloys at 500°C and 700°C. This may due to lack of visibility due to problems with the focused ion beam (FIB) sample quality or the resolution limit of TEM, which may not allow us to visualize bubbles with diameter less than 2nm.

In a follow-up study, we performed in-situ helium implantation on TEM samples of a high purity Fe-10Cr alloy and two nanostructured ferritic alloys (14YWT and CNA3) utilizing the Intermediate Voltage Electron Microscopy (IVEM) facility at Argonne National Laboratory.[2] Electro-polishing method was used to prepare the TEM samples in order to improve visibility of small features. The thin TEM foils (thickness ~ 100 nm) were irradiated with 10 KeV He ions at 600°C and 900°C, with a fluence near 10¹⁹ m⁻². After the 900-second implantation, based on the stopping and range of ions in matter (SRIM) calculation, the peak radiation damage and He concentration (at depth 60nm) were roughly 0.4 dpa and 12000 appm, respectively. For the 600°C experiments, when the peak dose reached 12000 appm, we stopped the implantation and subsequently performed post irradiation in-situ annealing to 900°C in 15 minutes. The annealing results were used to compare with the 900°C directly hot implanted experiments.



Figure 1. Unirradiated (a) Fe-10Cr, (b) CNA3 and (c) 14YWT materials.

The TEM images (Figure 1) of the unirradiated materials summarize the grain size difference of 82um, 30um and 150nm for Fe-10Cr, CNA3 and 14YWT, respectively. Previous APT and TEM results reported that the nanocluster density of 14YWT [3] and CNA3 [4] alloy was around 10^{23} m⁻³ and 10^{21} - 10^{22} m⁻³, respectively. The magnitude of the nanocluster density is also consistent with our previous EELS results [1]. The nanoclusters in 14YWT were mainly titanium/yttrium oxides, whereas MX precipitates (M: Ta/V, X: C/N) were the main nanocluster observed in CNA3.



Figure 2. 600°C He irradiated (a) Fe-10Cr, (b) CNA3 and (c) 14YWT materials.



Figure 3. 900°C He irradiated (a) Fe-10Cr, (b) CNA3 and (c) 14YWT materials.

As shown in Figure 2 and Figure 3, He bubbles were observed in all the irradiated materials at 600°C and 900°C. For the Fe-10Cr material, the average bubble radius significantly increased from 2.2nm at 600°C to 12.6nm at 900°C, and the corresponding bubble density decreased from $6x10^{22}m^{-3}$ to $8x10^{21}m^{-3}$. Bubbles were also visible in the implanted CNA3 alloy. For the same temperature range, the bubble size in CNA3 increased from 3.2nm to 5.1nm, and the density slightly decreased from $3x10^{22}m^{-3}$ to $1.5x10^{22}m^{-3}$ with increasing temperature. The variation in cavity parameters was more moderate for the 14YWT alloy. The bubble size slightly increased from 2.2nm to 3.1nm. However, at both 600°C and 900°C, the measured bubble density of 14YWT remained constant around $2x10^{23}m^{-3}$.



Figure 4. 900°C annealed irradiated (a) Fe-10Cr, (b) CNA3 and (c) 14YWT materials.

Figure 4. provides the TEM images of the cavities in the samples that were irradiated at 900°C and subsequently annealed to 900°C. The bubble size gradually increased from 600°C to 900°C. Comparing the 900°C post-implantation annealing results with the 900°C directly hot implanted experiments, the bubble size was only slightly larger in the annealed 14YWT and CNA samples, while the cavity radius of Fe-10Cr increased to 6 nm after annealing at 900°C instead of 12.6 nm as observed in the 900°C directly implanted sample (open symbol). On the other hand, the observed bubble density remained nearly constant for the annealed 14YWT and CNA alloys and the density decrease were less than the directly hot implanted experiments.



Figure 5. Comparison of the (a) cavity size and (b) density at 600-900°C.

The size and density of the He bubbles in all the sample are summarized in Figure 5. The open symbols represent the directly hot implanted experiments at 900°C, and the filled symbols are the 600°C implanted results followed by the post-implantation annealing process that was stopped at 750°C and 900°C for observation. In conclusion, from 600°C to 900°C, the general trend showed that bubble size increased, and the density decreased, as expected based on simple cavity coarsening expectations and numerous prior studies on gas-implanted materials. However, the variation was more moderate for the 14YWT alloy that had the highest nanocluster/precipitate density. At 600°C, the bubble number densities are comparable to the nanocluster densities for these two alloys (N_{He}/N_c ~1), suggesting that the nanoclusters in both alloys were effective at trapping He at elevated temperatures. Similar conclusions have been obtained by other researchers for dual ion irradiated ODS steels [5][6]. The results indicate that the 14YWT alloy appears to sequester the helium into smaller bubbles more effectively than the CNA3 alloy. This can be attributed to the much higher sink strength associated with the nanoclusters in the 14YWT alloy.

Future Plans

The decreasing of bubble density in CNA3 at 900°C need further EELS or APT examination to confirm if the nanocluster density also decreased. Furthermore, high resolution STEM-HAADF images with EELS mapping of bubbles and nanoclusters in the He implanted samples would be valuable to prove if the bubbles were really trapped by the nanoclusters.

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1.6 HYDROGEN ISOTOPE TRANSPORT PROPERTIES IN ADVANCED RAFM STEELS FOR FUSION REACTOR STRUCTURAL APPLICATION—Z. Chen, B.D. Wirth (University of Tennessee, Knoxville), Y. Katoh, X. Hu (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this project is to determine the permeability and diffusivity of deuterium in representative reduced activation ferritic-martensitic (RAFM) steels using gas-driven permeation testing technique.

SUMMARY

The fundamentals of hydrogen materials interactions are critically important for the design of a robust fusion breeding blanket enabling a self-sufficient tritium cycle. In this study, we utilized the gas-driven permeation testing techniques to determine the basic hydrogen transport properties in representative RAFM steels to expand the current database of hydrogen behavior in fusion materials. The experimental results are comparable to literature data. In addition, the correlation between sink strength and the deuterium permeability and diffusivity is briefly discussed. More work is needed to elucidate the correlation between the microstructures and the hydrogen isotope transport properties in fusion structural materials.

PROGRESS AND STATUS

Introduction

The RAFM steels have been developed as structural materials for fusion reactors since 1980's, with recent attention aiming for better mechanical properties, high temperature tolerance and radiation resistance [1, 2]. Castable nanostructured alloys (CNAs) [3], oxide dispersion strengthened (ODS) [4] ferritic steel are two promising advanced candidate systems. Nevertheless, the hydrogen isotope behavior in these materials is not well understood [5], especially in the service environment, which is critically important to realize fusion fuel self-sufficiency and concerns for tritium safety. The extremely hostile fusion environment, characterized by high temperature, high thermal flux, and intensive particle bombardment, significantly alters the microstructures and the resultant thermomechanical properties of the structural materials, thus, resulting in changes in the hydrogen isotope permeation characteristics of the materials.

In this project, we determined the deuterium transport properties of two RAFM steels (Eurofer 97 and G91), three ODS steels (M4 Fe-10Cr, 9YWT-PM2, 14YWTV-SM13) and two CNA (FTa1 and TT3mt) alloys in the as-fabricated state employing the gas-driven permeation testing technique.

Experimental Procedure

Disk-shape specimens were machined with a diameter of 12.6 mm and \sim 1 mm thickness. The specimens were polished to a surface finish of 1 μ m on both sides. The accurate thickness of each

specimen was measured by micrometer before testing. Permeation testing was performed using the Oak Ridge National Laboratory (ORNL) gas-driven permeation testing station. Figure 1 (a) shows a sketch of the gas-driven permeation system. The specimen is used as a VCR gasket, directly fixed by two VCR couplings. The effective permeation diameter is 7.7 mm (with effective permeation area of 46.5 mm^2), which is defined by the edges of the VCR coupling. The upstream and downstream chambers are separated by the sample. The whole sample fixture is heated in an electric resistance tube furnace with a maximum temperature of 1100°C. The ultra-high vacuum level in the upstream chamber is ~ 1×10^{-7} torr. When this vacuum level is reached, the manual gate valve connecting the vacuum chamber and the turbo pump is closed. Then ultrahigh purity (UHP) deuterium gas is introduced to the upstream vacuum chamber. In the downstream section of the system, the same vacuum level of $\sim 1 \times 10^{-7}$ torr is achieved using a pumping system that is identical to the upstream section. The diffusionlimited permeation process starts once high-pressure deuterium gas is introduced in the upstream chamber. A final steady permeation state is achieved after enough time. The gas pressure in the upstream chamber is measured by an MKS 909AR digital and analog hot cathode vacuum transducer. The permeating gas signal in the downstream chamber is captured by a quadrupole mass spectrometer (QMS) (Pfeiffer PrismaPlus OMG 220). Calibration of the QMS is made using a known deuterium gas leak system (VIT standard gas leaks) before the experiments.



Figure 1. (a) Sketch of the gas-driven permeation system. (b) Picture of the permeation system and sample fixture.

Results

Permeation Test

Deuterium permeation flux through sample materials as a function of applied D pressure (four pressures are applied at each temperature, with values of 140, 260, 380, and 500 torr) are measured at 5 or 6 temperature points ($350^{\circ}C-550^{\circ}C$ or $350^{\circ}C-600^{\circ}C$) with ΔT interval of $50^{\circ}C$. Figure 2 shows the permeation flux versus time during a permeation run for Eurofer 97. Following the measurement at each temperature, the upstream chamber is pumped to ultra-high vacuum prior to performing measurement at the next temperature.



Figure 2. A complete run of permeation flux vs. time at temperatures of 350, 400, 450, 500 and 550°C for a Eurofer 97 permeation sample.

An example (Eurofer 97) of the correlation between the steady state deuterium permeation flux and square root of upstream deuterium gas pressure is shown in Figure 3. A clear linear correlation is revealed, consistent with the assumption that the permeation process is diffusion limited. Theoretically, the steady state permeation flux is related to the square root of driving gas pressure through [6]:

$$J = \Phi \frac{p^{1/2}}{\delta} \tag{1}$$

Where Φ is the deuterium gas permeability in unit of mol $D_2 m^{-1}s^{-1}MPa^{-1/2}$, *J* is the permeation flux in unit of mol $D_2 m^{-2}s^{-1}$, *p* is the applied gas pressure in MPa and δ in the sample thickness in m.



Figure 3. Steady state deuterium permeation flux through Eurofer 97as a function of square root of upstream chamber deuterium gas pressure.

According to equation (1), the permeability of deuterium in materials can be calculated. The permeability of deuterium in the materials studied is summarized in Figure 4. The results indicate that the deuterium permeabilities of these tested samples are within a narrow range, one order of magnitude. However, it is observed that ODS steels have relatively lower permeability, especially the 14YWT-SM13, which has the largest sink strength due to the large number density of nanoparticles. Deuterium permeability of Eurofer97 is highest among all tested materials.

The temperature dependence of measured deuterium permeability in advanced steels is generally presented as an analytical expression:

$$\Phi = \Phi_0 \cdot exp\left(-\frac{E_0}{RT}\right) \tag{2}$$

The permeability coefficient Φ_0 and activation energy E_0 are obtained by fitting the experimental data and values are shown in Table 1. The obtained permeation activation energy varies in the range 34.6 kJ/mol to 39.67 kJ/mol, except for G91 (29.85kJ/mol). The activation energies of the two CAN steels and the M4 Fe-10Cr are similar. 14YWT-SM13 has the highest permeation activation energy, ascribed to its highest sink strength.



Figure 4. Deuterium permeability of advanced steels. Data for ODS-Eurofer and Eurofer97 from literature are also shown for comparison[7].

Category	Sample steel	Temperature	${oldsymbol{\Phi}_0}$	E ₀
		°C	$mol \cdot m^{-1} \cdot s^{-1} \cdot MPa^{-1/2}$	$kJ \cdot mol^{-1}$
RAFM	Eurofer 97	350-550	1.26×10^{-5}	35.22
	G91	350-550	4.22×10^{-6}	29.85
CNA	TT3mt	350-600	1.55×10^{-5}	37.30
	FTa1	350-600	1.46×10^{-5}	37.26
ODS	M4 Fe-10Cr	350-600	1.53×10^{-5}	37.75
	9YWTV-PM2	350-600	1.14×10^{-5}	35.51
	14YWT-SM13	350-600	1.73×10^{-5}	39.67
Reference	ODS-Eurofer ^[7]	/	4.22×10^{-5}	34.6
steels	Eurofer 97 ^[7]	/	1.53×10^{-5}	38.3

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Deuterium diffusivity

The deuterium permeation through RAFM steels before achieving steady permeation state is controlled by the Knudsen mechanism. By applying the boundry conditions for this system, the theoretical expression for pressure increase in the downstream chamber is obtained from Fick's second law [8]:

$$p_{L}(t) = \frac{AD_{K}C_{0}}{V\delta} \left[t - \frac{\delta^{2}}{6D} + \frac{2\delta^{2}}{\pi^{2}D} \sum_{n=1}^{\infty} \frac{(-1)^{n+1}}{n^{2}} exp\left(-\frac{Dn^{2}\pi^{2}t}{\delta^{2}}\right) \right]$$
(3)

where V is the effective volume of the downstream chamber. At the steady permeation state, the steady state term of eq. (3) can be written as:

$$p_{\infty}(t) = \frac{ADC_0}{V\delta} \left[t - \frac{\delta^2}{6D} \right]$$
(4)

At the steady permeation state, the downstream pressure linearly increases. Thus set $p_{\infty} = 0$, the time lag is obtained:

$$t_L = \frac{\delta^2}{6D} \tag{5}$$

Figure 5 shows the integral deuterium permeation flux vs. time for Eurofer 97 at 350°C. By extending the steady state line, the time lag is obtained as the x-axis intercept.


Figure 5. Experimentally measured permeation of deuterium through Eurofer97 at 350°C under a constant upstream pressure of 1 atm. Dashed line indicates the definition of the time lag.

In consideration of the trapping phenomena caused by the microstructures, which have a significant influence on the time lag, fresh samples were used for diffusivity tests at each temperature point. The upstream deuterium pressure is set to 1 atm. The results of diffusivity of the seven RAFM steels, together with the data for ODS-Eurofer and Eurofer97 from literature, are plotted in Figure 6. Generally, deuterium diffusivity in steels with higher sink strength is lower than that in steels with lower sink strength. The measured diffusivity in the current study is comparable to the literature data.



Figure 6. Deuterium diffusivity in advanced RAFM steels. Data for ODS-Eurofer and Eurofer97 from literature were also shown for comparison [7].

Diffusion coefficients and activation energies of the seven studied materials are obtained by fitting the experimental data, yielding values given in Table 2. The diffusion activation energy of CNA steels is clearly smaller than that of conventional RAFM steels and ODS steels. More work is needed to understand the correlation between the microstructure and the measured diffusivity.

Results

The gas-driven permeation testing station was used to measure the deuterium permeability and diffusivity of advanced steels for fusion structural application. The results indicate that higher sink strength leads to lower permeability and diffusivity. More work is needed to establish a robust understanding of the correlation between the microstructures and the hydrogen isotope transport properties in these advanced fusion steels.

Category	Sample	Temperature	D ₀	E ₀
	steel	°C	$m^2 \cdot s^{-1}$	kJ
				$\cdot mol^{-1}$
RAFM	Eurofer 97	350-550	8.89×10^{-7}	14.71
	G91	350-550	2.02×10^{-6}	15.82
CNA	TT3mt	350-600	3.15×10^{-8}	2.54
	FTa1	350-600	6.13×10^{-8}	4.30
ODS		350-600	1.04×10^{-7}	7.00
	9YwTV-	350-600	2.23×10^{-7}	13.46
	PM2			
	14YwT-	350-600	8.42×10^{-6}	20.86
	SM13			
Reference	ODS-	/	1.33×10^{-6}	30.4
	Eurofer ^[7]			
steels	Eurofer 97 ^[7]	1	1.50×10^{-7}	14.5

Table 2. Deuterium diffusivities of advanced RAFM steels

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1.7 DESIGN OF 3Cr-3WV BAINITIC STEEL FOR CROSS-WELD PROPERTY IMPROVEMENT—Y. Yamamoto (Oak Ridge National Laboratory)

OBJECTIVE

This work aims to develop new bainitic steels based on 3Cr-3WV(Ta) steels originally developed at Oak Ridge National Laboratory (ORNL). The goal is mechanical properties of both base metal and weldments superior to those of existing commercial bainitic steels or ferritic-martensitic (FM) steels, together with no requirement for post-weld heat treatment (PWHT). The target applications are high temperature structural components in fusion reactors such as the vacuum vessel, structural ring which supports the blanket modules, and magnet shields, to be used at or above the 400-500°C range. Improvement of long-term creep properties by introducing additional fine, stable second-phase dispersions, as well as maintaining good weldability, is targeted via optimization of alloy composition and thermo-mechanical heat treatment.

SUMMARY

Property evaluation of the base metal and the cross-weld specimens of the modified 3Cr-3WV base steels (MLC02 and MLC02B, high Mn + low C without and with 0.01 B, respectively) has been summarized. The hardness inhomogeneity across the weldment (the base metal and the heat affected zone [HAZ]) was reduced in the modified steels, as expected from the proposed alloy design strategy. The boron addition did not strongly impact the hardness. The modified steels exhibited a significant improvement of the base metal creep-rupture life at 600°C, compared to the original steel, at relatively low stress range. The cross-weld creep results also showed an improved performance in MLC02, but not much in MLC02B tested at 600°C and 170/200MPa. Cross-weld creep testing at low stress range is currently in progress.

PROGRESS AND STATUS

Based on previous results, new alloy design strategy has been proposed; high Mn, low C, low Si, and a small amount of B addition. The boron addition in ferritic-martensitic steels is known to refine the size of $M_{23}C_6$ type carbides and improve the thermal stability of the carbide which positively works in stabilizing the martensite laths at the service temperatures and therefore the creep performance [1]. There is a concern about the potential helium-bubble formation due to transmutation of B during irradiation, although the negative impact would be sufficiently small or even negligible because the expected doses are relatively low in the target components compared to the first wall/blanket components. In addition, the new alloys were based on Ta-free 3Cr-3WV steel in order to eliminate the combined effect of MC type carbide formation on the alloy modification. In this report, the evaluation of the cross-weld property of the modified steels, including the hardness and the creep performance, has been updated and summarized.

Table 1 summarizes the nominal compositions of two modified 3Cr-3WV base steels and the original 3Cr-3WV steel. The modifications include the additions of (1) high Mn ($0.4 \rightarrow 2 \text{ wt.\%}$), (2) low C ($0.1 \rightarrow 0.05 \text{ wt.\%}$), and (3) without and with 0.01 wt.% B. Note that the Si addition was kept the same as the original steel (0.16 wt.%) to avoid temper-embrittlement.

Alloy	Nominal composition, wt.%									Demerke
Alloy	Fe	Cr	Mn	Si	Та	V	W	С	В	Remarks
MLC02	91.59	3	2	0.16	-	0.2	3	0.05	-	High Mn, Iow C
MLC02B	91.58	3	2	0.16	-	0.2	3	0.05	0.01	High Mn, low C, with B
Original (heat #2750)	93.14	3	0.4	0.16	-	0.2	3	0.1	-	3Cr-3WV base, Ta-free

Table 1. Nominal and analyzed compositions of the steels

The cross-weld hardness distribution in three different steels are summarized in Figure 1. All base metals were normalized and tempered in standard conditions, and a gas tungsten arc weld (GTAW) was applied with a filler metal of the original 3Cr-3WVTa steel. The hardness inhomogeneity across the weldment (the base metal and the HAZ) was reduced in the modified steels, indicating that the potential negative impact from the repeated heating and cooling during weld procedure was successfully reduced. It should be noted that the boron addition did not strongly impact the hardness at room temperature.



Figure 1. (a) Color contour map of Vickers hardness in as-welded MLC02, MLC02B, and the original 3Cr-3WV steels, and (2) the hardness profiles across the weldments of the same materials.

The base metals exhibited improved creep-rupture life at 600°C, compared with the original steel. The trend becomes obvious in the relatively low stress range. The B containing steel showed a stronger dependence of the stress than the other steels. The cross-weld creep results also showed an improved performance in MLC02 compared to the original steel, but not much in MLC02B tested at 600°C and 170/200 MPa. Based on the trend in the base metal, the effect of modification would possibly be more apparent in low stress testing, so additional creep tests at 600 and 130 MPa are currently in progress.



Figure 2. Base metal and cross-weld creep properties of two modified steels and the original 3Cr-3WV steel, compared to that of Grade 91 (mod. 9Cr-1Mo steel) base metal, tested at 600°C.

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2. ODS AND NANOCOMPOSITED ALLOY DEVELOPMENT

2.1 INVESTIGATION OF THE EFFECT OF He IMPLANTATION ON α'**-FORMATION AND STABILITY OF ODS PARTICLES IN 14YWT**—Karen Kruska, Danny J. Edwards, Jing Wang, Richard Kurtz, Charles H. Henager Jr. (Pacific Northwest National Laboratory), T. Yamamoto and G.R. Odette (University of California-Santa Barbara)

OBJECTIVE

This work will focus on the effect helium injection in conjunction with neutron irradiation on the α' phase precipitates and nano-oxide dispersion in an oxide dispersion strengthened (ODS) ferritic alloys 14YWT.

SUMMARY

Atom Probe Tomography (APT) analysis was performed on the He implanted (ISHI) side of an irradiated 14YWT specimen. The initial indications point towards a lower volume fraction of α ' phase and a coarser distribution of ODS particles compared to neutron irradiation only. However, these results need to be confirmed with additional datasets due to local heterogeneities within the 14YWT alloy.

PROGRESS AND STATUS

Introduction

The ODS Fe-Cr alloys are a candidate material for plasma facing components in fusion reactors due to their ability to withstand the high temperatures and neutron irradiation environment. However, like many structural alloys, the large levels of helium produced during reactor operation are expected to lead to a degradation on the mechanical properties and swelling resistance. It is hypothesized that finely dispersed oxide clusters can act as effective traps for helium, hindering the formation of large helium bubbles that can eventually transition to void swelling. In this study, we interrogate the effects of high levels of helium on the overall microstructural evolution and relative stability of the various phases present under irradiation.

Experimental Procedure

The 14YWT is an Fe-14Cr ODS alloy with a fine dispersion of yttrium-titanium-oxide particles less than 2 nm in diameter. One side of a 3 mm Transmission Electron Microscopy (TEM) disc sample was coated with 4 μ m of NiAl prior to neutron irradiation in the High Flux Isotope Reactor (HFIR) at the Oak Ridge National Laboratory (ORNL) to a dose of 21.2 dpa at 500°C in the JP-27 experiment [1, 2]. Previous studies have shown APT to be particularly suited to analyze the size, density and composition of the nanofeatures in this alloy [3-5]. The APT specimen preparation was carried out in a Field Electron and Ion Company (FEI) Quanta dual-beam focused ion beam (FIB) using a standard lift-out method. The APT analysis was conducted using a laser-pulsed Cameca local electron atom probe (LEAP) 4000HR at temperature of 40 K, a pulse frequency of 200kHz and a pulse energy of 60 pJ with a laser wave length of 355 nm. A novel cluster search algorithm developed at Pacific Northwest National Laboratory (PNNL) (OPTICS) [6] was used to quantitatively evaluate ODS and α ' phase particles.

Results

The sample preparation from the ISHI was complicated by the 4 μ m thick NiAl coating at the surface which needed to be completely removed to obtain reliable data of the He implanted region. Of the two atom-probe needles that ran successfully, one contained high amounts of Al, indicating that the lift-out was produced from a region too close to the surface. Ten M ions were acquired from the second needle. Ion maps showing the distributions of α ' phase precipitates and ODS particles are shown in Figure 1. The data indicate that the α ' particles are smaller and lower in density than on the neutron irradiated side (Table 1). However, large variations in local precipitate densities were observed in the prior neutron irradiated sample through analysis of multiple needles, whereas the amount of data from the He implanted side is much more limited.

Further analysis is needed to confirm this observation that the helium injection impacted the formation of α' phase precipitates. The trend for the ODS particles in the He implanted sample indicated slightly larger particles at a lower density. Again, however – this could be a highly localized effect and more data is



Figure 1. A) APT ion map showing α' phase precipitates. The precipitates are visualized using Cr isosurfaces at 27 at. % Cr. B) APT ion map showing ODS particles. The particles are visualized using Y isosurfaces at 1 at. % Y.

 Table 1. Comparison of ODS and α' particle distributions in as-received, neutron irradiated and (ISHI) He implanted 14YWT

	ODS particle	ODS particle	α' density	α' mean size
	density	mean size		
As-received	6.27×10 ²³ ±	4.4 ± 1 nm	n/a	n/a
	0.32×10 ²³ m ⁻³			
Neutron irradiated	4.85×10 ²³ ±	4.2 ± 1 nm	5.18×10 ²³ ±	4.2 ± 1.9 nm
	2.7×10 ²³ m ⁻³		4.11×10 ²³ m ⁻³	
He implanted	1.52×10 ²¹ m ⁻³	4.9 ± 1.4 nm	3.97×10 ²¹ m ³	3.0 ± 1.1 nm

necessary to warrant a conclusion about the effect of He implantation on ODS particle size and density. If this tentative trend holds up under further investigation, this would mean that while He implantation has a positive effect preventing excessive formation of the α' phase, coarsening of the ODS particles would decrease the alloys resistance to bubble formation and swelling. The particle size histogram shown in Figure 2 reveals that most particles are between 3.5 and 5.5 nm, just like for the other two samples. The mean is higher because of the 3 outliers that have a diameter greater than 8 nm. Such outliers were also present in the other samples, but due to the overall larger number of particles analyzed in these cases, they were given less weight. If agglomeration of ODS particles takes place as an effect of He implantation, this should become more obvious if more data is collected.

Lastly the composition of the ODS precipitates in the He implanted sample was analyzed and compared to previous data. The initial analysis is shown in Figure 3. Similar to the neutron irradiated sample as shift towards CrO was observed in the ion make-up of the ODS precipitates. This could mean that Cr does not only form α ' particles as a result of temperature and irradiation, but also segregates to the ODS particles. The ion maps in Figure 1 show that α ' phase precipitates and ODS particles are usually well separated, thus ruling ODS particles out as a nucleation site for α ' phase formation. It appears that Cr is either bound in a Cr oxide shell or incorporated into a multi-component oxide.

Future work will be needed to confirm the impact of the helium on the α' formation and growth, as well as the distribution and chemistry of the oxide dispersion since these results are quite surprising.



Figure 2. ODS particle size distributions in as-received, neutron irradiated and (ISHI) He implanted 14YWT.



Figure 3. Ratios of major ions detected in each cluster. During the APT experiments, ions consisting of single atoms as well as fragments of multiple atoms are evaporated. The most common fragments contained in the ODS clusters are Y, CrO, and TiO. The ternary diagrams do not show the direct composition of Y, Cr, Ti, and O, but rather the ratio between these three fragments in the APT analysis.

Acknowledgements

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3. CERAMIC COMPOSITE STRUCTURAL MATERIAL DEVELOPMENT

3.1 HIGH-DOSE, INTERMEDIATE-TEMPERATURE NEUTRON IRRADIATION EFFECTS ON SILICON CARBIDE COMPOSITES WITH VARIED FIBER/MATRIX INTERFACES—Takashi Nozawa, Hirayasu Tanigawa (QST-Japan), Takaaki Koyanagi, Yutai Katoh (Oak Ridge National Laboratory)

Summary of a manuscript in Journal of the European Ceramic Society 39 (2019) 2634–2647

Silicon carbide (SiC) fiber reinforced SiC matrix composites are promising structural candidate materials for fusion reactor applications over high neutron damage (up to >~100dpa). Hence, tolerance to high dose neutron irradiation needs to be understood to ensure the performance of these composites. Radiation swelling and mechanical properties following irradiation to 11–44 dpa at ~600 °C were evaluated for Hi-Nicalon Type-S or Tyranno-SA3 fiber–reinforced SiC matrix composites produced by chemical vapor infiltration. The influence of various fiber/matrix interfaces, single layer pyrolytic carbon (PyC) monolayer interphase and multilayer PyC/SiC interphase, was evaluated and compared with the previous results for a PyC/SiC multilayer interphase with different layer thickness. Regardless of the fiber type, composites with single layer PyC interphase showed considerable reduction of flexural properties after irradiation to 11–12 dpa at 450–500 °C; and neither type showed the deterioration identified at the same dose level at higher temperatures (> 750 °C) in a previous study. After further irradiation to 44 dpa at 590–640 °C, the degradation was enhanced compared with conventional multilayer composites with a PyC thickness of ~20 nm. Irradiation-induced debonding at the F/M interface was found to be the major cause of deterioration of various composites.

4. HIGH HEAT FLUX MATERIALS AND COMPONENT TESTING

4.1 TENSILE TESTING OF STEEL AND TUNGSTEN FIBERS FOR APPLICATIONS IN COMPOSITE

MATERIALS—L. M. Garrison (Oak Ridge National Laboratory), Matthew Weinstein (University of Wisconsin-Madison)

OBJECTIVE

The goal of this project is to develop a technique for the tensile testing of steel and tungsten fibers to determine the mechanical properties of the fibers for use in composites.

SUMMARY

This report discusses the development of a method for tensile testing steel and tungsten fibers in order to understand the properties of fiber composite materials for use in fusion reactors. A new fixture was designed for tensile testing fibers. To cushion the sample in the fixture, each fiber must be precisely glued into a paper frame. Initial tests have been completed on steel fibers.

PROGRESS AND STATUS

Introduction

Tungsten fiber composites are being examined as a possible way to improve the toughness of bulk tungsten materials. The fibers can dissipate the crack energy in the composites, could increase their ductility, and lower their effective brittle to ductile transition temperature. In order to understand the mechanical properties of the tungsten fiber composites, it is important to understand the properties of the fibers that will be used to fabricate them. To accomplish this, a method for tensile testing of steel and tungsten fibers is being developed. This involves developing a new fiber testing fixture and procedure that will first be used on steel materials to ensure accurate results. The process of developing a tensile testing technique for the fibers was divided into three tasks. The first was the development of the fiber tensile fixture, second was the development of the fiber sample preparation procedure and third was the development of the fiber testing procedure.

Fiber tensile fixture

Fiber samples have opposing features in that they are intrinsically stronger than typical bulk tensile test samples while simultaneously being more prone to damage when handled in the laboratory. Because of this, a special tensile test fixture had to be designed to provide the necessary gripping strength to hold the fiber in place, while also ensuring that the grips will not damage the fibers and cause fracture under the grips, away from the observable part of the sample.

The fixture that was designed for these samples consisted of two sample holding plates with two rectangular indents for samples to be placed in and held down by clamps secured by four screws, one for each corner, as shown in Figure 1.



Figure 1. Different views of the fiber fixture with a) clamps removed b) clamps removed and sample in place, and c) sample in place and locked down.

The clamps provide the necessary force required to hold the fiber samples in place during the tensile tests. Additionally, the two guide rods ensure that the samples do not shift laterally as the samples are being mounted and tested. To ensure that the metal plates did not damage the samples, they were held between two pieces of paper sealed together by epoxy.

Fiber sample preparation

In order to collect accurate tensile test data, it was important to properly mount and prepare the samples so that they were not damaged prior to testing. Fiber samples of steel Alloy 304 at 203.2 μ m diameter were cut into 40 mm lengths while the tungsten samples of 150 μ m diameter were provided to us by a collaborator. These specific steel samples were chosen because they had a similar diameter to the tungsten fibers and were a readily available material with well-known mechanical properties.

First the sample testing papers were printed and cut so that the fiber samples could be sealed between them. Once the paper sample holders had been prepared, the epoxy for attaching the fiber samples to the paper was prepared in a fume hood with nitrile gloves. The epoxy used was the UHU PLUS Endfest 300. The hardener and binder were first mixed according to manufacturer's directions in a 1:1 ratio.

The epoxy was brushed onto the bottom paper sample holder and the steel wire was placed on top of it along the black guideline on the paper sample holder. The second paper sample holder was placed on top and aligned with the bottom one while gently steering the wire to stay in the correct position. The sample was then placed in a holder with a surface that the epoxy will not bond to so that the weight of the holder would help the epoxy bond and set the sample in place. The samples were than left for at least twelve hours for the epoxy to set and following that were ready for testing. Preliminary tensile tests on the steel samples showed varied success for this method of sample preparation with some samples slipping out of the bonded sample holder as discussed in the next section.



Figure 2. Completed steel wire samples in the paper frame, sitting on the gluing fixture.

Fiber testing procedure and Preliminary Results

Once the epoxy bond on the samples had set, tensile tests could then be performed. So far, only steel samples were tested in the tensile fixture. The samples were mounted in the fixture shown in Figure 1 and below in Figure 3. The fixture then had two bolts attached on either end for placement in the tensile frame. It was important during the setup process that the sample fixture was handled gently to ensure no damage to the fibers prior to testing. The extension rate for these tests was 8 μ m/s based on the ASTM standard for fiber testing in order to load through the elastic region for the steel alloy within 30 seconds. The sample fixture was then slowly raised via the crosshead to just below the start of applying force to the sample. After removing the side braces and cutting the paper on either side of the exposed fiber, the tensile test could begin. Multiple tensile tests were performed in order to verify that the data was repeatable, and that the tensile test procedure was giving reliable data.



Figure 3. Steel sample tensile test setup prior to testing before cutting the paper support strips.

The first tensile tests performed underwent failure successfully, with fracture occurring in the area between the grips. Additionally, load vs. extension curves, shown in Figure 5, show good repeatability of the tensile tests with the elastic region for both tests being very similar and the only major noticeable difference being in the plastic region of the test with marginally greater extension to failure in the first sample.



Figure 4. Fracture of successful steel sample tensile test.



Figure 5. Load vs Extension Curves for the first two steel samples.

While these initial results were promising, later attempts to repeat the experiment were not as successful. The next set of steel sample tests all exhibited a similar elastic region but upon reaching a threshold force of approximately 20 N, the samples began to slip from the fixture and slowly pull out of the paper sample holders until one end was no longer in the fixture. There has not been any correlation noticed for one side of the fixture vs the other for the wire pulling out from the fixture. With these inconsistencies in the test data of the steel samples, a few different methods are being evaluated to resolve this issue. New steel samples have been generated using greater amounts of epoxy to ensure that every interior surface between the two

paper sample holders can provide the greatest amount of force to prevent the sample from slipping out during testing. Once the necessary changes to the sample preparation have been made and are shown to be successful by repeated steel wire tests, tensile testing will then be able to begin using tungsten samples. Additionally, future work will involve the use of digital image correlation (DIC), in order to more accurately map the strain of the fiber samples during the tensile testing. This will allow accurate mapping of fiber mechanical property data enabling a better understand of how the constituent components of fiber composites interact.

4.2 ON THE EFFECT OF SPECIMEN SIZE, GEOMETRY AND DUCTILE PHASE CONTENT ON THE FRACTURE TOUGHNESS OF TUNGSTEN HEAVY METAL ALLOYS—M.E. Alam, G.R. Odette (University of California Santa Barbara)

OBJECTIVE

The objective of this research is to characterize the fracture toughness of tungsten heavy alloys as candidate divertor material.

SUMMARY

The tensile and fracture toughness ($K_{Jm/lc}$) properties of four ductile phase toughened (DPT) commercially available tungsten (W)-based heavy metal alloy composites (W-NiFe), reinforced with 3 to 10 (wt.%) of a NiFe ductile phase (DP), were previously thoroughly characterized from room to liquid nitrogen (LN₂) temperatures using small 3-pt, bend (3PB) specimens. The brittle-to-ductile transition temperature (BDTT) ranged from -25°C to -150 °C, depending on DP content. Above this temperature the specimens fail by stable crack growth, while at lower temperature elastic fracture occurs. However, in all cases the WHA had much higher $K_{Jm/lc}$ than monolithic W. Here we study specimen size and geometry effects on $K_{Jm/lc}$, depending on the amount of DP.

PROGRESS AND STATUS

Introduction

The high melting temperature, high temperature strength, good conductivity and low sputtering rate make the tungsten (W) and its alloys an attractive candidate for plasma facing component for future fusion reactor divertor application [1-4]. This application requires that W-based alloys have enough fracture toughness to withstand the severe thermal-mechanical environment of a divertor. It is likely that monolithic W is intrinsically too brittle for this task. Previously, toughness tests on small 3PB (B = 1.65mm, W = 2B and S = 4W) on a series of W-NiFe (90, 92.5, 95 and 97 wt.% W with 7:3 = Ni:Fe ductle phase) heavy metal alloys showed that all the WHA have much higher room temperature toughness (> 10x) and much lower BDTT temperatures (-150 to -25 °C) than monolithic W (several hundred °C). depending on their ductile phase NiFe content [5-6]. Three times larger specimens (B = 5mm) also show high toughness and stable crack growth at room temperature except for the highest W% alloy, while 6x larger specimens (B = 10mm) show mixed crack propagation behavior for the 95W-NiFe alloy [7]. In this study, additional tests were performed at room temperature on even larger 3PB specimens (\approx 8x) for the 95 and 92.5W alloys. The room temperature (RT) fracture toughness compact specimen (CT) toughness tests on the 95W alloy were also performed to investigate the effect of specimen geometry. Additional room temperature tests on the small 3PB specimens were conducted to improve the data statistics. Finally, the measured DP area fraction and ligament thickness were correlated to KJm/lc.

Experimental Procedure

Four commercial (Mi-Tech Metals, Indianapolis, Indiana [IN], United Staes of America [USA]) liquid-phase sintered (LPS) tungsten heavy metal alloys (WHA) were received in the form of plates that contain 90, 92.5, 95 and 97 wt. % W with a balance of an initially 70% Ni and 30 %Fe phase. In our previous studies, we have tested small specimens (B=1.65 mm) [5-6], and 3 to 6x larger specimens [7] in a 3PB bar fixture. In this study, even larger (B = 12.7 mm, W = 25.4 mm, S = 101.6 mm or ASTM Standard 0.5T specimen, dubbed as XL) specimens were tested. The experimental methods can be found in [5-6]. The CT specimens were prepared from the broken 6x larger 3PB bars of 95W. The dimensions of CT specimen are shown in Figure 1. Like 3PB specimens, the CT specimens are also precracked to a/W \approx 0.4- 0.5, and tested and analyzed as per ASTM Standard E-1921 [8]. Due to the local and specimen to specimen microstructural variations, additional and higher resolution scanning electron microscopy (SEM) was used

to measure the W-particle size, DP area fraction and DP skeleton thickness for better statistics. Electron probe microanalysis (EPMA) was also used to analyze DP constituents more precisely.



Figure 1. The compact tension (CT) specimen dimensions.

Results

Microstructural characterization

The tungsten particle size, DP area fraction, and their constituents (measured by EDS) for all four WHA alloys were reported previously [5-6]. However, due to local microstructural inhomogeneity of these alloys (see Figure 2), and after acquiring second batch of WHA plates from the same vendor (Mi-Tech Metals. IN), we analyzed additional and higher resolution SEM images to get better statistics of characteristics that directly or indirectly affect fracture toughness. The old and new results were combined, and their averages are shown in Table 1. For example, the average local DP area fraction for specimen 1 and 2, taken from the same plate, for 90W are 22.4 and 13.1%, respectively (Figure 2a,b); and those for 97W are 9.2 and 5.7% (Figure 2c,d). Similar results were found for plate 2. In contrast, the W-particles sizes, were similar (Table 1 and Reference [5]). As expected, W-W contiguity, Cw, increases, and t/W ratio decreases with increasing W (see Table 1). Interestingly, WHA area fraction has a minimal (or random, if consider average values) effect on DP skeleton thickness (t). This might be due to the line intercept method used that only considers where DP is present and ignores W-W interfaces (therefore, C_w is higher for 97W compared to 90W, but t is almost the same), EPMA was also performed on finely polished specimens to measure the percentage of ductile phase constituents more precisely (Table 1). The EPMA shows Ni/W/Fe constituents in DP are $\approx 55\%$ Ni, 25% W and 20% Fe (by wt.), compared to $\approx 50\%$ Ni, 30%W, and 20% Fe for EDS (Table 1 and Reference [5]).



Figure 2. Backscattered SEM images showing local microstructural variations scanned from two different specimens for: (a,b) 90W, and (c,d) 97W-NiFe alloys, respectively.

Table 1. The W particle size, and the area fraction, of	compositions and morphology of the NiWFe
honeycomb web structure	for all WHA alloys

WHA	W-particle size, (µm)	DP area (wt.%)	Ni/W/Fe in DP (%)	W-W contiguity, <i>C</i> _w	Thickness, <i>t</i> (μm)	<i>t/W</i> (μm/μm)
90W	17 ± 7	16.1 ± 3.8	54.0/24.9/21.1	0.161	5.9 ± 5.4	0.22
92.5W	18 ± 7	11.8 ± 2.2	54.3/25.2/20.5	0.197	4.0 ± 3.7	0.13
95W	26 ± 11	10.7 ± 1.3	55.0/24.8/20.2	0.224	5.1 ± 4.1	0.12
97W	38 ± 15	6.4 ± 1.5	57.4/22.7/19.9	0.315	4.5 ± 5.0	0.07

t = NiWFe thickness, W = tungsten fraction.

Specimen size effects on room temperature fracture toughness

We have previously reported maximum load fracture toughness (K_{Jm}) properties of small (B= 1.65mm) specimens from RT to liquid nitrogen (LN₂) temperature [5-6], and for medium (3x) to large (6x) 3PB specimen at RT [7]. In summary, previous results show that the RT K_{Jm} for 90 to 95W do not vary significantly for small specimens averaging \approx 102 MPa \sqrt{m} , compared to 97W is \approx 73 MPa \sqrt{m} [5-6]. However, standard deviation of small specimens for all four alloy compositions was found relatively high (up to ± 20%), which might be due to the local microstructural inhomogeneity (especially DP area %, see Figure 2). Therefore, additional tests were performed on small specimens to improve their statistics (now,

total 7 tests per alloy compositions at RT). However, after adding the new results, the average K_{Jm} still remains almost the same (\approx 100 MPa \sqrt{m} for 90-95W, and \approx 69 MPa \sqrt{m} for 97W) with a slightly improved standard deviation (Table 2). In all cases, small specimens show stable crack tearing. The small specimen's dimensions are nominally valid as per ASTM Standard E-1921 [8], however, we have tested a limited number of specimens that are 3x (M: 45x10x5mm) and 6x larger (L = 90x20x10mm) [7]. The results of all the medium-sized (B= 5mm) specimens, except 97W, show stable crack growth [7]. The toughness, KJm, slightly decreases with increasing W loading up to 95W, however, the averages are still within the range of standard deviations. However, K_{lc} for 97W is lower at 38 ± 4 MPa√m; note, it is still ≈ 5x higher than monolithic W ($\approx 8 \pm 2$ MPa \sqrt{m}) (see Table 2). The 6x larger (B = 10mm) 95W specimens showed mixed crack propagation responses. Two specimens, out of four, showed stable, ductile tearing, while other two failed unstably just after reaching maximum load, although the maximum load toughness is same (\approx 83 ± 9 MPa \sqrt{m}) as the medium size specimens (Table 2 and Reference [7]). Big oxide inclusions (~1mmx 0.7mm) near the crack tip were found in both brittle specimens and are likely the cause for the unstable crack propagation. However, in this study, we have also guantified the DP area% from the side surface of these large 95W alloys and found $\approx 8\%$ and $\approx 6\%$, marked the boundary between unstable crack propagation, even though they all are from the same plate-1 that averaged \approx 11% DP.

Specimen		K _{Jm/lc} (MF	Pa√m)	
geometry and size	90W	92.5W	95W	97W
3PB-Small	97 ± 17	96 ± 12	107 ± 14	69 ± 12
3PB-Med	92 ± 6	84 ± 11	75 ± 4	38 ± 4
3PB-Large	-	-	83 ± 9	-
3PB-XL	-	81 ± 7	80 ± 3	
CT-0.35T			63 ± 8	

Modified 0.35CT (or thickness \approx 9 mm, see Figure 1) specimens were fabricated from the tested large 3PB bars halves to ensure maximum utilization of materials. The P-d curves for CT specimens show most of the specimens developed unstable crack propagation after reaching maximum load (Figure 3a). The average K_{Jm} or K_{lc} is lower than the other 95W specimens tested in 3 or 4PB bending. Only limited process-zone microcracking was observed (Figure 3b), and the overall plastic process zone is much smaller than for the ductile tearing 95W. The SEM image from the fractured surfaces also reveals that the W-W particle separation and W-DP decohesion dominate the local fracture modes (Figure 3c). A lower amount of DP%, combined with weak, or no, W-DP interfacial bonding (Figure 3c,d) might be affecting the P-d behavior. System compliance might play a role for stable vs unstable crack propagation for 3PB large vs, CT specimens. Note, there was less tungsten cleavage (WC) in these CT specimens, which is normally a dominating factor for unstable crack propagation.



Fusion Reactor Materials Program



Figure 3. (a) Normalized load for a/W \approx 0.5, the P-d curves for the room temperature CT specimens; (b) side surface shows limited plastic process zone; (c) SEM fractograph shows WW and WD dominations; and (d) absence of DP between W particles, and at the surface that might be scruff off during mechanical polishing (due to weak interfacial bonding). Note, the image is from the unloaded sample, and not chemically etched.

Due to the inconclusive results for the large 95W specimens, we have tested two more 95W alloy with extra-large (XL) size (115 x 25.4x12.7mm, or ASTM 0.5T), fabricated from plate 2. Note, the average DP area % for the second 95W plate is slightly lower ($\approx 9 \pm 1.2\%$), than the average of 95W ($\approx 10.7 \pm 1.3\%$) for the first plate. Here, again, one of the specimens showed stable ductile tearing while the other fractured unstably (see Figure 4a). While stable crack growth 95W XL specimen shows all types of toughening mechanism, (i.e. dilatations, microcrack formation, blunt and opening, crack bridging, see Figure 4b), along with relatively similar local fracture mode fractions (i.e. WW, WC, WD, DP, see Figure 4c), as mentioned in previous reports for ductile tearing [6], the unstable 95W_XL shows WC followed by WW dominance of the local fracture mode (Figure 4d), which is responsible for cleavage fracture [6-7]. Some loosely bonded, small tungsten particles were also observed (see inset of Figure 4c). Whether stable or not, the elastic component of fracture toughness for all the large and extra-large (XL) 95W averaged ≈ 60 MPa \sqrt{m} , which is ≈ 7.5 times higher than pure W.

Fatigue precracked, XL size 3PB samples were also tested for 92.5W WHA at RT. Normalized P-d curves show very stable crack propagation in all cases (see Figure 4a). The average K_{Jm} is $\approx 81 \pm 7$ MPa \sqrt{m} , relatively unaffected by different specimen size (84 \pm 11 MPa \sqrt{m} for medium and 96 \pm 12 MPa \sqrt{m} for smaller specimens, Table 2). The SEM images taken from the side (Figure 4e), and fractured face (Figure 4f) show local fracture modes like those seen for the medium or smaller specimens for same alloy composition [6-7].



Figure 4. (a) Normalized for a/W = 0.5, RT load-displacement (P-d) curves for XL size 92.5 and 95W; (b) side surface of stable 95W_XL specimen showing the features of plastic process zone near the crack propagation path; local fracture modes for (c) stable, and (d) unstable fracture for 95W_XL. Figure (e) showing the arrays of microcracks that form on a W particle with load that helps to dissipate energy; and (f) local fracture mode for 92.5W XL sample.

Figure 5a shows normalized P/P_{max} vs d/S (displacement/span length) for all specimen size tested at RT. Note, only stable P-d curves are considered for 95W large and XL specimens. It can be easily seen that how specimens size play with the P-d response. The corresponding K_{Jm} (for stable fracture, filled symbol) or K_{Ic} (for unstable fracture, unfilled symbol) values are plotted at Figure 5b. Except for 97W medium or 95W CT specimens, all are within their respective standard deviations, although K_{Jm/Ic} slightly decreases with increasing W content. The percentage of DP area was also calculated from the bulk, undeformed specimens as well as from the tested specimens and are tabulated in Figure 5c and plotted in Figure 5d against K_{Jm/Ic}. It is estimated that $\approx 8\%$ DP is required for stable crack growth for medium to large specimens. Since, thickness (*t*) of the DP ligament does not vary significantly with higher W alloys (Table 1), it has minimal effect on stable crack growth.



(c)

Figure 5. (a) Normalized for sample size, the room temperature P/P_{max} vs d/S; (b) corresponding toughness values; (c) tabulated, and (d) plotted form of DP% vs toughness response for all sample size and alloys.

Ongoing and Future Work

- A controlled environmental set-up and Inconel fixture is nearly ready for the high temperature (600 to 800°C) toughness testing for medium size specimen.
- A multi-mechanism toughening model will be developed.
- Thermal shock tests will be performed on these WHA's.

Acknowledgement

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4.3 SIMULATION OF MECHANICAL PROPERTIES OF TUNGSTEN COMPOSITES—L. M. Garrison, N. Jensen (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this project is to perform a finite element analysis of a tensile test simulation of tungsten composites to understand and optimize their properties for use as plasma facing components in fusion reactors.

SUMMARY

As a first step toward modeling tungsten composites, simplified tensile tests of individual tungsten foils and composites were simulated. To realistically define the material properties required for modelling with ANSYS, experimentally measured stress-strain data from uniaxial tensile tests were imported. Strength, elasticity, and other relevant material properties were manually defined for tungsten, steel, and copper. The tensile frame contacts were simulated by modelling circular contact surfaces where the frame and specimen touched. One side of the contacts were selected to be fixed supports and a velocity of 0.1 mm/min in the axial direction was applied to the other. A finite element analysis of the uniaxial tensile test simulation was solved for the stress, strain, and deformation in ANSYS. The ANSYS modeling project that was begun last year was improved in several ways to better reflect the physical experiments.

PROGRESS AND STATUS

Introduction

For the plasma-facing components of fusion reactors, tungsten will be the interface between the plasma and the underlying structural component because tungsten has a low sputtering yield, high melting temperature, and relatively high thermal conductivity. However, because tungsten is brittle and has low fracture toughness, tungsten composites are required. The good thermal properties make tungsten composites promising, but their behavior under relevant fusion conditions is not yet predictable. For instance, initial tests of tungsten copper laminate composites had good unirradiated tensile ductility, but little to no ductility after neutron irradiation [1]. Joints between tungsten and other dissimilar materials are also required but are not the focus of this report.

There are several types of tungsten composites being considered in the fusion community, including sintered, laminate, and tungsten-fiber reinforced materials. Many of these options have been successfully fabricated on a trial basis, but they have not been optimized. However, simulation of tungsten composites facilitates easier parameter modification, anticipates material properties and behavior, and identifies trends while avoiding the costs of time and resource intensive fabrication.

Of the tungsten composites considered in the fusion community, the laminate composite was chosen to model first. To properly model a laminate with such complex interface properties, individual tungsten, steel, and copper foils must be accurately simulated to reduce error in modeling the composite material. Foils of varying thicknesses can then be combined in ANSYS Composite PrepPost (ACP) to model laminates. Individual foils and a variety of laminates will be tensile tested to compare material and mechanical properties.

Results

Input Parameters and Geometry

In this first round of testing, only the minimum material properties required to simulate a tensile test were defined for sample tungsten and steel materials in Tables 1 and 2, respectively. The simulated tungsten was tungsten foil with a thickness of 100 μ m. It was assumed that density would be unchanged during

fabrication, so the standard value of 19250 kg/m³ for tungsten metal was used. Isotropic elasticity was defined using Poisson's ratio and Young's modulus. In several previous studies, elasticity properties were found to be similar when tested for both tungsten metal and tungsten foil [2], [3], [4]. Values for the yield strength and ultimate tensile strength were taken directly from the experimental tensile tests for 100 μ m thick tungsten foil. Stress-strain data from uniaxial tensile tests performed at room temperature were imported to improve the material definition. These values defined the material "Tungsten Foil" in the Engineering Data for the project in ANSYS. The same process was followed to create the grade 92 steel foil.

Table 1. Material property input parameters to ANSYS model for 100 µm thick tungsten foil

Property	Value	Unit	Citation	Form
Density	19250	kg m ⁻³	[2]	Metal
Poisson's Ratio	0.28	-	[2], [3]	Foil
Young's Modulus	4.05E+11	Pa	[2], [3], [4]	Foil
Tensile YS	2167.91	MPa	Tungsten Foil Tensile Data W10a-1	Foil
Tensile US	2231.27	MPa	Tungsten Foil Tensile Data W10a-1	Foil

Tabla 2	Material	nroport	v input	narameters to	ANISVS m	nodel for 100) um thick sta	al grade 02 foil
i able 2.	Indrenat	property	y input	parameters to	ANS IS II		µnn unick sie	el glade 92 IUII

Property	Value	Unit	Citation	Form
Density	7800	kg m ⁻³	[Lizhen Tan]	Metal
Poisson's Ratio	0.29	-	[Lizhen Tan]	Foil
Young's Modulus	2.10E+11	Pa	[Lizhen Tan]	Foil
Tensile YS	1006.63	MPa	[Lizhen Tan]	Foil
Tensile US	1087.48	MPa	[Lizhen Tan]	Foil

The geometry for both the experimental and simulated tensile tests is referred to as SSJ2. This is a tensile bar with outer dimensions 16 x 4 x 0.5 mm. The design is shown in Figure 1. The materials properties for individual 100 μ m thick tungsten and steel foils were assumed to be the same when joined to build a 500 μ m thick layered composite.



Figure 1. SSJ2 geometry definition.

Mesh and Model Setup

The mesh, or division of the geometry into elements, that was automatically generated with ANSYS is shown in Figure 2. The size function used was "Proximity and Curvature". The mesh had a fine relevance center with 3265 elements, medium smoothing with 5 iterations, fast transition between element types, and a fine span angle center. These settings allowed for an adequately fine mesh and adequate precision solution for the general scope of this project without taking too much solving time or processing power.



Figure 2. Visual representation of geometry division into mesh elements.

The conditions for this first simulated tensile test were simplified and differ from the real experiment in several ways. There was only a fixed support (Figure 3 mark B) and one constant velocity of 0.1 mm/min (Figure 3 mark A), each applied at opposing contacts of the tensile bar. The modeled circular contacts were not a perfect representation of the real tensile testing procedure but are as accurate as possible within an finite element analysis (FEA) simulation. The velocity in the simulation was applied over 10 minutes, while all the experimental samples failed prior to that time.



Figure 3. A 0.1 mm/min constant velocity was applied to the right circular contact pieces over 10 minutes in the positive x direction with "A". A fixed support was applied across the faces of the left contact pieces marked with "B".

Results

Figures 4 through 8 show the results at 544 seconds which was when the specimen fractured, according to the stress-strain curves produced by the ANSYS software. The outline of the undeformed shape is only shown in Figure 4 and it is excluded in Figures 5 through 8 to better show the specimen. The complex, layered geometry required a fine mesh to make each layer have a thickness of more than one element. This resulted in a long solve time of approximately 3 hours. The points carrying the greatest load are predictably where the necking occurs, which is where failure will happen. The ANSYS is not able to simulate fracture accurately, and the large amount of necking is a result of instructing the simulation to run for longer

(larger strains) than would by physical for these materials. However, for initially evaluating the model, it is useful to have the drastic and noticeable change in the simulated material.



Figure 4. Total deformation, including undeformed model.



Figure 5. Equivalent (von-Mises) stress.







Figure 7. Equivalent elastic strain.



Figure 8. Maximum principal elastic strain.

Future Work

A basic understanding of a tensile test simulation was obtained and several routes for improving this model were identified. The tungsten layered composites will be modeled without using ACP for comparison and verification. Further investigation of the fracture location will also be performed. Due to the perfect conditions of an FEA analysis, it is not known at this time why the specimen did not fracture exactly at the middle point. The simulated tungsten material retains the same geometry as the tested SSJ2 tensile bars, but a singular tungsten foil will have different properties depending on its thickness. The simulated tensile bar needs to match the geometry of the material in question. Results from an updated model also must be validated with experimental data. Additional foil properties may need to be measured to be included in the model.

Once the model sufficiently reflects experimental results, mesh optimization should be explored, using relevant finite element analysis literature. The velocity also needs to be applied over the same amount of time as the corresponding experimental tensile test.

Once the tungsten-steel foil simulation results are satisfactory, the tungsten-copper composite will be modeled. Before an ANSYS simulation can be performed, however, experimental data for copper foil needs to be acquired to define the material data.

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4.4 ANALYSIS OF MICROCRACKING IN W-Ni-Fe DPT ALLOYS AS A FUNCTION OF STRAIN DURING ROOM TEMPERATURE TENSILE TESTING—Charles H. Henager Jr., Ba Nghiep Nguyen, Jacob Haag, Zoe Gotthold, Esther Mutesi (Pacific Northwest National Laboratory)

OBJECTIVE

Computational materials design efforts of advanced W-based composites require substantial benchmarking and validation before such designs can be realized via advanced manufacturing methods. The Pacific Northwest National Laboratory (PNNL) is exploring brick and mortar microstructures as part of our ductilephase toughening approach to tungsten divertor materials, and such designs include understanding basic deformation mechanisms. This report documents methods to quantify observed microcracking in miniature tensile specimens strained to varying amounts including tested to failure. The main purpose of this study was to develop measures to analyze the microcracking that occurs when W-Ni-Fe tensile bars are strained and plastically deformed. Scanning electron microscope (SEM) images were taken of strained samples, including *in situ* SEM tensile bars being strained over time. By analyzing this microcracking, hopefully the final fracture path can be predicted.

SUMMARY

This study examines the ability of W-Ni-Fe composites to withstand microcracking prior to final fracture. To visualize the distribution of cracking along tested miniature tensile specimens, a system to translate a sample image into a heat map of crack density was developed. Using code written in ImageJ, the original image was translated into black and white to identify the cracks. A heat map of crack density was then generated using the resulting crack image data. We also used an ImageJ plugin macro to measure the orientation of each crack with respect to the tensile axis and determined that the average crack orientation was approximately 90 degrees from the tensile axis and followed a normal distribution. By understanding the behavior of this distributed microcracking, we hope to develop a predictive model of the ultimate failure site within such composites. Currently, we are working on understanding whether existing cracks grow under stress or if new cracks form within the material.

PROGRESS AND STATUS

Introduction

The W and W-alloys are the solid materials of choice for plasma-facing components (PFCs) of future fusion reactors, such as the International Thermonuclear Experimental Reactor (ITER) and Demonstration Power Plant (DEMO), due to their high melting point, strength at high temperatures, high thermal conductivity, low coefficient of thermal expansion, and low sputtering yield [1-3]. However, W and most W-alloys exhibit rather low fracture toughness and a high ductile-brittle transition temperature (DBTT) that would render them as brittle materials during reactor operations [1, 3, 4]. The DBTT for unirradiated W-alloys typically ranges from 573K to 1273K (300° C to 1000° C), and in a reactor environment radiation hardening would further elevate this range [3, 5, 6]. The W-alloys toughened by engineered reinforcement architectures, such as ductile-phase toughening (DPT) are strong candidates for PFCs. Our efforts have focused on developing microstructures that exhibit improved fracture toughness based on hot-rolled W-Ni-Fe composites. We recently tested some of our 87%-rolled W-Ni-Fe composite materials previously reported using a miniature tensile test at room temperature. Additionally, a miniature tensile sample was tested in a SEM at Virginia Tech (VT) in an *in-situ* test apparatus. Post-test SEM examinations at PNNL revealed a distribution of microcracks in the specimen prior to final fracture. Similar results were observed at VT during SEM *in situ* tensile straining tests.

Computational Procedure

Crack Density (Heat) Maps

Starting with an original SEM image, we used ImageJ to split that image into 624 blocks, where each block corresponds to an area of $100 \times 100 \ \mu m^2$. We split the entire image into a binary mode, based on whether a given point is part of a crack or part of the surrounding material. These individual images were then analyzed for percent white, or the percent of cracking present in each pixel. The percent crack area for each pixel was fed into a JAVA program to be normalized to the values 1-6. These values were then saved in a 2D array and used to generate a graphical representation of the crack density across the sample. Figure 1 is an original image of a strained tensile bar of 87%-rolled W-Ni-Fe material with the tensile axis along the transverse rolling direction. The bar has been strained to a significant fraction of total elongation but prior to failure. Figure 2 is a binarized image (Black and White) showing the cracks as white pixels. Figure 3 is the crack density map from Figure 2. Figure 4 is all of the crack density maps from the four tested miniature tensile samples including the image in Figure 4d for a fractured sample.



Figure 1. The SEM image montage assemble from 300x magnification images such that the width of the tensile sample shown here is 1.1 mm.



Figure 2. Binarized image (Black and White) used in ImageJ analysis based on the tensile bar image in Figure 1.



Figure 3. Heat map of cracking density based off binarized image of cracking using 6 color values. Each pixel in this image represents 100x100 μ m² in real units from Figure 1.



Figure 4. Additional crack density maps for all tensile bars shown in order of increasing strain level (a) to (d), including final fracture in (d).

Crack orientation and centroid distributions

In addition to crack density, crack orientation with respect to the tensile axis was also determined using ImageJ. We fed the binary image shown in Figure 2 into an ImageJ plugin BioVoxxel that was created for advanced particle analysis to calculate the orientation of individual cracks. This BioVoxxel plugin allows for an expansion of the regular particle analysis capabilities. Specifically, it allows for the calculations of the orientations of irregular shapes, such as the object shown in Figure 5. This also works on cracks, which are not perfectly linear. A crack angular orientation histogram was created representing the distribution of orientation angles for each crack on sample shown in Figure 4c.



Figure 5. Representation of how the particle analysis works within the BioVoxxel macro. An average orientation angle is determined for an arbitrary shaped particle, which includes jagged-shaped cracks.



Figure 6. Crack angular orientation distribution using BioVoxxel macro and the binarized image for the sample shown in Figure 4c.

We then used ImageJ to find the center of a bounding box for each crack using cartesian coordinates. Using these coordinates, we map each crack on a scatterplot overlaid on the tensile sample image to determine the location of individual cracks. This differs from the heatmap as this method counts individual cracks, not just the density. In this method, smaller cracks will be overrepresented. Figure 7 illustrates the concept of a bounding box and how ImageJ represents the crack centroid based on the bounding box. Figure 8 shows the resulting crack location diagram where the crack centers are plotted with respect to the tensile image. This data will be used to characterize crack spatial distributions in future studies.


Figure 7. This image illustrates a bounding box around a crack, with the crack center selected.



Figure 8. Binarized image (Black and White) in ImageJ shown in (a) and resulting crack location in pixels plot shown in (b). This data will be used to further characterize crack spatial distribution in the future.

Future Work

We plan to continue working on analyzing the distribution of crack locations within each sample in order to understand microcracking as a function of tensile strain for these WNiFe materials. We plan to create heat maps over time of a single tensile sample being tested *in situ* in an SEM, in order to determine a prediction technique of the ultimate failure site.

Acknowledgements

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4.5 ELEMENTAL CHARACTERIZATION OF NEUTRON IRRADIATED TUNGSTEN USING THE GD-OES TECHNIQUE—L.M. Garrison, N. Reid (Oak Ridge National Laboratory)

OBJECTIVE

The depth dependent concentration of trace transmutation elements in neutron-irradiated tungsten will be measured as a part of the PHENIX collaboration.

SUMMARY

Tungsten undergoes a transmutation reaction chain to Re and subsequently Os under neutron irradiation. Precipitates that form in neutron-induced transmutation can cause changes in microstructure, mechanical, and thermal properties. Glow discharge optical emission spectroscopy (GD-OES) will be able to determine the concentration of Re and Os with respect to depth. To prepare this technique for irradiated tungsten samples, a mounting system was designed and machined. Glow discharge parameters were optimized for bulk W. Unirradiated model W-Re alloys were used for calibration of Re content to an internal standard value determined by third party elemental concentration measurements.

PROGRESS AND STATUS

The GD-OES utilizes a capacitively-coupled glow discharge argon plasma with a radiofrequency (rf) source and an optical emission spectrometer that consists of transfer optics, a grating, and a polychromator with detectors situated around a Rowland circle. It gives fast surface analysis and rapid depth profiling, allowing measurement of elemental quantities from surface to bulk. It can achieve nanometer-depth resolution, with parts-per-million (ppm) sensitivities for most of the elements in the periodic table as well as their isotopes. By sputtering the surface of the sample, ionizing the eroded material, and measuring the optical emission of the excited atoms, GD-OES can measure elemental information in a sample. An emission spectrum from electronic transitions occurring by sputtered atoms in the glow discharge plasma is picked up by the spectrometer. Each element is defined by the spectral lines that it produces when in the excited state. The GD-OES instrument is powerful, but required a special mounting system for small samples, a parameter optimization, and a calibration for it to successfully be used with our samples.

Tungsten samples that were irradiated in the RB*19J capsule in High Flux Isotope Reactor (HFIR) at temperatures between 450-1200°C to doses of ~0.2-0.7 dpa will be analyzed by the GD-OES technique using the glow-discharge (GD) profiler 2 instrument purchased from HORIBA Labs. This irradiation caused W transmutation reactions to occur, forming Re and Os ternary irradiation-induced precipitates. Due to the energy level of neutrons produced in HFIR, and the resonance peaks in the neutron energy spectrum for W, most of the transmutation reactions occurring are by neutron capture by the ¹⁸⁶W(n, γ)¹⁸⁷W reaction to produce Re-187, then ¹⁸⁷Re(n, γ)¹⁸⁸Re reaction to produce Os-188.

The GD Profiler 2 instrument is typically used for many other commercial or scientific needs where the sample size is relatively large, as compared to typical irradiated material sample sizes. The instrument can easily accommodate flat material pieces with diameter or rectangular dimensions of about 0.5 to 1 inch. For irradiation experiments, the size of the samples is minimized to limit the activated volume. For the RB*19J capsule of the PHENIX irradiation campaign, the samples of interest include 3-, 6-, and 10-mm diameter disks and SSJ-2 tensile end tabs (4 by 4 mm square). The GD Profiler 2 is designed such that the sample is placed directly in front of the plasma anode and must make a seal with an O-ring (Figure 1). The O-ring has an inner diameter of 11-mm, and so our smaller samples are mounted into an aluminum tab with larger dimensions than the O-ring to be vacuum-tight; otherwise, it will allow air impurities to leak into the Ar

plasma. The size of the anode determines how large a crater is being eroded into the sample surface. With smaller anode sizes, the main elements in the material will be observed. However, less light will be produced from low concentration elements, so they will not be detected as effectively. We can make use of a larger 4-mm diameter anode with the sample mounting tabs (Figure 1). A vacuum seal is made by using a thermally conductive substrate to affix the samples to. Aluminum was chosen for this purpose and was designed to center the sample precisely to the middle of the anode orifice (Figure 1). The sample is adhered to the aluminum using thermally conductive silver resin. When loaded onto the GD-OES (as in Figure 2), the sample must be flat enough to have no greater than 20 microns difference in height at any point in the surface. Polished samples work best to create the correct seal. A capacitively-coupled glow discharge plasma is ignited between a copper anode and sample, which acts as a cathode with the cooling block and rf source pressed against the back side. The front side (plasma-facing) is sputtered by ~50 eV Ar ions, and the depth is monitored by a differential interferometry profiling (DiP) laser. This allows simultaneous atomic emission and depth measurement.



Figure 1. Exploded view shows the sample glued to a custom-designed aluminum tab with silver conducting paint, a centering disk with tight tolerance over the ceramic, and the O-ring, anode, and DiP location.



Figure 2. Cross section illustration of the sample and plasma region of the GD-OES. The depth of sputtering is measured in-operando with sputtering of the sample by using the phase difference between a reference beam at the surface of the sample and an analysis beam inside the crater that is formed via sputtering.

The calibration procedure is critical for accurate quantification of trace elements and is a step in obtaining a compositional depth profile (CDP). Figure 3 shows a basic schematic of how CDP is obtained using the raw polychromator data, DiP, and calibration of standards. The standards being used are W-Re or W-Re-Os alloys with known bulk concentrations as measured by ICP-OES by an external company. These are inputs for data points on a calibration curve for each element of interest. Current concentration curves with four different Re concentration amounts are given in Figure 4 for the elements W, Re, and Os (zero for all W-Re alloys measured). The parametrization of raw data, DiP, and calibration curves are done by the software to produce a wt% versus depth plot, shown in Figure 5.



Figure 3. The CDP can be obtained with sputtering rate calibration. With the combination of GD-OES, DiP, and standards with known concentration, we can observe spatial heterogeneity of Re and Os transmutation products.



Figure 4. a) W, b) Re, and c) Os calibration curves using a continuous rf plasma measurement. The W-Re-Os alloys will be used for calibration of Os in future work, as well as alloys with concentrations in the middle range of 1-15 wt%. With more standard materials used, higher-order polynomial calibration fits will be obtained, statistical and propagation error reduced, and less contribution from statistical outliers.



Figure 5. Pulsed and continuous rf sputtering of an unalloyed W sample done in series to obtain a CDP. Sputtering rate can be controlled to get surface-sensitive quantification. The radiofrequency (RF) source was pulsed at 100 Hz for 5 min and then run continuously for 5 minutes. Both sputtering methods require independent calibration.

The W and Re signals measured in Figure 5 demonstrate two different rf sputtering modes that GD-OES can perform. The rf plasma can be run continuously (normal) or pulsed at a user-specified frequency and duty cycle. The advantages of running the plasma pulsed is that the sputtering rate of the surface is done at a controlled pace and allows time for sputtered atoms to be pumped out, as well as preventing certain other artifacts such as self-absorption. The sacrifice of running pulsed is that less light is emitted from the sample as the frequency is reduces, degrading the sensitivity of the measurement. Running pulse mode sputtering also relies on the user to specify the rf capacitance beforehand, which may lead to higher reflected rf power and thus less light emission and degraded sensitivity. Any changes in the rf parameters creates a plasma with different properties, and hence a calibration needs to be done for any case where the pulse frequency, duty cycle, power, or ambient gas pressure is changed. Pulsed mode was calibrated only for the unalloyed W sample and WRE5, giving it a larger error band but resulting in much less noisy data (Figure 5). More standards can be calibrated to give a transition from pulsed to normal without any breaks.

The pulsing conditions of 100 Hz at a 0.5 duty cycle are preferred for measurement of PHENIX sample transmutation elements. Calibration will be done to capture a sufficient set of Re and Os concentrations on the calibration curve ranging between ppm amounts to near the solubility limits of these elements in W. The hot samples will be affixed to aluminum substrates and centered with the procedure to achieve a vacuum-tight measurement.

4.6 COUPLED IRRADIATION INDUCED GRAIN GROWTH AND DAMAGE EVOLUTION IN SOLUTE STABILIZED NANOCRYSTALLINE TUNGSTEN—W.S. Cunningham, J.R. Trelewicz (Stony Brook University), K. Hattar (Sandia National Laboratory)

OBJECTIVE

The unique thermodynamic state occupied by nanocrystalline alloys presents opportunities for designing materials against instabilities while simultaneously enhancing their radiation tolerance through the deliberate introduction of defect sinks. The objective of this study is to investigate evolution of the defect microstructure in nanocrystalline W-20 at.% Ti subjected to heavy ion irradiation and compare the response to damage trends for unalloyed nanocrystalline W. Focus is on the coupling between nanostructure stability and defect state, which are collectively mapped through in situ measurements and bridged to high-dose stability using ex situ experiments.

SUMMARY

In this study, we probe the coupling between microstructural evolution and irradiation damage state in nanocrystalline W-20 at.% Ti using ion irradiation experiments. Defect evolution is mapped up to 20 dpa through in situ measurements and bridged to high-dose stability using ex situ experiments up to 400 dpa. The nanostructure is shown to exhibit a transient peak damage state followed by a reduction in defect density, which is attributed to the interaction of defect loops with migrating grain boundaries during irradiation induced grain growth. Grain size stabilizes around 50 nm, still well within the nanocrystalline regime, and is shown to be accompanied by a saturation of the loop density that continued out to the maximum dose of 400 dpa.

PROGRESS AND STATUS

Nanocrystalline W (NC-W) and W-20 at.% Ti (W-Ti) films were magnetron sputtered to a thickness of 50 nm. The W-Ti samples were annealed ex situ at 1000 °C for 1 hour whereas NC-W was annealed in situ at the same temperature, but with time limited to avoid coarsening of the microstructure. In situ heavy-ion bombardment was performed using the in-situ ion irradiation transmission electron microscopy (TEM) facility at Sandia National Laboratories (SNL). A 6 MV HVE Tandem Accelerator was employed to bombard the films with 1.7 MeV Au+1 ion in a JEOL 2100 (HT) TEM. The damage range and average production rate were determined using the Stopping and Range of Ions in Matter (SRIM) program2 with inputs matched to experiments. Diffraction contrast imaging-scanning electron microscopy (DCI-STEM) was employed in quantifying defect microstructures.

Images of the W-Ti alloy are shown in Figure 1a for the pristine condition and Figure 1b after 20 dpa. Precession electron diffraction maps overlaid on each figure and shown magnified in Figure 1c were used in identifying grains under favourable diffraction conditions for quantifying loop density and area, which are depicted in Figure 2a and 2b, respectively. A transient regime was evident in the W-Ti alloy and followed by a plateau after 20 dpa with defect densities greater than NC-W but average loop areas generally 25% smaller. The reduced loop size produced less nondimensional total damage (density scaled by area) in W-Ti relative to W as shown in Figure 3a, and an increase in the mean grain size in Figure 3b was observed to align with the transient damage regime. The underlying mechanism relates to defect loop annihilation at grain boundaries as evidenced in Figure 4, which is further substantiated by the coupling between the reduction in the defect density and growth of a single grain as illustrated in Figure 5.

Acknowledgements

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FIGURES



Figure 1. The DCI-STEM images of (a) pristine W-20 at.% Ti and (b) after in situ bombardment with 1.7 MeV Au⁺¹ ions to 20 dpa; overlaid precession electron diffraction (PED) maps are also shown with an inverse pole figure colormap in (b). (c) Magnified view demonstrating the selected area diffraction patterns for specific grains in the PED map (labeled) with defect loops also evident.



Figure 2. (a) Average loop density and (b) average loop area as a function of dose (logrithmic scale) for the present nanocrystalline W-20 at.% Ti and W films (solid symbols) with literature data summarized for other nanocrystalline W samples (open symbols).



Figure 3. (a) Nondimensional total damage (density scaled by the area) and (b) average grain size as a function of dose. The transient damage and grain growth regimes occur over nominally identical dose ranges.



Figure 4. Bright-field TEM images demonstrating a defect loop migrating to a grain boundary over time from (a)-(d).



Figure 5. Bright-field TEM images of a grain growth event in (a)-(d), which is mapped in (e) as a function of x-y position in the image. (f) Grain area and defect density mapped as a function of dose for the demarcated grain demonstrating a coupling between the two processes.

4.7 HELIUM ION IRRADIATION OF POLYCRYSTALLINE AND DUCTILE-PHASE TOUGHENED TUNGSTEN AT 973 K—Weilin Jiang, Alan Schemer-Kohrn, Giridhar Nandipati, Wahyu Setyawan, Charles H. Henager Jr., Richard J. Kurtz (Pacific Northwest National Laboratory)

OBJECTIVE

The aim of this experimental work is to study the void properties (diameter, number density and spatial distribution) in polycrystalline tungsten (poly W) foils irradiated with He ions in transmission geometry. The data will be used to validate predictions obtained from Object Kinetic Monte Carlo (OKMC) simulations. Helium cavities in poly W bulk irradiated under identical conditions will also be investigated and their properties will be compared to those of the voids. In addition, irradiation effects at W and W-Ni-Fe interfaces in ductile-phase toughened (DPT) W will be examined.

SUMMARY

Poly W foils of ~10 μ m thickness was prepared using mechanical grinding and lapping. Further thinning of the foils to ~4 μ m was achieved using an electrochemical method. Transmission electron microscopy (TEM) grid holders for vacuum heating irradiation were fabricated from low-carbon 316L stainless steel. Simultaneous irradiation of one poly-W foil, one poly-W bulk, and one DPT W bulk (90W-7Ni-3Fe) was performed at 973 K using a defocused 4 MeV He²⁺ ion beam to a local dose of 0.25 dpa at a depth of 2 μ m. The dose rate was ~8.3×10⁻⁷ dpa/s for an irradiation time of ~84 h. Helium ion microscopy (HIM) was performed for the irradiated bulk samples. Some nanoscale features were observed, but the data are not conclusive at this time. Twin-jet electropolishing of the irradiated foil will be performed. Liftouts for the irradiated bulk sample will be also prepared using focused ion beam (FIB), followed by flash polishing (FP) to electron transparency. Scanning TEM (STEM) will be used to characterize the samples.

PROGRESS AND STATUS

Introduction

Our recent study [1] of self-ion irradiation of monocrystalline tungsten (mono-W) and poly-W has suggested that the defect production rates in mono-W irradiated to 1 dpa at 900 K are ~6 times lower at 10⁻⁴ than for 10⁻³ dpa/s; the average void diameter is ~60% larger at 10⁻⁴ than for 10⁻³ dpa/s in mono-W, while it is ~40% larger in poly-W. The results from the study show a considerable dose-rate effect on both defect production rate and void growth even in the relatively small range of dose rates at 900 K. In order to better emulate neutron irradiation effects (typically dose rates on the order of 10⁻⁷ dpa/s or lower) by ion beams, a significantly lower dose rate is needed, which is, however, not achievable by self-ion irradiation due to the large mass of W ions. This study employed light-ion irradiation to achieve a lower dose rate. As in our previous report [2], stopping and range of ions in matter (SRIM) simulations suggest that MeV light-ion irradiation in W can readily provide dose rates of 10⁻⁶ to 10⁻⁷ dpa/s. To minimize the effects from the implanted species, a transmission irradiation experiment is performed for this study. A careful selection was made for the ion species and energy as well as the poly W foil thickness based on multiple beam tests at the 3.0 MV tandem ion accelerator facility used in this study. A poly W bulk sample was also irradiated under the same conditions for comparison. Also included in the irradiation was a DPT W bulk sample to study possible modifications in the microstructure and composition at the interfaces between W and W-Ni-Fe phases.

Experimental Procedure

Experimental procedures in this study include (1) the design of a transmission irradiation experiment, (2) fabrication of the TEM grid holder, (3) preparation of poly W foils, (4) electrochemical thinning of the foils, (5) ion irradiation, (6) twin-jet electropolishing of the irradiated poly W foil, (7) FIB of the poly W and DPT W bulk samples, followed by FP to electron transparency, and (8) sample characterization using STEM and STEM-EDS mapping. Multiple beam tests at the Texas A&M University (TAMU) 3.0 MV NEC tandem ion

accelerator facility were performed and the optimal irradiation conditions were determined to be 4 MeV He²⁺ with a defocused beam current of ~300 nA over an area of 6 × 6 mm² at 973 K. Figure 1 shows the results from a quick Kinchin-Pease (KP) SRIM simulation for 4 MeV He ion irradiation in W. For a 4 µm thick W foil, there is a relatively flat region of displacement rates from 2.2×10⁻¹⁹ dpa/(ions/cm²) at the surface to 5.6×10⁻¹⁹ dpa/(ions/cm²) at 4 μ m. Nearly all of the He ions pass through the foil. From Figure 1, the average displacement rate in the middle of the foil or at the depth of 2 μ m is 3.2×10⁻¹⁹ dpa/(ions/cm²), where retention of the implanted He atoms is 9.1×10-21 at%/(ions/cm²). For a He²⁺ ion beam current of ~300 nA over an area of 6 × 6 mm², ion flux is ~2.6×10¹² (He²⁺/cm²)/s and the corresponding



Figure 1. Quick Kinchin-Pease SRIM13 simulation results of 4 MeV He ion irradiation in tungsten for depth profiles of displacement rates and He atoms. E_d : threshold displacement energy; E_b : lattice binding energy.

dose rate at the depth of 2 μ m is ~8.3×10⁻⁷ dpa/s. For a total dose of 0.25 dpa at 2 μ m or an ion fluence of 7.8×10¹⁷ He²⁺/nm², the required irradiation time is ~84 h. Under the irradiation conditions, the retained He concentration at 2 μ m is only ~70 appm. The actual concentration could be even lower due to likely He out-diffusion and release during irradiation at 973 K.

A high-temperature sample holder was fabricated for the experiment. The holder was initially made of yellow brass (~60% Cu, ~33% Zn, ~3% Mn, ~2% Fe, ~1% Al and ~1% Sn) with a melting point of 1173 K, thermal conductivity of 109 W/(m·K), and Brinell hardness of 100. Tests showed significant Zn out-diffusion and evaporation occurred during heating in vacuum at high temperatures (\geq 650 °C). This behavior has also been reported in the literature [3]. For this reason, vacuum compatible low-carbon 316L stainless steel was



Figure 2. (a) TEM grid holder made of low-carbon 316L stainless steel, and (b) tungsten foil of ~10 μ m in thickness on a Au ring.



Figure 3. Electrochemical thinning and FP equipment with (a) electrical apparatus: ammeter, timer and power supply, (b) chemical apparatus: beakers, cooling baths, and stir plates, and (c) tweezer jig. (d) Grainger point micrometer and a micrometer stand for foil thickness measurement.

chosen for fabrication of the TEM grid holder, as shown in Figure 2a. Up to 4 W foils can be mounted on the holder for simultaneous irradiation. The opening area for holding thin foils has a diameter of 2.1 mm. The overall diameter of the holder is 18 mm. A number of ~10 μ m thick poly W foils have been prepared using mechanical grinding and lapping methods. An example of the tungsten foil (B5) on a 3 mm diameter Au ring is shown in Figure 2b.

Further thinning of the foil to ~4 μ m was accomplished using an electrochemical method consisting of a 1% NaOH solution at a voltage up to 30 V for 1 h at room temperature. Figures 3a, 3b and 3c show the thinning equipment, including an electrical apparatus, a chemical apparatus, and a tweezer jig. The timer has not yet been used, but will be needed for FP. Shown in Figure 3d is the Grainger point micrometer and micrometer stand for foil thickness measurement. Repeated sequences of thinning and measurements were performed until the poly W foil thickness was ~4 μ m.

The schematic diagram in Figure 4 shows how the three samples were arranged on the 316L holder for the elevated temperature ion irradiation. The samples are one poly W foil (B5) of ~4 μ m in thickness with a circular area in the holder opening of diameter 2.1 mm exposed to the beam, one poly W bulk of 5 × 10 × 1 mm³ in dimension with an area of 3 × 5 mm² exposed to the beam, and one DPT W (90W-7Ni-3Fe; while W grains are nearly pure, the W-Ni-Fe matrix phase is a solid solution with ~23 wt.% W, ~55 wt% Ni, and ~22 wt.% Fe [4]) bulk of 4 × 13 × 2 mm³ in dimension with an area of 3 × 3 mm² exposed to the beam. The irradiation area was 6 × 6 mm², as indicated in Figure 4. Also included in Figure 4 is a picture of the foil mounted in one of the holder openings and two bulk samples clamped on the holder after irradiation. The irradiated area is marked in the figure. Irradiation was performed with a broad continuous (defocused) 4 MeV He²⁺ ion beam at normal incidence. The average ion flux was ~2.6×10¹² (He²⁺/cm²)/s, corresponding to a dose rate of ~8.3×10⁻⁷ dpa/s at 2 µm depth. A total ion fluence of 7.8×10¹⁷ He²⁺/nm² or 0.25 dpa at 2 µm was applied over ~84 h, as designed. The irradiation was performed at 973 K. A resistance heater was



Figure 4. Ion irradiation of a poly W foil, poly W bulk, and DPT W bulk at 973 K using a defocused 4 MeV He²⁺ ion beam over an area of 6×6 mm² to a dose of 0.25 dpa at the depth of 2 μ m.

located behind the copper plate on which the sample holder was mounted. The temperature was monitored using a thermocouple on the copper plate.

Results

The irradiated samples were examined using HIM at the Pacific Northwest National Laboratory (PNNL). Figure 5 shows two representative images at different resolutions for the irradiated poly W bulk. There are regions of dark contrast in Figure 5a. Similar features were also observed in our previous study of self-ion irradiated mono-W at 900 K. Previous STEM-EDS mapping data suggested local surface oxidation in the darker-contrast region. There are particles of a few hundred nanometers in size on the surface, which could potentially originate from surface polishing process or other sources. The smaller surface features (a few to tens of nanometers) shown in Figure 5b are not identified at this time. It remains to be further investigated whether they are small particles or He blisters. Similar features were found to be H blisters near the surface of a poly W sample irradiated with 6 keV H₃⁺ to 10^{19} H⁺/cm² or 80 keV H₂⁺ inradiated poly W sample after thermal annealing up to 873 K for 1 h [6].

Further thinning of the irradiated poly W foil to electron transparency will be performed using a twin-jet electropolishing technique. The sample will be examined using a Cs-aberration corrected JEOL ARM 200cF STEM at PNNL. Void properties in W grains with different sizes will be studied, including diameter, number density and spatial distribution. A particular focus will be on small grains less than 1 μ m in diameter with the intent of validating the OKMC predictions.

In addition, FIB will be used to prepare liftouts from the poly W bulk sample. The FP process will be applied to remove the layer of Ga⁺ ion damage due to FIB. The samples will also be examined using STEM and He cavity properties will be determined as a function of depth. The properties will be compared to those of the voids in the poly W foil under the same irradiation conditions. The effort is intended to provide experimental data and study He effects on cavity formation and growth in W. Research on He and vacancy interaction in W has been reported in the literature [7-9]. Future investigations may also include determination of the He depth profiles in He ion implanted W.

The irradiated DPT W bulk sample will follow similar steps as for the poly W bulk sample to study He²⁺ induced damage at the W and W-Ni-Fe interfaces. The STEM and STEM-EDS mapping will be performed for both unirradiated and irradiated DPT W. Lattice disordering and possible atomic intermixing at the W



Figure 5. HIM images of poly W irradiated at 973 K with 4 MeV He²⁺ ions to a dose of 0.25 dpa at the depth of 2 μ m.

and W-Ni-Fe interfaces will be investigated. Several intermetallic phases of W, Fe and Ni at elevated temperatures have been reported [10]. In addition, possible He cavities in the grain interiors and/or along the grain boundaries will be examined. The data from this study will also be valuable for studies of irradiation effects at interfaces between W and stainless steel.

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4.8 MECHANICAL PROPERTIES OF IRRADIATED TUNGSTEN - PHENIX COLLABORATION—L.M. Garrison, J. R. Echols, T. Miyazawa, N. Reid (Oak Ridge National Laboratory)

OBJECTIVE

The PHENIX collaboration on tungsten irradiation aims to expand the database on neutron irradiation data for tungsten materials.

SUMMARY

The RB*19J capsule irradiated in HFIR included over 20 varieties of tungsten in three temperature zones, nominally 500, 800, and 1200°C. Tensile tests on eight materials from the 500 and 800°C zones have been completed at 500 and 700°C respectively. Fracture surface analysis was completed in the scanning electron microscope (SEM). A new alumina fixture for the tensile frame was designed and will allow testing up to 1100°C. The new fixture was installed and the tests from the 1200°C zone have begun.

PROGRESS AND STATUS

Tensile Results

Tensile tests of the eight US materials irradiated in the 500 and 800°C zones were completed (Table 1). The range of irradiation temperatures in the "500°C sub capsule" was 432 to 671°C, and the tensile tests were all performed at 500°C. The range for the "800°C sub capsule" was 739 to 979°C and tensile tests of samples from this sub capsule were performed at 700°C. In addition to the irradiation temperature difference, the 800°C sub capsule received a higher neutron dose than the 500°C sub capsule as it was closer to the centerline in HFIR. The average dose to the tensile samples in the 500°C sub capsule was 0.25 dpa while the average was 0.72 dpa in the 800°C sub capsule. It is postulated that the increased dose was the dominant factor in the tensile behavior rather than the irradiation temperature. More samples had a ductile failure mode after irradiation to ~0.25 dpa and testing at 500°C, and more samples had a brittle failure after irradiation to ~0.72 dpa and testing at 700°C. Further analysis of the tensile curves and further testing where initial tests were invalid is underway.

Material Type	Sample ID	Irradiation Temperature (C)	Fast Fluence (n/m ² , E>0.1	Dpa	Test Temperature (C)	Type of failure
Thick plate orientation A	AT00	514	1.68E25	0.33	500	Ductile
Thick plate orientation B	BT00	503	1.57E25	0.31	500	Invalid
Thick plate orientation C	СТ00	495	1.48E25	0.29	500	Ductile
Alfa Aesar plate orientation B	3E00	437	9.77E24	0.19	500	Ductile
Alfa Aesar 2 mm foil	EE00	455	1.10E25	0.21	500	Brittle
Alfa Aesar plate orientation A	GE00	510	1.04E25	0.20	500	Ductile

Table 1. Tensile test results for tungsten materials irradiated in HFIR-RB*-19J

Single crystal (100)	HE00	466	1.18E25	0.23	500	Ductile	
Single crystal (110)	Single UE03 481 rrystal (110)		1.31E25	0.26	500	Ductile	
Single crystal (100)	JE00	464	1.16E25	0.23	500	Ductile	
Thick plate orientation A	AT08	776	3.54E25	0.69	700	Brittle	
Thick plate orientation B	BT0A	793	3.62E25	0.71	700	Brittle	
Thick plate orientation C	c plate CT0A 820 Itation		3.78E25	0.74	700	Invalid	
Alfa Aesar plate orientation B	3E05	745	3.49E25	0.68	700	Brittle	
Alfa Aesar 2 mm foil	EE05	793	3.72E25	0.73	700	Ductile	
Alfa Aesar plate orientation A	GE05	791	3.67E25	0.72	700	Ductile	
Single crystal (110)	UE0G	830	3.82E25	0.74	700	Brittle	
Single crystal (100)	JE05	825	3.78E25	0.74	700	Brittle	

High temperature tensile fixture

The irradiation temperatures ranged from 882 to 1085°C in the third region of the RB19J (the 1200°C sub capsule), and the tensile test temperature will be 900°C. To allow testing at temperatures of 800-1100°C, a new fixture was needed for the tensile frame in LAMDA. The fixture was designed and machined from alumina (Figure 1) so that it would have enough strength at these elevated temperatures. Because of the brittle nature of alumina ceramics, the fixture was designed with a pin connection to the pull rods instead of the typical screw joints that are used for metal fixtures. New pull rods for the tensile frame were machined from the molybdenum alloy TZM. The new fixture and rods have been installed in the tensile frame and testing is ready to begin on the next group of samples.



Figure 1. Alumina fixture with example tensile bar in place. The holes at top and bottom have pin connections to molybdenum alloy TZM pull rods.

SEM Fracture Surface Images

The samples tested so far (Table 1) were imaged in the SEM. One full width and one close image is included for each sample that was imaged. The fracture surfaces of the different tungsten materials are distinct. Sample HE00, a single crystal, has a nearly uniform flat cleavage across the gauge section. The elongated grains and partial delamination failure of the Alfa Aesar 2 mm foil can be seen. The Alfa Aesar plate tensile orientation A has clear horizontal elongation of grains, while Alfa Aesar plate tensile orientation B has vertical. The fracture surface gives useful information about the failure character and will be examined in more detail.

Note that for all the following fractography the captions are ABOVE the images they relate to.

JE00 – Single crystal tungsten (100) – 500°C Capsule; T_{irr} 464°C; 1.42E14 Fast Flux (n/cm² sec); 1.16E25 Fast Fluence (n/m²)



UE03 - Single crystal tungsten (100) - 500°C Capsule; T_{irr} 481°C; 1.62E14 Fast Flux (n/cm² sec); 1.31E25 Fast Fluence (n/m²)



JE05 (side a) - Single crystal tungsten (100) - 800°C Capsule; T_{irr} 825°C; 4.66E14 Fast Flux (n/cm² sec); 3.78E25 Fast Fluence (n/m²)



JE05 (side b) - Single crystal tungsten (100) - 800°C Capsule; T_{irr} 825°C; 4.66E14 Fast Flux (n/cm² sec); 3.78E25 Fast Fluence (n/m²)



UE06 - Single crystal tungsten (110) - 500°C Capsule; T_{irr} 476°C; 1.54E14 Fast Flux (n/cm² sec); 1.25E25 Fast Fluence (n/m²)



HE00 - Single crystal tungsten (100) - 500°C Capsule; T_{irr} 466°C; 1.45E14 Fast Flux (n/cm² sec); 1.18E25 Fast Fluence (n/m²)



AT00 – Thick plate tungsten orientation A - 500°C Capsule; Tirr 514°C; 2.07E14 Fast Flux (n/cm² sec); 1.68E25 Fast Fluence (n/m²)



AT08 - Thick plate tungsten orientation A - 800°C Capsule; Tirr 776°C; 4.36E14 Fast Flux (n/cm² sec); 3.54E25 Fast Fluence (n/m²)



BT0A - Thick plate tungsten orientation B - 800°C Capsule; T_{irr} 793°C; 4.46E14 Fast Flux (n/cm² sec); 3.62E25 Fast Fluence



CT00 - Thick plate tungsten orientation C - 500°C Capsule; Tirr 495°C; 1.82E14 Fast Flux (n/cm² sec); 1.48E25 Fast Fluence (n/m²)



EE00 - Alfa Aesar 2mm foil - 500°C Capsule; T_{irr} 455°C; 1.35E14 Fast Flux (n/cm² sec); 1.10E25 Fast Fluence (n/m²)



EE05 - Alfa Aesar 2mm foil - 800°C Capsule; Tirr 793°C; 4.58E14 Fast Flux (n/cm² sec); 3.72E25 Fast Fluence



GE05 – Alfa Aesar plate tensile A - 800°C Capsule; T_{irr} 791°C; 4.52E14 Fast Flux (n/cm² sec); 3.67E25 Fast Fluence (n/m²)



GE00 – Alfa Aesar plate tensile A - 500°C Capsule; T_{irr} 510°C ; 1.28E14 Fast Flux (n/cm² sec); 1.04E25 Fast Fluence (n/m²)



3E00 – Alfa Aesar plate tensile B - 500°C Capsule; T_{irr} 437°C; 1.20E14 Fast Flux (n/cm² sec); 9.77E24 Fast Fluence (n/m²)



3E05 – Alfa Aesar plate tensile B - 800°C Capsule; Tirr 745°C; 4.29E14 Fast Flux (n/cm² sec); 3.49E25 Fast Fluence (n/m²)



4.9 THERMAL PROPERTY EVALUATION OF NEUTRON IRRADIATED TUNGSTEN MATERIALS -PHENIX COLLABORATION—L. M. Garrison, S. Copp, S. Curlin, H. Wang, A. Masafumi (Oak Ridge National Laboratory)

OBJECTIVE

The purpose of this project is to collect thermal property data on tungsten-based materials to aid in future fusion reactor design.

SUMMARY

Eight tungsten samples of different diameters and thicknesses are being tested to confirm that their variable geometries ultimately have no effect on the measured thermal diffusivity. Additionally, sixteen unirradiated tungsten disk samples will be tested for thermal diffusivity using the LFA467 device. The companion irradiated tungsten samples from the RB*19J capsule irradiated in High Flux Isotope Reactor (HFIR) as part of the PHENIX collaboration will also be tested using the LFA467 at test temperatures up to their nominal irradiation temperature.

PROGRESS AND STATUS

The PHENIX US-Japan collaboration is evaluating tungsten for use in future fusion reactor divertors and is organized into three interrelated tasks: Task 1 to evaluate high heat flux effects; Task 2 to evaluate microstructure, thermal, and mechanical properties; and Task 3 to investigate plasma-surface effects. The thermal properties of neutron irradiated tungsten are measured in Task 2 at Oak Ridge National Laboratory (ORNL).

Sample Size Testing

Before any other tests are conducted, the size effects study must be completed. Each of the eight samples are unirradiated tungsten of various diameters and thicknesses cut from a single block of Alfa Aesar brand unalloyed tungsten. Each sample tested in Table 1 should have data points that fall along the same curve in the thermal diffusivity vs. temperature graph. It is critical that the data being collected in this first step is consistent for all sizes and shapes of the sample being tested before we move on to the irradiated samples. Samples will be tested from room temperature up to a maximum temperature of 800°C in 50°C increments using the LFA467 device.

Table 1. Unirradiated Alfa Aesar-manufactured unalloyed tungsten samples used for size effect study

Sample Type	Sample Diameter	Sample Thickness
Tensile Bar	n/a	0.5 mm
Disk	3 mm	0.5 mm
Disk	3 mm	1 mm
Disk	6 mm	0.5 mm
Disk	6 mm	1 mm
Disk	6 mm	2 mm
Disk	12.7 mm	2 mm
Disk	10 mm	1 mm



Figure 1. Pictured above is the LFA467, the device that will be used to measure the thermal diffusivity of all the samples in this study. (https://www.netzsch-thermal-analysis.com/us/products-solutions/thermal-diffusivity-conductivity/lfa-467-hyperflash/)

The LFA467 works by applying a short laser pulse to one face of the sample and measures the temperature difference on the opposite face using a detector. The LFA software will calculate a thermal diffusivity value based on the time it takes the sample surface to reach one half of its final temperature. Because of the device's four individual heaters and sample holders, along with its extremely fast laser pulse, it can test smaller samples – a feature extremely useful for our purposes as some of the samples being tested are as small as 3 mm diameter.

To date, six of the eight samples from Table 1 have been tested (Figure 2). The thermal diffusivity values of the samples lie on a fairly tight curve, with the exception of the sample D6T.5. Originally, this inconsistency was suspected to be due to the geometry of the sample since it is so thin and there have been difficulties with samples of this thickness in the past. However, the tensile sample (denoted by green circles in the graph) is also of 0.5mm thickness and has data that fits in the curve of the other samples. The LFA 467 does not have built in settings for samples that are 0.5 mm thick or for non-round samples. There are many options that can be tuned including the pulse width and half time, so these must be adjusted to find the accurate settings for each geometry. A set of parameters specific to each sample geometry is being developed.



Figure 2. Thermal diffusivity data collected on six of the eight samples listed in Table 2. The legend at the bottom of the graph lists first the diameter followed by the thickness of the sample.

Thermal Properties of Unirradiated Tungsten Samples

Once the size effect study has been completed and the results conclusive, the next step in Task 2 of the PHENIX project can be initiated.

The samples listed in Table 2 were chosen as companion samples to the materials that have already been irradiated at 500°C, 800°C, and 1200°C in the HFIR at the ORNL. These unirradiated samples will be tested for thermal diffusivity using the LFA467. Much like the samples used in the size effect study, the unirradiated tungsten samples will be tested up to no higher than 800°C from room temperature in 50°C increments in Oak Ridge's Low Activation Materials Development and Analysis (LAMDA) laboratory. The data collected from this test will be used to quantify the effect of neutron radiation damage on thermal diffusivity for a variety of tungstens and tungsten-rhenium alloys.

Table 2	2. Unirradiated	samples con	prised of the san	ne materials as	those that were	irradiated
		Sumples con	iphoed of the out	ne materiale as		madiated

Material Code	Disk Size	Engraved ID
00	6mm	To Be Determined
60	3mm	6007
80	3mm	800H
EE	3mm	EE01
FR	6mm	FRO6
KE	3mm	KE04
СТ	3mm	СТОС
P0	3mm	РООН
UE	3mm	UEOE
ZE	10mm	ZEOF
3R	6mm	3R0T
41	3mm	410H
5E	6mm	5E05
8E	10mm	8E0E
71	6mm	7120
YO	6mm	YOOM

PHENIX Irradiated Tungsten Thermal Properties

As part of Task 2 of the PHENIX project, the thermal diffusivity of the tungsten materials from the HFIR RB*19J capsule will be analyzed (Table 3). The LFA467 is to be used for these measurements because it has a fast-enough laser pulse that disk samples measuring 3 mm in diameter and 0.5 mm in thickness will be able to be measured. There are more than twenty types of tungsten-based materials included in the PHENIX irradiation program. Some materials with different identification codes in the irradiation matrix are, in fact, the same base material, which is denoted by the first two characters in the materials' ID code. The samples listed as "TBD" will be selected for testing once the inventory of irradiated samples in LAMDA is complete.

			Nominal Irradiation Temperature (°C)					
Material Type / Condition	Provider	Code	500		800		1200	
			ID	shape	ID	shape	ID	shape
Pure W (Rolled, 80%, X- direction)	Japan	P0	P00A	D3TH	P00C	D3TH	P00G	D3TH
K-doped W (Rolled, 80%, X-direction)	Japan	60	6009	D3TH	600E	D3TH	600G	D3TH
K-doped W-3%Re (Rolled, 80%, X- direction)	Japan	80	8009	D3TH	800E	D3TH	800G	D3TH
W-3%Re (Rolled, 80%, X-direction)	Japan	41	4109	D3TH	410E	D3TH	410G	D3TH
Alfa Aesar 2mm foil	US	EE	EE02	D3TH	EE05	tensile	TBD	tensile
Alfa Aesar plate*	US	FR	FR00	D6T1	FR01	D6T1	FR02	D6T1
PCW-0%Re (ORNL)*	US	KE	KE00	D6T1	KE01	D6T1	KE02	D6T1
W-0.2%Re (ORNL)	US	3R	3R0L	D6T1	3R0M	D6T1	3R0R	D6T1
W-3.4%Re (ORNL)*	US	5E	5E00	D6TH	5E01	D6TH	5E02	D6TH
Thick plate W	US	СТ	CT02	D3TH	CT05	D3TH	CT09	D3TH
Single crystal tungsten [110]	US	UE	UE02	D3TH	UE05	D3TH	UE08	D3TH
W PIM	US	ZE	ZE00	D10T1	ZE05	D10T1	ZE04	D10T1
W TiC	US	8E	8E01	D10T1	8E08	D10T1	8E0G	D10T1
Allied W	Japan*	00	TBD	D6TH	TBD	D6TH	TBD	D6TH
W-5Re*	Japan*	71	TBD	D6TH	TBD	D6TH	TBD	D6TH
UFG-W*	Japan*	Y0	TBD	D6TH	TBD	D6TH	TBD	D6TH

Those samples with a * in Table 3 indicate they will be needed for one or more tests in addition to thermal diffusivity. The order of testing on these samples is important.

As seen above in Table 3, each of the irradiated samples are grouped together by their nominal irradiation temperatures (°C). These irradiation temperatures will determine what the maximum test temperature will be for the sample's thermal diffusivity test. Those samples that have a nominal irradiation temperature of 500°C will be tested in the LFA467 for thermal diffusivity up to 400°C in 50°C increments from room temperature. The samples that were irradiated at 800°C will be tested for thermal diffusivity up to 700°C in 50°C increments from room temperature. Lastly, those having a nominal irradiation temperature of 1200°C will be tested in the LFA467 up to no higher than 800°C in 50°C increments from room temperature. The data from these tests will be compared amongst each other, as well as with their companion unirradiated samples listed in Table 2.

Procedure for Flash Thermal Diffusivity Testing of Irradiated Tungsten

This test procedure is developed for testing irradiated tungsten materials in the LAMDA lab at ORNL. The materials have been irradiated at the HFIR under the PHENIX collaboration. Typical flash diffusivity tests and analysis follow the ASTM 1461 Standard. Special procedures are developed to measure very thin (0.5 mm) and small (3 mm diameter) samples which are also prone to oxidation above 300°C.

Instrument

NETZSCH LFA467 Hyper Flash HT: 20-1200°C

Sample Preparation

- Disk samples 3 mm, 6 mm or 10 mm in diameter should be machined for SiC holders.
 - 10 mm diameter samples should use a 10 mm graphite insert ring that fits into the 12.7 mm diameter standard SiC holder. Another 10 mm diameter ring and SiC shield should be placed on top of the sample to prevent light leaks from the flash.
 - ➢ 6 mm diameter samples should use the SiC holders from Netzsch.
 - 3 mm samples should use special sample holder assembly designed in Japan and made from graphite or tantalum. It fits into the 12.7 mm diameter SiC holder (a 10 mm graphite ring is needed for the tantalum holder). SiC shield is placed on the top.
- 4 mm x 4 mm square samples or broken tensile specimens.
 - Special graphite sample holder should be used. The holder fits into the 12.7 mm diameter SiC holder. The SiC shield is placed on the top.
- <u>Thickness measurement</u>: Sample thickness should be measured with a micrometer to <u>+</u> 0.001 mm. Multiple readings are needed to confirm the sample surfaces are parallel. Point-tipped micrometer is needed to read the thickness of 3 mm diameter samples.
- <u>Surface preparation</u>: For polished samples, a thin coating is applied to both surfaces. Special Netzsch graphene spray should be used for the tungsten samples, especially the 0.5 mm thick samples. The spray contains nanoplatelets graphene and is available from NETZSCH under the name GRAPHITE by CRAMOLIN®. Only a very thin layer of spray is needed on each side. (proper spray technique is needed: no direct spray to the specimen; the nozzle should be pointed 10 cm above the sample and let the graphite "drop" to the surface, and the minimum distance of the nozzle from the sample should be 25 cm.

Loading Tungsten Samples

- The LFA467 detector/furnace shield assembly can only be removed when the system is at atmosphere pressure (the UNLOCK light should be green). The assembly can be moved back by holding the interlock release button (on the right side of the system) while pushing down the "OPEN" button at the from control panel.
- The LFA467 has 4 stand-alone furnaces. The top window should be opened using the special tool from Netzsch. Samples (with holder and shield) described in the last section should be placed inside the furnace using the special tweezer. The top window is then placed back and tightened (no excessive force is needed, "finger-tight" should be enough).
- The detector/furnace shield assembly is move back by holding the interlock release button and "CLOSE" button in the front panel simultaneously.

Preparing the LFA467 for Testing

- Liquid nitrogen should be filled to the InSb IR detector using the funnel. Slow/multiple-step filling is recommended when the detector is empty to avoid pressure build-up inside the detector. Gloves and eye-shield are required while filling the liquid nitrogen.
- The LFA467 Hyper Flash HT is connected to a UHP Argon tank and equipped with a rotary pump (RP) and a turbo-molecular pump for vacuum operations.
- Purging the system:
 - Open the test control software (Proteus®) and open the windows for "Vacuum Control" and "Multi-channel Gas Controller".
 - Open the valve between the RP and turbo pump manually and click on "Evacuate" button from the vacuum control window.

- Monitor vacuum level from the controller screen (No. 340). When the vacuum level is <2 x 10⁻¹ hPa, manually close the valve and click "Refill" in the software.
- When the vacuum percentage become 0%, and Protective gas flow level dropped to 0 ml/min, the vacuum control window will show "Exit" and the "Evacuate" button will change from "grayed-out" to black (active).
- > Repeat the above Evacuate/Purge cycle two more times.
- At the end of the 3rd cycle, when the RP vacuum level is reached, close the Ar tank supply valve on the wall and "Refill" the system with the remaining Ar in the purge line (this is a special step due to the uncertain position of the vacuum/purge gas valve). Make sure there is no residual gas flow (the flow gauge should read "0" instead of a small number from 2-5 ml/min).
- Testing in Vacuum/purge gas:
 - After the final cycle, the manual valve should be opened to allow RP to pump the system to <2x10⁻¹ hPa.
 - Turn on the power of the turbo pump and monitor the speed of the pump to increase to 1500Hz. Vacuum level should reach <10⁻³ hPa in 3-4 minutes and reach <2x10⁻⁴ hPa after 10-20 minutes.
 - > The system should now be ready for testing.

Testing Tungsten Samples

- Open the Proteus® testing software and select "New" under Measurement menu.
 - ✓ Put in test file name and header information.
 - ✓ Select sample holders to each furnace by dragging the holder picture (on the right side) to the selected furnace position (on the left).
 - ✓ Program each sample by clicking on the furnace picture: input sample name, beam size, spot size, thickness, scan time, pulse width and amplifier gain levels.
 - ✓ Program test profiles: initial temperature, temperature steps, intervals, number of shots for each sample, purge gas or vacuum condition, emergency limits (for this system, the final temperature should be at 10°C to avoid automatic refilling of the purge gas).
 - ✓ The system should be ready for testing by clicking the "Run" button.
- Special testing conditions:
 - ✓ Due to the temperature differences observed during testing among the four furnaces, the test should be carried out only in furnace A, B, C up to 500°C and A, B to 800°C.
 - ✓ Heating rate: for vacuum test, the heating rate was selected at 5K/min (make sure it is the case by checking the "configuration" screen.
 - ✓ Furnace control parameters: Special parameter have been tested and selected for vacuum runs and purge gas runs:
 - Argon Purge Gas: Furnace Xp=4, Tv=4, Tn=4; Sample: Xp=4, Tv=4, Tn=4.
 - Vacuum: Furnace Xp=6.5, Tv=6.5, Tn=4; Sample: Xp=6.5, Tv=6.5, Tn=4.

Finishing the Test

- The manual valve should be closed, and turbo pump power should be turned OFF.
- Backfill the furnace with Ar gas.
- The front panel light should change from "Locked" to "Unlocked".
- Open the Detector/Furnace shield assembly and retrieve the samples from furnaces.

4.10 ELECTRICAL CONDUCTIVITY OF NEUTRON IRRADIATED TUNGSTEN MATERIALS - PHENIX COLLABORATION—L. M. Garrison, J. R. Echols (Oak Ridge National Laboratory)

OBJECTIVE

The purpose of this project is to measure electrical conductivity of tungsten-based materials. The PHENIX collaboration's goal is to expand the database on irradiation effects in tungsten.

SUMMARY

Two new fixtures were developed for measuring the electrical conductivity of miniature tensile samples and 3 mm disks.

PROGRESS AND STATUS

The high thermal conductivity of tungsten is critical for its use in future fusion reactors and is one of the reasons tungsten is the leading candidate material for high heat flux regions of reactors. Neutron irradiation in a reactor environment degrades the thermal conductivity. The mechanism by which thermal conductivity is reduced is phonon transport inhibition by generation of point defects and trans mutant elements rhenium and osmium. However, the electron transport aspect of thermal conductivity is also affected - albeit to a lesser degree - by neutron irradiation. Additionally, the thermal conductivity is affected by the location of the transmutation products rhenium and osmium, which can remain in solution or form precipitates. To separate the phonon, electron, and trans mutant element contributions to tungsten thermal conductivity, electrical diffusivity measurements can be compared against thermal conductivity measurements.

The US-Japan collaboration, PHENIX, has a goal of investigating tungsten and tungsten-based materials response to neutron irradiation for use in future fusion reactors. To this end, the PHENIX collaboration exposed over 1500 single crystal, polycrystalline, and W-Re alloy samples in the RB*19J irradiation capsule in the High Flux Isotope Reactor (HFIR) to doses of ~0.2-0.7 dpa. Nominal temperatures of irradiation are 500, 800, and 1200°C. A gadolinium shield was included in the irradiation capsule to reduce the thermal neutron flux and therefore the amount of fusion-relevant transmutation atoms.

The HFIR geometry and radioactivity levels of the post-irradiated samples severely limit the size of the samples which are used. Therefore, to test the electrical conductivity of the post-irradiation samples, two different miniature electrical resistivity fixtures were designed and constructed: one for 3 mm diameter disks and one for SSJ tensile bars. These fixtures are shown in Figure 1.

The resistance measuring system utilizes a Keithley Model 182 Sensitive Digital Voltmeter and a Model 237 High Voltage Source Measure Unit along with the custom fixtures. The general test procedures for electrical resistivity testing of metals are given in ASTM B 193-87, Standard Test Method for Resistivity of Electrical Conductor Materials. Calibration tests with tungsten samples are currently underway.



Figure 1. Electrical resistivity measurement apparatuses. For each image, major gridlines are 1cm apart. a) Disassembled 3 mm disk holder. Apparatus on left, example 3 mm disk and plug on right. b) Assembled 3 mm disk holder. The plug is clamped in the apparatus to ensure proper contact with the electrical contact pins. c) Tensile bar fixture.

4.11 DAMAGE MECHANISM INTERACTIONS AT THE PLASMA-MATERIALS INTERFACE (Early Career Award)—C.M. Parish, D. Morrall (Oak Ridge National Laboratory)

OBJECTIVE

This work intends to develop the fundamental scientific basis for modeling and predicting the behavior of helium bubbles in refractory materials, to provide support for the science and engineering of the tokamak plasma-facing-material environment. We have engaged in collaborations with University of California – San Diego (Drs. Tynan, Doerner, and Baldwin), and with Dr. J-P Allain (Now at Pennsylvania State University). We have hosted one summer student from Colorado School of Mines. We have brought onboard a new post-doctoral scholar, Dr. Daniel Morrall (Kyoto University).

SUMMARY

In this reporting period, we have engaged in several parallel efforts to develop the scientific basis of the plasma-materials interaction at the microscopic scale.

First, we have collaborated with Dr. J-P Allain and performed post-irradiation examination on the gadolinium-shielded tungsten specimens from the HFIR-RB*-19J irradiation experiment. The EBSD was performed using the new Tescan instrument in LAMDA, which allowed unprecedented statistics (20,000-40,000 grains / specimen) to be acquired, which provided strong statistical power to examine if different temperatures/doses resulted in changes to the grain size distribution. Preliminary results indicate that the examined samples did not suffer recrystallization or grain growth. The STEM found that the ~0.7 dpa, 800°C samples showed dense populations of irradiation-induced voids (~few nm in diameter) and large numbers of small Re-enriched clusters.

Second, in collaboration with Drs. Doerner and Baldwin, polycrystalline tungsten was exposed to helium plasma at low temperature (~30 to 300°C). This study intends to determine how fine bubbles will increase or decrease fuel retention. We have discovered that thin native surface oxide seems to interfere with the near-surface bubbles at these low temperatures, and we are working to both quantitatively analyze the bubbles in the near-surface region and determine the effect of the oxide on the bubble behavior.

Third, work on micro- and nano-indentation continues in single crystal, polycrystal, and recrystallize largegrain tungsten on order to determine the effects of different grain orientations and helium loading on the near-surface mechanical behavior. **4.12 HIGH HEAT FLUX TESTING OF NEUTRON IRRADIATED TUNSTEN**—L.M. Garrison, A. Sabau, B. Gregory, J.W. Geringer, Y. Katoh, S. Copp (Oak Ridge National Laboratory), Y. Hamaji (National Institute of Fusion Science), A. Hasegawa (Tohoku University)

OBJECTIVE

The aim of this work is to understand how neutron irradiation and high heat flux testing change the morphology and properties of tungsten.

SUMMARY

Samples of K-doped W-3Re and thick plate unalloyed W, each with grains elongated perpendicular to the surface, were exposed to high heat flux (HHF) cycles in the Oak Ridge National Laboratory (ORNL) Plasma Arc Lamp (PAL). Both unirradiated and neutron irradiated samples of the two materials were exposed. The surfaces were imaged with scanning electron microscopy before and after HHF exposure. A paper focused on the thick plate unalloyed W was submitted. Many of the additional scanning electron microscope (SEM) images are presented here and will be used for further analysis.

PROGRESS AND STATUS

Abstract of a submitted paper

Thick plate, unalloyed W was neutron irradiated in the High Flux Isotope Reactor (HFIR) at 550°C to a fast fluence of 1.24×10²⁵ n/m² E>0.1 MeV (~0.24 dpa). Unirradiated and irradiated specimens of the material were HHF tested in the PAL facility. The PAL uses a high-power photon source to provide a broad and even heat distribution on the sample surface. The samples were exposed to approximately 800 cycles at 4.73 MW/m² absorbed heat flux (incident heat fluxes of 10.95 MW/m²). After PAL exposure, slight changes were observed on the surfaces of the samples with scanning electron microscopy. The samples showed some annealing in the near surface polished region, but they were all below the damage threshold for cracking or other destructive features. The PAL has a large parameter space for future testing. The use of the HFIR and PAL to sequentially expose neutron irradiated samples to HHF will be a powerful tool for understanding materials behavior in a fusion-like environment.

Reference: L.M. Garrison, A.S. Sabau, B. Gregory, J.W. Geringer, Y. Katoh, Y. Hamaji, and A. Hasegawa "Plasma-Arc Lamp High Heat Flux Cycling Exposure of Neutron Irradiated Tungsten Materials." Submitted to *Physica Scripta*. June 2019

Scanning Electron Microscopy Data

More SEM data was collected than could be included in the above paper. The full set of images is included here for future reference. For each sample, multiple locations were imaged before and after HHF exposure. Where possible, the same locations were imaged for both conditions, but in some cases only representative images were taken. Sample 500G was scratched with a diamond tipped scoring tool on the reverse side so that the scratch would serve as a landmark after the HHF testing. Samples A00C and A001 are K-doped W-3Re with grains elongated perpendicular to the surface. Samples 500G and 5002 are thick plate unalloyed W with grains elongated perpendicular to the surface.

Images are included for these samples and conditions:

- A00C-unirrradiated, before HHF, imaged on engraved side.
- 500G-unirradiated, before and after HHF, imaged on engraved side and scratched side.
- 5002-irradiated, before and after HHF, imaged on engraved side.
- A001-irradiated, before and after HHF, imaged on engraved side.






Sample A00C Unirradiated; Before HHF Location 5























































4.13 PROPERTIES AND CHARACTERIZATION OF NOVEL COPPER ALLOYS FOR FUSION ENERGY

APPLICATIONS—Ying Yang (Oak Ridge National Laboratory), Ling Wang and Steven J. Zinkle (University of Tennessee)

OBJECTIVE

This study aims at developing high strength, high conductivity Cu alloys with improved thermal creep strength for long pulse fusion high heat flux structures, through an accelerated approach of computational thermodynamics guided alloy design.

SUMMARY

Work performed during this reporting period investigated the creep properties of the newly developed CuCrNbZr alloys and the reference CuCrZr alloys at 500°C in air and protective atmospheres under various applied stress levels.

PROGRESS AND STATUS

In FY2018, we did preliminary evaluation on the creep property of the newly developed CuCrNbZr (CCNZ) alloys, compared with the reference CuCrZr (CCZ) alloy fabricated by Kabelmetal. Figure 1 shows the creep strain vs time for both alloys tested at 500°C in air under an applied stress of 90 MPa, and the optical images in Figures 2(a) and (b) show the entire fracture and Figures 2(c) and (d) show crack propagation as marked by yellow arrows. Two low-magnification scanning electron microscope (SEM) images in Figures

2(a) and (b) show the ductile fracture behavior of the CCZ and CCNZ alloys at 500°C in air and 90 MPa. The blue rectangle regions were magnified in Figures 2(c) and (d). Cracks were observed in both alloys. Large cracks were developed along grain boundaries where no Laves phase is present. Most cracks in the CCNZ alloys are small when they were surrounded by Laves phases, while the cracks in the CCZ alloys, due to the absence of Laves phases, are generally larger than those in the CCNZ alloys. This finding strongly indicated that the presence of Laves phase in the CCNZ alloys effectively blunts the crack propagation, resulting in longer creep life.



Figure 1. Plots of creep life of the CCNZ alloy compared to the CCZ alloy at 500°C in air under 90 MPa.



Figure 2. Optical images in coper alloys tested at 500°C in air under 90 MPa. Low magnification (a) and (b) showing the entire fracture and high magnification (c) and (d) showing crack propagation as marked by yellow arrows in the CCZ (left) and CCNZ (right) alloys. Laves phases are marked by white arrows.

As the creep test in air had led to significant oxidation of samples, subsequent creep tests were performed in a protective atmosphere (flowing high purity argon). To fully understand the creep property of the CCNZ alloy at 500°C, systematic creep tests were conducted under various applied stress levels, i.e., 90, 100, 110, 125, 140 MPa. All tests used the type SS3 sheet tensile specimens (0.76 mm gage thickness, 1.52 mm gage width, 7.6 mm gage length), in accordance with the ASTM Standard E139-11, Standard Test Methods for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials.

Creep strain vs. time curves of the CCZ and CCNZ alloys are shown in Figures 3 (a)~(e) for applied stress levels of 90, 100, 110, 125, 140 MPa, respectively. At 500°C and 90 MPa, the creep life of the alloys in protected atmosphere was significantly longer than that in air, 640 h (2.3×10^6 s) vs. 120 h (4.3×10^5 s) for CCNZ and 390 h (1.4×10^5 s) vs. 75 h (2.7×10^5 s) for CCZ. The creep rupture time of the CCNZ alloys exceeded that for the CCZ alloys over all the applied stresses, as shown in Figure 3(f). They typically exhibited a short primary stage and a brief steady state stage, followed by a long tertiary stage of creep, except for CCZ under 100 MPa and 140 MPa. The minimum creep strain rate values (Table 1) were obtained by the polynomial fitting method applied to the creep strain curves. For the CCZ alloys at 500°C at 140 MPa, the creep life was only a couple of hours without apparent accelerating strain curves, which is not reliable to derive the minimum strain rate. Despite longer creep life, the CCNZ alloys exhibit larger creep strain rates through all the creep tests than that of the CCZ alloys. This might be due to the difference in the volume fraction, size or coarsening behavior of the matrix Cr precipitates in the two alloys. On the other hand, the Laves phase appears to be beneficial in pinning the grain boundary sliding and extending the creep life of the CCNZ alloys. Additional analysis is ongoing to understand the relationship among Nb addition, Cr-rich precipitate coarsening, and creep properties.



Figure 3. Creep tests at 500°C for applied stresses of (a) 90 MPa; (b) 100 MPa; (c) 110 MPa; (d) 125 MPa; (e) 140 MPa on CCZ (blue) and CCNZ alloys (red); (f) plots of applied stress versus time for CCZ and CCNZ alloys using a log-log scale.

Table 1. Minimum creep strain rates obtained from polynomial fitting method for CuCrZr and CuCrNbZralloys under applied stresses of 90, 100, 110, 125 and 140 MPa at 500°C

Applied Stress (MPa)	Strain rate $\dot{\varepsilon}_{min}$ (s ⁻¹)			
	CuCrZr, Kabelmetal	CuCrNbZr		
90	8.4E-10	6.1E-9		
100	3.0E-9	8.2E-9		
110	3.8E-9	3.5E-8		
125	1.5E-8	3E-8		
140		1.3E-7		

Future work

- 1) Understand the relationship among Nb addition, minimum strain rate and the creep properties.
- 2) Fabricate a new Cu-Cr-Nb-Zr alloy that has an increased Zr content and decreased Nb content to optimize the distribution of Laves phase and boost the volume fraction of Cu-Zr intermetallic compounds.

4.14 PHASE STABILITY OF PRECIPITATES AFTER ION IRRADIATION IN DILUTE COPPER-BASE BINARY ALLOYS—Ling Wang, Steven J. Zinkle (University of Tennessee), Arunodaya Bhattacharya, Chad Parish (Oak Ridge National Laboratory)

OBJECTIVE

This study focuses on fundamental understanding of phase stability of nanoscale precipitates within and beyond the ion displacement damage region in irradiated dilute Cu-base binary alloys. The primary goal is to correlate the stability of precipitates to irradiation-induced defect migration behavior through various irradiation conditions with respect to temperature and damage dose.

SUMMARY

Transmission electron microscopy (TEM)-EDS analysis showed good phase stability of Co- and Fe-rich precipitates at 25 and 350 °C after heavy ion irradiation up to 12 dpa, indicating no significant irradiation induced dissolution or coarsening effects. The initially coherent interface between the precipitates and matrix became incoherent due to absorption of point defects. The TEM observations showed that in Cu-1at.%Co binary alloys with lower precipitate sink strength, loss of coherency occurred well beyond the nominal maximum 600 nm depth of irradiated region due to the migration behavior of SIAs clusters under irradiation condition at 25 and 350 °C with dose of 1 and 12 dpa. Evidence for pronounced defect migration beyond end of ion irradiation region (much larger ranges than can be attributed to 3D motion of mobile radiation defects) suggests significant 1D migration of defect clusters.

PROGRESS AND STATUS

Initial coherent precipitates

Here, we use coherent 'vacancy' type precipitates to serve as a diagnostic monitor to provide an indicator of interstitial cluster migration behavior beyond the irradiated region. In this study, two typical Cu-base binary alloys (1%Co and 1%Fe, at.%) were selected and, after a solution anneal treatment at ~970°C followed by water quenching, a series of thermal aging conditions were applied to produce uniformly distributed coherent Co- and Fe- rich precipitates (lattice parameter of Co and Fe are both smaller than the Cu matrix) with different sizes and number densities. Precipitate number density and diameter values were calculated through TEM-BF images and EDS mappings. Figure 1 shows the coherent Co- and Fe-rich precipitates with "no contrast lines" normal to the *g* vectors; the inset images in this figure show SAED patterns for the TEM BF images. Since there are two different thermal treatment processes on Cu-Co alloys, we named sample #1 as Cu-Co aged at 625 °C for 12 h, sample #2 as Cu-Co aged at 600 °C for 12 h, and sample #3 as Cu-Fe aged at 600 °C for 12 h. These aging treatments produced a relatively low point defect sink strength due to the precipitates in sample #1 (2pNd~5x10¹³/m²) where N and d are the precipitate concentration and diameter, respectively, and a relatively high sink strength of ~3x10¹⁴/m² for samples #2 and #3.

Thermal stability after ion irradiation

Figure 2 shows TEM-EDS mappings of sample #1, #2, #3 irradiated with 1MeV Ni ions to a peak dose of 12 dpa at 25 and 350 °C. The white dotted line is separating the irradiated regions from non-irradiated regions, as determined from SRIM computer calculations. Sample #1, #2 and #3 show stable precipitate sizes in the irradiated regions, indicating that ballistic dissolution is not predominant and that there's not much coarsening effect on Co- and Fe-rich precipitates for the selected irradiation conditions. Precipitate dissolution tends to become pronounced for energetic displacement cascade irradiation conditions when the precipitate size is comparable to or smaller than the displacement cascade size (~5 nm) and when solute diffusion is sluggish (e.g. when the irradiation is performed at temperatures near or below the onset

temperature for vacancy migration). Similarly, precipitate coarsening would be expected for conditions where there is very high solute mobility.

Defect cluster migration behavior

Table 2 compares the observed beyond end of range (BEOR) loss of precipitate coherency with computational predictions by using the pure 3D and 1D defect diffusion equations from Reference 1. At 350 °C (recovery "stage V" temperature in Cu), vacancy clusters are thermally unstable and evaporate to release vacancies to the matrix, resulting in the initially coherent precipitates in these Cu alloys acting as the predominant point defect sinks with the sink strength calculated as $2\pi Nd$. However, at 25 °C (well below recovery "stage V" temperature), thermally stable vacancy-type stacking fault tetrahedra (SFT) along with dislocation loops and network dislocations play the key role of defect sinks in irradiated regions. The corresponding values of sink strength for irradiation at 25 °C chiefly from irradiation-induced defect clusters) are about 200 times higher than for 350 °C ion irradiation (chiefly from initial precipitates) based on data collected from Reference 2, causing fewer surviving defects to escape into the BEOR. The observed extent of the loss of coherency regimes of sample #1 are consistent with 1D defect cluster motion, since as summarized in Table 2 at 350 °C (~1700 nm BEOR for 12 dpa and 800 nm for 1.2 dpa) and 25 °C (~550 nm for 12 dpa and 220 nm for 1.2 dpa) both BEOR values for loss of precipitate coherency are close to their calculated 1D motion mean free paths, and significantly larger than 3D motion mean free paths, indicating that a significant fraction of the radiation-produced clusters exhibit 1D motion. No coherency loss of precipitates in the BEOR was observed at 0.1 dpa for sample #1 at either of the temperatures, indicating there's an insufficient concentration of surviving defect clusters that migrate beyond the ion irradiated regions to induce loss of coherency at this dose. Also, BEOR loss of coherency was not observed for samples #2 and #3 at any ion irradiation conditions, indicating the BEOR precipitate loss of coherency is related to initial precipitate sink strength. When the precipitate sink strength is over $\sim 10^{14}$ m⁻², it appears that relatively few 1D migrating defect clusters can diffuse significant distances, i.e., the effective diffusion mean free path of the gliding defect clusters is less than that estimated (Reference 1) for pure 1D diffusion. Conversely, when the precipitate sink strength is below ~10¹⁴ m⁻², the extent of the observed BEOR loss of precipitate coherency matches well with 1D diffusion behavior. Figure 3 shows two profiles of BEOR versus dose for the two temperatures (25 and 350 °C) of the observed loss of coherency in sample #1.



Figure 1. TEM BF images and the corresponding EDS mappings of element Cu, Co and Fe.



Figure 2. The TEM-EDS mappings of sample #1, #2, #3 irradiated with 1 MeV Ni ions to a peak dose of 12 dpa at 25 and 350 °C.

Table 2. Comparison of observed precipitate loss of coherency beyond end of range in alloys irradiated atroom temperature and 350°C with predicted radiation defect mean free path diffusion ranges for 1D and3D migration

Alloys	Majority	Majority	Majority sink	3D motion	1D motion	Observed
	sink mean	sink	strength	λ~(4πrN)⁻	λ~(6π ²r ⁴ N²) ^{- 1/2}	BEOR loss of
	size	density (m [.]	~4πNR	1/2	(nm)	coherency
	(nm)	³)	(m ⁻²)	(nm)		(nm)
#1	22	4.3×10 ²⁰	5.6×10 ¹³	129	2498	~1700 (12 dpa)
(350 °C)						~800 (1.2 dpa)
#1	2	9×10 ²²	1.1×10 ¹⁵	129	2498	~550 (12 dpa)
(25 °C)						~220 (1.2 dpa)
#2	14	3.3×10 ²¹	2.9×10 ¹⁴	59	804	None
(350 °C)						
#3	12	4.3×10 ²¹	3.3×10 ¹⁴	55	839	None
(350 °C)						





Results

The investigation of phase stability of coherent precipitates after ion irradiation will be extended to newly fabricated dilute Cu-1%Fe and Cu-0.4%Cr binary alloys. The thermal aging conditions for these two Cubase alloys will be selected with the purpose of producing coherent precipitates with low sink strength (~1 to 5x10¹³ m⁻²). The potential effect of different chemical compositions (and different initial degrees of coherency) of coherent precipitates on the interaction with glissile defect clusters will be evaluated.

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5. MAGNETIC AND DIAGNOSTIC SYSTEM MATERIALS

No contributions this reporting period.

6. FUSION CORROSION AND COMPATIBILITY SCIENCE

No contributions this reporting period.
7. MAGNETIC AND DIAGNOSTIC

No contributions this reporting period.

8. ADVANCED MANUFACTURING

8.1 ODS FeCrAI PRODUCTION USING ADDITIVE MANUFACTURING WITH *IN SITU* OXIDATION—Ty Austin, Steven J. Zinkle (University of Tennessee, Knoxville), Niyanth Sridharan (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this task was to produce oxide dispersion strengthened (ODS) FeCrAl + Mo + Y alloy parts using a laser directed energy deposition (DED) additive manufacturing (AM) process in an oxygen-rich environment in order to achieve high-performance, geometrically complex ODS alloy components.

SUMMARY

An Fe - 12Cr - 6AI - 2Mo - 0.18Y (wt%) alloy powder was consolidated using a DED AM process in an oxygen-rich environment using a pulsed laser raster and continuous laser raster pattern at 350W and 400W laser powers. The oxygen content of as-built parts varied from about half to double the as-received powder content depending on the deposition conditions, with the highest oxygen content observed for 400 W pulsed laser raster conditions. This indicated that the oxygen absorption and retention was controlled by a solute trapping mechanism due to the pulsed laser raster also induced increased thermal cycling in the sample which lead to increased residual stress, intergranular cracking, and trans granular cracking compared to the continuous raster samples. This deleterious cracking indicated that the processing parameters need to be refined in order to improve the mechanical properties of the parts and reduce cracking.

PROGRESS AND STATUS

20mm x 20mm x 20mm sample cubes were built in an oxygen-rich environment using a powder-blown, DED AM system built by DM3D Technology with gas atomized metal powder (Fe – 12Cr - 6AI - 2Mo - 0.18Y wt%) provided by ATI Powder Metals. The build chamber was maintained at approximately 16 at% oxygen during the build process for all samples. The ambient oxygen present in the build chamber was used as the source of oxygen for our alloy. A schematic of this process is shown in Figure 1 where the oxygen present diffuses into the hot melt pool after the laser has passed by.



Figure 1. Schematic of DED AM Process Using In Situ Oxidation.

For this first set of experiments, samples were built under four different laser conditions: 350W pulsed laser raster, 350W continuous laser raster, 400W pulsed laser raster, and 400W continuous laser raster. For a

pulse laser raster, the nozzle moves to the start point on the substrate, the laser fires for the duration of the set dwell time, the laser is shuttered, the nozzle steps over a set travel distance, and the process repeats. The remaining operating conditions are described in Table 1.

Build	1	2	3	4
Laser Power (W)	350	350	400	400
Laser Raster Pattern	Pulsed	Continuous	Pulsed	Continuous
Powder Flow Rate (grams / min)	5	5	5	5
Atmosphere Oxygen Concentration (at %)	16	16	16	16
Tool Speed (mm / min)	720	720	720	720
Laser Diameter (mm)	1.2	1.2	1.2	1.2
Pulse Dwell (s)	0.1	n/a	0.08	n/a
Nozzle Step Size (mm)	0.6	0.6	0.6	0.6
Final Layer Height (mm)	01.56	0.44	1.45	0.5

Table 1. Sample Processing Parameters

After fabrication, samples from each specimen and the as-received powder were sent to Dirats Laboratories for compositional analysis using inductively coupled plasma optical emission spectroscopy (ICP-OES) and combustion analysis using inert gas fusion (IGF). The compositional results are shown in Figure 2 along with a conventionally produced (ball milled, HIP'd/extruded) ODS-FeCrAI alloy included for Reference [1]. As shown in Figure 2, the pulsed laser raster samples (dark blue and yellow) were more successful at absorbing and retaining oxygen during fabrication. This increased oxygen absorption and retention for the pulsed laser is attributed to solute trapping. Solute trapping occurs when a liquid phase containing the solute (oxygen) solidifies to a solid phase with reduced solubility of the solute. This creates a driving force for diffusion of the supersaturated solute from the solid to the liquid phase. If the solidification rate or solid-liquid interface velocity is larger than the rate of solute diffusion, the solute can be trapped in the solid and form a supersaturated solution [2]. While the oxygen concentration is about one-fifth of the conventionally manufactured alloy, there was a doubling in oxygen content in the pulsed 400 W build compared to the as-received powder.



Figure 2. Compositional Analysis of As-Received Powder, As-Built Specimens, and Conventionally Fabricated ODS FeCrAI [1].

In addition to compositional analysis, the specimens were cross-sectioned, mounted, and polished using conventional metallography techniques to prepare for microstructural characterization using a scanning electron microscope (SEM) followed by etching for optical microscopy. The samples were etched using glyceregia (20 mL glycerol, 30 mL HCl, and 10 mL HNO₃) followed by characterization using a Leica DM4000M optical microscope. The optical images of the specimens are shown in Figure 3.



Figure 3. a) 350W Pulse, b) 400W Pulse, c) 350W Continuous, d) 400W Continuous (white arrows indicating lack of fusion defects; black arrows indicating cracking.

As shown in the stitched optical images, significant cracking and porosity occurred in the as-built samples (particularly in the pulsed raster specimens). The defect density was calculated using ImageJ in order to quantify the specimen porosity and cracking, as described in the literature [3]. The results, shown in Figure 4, agreed with the qualitative behavior found in the optical images in Figure 3. The highest porosity and cracking occurred in the pulsed samples, and the lowest overall defect density occurred in the 400 W continuous raster sample.





The increase in defect density for the pulse laser raster specimens compared to the continuous laser raster specimens is attributed to an increase of lack of fusion defects and residual stresses for the pulsed laser builds. The pulsed laser raster contributes to an increase of fusion defects by significantly increasing the amount of material deposited as the laser dwells on a given point such that the energy input is not enough to completely fuse the new layer with the previous one.

To better understand the cracking mechanisms at work, electron backscatter diffraction (EBSD) was performed using a Zeiss EVO SEM equipped with an EBSD detector. The EBSD maps shown in Figure 5 demonstrated that both intergranular and trans granular cracking occurred in the 400W pulsed laser build. Intergranular cracking indicates that stress buildup may be pulling the molten melt pool apart prior to complete solidification [4, 5]. Trans granular cracking implies the occurrence of large residual stresses on the solidified material exceeding the yield strength. We believe that the pulsed laser raster contributed to increased residual stress by increasing the amount of localized thermal cycling and expansion/contraction. Future work will aim to reduce these thermal gradients that cause increased residual stress and cracking.



Figure 5. Trans granular and Intergranular Cracking in the 400W Pulsed Laser Raster Specimen.





The Zeiss EVO was also equipped with an energy dispersive x-ray spectrometer. This was used to perform local compositional analysis on the as-built specimens to identify any possible precipitation. As shown in

Figure 6, a relatively large Y-Al-O precipitate formed during fabrication (400W pulsed laser). It should be noted that the observed precipitate was much larger than desired. Further analysis such as transmission electron microscopy is needed to evaluate whether any nanoscale precipitation may also be occurring.

To better examine the potential for nanoscale precipitation, the as-built specimens were heat treated for one hour at 600°C as suggested by the work done by Massey et al. [1]. Their work suggested that oxide nanoparticle precipitation in a similar ODS FeCrAl alloy system was completed at 600°C in as little as 15 minutes. Heat treating for one hour at this temperature should ensure that precipitation was completed and moved to the growth stage. Using a Hitachi S4800 SEM equipped with an EDAX energy dispersive x-ray spectroscopy detector, these mounted and polished heat-treated specimens were characterized. Figure 7 showed finer-scale precipitates formed in the heat-treated 400W pulsed laser raster. Further high-magnification microstructural characterization is needed to quantify the magnitude and size distribution of the oxide precipitation.





Future Plans

Transmission electron microscopy (TEM) will be used to examine the possibility that nanoscale oxide precipitation occurred. Additionally, the experimental parameters and experimental setup will be modified in order to simultaneously reduce the buildup of residual stress and encourage further oxygen absorption. These newly built samples will be characterized by the same techniques as the current set of experiment. They will also be examined using atom probe tomography (APT) in order to better understand the morphology and composition of the oxide phase formed during the AM process.

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8.2 ADDITIVE MANUFACTURING OF TUNGSTEN DIVERTOR COMPONENTS THROUGH ELECTRON BEAM MELTING AND BINDER JETTING—John Echols, Betsy Ellis, Lauren Garrison, Michael Kirka, Yutai Kato, Ryan Dehoff, Timothy Horn, Chris D. Rock, Christopher Ledford, Sullivan Figurskey, Kurt Terrani, Brian Jolly, Amy Elliott (Oak Ridge National Laboratory)

OBJECTIVE

The purpose of this project is to demonstrate viable tungsten divertor part production by additive manufacturing technology.

SUMMARY

Investigation has begun into electron beam melting and binder jetting production of tungsten divertor parts. Parameter-space studies of electron beam melting are required due to the immaturity of the technique for manufacturing with tungsten. This study has shown the benefit of a contoured scan strategy to allow for Ti build plate adhesion of the W. Additionally, scan speed and spot size have been investigated to minimize post-print porosity. Chemical vapor infiltration for binder jet manufactured preforms have shown densification potential but must be further optimized.

PROGRESS AND STATUS

Additive Manufacturing (AM) has recently been acknowledged as "one of the most promising transformative enabling capabilities for the U.S. to pursue that could promote efficient advance toward fusion energy" by the U.S. Fusion Energy Sciences Advisory Committee¹. Because the plasma-facing components of fusion reactors can be composed of many small parts, require complex shaping, internal channels, and dissimilar material joins, they make ideal candidates for AM. Tungsten is a particularly good candidate for this work since the high melting point and brittleness at room temperature makes it difficult to fabricate by traditional machining methods. Two methods of additively manufacturing tungsten are being investigated: Electron Beam Melting (EBM) and Binder Jetting (BJ). Both methods involve assembling parts from powered tungsten, but their methodologies differ significantly.

Electron Beam Melting

Electron beam melting (EBM) functions by locally melting the tungsten powder in an electron beam with a small spot size, creating a series of small melt pools from which the part is constructed. The process described here uses an ARCAM Q10 machine and plasma-atomized tungsten powder with ~90-micron particle diameter. The EBM process is ideal for higher melting temperature materials such as tungsten, but the process is still not well characterized for tungsten and significant effort has been devoted to optimizing the parameter-space for tungsten prints.

The parameter-space characteristics which have been investigated so far are:

- Build plate adhesion.
- Porosity of the printed material / density optimization.

During part production, proper adhesion to the build plate is of paramount importance - lack of adhesion can lead to a failure of an entire print. Differing scan strategies were investigated to determine if a raster or contouring strategy was more optimal when using a Ti build plate. Cross-sections of prints with different scanning strategies are shown in Figure 1. Contouring was shown to significantly reduce delamination in the first few mm of a tile print.

Porosity of the printed material is also of importance in maintaining the required material properties, such as high thermal conductivity of W in a divertor application. Beam speed and spot size are important for controlling porosity, with higher spot sizes and lower beam speeds generally leading to more dense prints, as shown in Figure 2. Additionally, it was found that higher analysis temperature (the temperature at which

the printing algorithm attempts to keep the print) leads to smoother print surfaces with less blobbing and balling of melted material.



contours

Figure 1. Exploratory tungsten divertor tile prints showing rastering and contouring scanning strategies. Optical micrographs are shown on the right.



Figure 2. Optical micrographs of cross-sections of exploratory prints from a parameter search matrix for EBM printing of W on a Ti build plate.

Currently the dense tungsten prints are undergoing hot isostatic pressing and will then be sectioned and tested for tensile properties, thermal conductivity, hardness, roughness, and density.

Binder Jetting

Binder jetting produces additively manufactured parts by selectively depositing a liquid binding agent to join powder particles. Layers of material are bonded to produce an object. Prints were produced on a X1-Lab with solvent binder. The powder material was Tekna W-25.

Chemical vapor infiltration (CVI) is being investigated for its potential to densify BJ W prints. The process occurs by passing gaseous tungsten hexafluoride and hydrogen over the W preform at elevated temperature. The following reaction results in W growth in the porous structure.

$$3H_2 + WF_6 \rightarrow W + 6HF$$

An initial run conducted at 625°C successfully deposited W in the preform. Trials are ongoing to optimize the deposition rate for optimal densification.

References

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8.3 COMPUTATIONAL DESIGN OF W-BASED BRICK AND MORTAR MODELS FOR FUTURE ADDITIVE MANUFACTURING CONCEPTS: ELASTIC MODUL—Charles H. Henager Jr., Ba Nghiep Nguyen, Cleopatra Howell, Lay Paw, Zoe Gotthold, Esther Mutesi (Pacific Northwest National Laboratory)

OBJECTIVE

Computational materials design efforts of advanced W-based composites require substantial benchmarking and validation before such designs can be realized via advanced manufacturing methods. Pacific Northwest National Laboratory (PNNL) is exploring brick and mortar microstructures as part of our ductile-phase toughening approach to tungsten divertor materials, and such designs start with an understanding of basic composite properties such as the composite elastic modulus. This report documents the computation of the plain strain and plane stress elastic moduli of a variety of brick and mortar structures and a comparison to a published analytical equation for the same.

SUMMARY

Success is demonstrated towards making computational models of deformation applied to W-Ni-Fe as well as W-Cu composites of bricks and mortar (BAM) structures using an open source program called OOF2¹. OOF2 can import images of BAM structures, generate a dual-phase finite element (FE) mesh, and apply various tests to the meshed microstructures. The program was used to compute composite elastic moduli of a wide variety of BAM structures with varying component elastic moduli. OOF2 also outputs FE meshes that can be read into Abaqus to compute composite moduli, which resulted in similar values. The goal of this initial study was to correlate our FE results to an analytical equation for the plane strain elastic moduli of BAM structures. We determined that the analytical expression exhibited the same trends as the FE models but that the equation began to increasingly overpredict the modulus with increasing brick aspect ratios as expected. Our results demonstrated that BAM structures can have tailored elastic moduli over a range that depends strongly on the modulus difference between the brick and mortar phases and also on the brick aspect ratio. The brick aspect ratio has a strong effect when the mortar modulus is small compared to the brick modulus but has very little effect when the two moduli are similar. In all cases it is clear that there will be a competition between brick failure and vertical mortar failure when determining that part of the BAM structure that controls ultimate tensile strength.

PROGRESS AND STATUS

Introduction

The W and W-alloys are the solid materials of choice for plasma-facing components (PFCs) of future fusion reactors, such as the International Thermonuclear Experimental Reactor (ITER) and Demonstration Power Plant (DEMO), due to their high melting point, strength at high temperatures, high thermal conductivity, low coefficient of thermal expansion, and low sputtering yield [1-3]. However, W and most W-alloys exhibit rather low fracture toughness and a high DBTT that would render them as brittle materials during reactor operations [1, 3, 4]. The DBTT for unirradiated W-alloys typically ranges from 573K to 1273K (300°C to 1000°C), and in a reactor environment radiation hardening would further elevate this range [3, 5, 6]. Wallovs toughened by engineered reinforcement architectures, such as ductile-phase toughening (DPT) are strong candidates for PFCs. Our efforts have focused on developing microstructures that exhibit improved fracture toughness based on W-Ni-Fe composites and that could be potentially manufactured using additive manufacturing methods, such as 3D printing. Brick and mortar microstructures have been studied recently and appear to have attractive mechanical properties [7]. Our studies begin with understanding the fundamental mechanics of such structures as a function of microstructural parameters, such as brick aspect ratio and relative volume fractions of the mortar and brick phases. The composite elastic modulus for BAM structures is one such fundamental materials property and represents a convenient model benchmark to compute. We use FE methods and compare these computations to analytical expressions for the moduli of these structures.

¹ OOF2 was developed at the National Institute of Standards and Technology (NIST).

Computational Procedure

We decided to simulate the elastic deformation that this material would undergo using OOF2, which is software that can take an image, turn it into a microstructure with different properties and values that can be assigned to a mesh, and that can be deformed using specific fields and equations with certain boundary conditions. In order to use this software, we first needed to create images that OOF2 could read and assign properties to. The first step in this process was to create an image that was designed as a BAM structure, where the brick could represent the tungsten (W) and the mortar could be the nickel-iron phase (Ni-Fe). We also wanted to be able to vary the length and width these bricks and with a certain thickness, t, of the mortar to get a specified volume fraction of the W (f_b) and Ni-Fe (f_m). So first we decided what our unit cell would look like. The unit cell should be the basic foundation of the microstructure we would be creating, so that using the unit cell the BAM microstructure could be expanded by simply repeating the unit cell horizontally and vertically. We could create a microstructure that consisted of 2x2 unit cells or 5x5 unit cells or larger. Our unit cell consists of two full bricks for the so-called half-stagger model. One of these bricks makes up the first row without any mortar layer in between it. The second brick, one mortar thickness below the first, would be split into two, separated by a vertical mortar segment, with the mortar segment centered under the first brick. In this unit cell there are two layers of mortar vertically, and two layers horizontally. The image in Figure 1 represents this unit cell.



Figure 1. A single unit cell of the BAM models used in this study showing *L*, *W*, and *t*. *L*/*W* is the BAM brick aspect ratio and *t* is the mortar thickness, which is uniform vertically and horizontally for all models tested here. The BAM stagger is $\frac{1}{2}$.

Since these are 2D models, the area fraction is equal to the volume fraction. By inspection, $A_b = 2LW$ and $A_m = 2Wt + 2t(L + t)$ such that f_m is $A_m/(A_m+A_b)$ or:

$$f_m = \frac{2t(L+t) + 2tW}{2t(L+t) + 2LW + 2tW}$$
(1)

Equation 1 can be used to solve for t given a specified L, W, and f_b (or f_m) as:

$$t = \frac{L - f_m L + W - f_m W - \sqrt{4(1 - f_m) f_m L W + (L - f_m L + W - f_m W)^2}}{2(f_m - 1)}$$
(2)

where f_m is the fraction of mortar, f_b is the fraction of brick, and t is the mortar thickness (see Figure 1). We used Mathematica to solve these equations and created brick model parameters for W weight percentages of 90, 95, and 97%. Models were built based on a W weight percentage of 90%, where f_m was 0.2 Ni-Fe and f_b 0.8 W. From this, we were able to produce models with varying *L/W* of 4.8, 5, 10, 20, 30, 40, and 50. An example of some of these models are shown in Figure 2.

Because OOF2 is a pixel-based system, we required integer values for *t*, *L*, and *W* for each brick. If the models were not integer numbers of pixels for *L*, *W*, and *t* then it would not be possible to create a mesh on OOF2 that is completely homogenous, which was greatly desired for comparison between models and with Abaqus results. Because of this, we had to find integer values for *L* and *W* and *t*. The problem with this is that there are only a few *t* values that give an exact f_m of 0.2 NiFe. We only found one *L/W* of 4.8 with a *t* of 2 that could give us exactly 0.2 NiFe and 0.8 W. However, we wanted to test more models with varying *L/W*. The only way we could do this is if we allowed the volume fraction to vary slightly from model to model. To see if this made a big difference, we created two models. The first had an *L/W* of 4.8 with a *t* of 2 and the L and W of this brick was 48x10 pixels. We made this model with a 2x2 unit cell design. The second model had an *L/W* of 5 with a *t* of 2 and bricks that were 50x10 pixels. This model also had a 2x2 unit cell. The difference was that in the first model, the volume fraction was exactly 0.2 NiFe and 0.8 W, and in the second the volume fraction was 0.1987 NiFe and 0.8013 W.



Figure 2. BAM models with L/W of 5 in (a) 2x2, (b) 5x5, (c) 10x10, and (d) 20x20 sizes, respectively.

Both designs were tested in OOF2 for comparison. OOF2 provides the ability to create a microstructure from an image by assigning a name and material to each pixel. In our microstructures, we had two-pixel groups: BAM. Each group was assigned a mechanical elasticity property with varying elastic moduli. The brick was always assigned an elastic modulus of 383 GPa (the elastic modulus of W) and a Poisson ratio (v) of 0.28, while the Mortar had varying moduli assigned to it. For our first trials, we assigned the mortar an elastic modulus of 203.57 GPa (the elastic modulus of Ni-Fe) and a v of 0.30364. We then used OOF2 to build a skeleton and a mesh for the microstructure with the given properties. Meshes built on OOF2 can also be saved for use on other applications such as Abaqus. We saved meshes for OOF2 for each microstructure as well as meshes for Abaqus. Next, we used Dirichlet boundary conditions to fix the left and bottom sides of the model, applying a boundary condition of 0.1 on the right side of the mesh. A diagram of this can be seen in Figure 3 below.



Figure 3. Fixed and applied displacement boundary conditions used in OOF2 and Abaqus to determine the composite elastic modulus in both plane strain and plane stress.

Given these applied boundary conditions, we solved for the mesh on OOF2 and using the analyzing capabilities of the software, we calculated an average stress and strain from the deformed mesh. We compared the results from the brick model with an L/W of 4.8 to an L/W of 5 which had slightly different volume fractions as earlier stated. We found that the composite modulus for the brick model with L/W 4.8 and volume fraction of 0.2 Ni-Fe was 343.42 GPa and the composite modulus for the brick model with an L/W of 5 and volume fraction of 0.199 Ni-Fe was 344.34 GPa. This difference was less than 0.3% and didn't seem to make much of a difference when comparing the models' ratio of its composite moduli to the elastic modulus of the brick (383 GPa), which was 0.897 for the L/W of 4.8 and 0.899 for the L/W of 5. We also created a contour map of the stress and strain for these solved meshes shown in Figure 4. The maps of these models were not very different, so we were able to tell that the small change in volume fraction had little effect on the computed modulus values.



Figure 4. Contour maps of stress and strain for L/W = 4.8 and 5 with slightly varying values of f_m . At the top are the maps for L/W = 4.8 with stress maps on the left, strain on the right. The maps are nearly identical.

Comparison with Analytical Expression for BAM structure moduli

Next, we wanted to test the models for a wider range of modulus ratio $E_m/(f_mE_b)$ and to compare our FE results with Reference [7]. For W-Ni-Fe, we had a modulus ratio of 2.7, but we wanted to compare results to ratios of 0.1, 0.01, or 0.001 to test the analytical expression (Equation 9) in Reference [7]. The only way we could get these ratios was by reducing the elastic modulus of the mortar material. To get a modulus ratio of 0.1, we reduced the mortar's elastic modulus (E_m) to 7660 MPa. For an aspect ratio of 0.01, we reduced E_m to 766 MPa, and for 0.001, E_m reduces to 76.6 MPa. We then put these microstructures and modulus values into OOF2, testing L/W's of 4.8, 5, 10, 20, 30, 40, and 50. The results for these trials are seen below in Figure 5. From the comparison of the different structures (2x2 vs 5x5), we could see a problem with the smaller 2x2 structure that was used successfully when the modulus ratio was large or when E_m is close to E_b . However, for smaller ratios the periodic nature of the FE mesh solution requires the use of a larger model to avoid numerical averaging errors. With more bricks and mortar cells, there is an improved periodicity and improved numerical averaging over the FE cells.

Begley et al. [7] derived an analytical expression for the elastic modulus of BAM structures based on $\frac{1}{2}$ -stagger. Their expression for the elastic modulus of a BAM structure is given in Equation 9 in Reference [7] and is reproduced here as Equation 3 with κ_1 and κ_2 given in Equations 4 and 5.

$$\frac{\overline{E}_c}{\overline{E}_b} = \frac{2(\sinh[\kappa_2]\kappa_1 - 2\sinh[(-1 + \overline{s})\kappa_2]\sinh[\overline{s}\kappa_2]\kappa_2)}{2\sinh[\kappa_2](1 + \kappa_1) + (\cosh[\kappa_2] - \cosh[(-1 + 2\overline{s})\kappa_2])\kappa_2}$$

(3)



Figure 5. Stress Map Comparison of *L/W* ratios of 5 through a 2x2 and 5x5 structure for $E_m/(f_m E_b)$ ratios of 0.1, 0.001, and 0.001, respectively.

$$\kappa_1 = \frac{\overline{E}_m w}{\overline{E}_b t_1} \tag{4}$$

$$\kappa_2 = \sqrt{\frac{(1 - \nu_m)\overline{E}_m w^2}{2\overline{E}_b t_2 h}} \tag{5}$$

where E_m is the mortar elastic modulus, *w* is the brick length, E_b is the brick elastic modulus, *t* is the mortar thickness (and $t = t_1 = t_2$), v_m is Poisson ratio of the mortar, and *h* is the brick width. In Equation 3, we also see the variable *s*, which represents the how the brick is broken into mortar. We used an *s* value of 0.5 because the mortar of the bricks began at $\frac{1}{2}$ the length of one brick (Figure 1).

We input Equations 2, 3, 4, and 5 into Mathematica and were able to calculate E_o/E_b analytically and compared the results from the FE model results from OOF2 using both plane strain and plane stress. We note that Equation 3 was derived under plane strain assumptions and that our Mathematica expression was able to reproduce the results shown in Figure 2 of Reference [7] only by using Equation 2 to compute the mortar thickness for a given f_m . From the stress and strain values calculated by OOF2, we directly computed a composite modulus, E_c , from the stress divided by the strain of each solved model. With this composite modulus we could find E_o/E_b , using E_b for W equal to 383 GPa. The results of these OOF2 computations and the comparison to Equation 3 (or Begley Equation 9) are shown in Figure 6.



Figure 6. Shown are the plane strain and plane stress moduli computed in OOF2 using BAM structures with *L/W* varying from 4.8 to 50 and with varying modulus ratios as shown. In all cases the analytical expression increasingly overpredicts the BAM moduli for increasing brick aspect ratios.

WNiFe with varying weight percent W

To match known ranges of WNiFe materials, we created BAM structures containing 95%-W and 97%-W in addition to the 90%-W materials discussed above. For each of the results there was an increasing elastic modulus with increasing volume fraction of W. The plane stress composite modulus increased for the different percentages: with 90%, we saw that the plane stress was 346.6 GPa for the L/W = 5 while the 97%-W increased to 374.7 GPa which is an increase of 28.2 GPa. From the data, we concluded that increased percentages of W result in larger values of E_c and that E_c also increases with increasing brick aspect ratio increases are small compared to increases when the E_m is

small compared to E_b . In general, BAM structures become insensitive to L/W as E_m approaches E_b . These trends are shown in Figure 7 for L/W varying from about 2 to 50.



Plane stress moduli for WNiFe BAM structures with increasing W

Figure 7. Computed plane stress elastic moduli for BAM structures of WNiFe from OOF2 for 90%-W, 95%-W, and 97%-W and *L/W* ranging from about 2 to 50.

WCu compared to WNiFe

After testing microstructures with various weight percentages of W, we began testing on models with an elastic modulus of 121 GPa and v = 0.34, giving the mortar phase the property of copper (Cu). We tested 21 different models. Seven of them were composed of 90%-W and 10%-Cu, with seven different L/W values (4.8, 5, 10, 20, 30, 40, and 50). Another seven were composed of 95%-W with L/W values of 5, 6, 10, 20, 30, 40, and 50. The last seven were composed of a 97%-W with varying L/W values of 2.75, 5, 10, 20, 30, 40, and 50. We again made meshes for all of these models in OOF2. We solved for these meshes and created contour maps to display these copper models. Figure 8 below showcases two models of the same L/W of 5 with Cu mortar. The only difference is that they are composed of different amounts of W. The map on the right has a f_b of 0.801 W and f_m of 0.199 Ni-Fe, and the map on the right is 0.894 W and 0.106 Ni-Fe. Figure 9 shows the range of composite moduli as a function of L/W for the cases of WNiFe and WCu for 90%-W and 97%-W BAM models. We observe that the composite modulus is reduced, as expected, and the modulus range is wider for the WCu BAM structures. This plot provides some information about how far the composite modulus of BAM structures can be tailored using common metallic materials as the mortar phase.



Figure 8. Stress contour maps of L/W = 5. 90%-W 10%-Cu on the left and L/W = 5. 97%-W 3%-Cu on the right. The mortar is thinner, and the stresses are more homogeneous for the 97%-W models. The W-brick phase carries most of the load, but the Cu vertical mortar segments are also highly stressed.



Figure 9. Computed plane stress elastic moduli for BAM structures of WCu and WNiFe from OOF2 for 90%-W and 97%-W and *L/W* ranging from about 2 to 50.

Discussion of stress contour maps for BAM structures

Although this study focused on the elastic moduli of various BAM structures it is obvious that average values do not completely or accurately explain the deformation of the BAM structures treated here. For example, Figures 4, 5, and 8 contain detailed spatial information regarding stress and strain distributions that are not captured by average values, such as elastic moduli. Some of this is discussed in more detail in Reference [7] but our data reveals that, in general, the horizontal mortar regions have the lowest stresses, with the centroids of the bricks having the highest stresses and the vertical mortar joints also highly stressed but less than the bricks. It seems apparent that final failure of these BAM structures will be a competition between brick failures and vertical mortar failures. Our work with continuum damage modeling of BAM structures confirms this and will be the focus of much of our future work with BAM architectures.

After trying various simulations of different specific aspect ratios and percentages of W and different mortar properties, the experiment of changing the specific aspect ratios to 0.1, 0.01, or 0.001 can be seen in Figure 6. For the most part, the plots align closely with Begley's data [7]. The trend is similar, and the numbers are not too far off, especially for the smaller aspect ratios. Begley explains in his paper that as the aspect ratio gets higher, his data would not be as accurate. So, the results from OOF2 look good in this regard. When the W fraction increases, the computed moduli increase linearly as captured by Begley's Equation 9 [7]. The changes can be seen such that the moduli increase roughly in agreement with the rule of mixtures. From 90%, 95%, and the 97%, there is an increasing trend of all the outcome variables. For the WCu case there is a significant difference in the moduli compared to the WNiFe BAM structures.

Results

The results for this computational work can be used for future work. For ongoing experiments, we will still be referring to OOF2 as a source for simulations, but the BAM structures will move towards 3D structures using OOF3D, which is like OOF2 but with an additional dimension.

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8.4 COMPUTATIONAL DESIGN OF W-BASED BRICK AND MORTAR MODELS FOR FUTURE ADDITIVE MANUFACTURING CONCEPTS: ELASTIC-PLASTIC DEFORMATION—Ba Nghiep Nguyen, Charles H. Henager Jr., Cleo Howell, Lay Paw, Zoe Gotthold, and Esther Mutesi (Pacific Northwest National Laboratory)

OBJECTIVE

Biological materials such as nacre, teeth, and shells have been found to exhibit attractive and unique combinations of stiffness, strength, and fracture toughness. Research has already been conducted in various engineering areas to mimic the kind of naturally hierarchical microstructures or nanostructures of biological materials to produce materials with optimum stiffness, high strength and fracture toughness for their intended structural applications. In the same objective, we investigated the deformation and fracture behavior of ductile phase toughened tungsten (W) materials such as tungsten-nickel/iron (W-Ni/Fe) composites that possess lamellar-like and hierarchical "brick-and-mortar" (BAM) microstructures. During the current reporting period, we developed BAM microstructure models and determined the effects of microstructural features on the overall composite elastic stiffness, nonlinear stress-strain response up to failure, and damage and fracture patterns. The developed modeling capability can be used as a tool to design and tailor hypothetical W-Ni-Fe microstructures to increase strength and ductility of these composites.

SUMMARY

Hierarchical BAM microstructures have been shown to be able to combine high stiffness, strength and fracture toughness [1-2]. A series of regular W BAM microstructures were generated to evaluate the potential of such microstructures to achieve improved stiffness, strength, and ductility in comparison to the lamellar-like W-Ni/Fe microstructure. After studying the microstructural effects on the composite elastic stiffness, we applied the multiscale microstructural approach developed in [3-4] to simulate tensile loading of W-Ni/Fe specimens cut out from rolled W-Ni/Fe materials. The predicted material stress-strain response and crack patterns were then compared to the corresponding experimental results to identify the elastic-plastic damage model parameters for W and Ni/Fe phases in this domain. Finally, the same model parameters were used in the analyses of W-Ni/Fe BAM microstructures to investigate the effects of microstructural features on the composite stress-strain response, strength, damage, and fracture patterns.

PROGRESS AND STATUS

Background

The W-alloys and W-composites are the solid materials of choice for plasma-facing components (PFCs) of future fusion reactors such as ITER and DEMO due to their high melting point, strength at high temperatures, high thermal conductivity, low coefficient of thermal expansion, and low sputtering yield [5-8]. However, W and most W-allovs exhibit rather low fracture toughness and a high DBTT that would render them as brittle materials during reactor operations [6,8-9]. The DBTT for unirradiated W-alloys typically ranges from 573K to 1273K (300°C to 1000°C), and in a reactor environment radiation hardening would further elevate this range [8.10-11]. The W-allovs toughened by engineered reinforcement architectures are strong candidates for PFCs. One of the key toughening mechanisms is by ductile-phase toughening (DPT). In DPT, a ductile phase is included in a tungsten matrix to increase the overall work of fracture for the composite material. Our previous work [3-4] modeled the DPT mechanism in W-Cu and W-Ni/Fe specimens by a multiscale microstructural approach using a dual-phase domain in which the constitutive behavior of W and of the ductile phase (i.e., Cu or Ni/Fe) was described by an elastic-plastic continuum damage model. This approach captured the bridging of failed ductile phase representing the DPT mechanism. During this reporting period, by exploring the damage mechanisms observed in the actually tested W-Ni/Fe composite, the modeling predictions show that the regular "brick" lattice microstructures that experience the bridging mechanism combined with crack penetration across W-phase regions exhibit significantly higher strengths than the rather random lamellar-like microstructures. However, adjusting the

brick's length-to-height ratio for allowing more distributed damage leads to increased strength, ductility, and fracture energy significantly.

Model Development

Brick-and-Mortar Microstructures

A FORTRAN script was developed to generate a series of regular W-Ni/Fe BAM microstructures to evaluate the potential of such W-composite microstructures to achieve improve strength, ductility and fracture energy in comparison to the baseline lamellar-like microstructure analyzed previously [4]. All the generated BAM microstructural domains contain the same volume fraction of W (~0.8) and have the same dimensions (0.584 x 0.584 x 0.025 mm) as the baseline microstructure. Figure 1 shows an example of a BAM microstructure having two hierarchical levels: Level 0 corresponds to a single W "brick" with length *L* and height *H*; Level 1 includes the representative W-brick-and-Ni/Fe "mortar" unit cell that is repeated in both horizontal (x) and vertical (y) direction to build the microstructural domain.



Figure 1. An example of BAM microstructure for W 'bricks' with a ductile mortar phase such as Ni-Fe showing two hierarchical levels.

There are myriad ways to generate BAM microstructures as functions of relevant geometrical features. In this work, all the generated BAM microstructures have the same topology but are different in the W "brick" aspect ratio. In addition, while fixing the W volume fraction about 0.8 and the dimensions of the modeling domain (0.584 mm x 0.584 mm x 0.025 mm), the W "brick" aspect ratio is chosen as a design parameter and is varied to achieve BAM microstructures with L/H = 5, 9.7, 20.75, 31.25 and 41.5 (typically L/H=5,10, 20, 30, 40). The as-generated microstructures are presented in Figures 2(b-f) compared to the baseline lamellar-like microstructure shown in Figure 2a. They were also discretized in 2D finite elements (about 250,000 elements) working under plane-stress or plane-strain assumptions.

First, we studied the evolution of the elastic modulus in the reinforcement direction of interest (which is the x-direction in Figure 1). To this end, tensile deformation in the x-direction was applied to each generated

microstructure to compute the composite elastic modulus in this direction. Figures 3a and 3b report the evolutions of the normalized composite elastic modulus, E_{xx}^c/E_b (E_b being the W brick elastic modulus) with the brick aspect ratio L/H for the prescribed values of the constituent modulus ratio, E_m/f_mE_b . If the brick modulus (i.e., E_b = 383 GPa) and the brick volume fraction (i.e., f_b = 0.8) are given, the mortar elastic modulus, E_m is determined by the modulus ratio and $f_m = 1 - f_b$. However, for the W-NiFe and WCu composites with the same W volume fraction and known constituent elastic modulus, the modulus ratios are 2.66 and 1.44, respectively. Figures 3a and 3b show that if the modulus ratio, E_m/f_mE_b is small (<< 1), the composite modulus increases substantially with the increasing brick aspect ratio and tends to saturate for large aspect ratios (> 90). The plane-strain FE solutions agree very well with the numerical predictions by an Eshelby-Mori-Tanaka approach (EMTA) assuming ellipsoidal brick inclusions for small modulus ratios, but the plane-stress FE results agree globally well with the EMTA predictions for all constituent modulus ratios considered including the values for the W-NiFe and WCu composites. These figures also show that the brick aspect ratio only has a very little effect on the composite modulus for the W-NiFe and WCu composites with large values of E_m/f_mE_b .



Figure 2. (a) Baseline lamellar-like microstructure - Generated BAM W/Ni-Fe microstructures: (b) L/H=5, (c) L/H=10, (d) L/H=20, (e) L/H30, and (f) L/H=40.

To further investigate the elastic modulus computations, our FE plane-strain results and EMTA solutions presented in Figure 4 are compared to the Begley et al.'s plane-strain micromechanical solutions [12] for the same modulus ratios (0.02, 0.05 and 0.1). It is seen in Figure 4 that Begley et al.'s results agree fairly with the FE solutions for small to moderate brick aspect ratios (L/H < 20), but the results diverge for larger brick aspect ratios. The same findings are found when comparing EMTA's solutions to Begley et al.'s model, and that explains the significant divergence of our solutions compared to Begley et al.'s for large aspect ratios.



Figure 3. Normalized composite modulus as a function of the brick aspect ratio: (a) Plane-strain FE and (b) Plane-stress FE results compared to EMTA solutions.



Figure 4. Normalized composite modulus as a function of the brick aspect ratio: plane-strain FE results and EMTA predictions compared to Begley et al.'s results [12].

Stress-Strain Response and Damage Predictions

The 2D plane-stress model of the W-NiFe specimen subjected to tensile loading was analyzed using ABAQUS and the elastic-plastic damage model described in [3] to depict the constitutive behaviors of W

and NiFe phases in the microstructural domain shown in Figure 1b. A series of FE analysis was first conducted to identify the constitutive model parameters for W and NiFe by reverse engineering. The identification process started from using typical mechanical properties of W and of Ni alloys as initial guests. The constitutive parameters were then adjusted through FE analyses until the predicted stress-strain response and loading-dependent damage pattern development agree with corresponding experimental results. Figure 5a shows the predicted engineering stress-strain response that agrees well with the corresponding experimental data also reported on this figure. These same model parameters were then used for W and NiFe in all the FE analyses of the BAM microstructural models (Figures 2b to 2f) to investigate the W brick aspect ratio and microstructural effects on the stress-strain response of the created BAM composite. Figure 5b reports the stress-strain curves up to failure of these composites compared to the predicted response of the baseline composite shown in Figure 2a.

Figure 5b shows that increasing the brick aspect ratio L/H from 10 to 40 results in significant increase in strength but leads to reduction in fracture strain. In addition, strength saturation with increasing L/H from L/H=30 has been found. For small brick aspect ratios such as L/H=5 (Figure 2b), the model predicts a significant increase in strength combined with an important increase in fracture strain. The damage and fracture patterns for all the cases presented in Figures 6(a-f) elucidate the stress-strain responses in Figure 5b. Damage is quantified by a damage indicator that varies from 0 to 1. If the failure indicator is equal to 1, total failure (or fracture) causing crack propagation occurs and is captured by a vanishing element method [13, 3]. Figure 6a show the damage and fracture patterns for the baseline material already discussed in our previous report [4]. Figures 6(b-f) illustrate the damage and fracture patterns in the BAM microstructures with aspect ratios 5, 10, 20, 30 and 40, respectively. In addition to the DPT mechanism caused by failed NiFe elements, there are two other mechanisms: crack penetration across tungsten phase and multiplication of cracks over the domain. More importantly these mechanisms combined occur during loading, more facture energy is dissipated leading to higher strength and fracture strain. This is realized with the L/H=5 BAM microstructure. The fracture patterns in the other BAM microstructures are guite similar: increasing L/H has led to increased strength. More damage distributed in the L/H=10 microstructure has vielded higher fracture strain for this case. All the BAM materials exhibit higher strength than the baseline composite.



Figure 5. (a) Predicted tensile stress-strain responses for (a) the baseline W-Ni/Fe composite compared to the corresponding experimental data, and (b) the BAM composites shown in Figures 2(b-f).



Figure 6. Predicted damage distribution and fracture patterns at final failure for: (a) the actual lamellar-like microstructure, (b) L/H=5 (c) L/H=10, (d) L/H=20, (e) L/H=30, and (f) L/H=40 BAM microstructures.

Results

During this report period, important progress has been made on advancing our predictive capability for modeling ductile phase-toughened tungsten for PFC applications by our study of BAM microstructures. These microstructures appear to be promising to achieve the mechanical properties desired through material selection, tailoring, and optimization of their topology and architecture. Our approach that offers a new way to design PFCs can assist additive manufacturing to produce a structure or component having a BAM microstructure.

Future Work

We will perform additional work on W composites by investigating material combination, other BAM microstructures to increase strength and ductility of W composites.

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9. MECHANISMS AND ANALYSIS

9.1 DEFECT DIFFUSION AND ATOMIC TRANSPORT IN W-BASED ALLOYS—Yury Osetskiy (Oak Ridge National Laboratory)

OBJECTIVE

The purpose of this research is to understand the mechanisms of defects diffusion, atomic transport and phase transformations operating in W-based alloys under irradiation conditions.

SUMMARY

Atomic transport by interstitial atom diffusion in W-Re system is strongly affected by interaction with solute atoms. We have modeled the transport mechanisms in W-Re system to allow more accurate prediction of Re precipitates properties and formation mechanisms. Results show that:

- High interaction energy decreases the total defect mobility and tracer diffusion coefficients, and
- Strong chemically biased diffusion is observed via Re-atom drag by migrating interstitial defects.

PROGRESS AND STATUS

Introduction

We use molecular dynamics (MD), kinetic Monte Carlo (kMC) and diffusion theory to calculate diffusion and transport coefficients in pure metals and alloys. Due to availability of the reasonably accurate interatomic potentials (IAPs), we considered W-Re system. Further development of kMC models will allow us to extend this study to tungsten and other alloys using *ab initio* calculations of necessary parameters such as defects formation, migration and binding energies.

Molecular dynamics is an ideal tool for studying dynamic properties of atomic systems evolving at application temperatures with internal and external driving forces (see e.g. [1] for general information). However, currently only classical MD methods based on Newtonian equations, in conjunction with empirical IAPs, can model point defect diffusion over times long enough to provide enough statistics of elementary migration events – that is, defect jumps. Microsecond-scale MD simulations have been applied [2, 3] to the study of point defect diffusion in fcc-type Ni-Fe alloys. Concentrated alloys impose new diffusion mechanisms that a) decelerate defect diffusion, b) introduce new strong correlations of defect jumps and c) demand much greater statistics of defect jumps for results to converge.

Results

Here we present results of extensive modeling of interstitial defect diffusion in W-based alloys. We have modeled thermally activated migration of interstitial atoms in bcc pure W, W-2.5 at.% Re and W-5 at.% Re alloys. The general approach to treating MD data is described elsewhere where we operated mainly with self-diffusion coefficients, D^* , D^*_{Ni} and D^*_{Fe} , that are defined by treating atomic square displacements (ASD) as function of simulated time, t (see [4] for details). For the tungsten materials we treated long trajectories of ~10⁶ jumps of the interstitial atom modeled over a few hundred *ns* physical time. Interatomic interactions were described using IAPs developed for the W-Re system in [5]. These IAPs are fitted to a wide range of properties of pure metals and alloys and ensure qualitatively correct behavior of point defects in the W-Re bcc alloys.

Currently we are studying the interstitial atom (IA) diffusion mechanism. IA in pure W has a ground state configuration as a <111>-crowdion and perform fast, preferentially one-dimensional, diffusion along <111> direction. At high temperature the frequency of crowdion rotations increases making the total diffusion three-dimensional. This motion changes dramatically when Re is alloyed with the W matrix, with binding



Figure 1. Temporal evolution of atomic square displacement (ASD) in a 4396-atom crystal of W-5 at.%Re initially random alloy during IA diffusion at different temperatures.

energy between IA and Re in the W matrix of 0.8 eV. This high energy tightly binds the Re solute to the IA, and very much affects its diffusion. An example of temporal evolution of atomic square displacement in crystal of W-5 at.%Re random alloy during IA diffusion at different temperatures is presented in Figure 1. This shows that the ASD(t) dependence is quite rough and consist of segments with either complete immobility or fast motion (increasing ASD). The former is related to strong biding between Re solute and interstitial atom configuration, the later reflects a fast IA migration towards the next Re solute atom. Only at the highest temperature studied, 1500K, does ASD(t) come close to a linear function.

Tracer diffusion coefficients calculated for W, W-2.5 at.%Re and W-5 at.%Re alloys are presented in Figure 2 as Arrhenius-type plots with the effective activation energies indicated. Note the dramatic increase of the tracer diffusion activation energy from 0.101 eV in pure W to >1 eV in alloys. This reflects the high binding energy between the Re solute and an interstitial defect in the W matrix. This is a very important feature of W-Re system for it strongly affects alloys behavior under irradiation. For example, Re segregation should be strongly controlled by interstitial diffusion. We have studied this mechanism and revealed that an interstitial atom drags Re along its diffusion trajectory. The evidence of this is presented in Figure 3 where we plot the mean ratio Re-to-W atoms in each interstitial configuration that appears during migration in both alloys studied. Note that we treated $\sim 10^6$ instant configurations that appeared after each IA jump. At low T<1200K in both alloys this ratio is close to unity, indicating that the IA is a mixed W-Re dumbbell. This is in principle fully consistent with a high stability of this dumb-bell configuration calculated by ab initio and reproduced with the IAPs used here [5]. At higher temperatures the dumb-bell chemistry evolves differently in each alloy. In dilute 2.5 at.% Re alloys purer W-W interstitials appear due to thermal dissociation of W-Re configurations: then the Re/W ratio drops below unity. In the more concentrated 5 at.%Re alloy IA chemistry evolution is different; the Re/W ratio increases above unity. This means that the number of Re-Re interstitial configurations increases because of the local rearrangement due to more frequent migration of interstitial defects. Note that we modeled $\sim 10^6$ defect jumps. This high number of jumps help reveal the



Figure 2. Tracer diffusion coefficients calculated for pure W and 2.5 at.% and 5 at.% Re alloys as a function versus the reciprocal temperature. Inserts indicate the corresponding effective activation energies.

certain clustering of Re due to their strong attraction to interstitial defects. This is clear evidence that even at the molecular dynamics time scale, random W-Re alloys are unstable and undergo Re precipitation. A possible mechanism of formation and growth of Re precipitates due to interstitial defects diffusion was studied recently [6] using kMC technique at the mesoscale.

More accurate mesoscale study of precipitation and phase transformations in W-Re alloys under irradiation



Figure 3. Mean ratio of Re-to-W atoms in each interstitial configuration that appeared during migration in the both alloys as a function of ambient temperature. Error bars indicate standard deviation of the mean estimated over $\sim 10^6$ instant configurations that appeared after each IA jump.

conditions will be performed in future using diffusion theory and kMC modeling and utilizing the mechanisms and diffusion parameters obtained by molecular dynamics modeling. However, although mesoscale approaches allow the expansion of time and spatial scales for phenomena studied such as diffusion, segregation, precipitation and phase transformations, the detailed atomic-scale mechanisms still need to be identified by atomistic modeling. Further progress along this direction depends mainly on the availability of interatomic potentials for the alloys and compositions to be studied.

Application

This knowledge is necessary for predictive modeling of phase stability in W-Re systems under irradiation. This work is currently continuing.

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9.2 DENSITY FUNCTIONAL THEORY STUDY OF HELIUM AND HYDROGEN PROPERTIES IN DUCTILE-PHASE-TOUGHENED W-NI-FE ALLOYS—W. Setyawan, Charles H. Henager Jr. (Pacific Northwest National Laboratory)

OBJECTIVE

The overall objective is to provide scientific understanding of thermo-mechanical properties of ductilephase-toughened (DPT) W-Ni-Fe alloys under neutron irradiation, including the effects of gas species (He and H isotopes), as well as understanding of gas retention in these DPT alloys. The specific objective in this report is to calculate the formation energies of He and H in the alloys.

SUMMARY

Density functional theory (DFT) method is employed to calculate the formation energies of He and H atoms in the ductile phase of a DPT W-Ni-Fe alloy. The ductile phase is modeled as a face-centered cubic (fcc) Ni-Fe-W random solid solution. The formation energies of He and H at various octahedral and tetrahedral interstices have been obtained. The data will be used to construct a thermodynamic model of He and H distribution in the alloy (i.e. within W particles, W/Ni-Fe-W interphase boundaries, and within Ni-Fe-W ductile phases) as a function of concentration and temperature. The DFT calculations also reveal that He induces considerable relaxation with respect to its initial interstitial location. Helium prefers to relax closer to Fe atoms and away from W atoms. On the other hand, H does not induce any significant relaxation.

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The DPT W-Ni-Fe alloys are being developed as potential plasma-facing materials. W-Ni-Fe alloys are cheaper than W and commercially available. The alloys typically consist of essentially pure body-centered cubic (bcc) W particles embedded in ductile face-centered cubic Ni-Fe-W solid solution matrix (binder) phase [1-2]. Cohesion of the interphase boundary between the particle and the binder is key to the excellent ductile-phase toughening in these materials. For instance, tensile tests of 95W-3.5Ni-1.5Fe alloy (wt. ratio) alloy (referred to as 95W) [3] and 97W-2.1Ni-0.9Fe alloy (97W) [4] show that the interphase cohesion results in an ultimate strain of ~12% at room temperature. Further evidence of the toughening is demonstrated in bend tests of 90W-7Ni-3Fe alloy (90W) [1] which show a peak stress intensity of ~52 MPa/ \sqrt{m} at room temperature (with a displacement rate of 0.2 mm/min), significantly higher than 8 MPa/ \sqrt{m} of monolithic W.

For use as plasma-facing materials, the effects of neutron irradiation, helium, and H isotopes accumulation need to be understood. For example, the use of Ni may cause a concern that the activation of Ni may result in unsafe dose rate. To address this concern, a study has been performed to assess the activation of the 97W as a first wall material in a DEMO reactor [2]. The study shows that since 97W contains only a small amount of Ni (2 wt.%), the dose rate of the irradiated 97W after one year of operation is very similar to pure W, and it is less than two times higher than W after 10,000 years. The analysis suggests that W-Ni-Fe alloys with a small amount of Ni is acceptable.

In this report, the DFT method is employed to explore the formation energy of He and H in the binder phase. Results in un-irradiated alloys are presented. Table 1 shows the chemical composition of the binder phase for 90W, 93W, 95W, and 97 W determined using different experimental techniques as indicated in the table. The data obtained from electron diffraction spectroscopy (EDS) show that the composition of the binder among the different alloys is similar, that is about 66 at.% Ni, 25 at% Fe, and 9 at% W.

	Overall Composition (wt.%) in Alloy			Composition (at.%) in Binder			Method
	Ni	Fe	W	Ni	Fe	W	
90W	7	3	90	64.5	27.1	8.4	EDS [5]
93W	4.9	2.1	93	65.7	25.6	8.7	EDS [5]
95W	3.5	1.5	95	66.2	24.3	9.5	EDS [5]
95W	3.5	1.5	95	61.2	32.2	6.6	Magnetization [6]
97W	2.1	0.9	97	64.1	25.1	10.8	EDS [7]

Table 1	. Chemical	composition	of binder	phase in	W-Ni-Fe alloy	s. 1	The composition is determined based
	on e	electron diffra	ction spec	ctroscopy	(EDS) or mag	gne	tization measurement.



Figure 1. Model structure of Ni-Fe-W fcc solid solution obtained with special quasi-random structure (SQS) method. The model contains 72 Ni (red), 27 Fe (blue), and 9 W (yellow) atoms.

For DFT calculations, the binder phase is modeled as a random fcc solid solution in a 3x3x3 supercell containing 108 atoms. The SQS [8, 9] method as implemented in the ATAT code [10] is employed to optimize the randomness of the structure. The SQS selects the structure that best approximates the radial correlation functions of a perfectly random structure. A cutoff radius of 5.25 Å is used in the SQS calculations. The SQS model contains 72 Ni, 27 Fe, and 9 W atoms in the 108-atom simulation cell. Figure 1 shows the optimized structure of the solid solution model.

The DFT calculations are performed with the VASP software. The standard pseudopotentials for Ni, Fe, W, He, and H from the VASP's library paw_PBE.54 are employed. The electron-ion interactions are modelled within the projector-augmented-wave (PAW) method. The number of electrons treated as valence is 10 (Ni), 8 (Fe), 6 (W), 2 (He), and 1 (H). The electronic exchange-correlation is treated within the generalized-gradient approximation (GGA) with the Perdew-Burke-Ernzerhof (PBE) functionals. The energy cutoff of the wave functions is taken as 624 eV. The Brillouin zone is sampled with a *I*-centered **k**-point mesh. The **k**-point mesh for all calculations in the solid solution is 3x3x3. Magnetization experiment measurements show that the binder phase is only weakly ferromagnetic [11]. Therefore, all calculations are performed as non-spin polarized calculations. All calculations are fully relaxed with a force tolerance of 0.01 eV/Å. Such an accurate force tolerance is needed for phonon calculations to obtain zero-point energy (ZPE) associated with H. The phonon calculations are performed within the frozen phonon approximation.

To study the formation energy of He and H in the binder phase, octahedral and tetrahedral interstices are determined. In the model, there are 27 octahedral and 216 tetrahedral interstices. Ten octahedral and 15
tetrahedral sites are randomly selected to sample the overall interstices. Figures 2 and 3 show the atomic environment of these randomly selected octahedral and tetrahedral interstices, respectively.



Figure 2. Atomic environment of the 10 randomly selected interstices (black) out of 27 octahedral interstices in the fcc NiFeW solid solution model. Atoms are Ni (red), Fe (blue), and W (yellow). Bonds are drawn between atoms within 2.8 Å, and between interstice and atom within 2.3 Å.



Figure 3. Atomic environment of the 15 randomly selected interstices (black) out of 216 tetrahedral interstices in the fcc NiFeW solid solution model. Atoms are Ni (red), Fe (blue), and W (yellow). Bonds are drawn between atoms within 2.8 Å, and between interstice and atom within 2.3 Å.

Figure 4 shows the relaxed configurations after He is inserted at the 10 octahedral interstices. It can be seen that He no longer stays at its initial position (the center of the cubic cluster), but rather it moves away from W and towards Fe atoms. The displacement of the He atom upon relaxation is significant enough that the structures become complex and is no longer octahedrally coordinated. The formation energy of He associated with these configurations is shown in the figure. The formation energy is calculated with respect

to a monoatomic He gas. Figure 5 shows the relaxed configurations after He is inserted at the 15 tetrahedral interstices. Compared to the octahedral cases, the relaxation in the tetrahedral cases is considerable smaller, nevertheless the structures exhibit distorted tetrahedral configurations.



Figure 4. Relaxed configuration of atoms in the vicinity of interstitial He (black) that is initially placed at the corresponding octahedral sites as depicted in Figure 2. The atoms are Ni (red), Fe (blue), and W (yellow). Formation energy of He is shown.



Figure 5. Relaxed configuration of atoms in the vicinity of interstitial He (black) that is initially placed at the corresponding tetrahedral sites as depicted in Figure 3. The atoms are Ni (red), Fe (blue), and W (yellow). Formation energy of He is shown.

Unlike He, H causes only minor displacements in octahedral as well as tetrahedral interstices (not shown). The formation energy of H is calculated with respect to H_2 diatomic gas. Due to the light mass of H, the ZPE is not negligible. The calculated ZPE of an H_2 gas is ~0.29 eV. Considering that the atomic mass of

Ni, Fe, and W is much higher than H, only the vibrations of H are included in the ZPE calculation of the solid solution systems. The ZPE of H in the solid solution varies depending on its location. The smallest ZPE is found for H at the octahedral site c, while the largest ZPE is for H at the tetrahedral site o. The formation energies of H initially placed at the 10 octahedral 15 tetrahedral interstices are plotted in Figure 6a and 6b, respectively. Octahedral sites are the preferred interstices. In fact, the formation energies at sites b, c, and g are negative. On the other hand, the formation energy at tetrahedral sites ranges from ~0.3 to 0.55 eV, with the least preferred location at site o which is also to the site with the highest ZPE.



Figure 6. Formation energy of H initially placed at a) 10 octahedral sites a-to-j as depicted in Figure 2a) to 2j), and b) 15 tetrahedral sites a-to-o as depicted in Figure 3a to 3o.

Future research includes 1) study of segregation behavior of He and H towards the interphase boundary, 2) development of thermodynamic model of segregation and retention as a function of temperature and concentration of He and H, 3) study of influence of He and H on the cohesion of the boundary, 4) calculation of migration energy barriers of H and He in the ductile phase, and 5) eventually include the concurrent effects of vacancies and interstitials.

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10. MODELING PROCESSES IN FUSION SYSTEM MATERIALS

10.1 A MULTI-SCALE MODEL OF HELIUM TRANSPORT, FATE AND CONSEQUENCES IN IRRADIATED TEMPERED MARTENSITIC STEELS: VOID SWELLING AND DPA RATE EFFECTS—T. Yamamoto, G.R. Odette (University of California Santa Barbara)

OBJECTIVE

The objective of this research is to develop a multiscale model to exploring He transport, fate and consequences (HTFC) under fusion relevant irradiation conditions.

SUMMARY

The synergistic effects of displacement damage and He produced by high energy neutrons in the fusion reactor environment can cause severe degradation of structural materials. Here we develop a multiscale master model for exploring He HTFC for fusion relevant irradiation conditions. The model development is integrated with well-designed in situ He implanter (ISHI) experiments in High Flux Isotope Reactor (HFIR), as well as dual-ion irradiations (DII). This report focuses a critical bubble model (CBM) based void nucleation mechanism, and effects of dose rate on void swelling. The model predictions are in promising agreement with experiment.

PROGRESS AND STATUS

Introduction

One of the challenges that fusion reactor materials development faces is to predict and mitigate the effects of transmutation He on the mechanical properties of first wall structural materials [1]. Following up on the He transport and fate model reported previously [2], the objective of the present work is to develop prediction models for void-swelling in TMS alloys as a function of material and environmental variables, such as microstructure, displacement damage dose, He/dpa ratio and displacement dpa rate (dpa/s). Here we describe the implementation of CBM based void nucleation mechanism and explore the effects of dose rate on the void swelling.

Multi-scale Modeling

The interaction of He atoms, vacancies, and He-vacancy clusters with each other and key microstructural features such as dislocations, nano-features (NF) and grain boundaries (GB) are modeled using rate theory (RT) describing the HTFC as illustrated in Figure 1.

Void Nucleation on He Bubbles

Void nucleation on the bubbles modeled based on the critical bubble concept [1-3]. Bubbles grow stably by capturing He, while absorbing/emitting vacancies to maintain balance between excess vacancy flow, surface tension and He pressure. When the bubbles reach a critical size at a critical number of He atoms, the net (minus interstitial) vacancy absorption rate exceeds the emission rate and the cavity begins to grow as a void. The critical size depends on the excess vacancy flux, which in turn depends of the overall sink microstructure and displacement per atom (dpa) rate. The model evaluates the He bubble size distributions and excess vacancy fluxes to convert those above the critical size to growing voids.

Companion Experiments Using Two He-implanter Techniques

The model calculations are compared to our systematically designed and carefully controlled Heimplantation experiments. One He-implantation method is ISHI in a mixed spectrum reactor like HFIR. While the details are given elsewhere [4-6], briefly the ISHI technique uses NiAl coating deposited on transmission electron microscope (TEM) samples of tempered martensitic steels (TMS) and nano-structured ferritic alloys (NFA). The isotope, ⁵⁸Ni, which is about 68% of natural Ni, undergoes a two-strep reaction with neutrons to emit 4.71 MeV He α -particle. Depending on the thickness of the coating, He is implanted uniformly over about a 6 to 8 μ m thick depth. The He-to-dpa ratio can be adjusted from \approx 5 to 50 appm He/dpa, by adjusting the coating thickness. A second He-implantation method is DII, which we carried out in DuET facility at Kyoto University in Japan. The major difference between the techniques is the dpa rate, that is \approx 10⁻⁶ and 10⁻³ dpa/s for ISHI and DII, respectively.



Figure 1. Schematics of the rate-theory integration code structure for HTFC models.

Model calculations have been performed for the typical TMS microstructural parameters: dislocation density = $3 \times 10^{14} \text{ m}^{-2}$, prior-austenite grain size = 20 um and lath width = 600 nm. The corresponding values of the thermodynamic parameters used in the model are shown in Table 1.

	Parameter	Value
	i arameter	value
Binding energy of He (s)	To dislocations (disl.)	0.9 eV
	To nm-scale features (NF)	0.59 eV
	To grain boundary (GB)	0.5 eV
	To disl. Intersection vs. disl.	0.6 eV
	To deep trap on GB vs. GB	0.6 eV
Migration energies	He(i) in matrix	0.08 eV
	He(s) in matrix	2.35 eV
	V in matrix	1.40 eV
	SIA in matrix	0.08 eV
	He(s) on dislocations	1.18 eV
	He(s) on GB	1.18 eV
Formation energy of	Vacancy	1.6 eV

|--|

Results

Void swelling as a function of dpa, He/dpa and dpa/s with comparisons to ISHI and DuET experiments

Figures 1a and b summarize the model predictions of void swelling (f_v) at dose rates from 10⁻⁶ to 10⁻³ dpa/s and He/dpa ratios of 15 and 45 appm/dpa. Figure 1a plots f_y up to 100 dpa, while Figure 1b shows a low dpa blowup, where bubble to void transitions mainly occur. The fv is largest at 10⁻⁵ dpa/s and decreases with both lower and higher dpa rates. The effects of dpa rate on void swelling are complex. One effect of a higher dose rate is to enhance point defect recombination, that reduces radiation enhanced diffusion (RED). The effectiveness of RED can be measured by gsv, the fraction of irradiation-induced vacancies (V) that reach permanent sinks, given as $q_{sv} = D_v X_v S_t / G_v$ where G_v . D_v and X_v are the generation rate, diffusion coefficient and concentration of V, and S_t is the total sink strength. The g_{sv} for the starting microstructure decreases with increasing dose rate from 2x10⁻² at 10⁻⁶ dpa/s to 10⁻⁴ at 10⁻³ dpa/s. This generally delays microstructure evolution involving diffusion process such as bubble formation and growth as well as void growth. Another opposing effect of a higher dpa rate is to enhance bubble to void conversion. As shown in Figure 2a, the higher dpa rate increases vacancy supersaturation Λ (= [D_vX_v-D_iX_i]/D_vX_{ve}), here X_{ve} is thermally equilibrium vacancy concentration and Di and Xi are diffusion coefficient and concentration of selfinterstitial atoms. The Λ then reduces critical bubble radius, r* according the relation for a given temperature [1] (Figure 2c). The initial r* at 0 dpa decreases by a factor of \approx 3 from 10⁻⁶ to 10⁻³ dpa/s. Clearly this accelerates bubble to void transformation at higher dose rates. These opposing effects lead to a complex dependence of void swelling on the dose rate.

Figure 2 also shows that the Λ decreases (Figures 2a and b) and r^{*} increases (Figures 2d and e) with dose with increasing total sink strength, S_t, at various dpa rates for the 45 appm He/dpa case. The evolution of



Figure 1. a) The dpa dependences of void volume fraction (f_v) for various dpa rates from 10⁻⁶ to 10⁻³ dpa/s and He/dpa ratios of 15 and 45 appm/dpa; b) The low dose blow-up; comparisons with c) ISHI and d) DII experiments.

the bubbles at various microstructural sites is discussed in the next section. Note, recent work by Wirth and co-workers suggests that small bubbles are biased sinks [7], but here they are the treated as neutral sinks.

The Figures 1a and b also show systematic effects of the He/dpa ratio. High He/dpa decreases the incubation dpa for swelling, but a subsequent effect is that the swelling curves layover at higher dpa. The reasons for this behavior are also discussed in the next section.

Figures 1c and d compare the coordinated ISHI and DII experimental void swelling data with the models of corresponding dpa rates, 10⁻⁶ and 10⁻³ dpa/s, respectively. The ISHI data are shown in two He/dpa groups and DII in six groups, while models are for He/dpa of 15 and 45 appm/dpa in red solid and green dashed lines, respectively. In both types of experiments the models are in reasonable agreement with experimental data of the corresponding He/dpa groups.



Figure 2. Vacancy supersaturation, Λ , as a function of a) dpa and b) the corresponding sink strength, S_t, for various dpa rates at 45 appm He/dpa; as well as resulting r^{*} as a function of c) Λ ; d) dpa; and e)S_t.

Effects of dpa rate and He/dpa on cavity microstructure evolution

Figure 3 shows the evolution of cavity microstructure – the average size <r> and total density X (per atom) of bubbles at various sites, as well as those for voids. Figure 4 shows corresponding blow-ups for the initial 20 dpa, where void nucleation mainly takes place. Figure 5 shows the breakdown of voids by nucleation site. The figures are in lower to higher dpa rate order, from 10⁻⁶ dpa/s at the top to 10⁻³ dpa/s at the bottom, and for 15 and 45 appm He/dpa left to right, respectively.

The solid lines in Figures 3 and 4 show the bubble $<r_b>$ in the matrix (black), on dislocations (green), on grain boundaries (GB) (blue) and on lath boundaries (LB) (red), along with the void $<r_v>$ (purple), on the left



axis; the dashed lines are the corresponding $X_{b/v}$ on the right axis. At the lowest dpa rate of 10⁻⁶ dpa/s, void nucleation occurs only on dislocations. The dislocation model includes a wide variation of bubble nucleation

Figure 3. The average bubble and void sizes and number density as a function of dpa for dpa rates of 10^{-6} (top) to 10^{-3} dpa/s (bottom) and He/dpa of 15 (left) and 45 (right) up to 100 dpa.



Figure 4. Low dpa blow-ups of the size and number density plots shown in Figure 3.



Figure 5. The void number densities by nucleation site as a function of dpa for the models.

nucleation site spacing, so that larger bubbles form at the sites with larger spacings. The largest end of the bubble distribution in the group first reaches the critical size ($r^* \approx 1.7$ nm). Void nucleation continues by converting the bubble with smaller spacings on dislocations as they gain more He. This is indicated by the gradual decrease in dislocation bubble number density and corresponding increase in void number density. The nucleation stage ends by ≈ 8 and ≈ 3 dpa at 15 and 45 appm He/dpa, respectively. Thus, the higher 45 He/dpa accelerates void nucleation, while leading to a much higher number density of matrix bubbles (black dashed line) at high dpa; the matrix bubble sinks slow void growth compared to lower 15 appm He/dpa condition, resulting in a layover of the swelling curves at higher dpa.

At 10⁻⁵ dpa/s, void nucleation on dislocations occurs at a much lower < 1 dpa due to the smaller r*. The bubbles on LB also convert to voids in the dose range below \approx 10 dpa and \approx 30 dpa for 15 and 45 appm He/dpa, respectively. The faster void nucleation at 10⁻⁵ dpa/s than 10⁻⁶ dpa/s results in a higher number density of voids which grow at low dpa region. While the final average <d> are similar or slightly larger at 10⁻⁶ dpa/s, the higher number density results in larger swelling at 10⁻⁵ dpa/s.

At the even higher dpa rates of 10⁻⁴ and 10⁻³ dpa/s, void nucleation on dislocations occur even faster, but the contribution of the nucleation at LB becomes more significant. Void nucleation on matrix bubbles dominates at the highest dose rate and He/dpa. The huge sink unbalance between bubbles-voids and dislocations, leads to much lower void growth rates and swelling as well as larger loss of point defects on recombination.

Future Research

The model will be refined to include recent findings on sink bias on cavities and dislocations as well as be expanded to explore microstructure variations to compare with experimental data in more details.

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10.2 THREE-DIMENSIONAL MODELING OF THE EFFECTS OF HELIUM-VACANCY CLUSTERS ON THE STRESS-STRAIN BEHAVIOR OF AN IRON BICRYSTAL AT 600 K—B.N. Nguyen, R.J. Kurtz (Pacific Northwest National Laboratory)

OBJECTIVE

The objective of this study is to investigate the effects of helium (He) vacancy clusters on the stress-strain behavior of an iron (α -Fe) bicrystal by a mechanistic finite element (FE) approach using a continuum damage mechanics (CDM) description of the material behavior informed by molecular dynamics (MD) data. The approach models an α -Fe bicrystal system in which the elastic-plastic crystals finely discretized in three-dimensional (3D) FE are connected to each another by cohesive elements. The He-vacancy clusters at the GB are explicitly modeled through an equivalent hollow sphere under internal pressure and located in the middle of the modeling domain. Previously, MD and FE analyses of this bicrystal system subjected to uniaxial tensile loading and internal He pressure at 5 K and room temperature (RT) were performed using the LAMMPS software and ABAQUS FE package, respectively. During this reporting period, the effects of the He_nV₂ clusters on bicrystal stress-strain behavior at 600 K was studied to complete the validation of this modeling approach for a representative operating temperature.

SUMMARY

First, MD analyses of the single crystal and bicrystal lattice configurations ({ $\Sigma 11 < 110 > {332}$ orientation) were performed to compute the uniaxial tensile responses of the α -Fe single crystal and GB. The MD results were then used in FE analyses of the same systems to identify parameters for the CDM constitutive relations for the crystal and the traction-separation law for the GB modeled by cohesive elements. Next, a 3D FE model of the α -Fe bicrystal system with an imperfect GB subjected to uniaxial tensile loading was developed. This model includes an equivalent hollow sphere representing the system with two vacancies under internal pressure in the middle of the GB to model the effects of pressurized He-vacancy clusters at 600 K on stress, strain and damage distributions. Finally, MD stress/strain data of the same bicrystal system with He-vacancy clusters were compared to the corresponding FE results to further validate this approach that had been validated previously for the same system at 5 K and RT.

PROGRESS AND STATUS

Introduction

Ferritic/martensitic steels are considered to be prime candidate materials for structural applications in future fusion reactors [1]. In such applications, these materials are exposed to high-energy neutrons leading to He generation due to transmutation reactions. Formation of He is of particular concern because it can cause hardening and increases in the ductile-to-brittle transition temperature (DBTT) [2-3] and swelling as a result of nucleation and growth of He bubbles [4]. The He also weakens GBs by lowering the GB cohesive stress or by promoting nucleation, growth and coalescence of GB cavities, which can lead to intergranular fracture at high temperature [5]. As it is very difficult to experimentally quantify the effects of nano-scale He bubbles on material integrity, computational methods such MD simulations have been very helpful to elucidate the degradation mechanisms associated with He bubble formation. Our previous reports [6-7] have shown that a promising and efficient approach to model the effects of He on material integrity is by FE modeling of an α-Fe bicrystal system in which CDM is used to describe the constitutive behavior of α-Fe and cohesive elements are used to model the GB behavior. In [6-7], this mechanistic approach was validated through full 3D MD and FE analyses of such a system with perfect and imperfect GB at 5 K and RT and subjected to uniaxial tensile loading. In this report, we have completed the validation of the developed approach by analyses of the stress-strain behavior of the same bicrystal system subjected to pressurized He-vacancy clusters and uniaxial tensile loading at 600 K.

Model Development

We refer to our previous reports [6-7] for the details of the FE models developed for analyses of the α -Fe bicrystal systems containing the clean GB (without He clusters) (Figure 1a) and the GB involving two vacancies depicted by an equivalent hollow sphere occupied by He at a given pressure (Figures 1b and 1c). In these models the two crystals were connected one to another by cohesive elements. The dimensions of the modeling domains are 8 nm x 8 nm x 25 nm. In these models, the vertical displacements of the bottom boundaries were fixed while uniform vertical displacement loadings were incrementally applied on the top surfaces. The loading for the model with He-vacancy clusters (Figure 1b) also involved the prescribed internal pressure ramping up to a maximum value at the first loading step before application of the vertical displacement at the next loading step. Figure 1b illustrates a vertical section through the model with an imperfect GB showing the location of the equivalent hollow sphere that is further illustrated in Figure 1c. Similar to the 5 K and RT cases previously reported [6-7], the constitutive parameters of the elasticplastic damage model describing the single crystal α-Fe stress-strain behavior at 600 K were identified using the corresponding MD results for this temperature. In addition, the model with the clean GB (Figure 1a) was used to identify the material parameters for the traction-separation law describing the GB. Subsequently, the same set of model parameters for the crystal and GB was used in all the analyses to determine the He bubbles effect on the stress-strain response of the as-formed bicrystal system at 600 K.



Figure 1. The 3D FE models for the α -Fe bicrystal system subjected to uniaxial tensile loading in the vertical direction (z-direction) – (a) model with clean GB, (b) a vertical cross section of the model with two vacancies depicted by an equivalent hollow sphere occupied by He, and (c) a magnified view showing the hollow sphere region.

Results

A series of ABAQUS FE analyses had first been conducted for the single crystal model and the bicrystal model with clean GB to identify the material parameters for the constitutive laws at 600 K, which were subsequently used for the analyses of the bicrystal system with imperfect GB. In this work, the elastic-plastic model with isotropic hardening and isotropic damage available in the ABAQUS material model options was used for the crystals while cohesive elements were used to describe the behavior of the GB. Figure 2 reports the longitudinal stress-strain (in the z-direction) results at 600 K predicted by CDM modeling compared to the corresponding MD data. Good correlations between CDM and MD results allowed

identification of the material parameters at these temperatures for use in subsequent analyses of the bicrystal model with He-vacancy clusters.

In the FE model with an imperfect GB, He pressure was applied inside the hollow sphere incrementally to a prescribed level during the first loading step. At the next loading step, while maintaining the He pressure at the maximum prescribed level, uniform vertical displacements were applied on the top model boundary incrementally until the system completely failed.



Figure 2. Longitudinal stress-strain responses at 600 K predicted by FE analysis using CDM compared to the corresponding MD data. SC and BC denote single crystal and bicrystal, respectively.



Figure 3. (a) Damage distribution (viewed through a central cross section along the z-direction) depicted by the failure indicator for 0 GPa He pressure, and (b) Contour of the normal traction stress on the GB at the onset of total failure in the bicrystal system with 2 vacancies at 600K, and (c) Damage distribution for 25 GPa He pressure at 600 K.

Figure 3a shows the damage distribution at the onset of total failure (failure stress $\sigma_r = 10.56$ GPa) of the bicrystal system with imperfect GB subjected to 0 GPa He pressure and uniaxial loading at 600 K. Before final failure, damage in the crystals was still moderate but significant as indicated by the failure indicator about 0.53 near the hollow sphere and ~0.4 elsewhere. Total failure of the bicrystal was mainly caused by failure of the GB as illustrated in Figure 3b that shows the GB normal traction stress (S_{zz}) reached the fracture level of 10.56 GPa and started dropping to zero in the region surrounding the hollow sphere. At 25 GPa He pressure (Figure 3c), we observe failure of the crystals around the hollow sphere (failure indicator=1) at the onset of final failure caused by a total rupture of the GB.

The MD analyses revealed that GBs loaded at 300K and 600K failed by deformation twinning, rather than nucleation and propagation of <111> type lattice dislocations. Figure 4a shows the atomic configuration for the GB with a He₃V₂ cluster (two vacancies) loaded at 300K just beyond the UTS. The He pressure attained was 24.7 GPa. Deformation twins nucleated in the vicinity of the cluster and propagated throughout the specimen. Nano-crystalline Fe is known to deform by twinning rather than dislocation emission as discussed by Tong et al. [8]. Evidently, the GB investigated in this work enabled deformation twinning relative to dislocation emission. Note MD results for the 600 K case were similar to the 300 K case. For a comparison of results for the 600 K case, Figure 4b presents the damage distribution at the onset of final failure predicted by CDM for 25 GPa He Pressure in the bicrystal system and in the same plane (XZ plane) as the MD section shown in Figure 4a. As expected, CDM predicts continuum plasticity coupled to damage

that was progressively accumulated around the hollow sphere before final failure, but the collapse of the bicrystal was predicted by CDM as a result of GB rupture.



Figure 4. (a) Atomic configuration for a $\sum 11$ {332} grain boundary at 300 K with a H₃V₂ cluster at the center of the GB beyond peak load. White and black atoms are different {110} Fe layers normal to the plane of the paper. The He atoms are in red. The GB fracture was not observed, but rather twinning deformation. (b) CDM Prediction of damage accumulation at the onset of failure in the same system subjected to 25 GPa He pressure.

Finally, the effects of He pressure on the stress-strain response, strength and fracture strain of the bicrystal system at 600 K are illustrated in Figures 5a, 5b and 5c, respectively. The presence of vacancies at the GB reduces the system strength and ductility only slightly. However, such reductions are more important at 5 K and RT as previously reported [6-7]. With increasing He pressure up to 24 GPa, a negligible reduction of both failure strain and strength is found at 600 K as illustrated in Figures 5b and 5c, but beyond 24 GPa He pressure, CDM model predicts more significant reductions in strength and failure strain than the MD model for the bicrystal. Figures 5b and 5c show a good agreement between CDM and MD results up to 24 GPa.



Figure 5. (a) Uniaxial stress-strain responses predicted by CDM FE analysis, (b) strength and (c) failure strain as a function of the He bubble pressure for the α -Fe bicrystal system at 600 K predicted by CDM FE and MD analyses.

Conclusions

During this report period, the mechanistic FE approach informed by MD data was further validated to investigate the effects of He bubbles on the stress-strain behavior of an α -Fe bicrystal system at 600K, a representative operating temperature. The CDM predictions agree well with MD results for He pressure up to 24 GPa. However, above 24 GPa, CDM modeling predicts more significant reductions in strength and fracture strain than MD analysis. Our CDM predictions show as fairly good agreement with MD results at 600 K as at 5 K and RT previously reported [6-7]. The developed approach appears to be efficient in terms of computation time compared to MD simulations alone. Thus, it can serve as a good complementary approach to MD simulations to study the effects of He bubbles or other radiation-induced effects on material integrity.

Future Work

We are revising a journal article submitted to Journal of Nuclear Materials to describe the approach and initial modeling results. The approach will be further developed to include MD simulations at lower loading rates to identify other failure mechanisms that might be triggered at lower loading rates.

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10.3 POTFITML: A COMPUTATIONAL FRAMEWORK TO FIT INTERATOMIC POTENTIALS WITH MACHINE LEARNING—W. Setyawan, Charles H. Henager Jr. (Pacific Northwest National Laboratory)

OBJECTIVE

The objective of this research is to develop a computational framework to explore the feasibility of using machine learning to improve fitting of interatomic potentials.

SUMMARY

A computational framework (POTFITML) has been developed that utilizes machine learning (ML) technique to model property-to-parameter relationship of interatomic potentials. In this report, POTFITML is employed to fit an embedded-atom method (EAM) potential for Re to demonstrate the feasibility of using ML to improve the Re potential previously fitted with a force-matching (FM) method. A total of 2000 potential sets have been fitted with the FM method. The sets are used to test POTFITML. The results show that POTFITML can consistently produce potentials that are more accurate than the initially best set from the FM fit. In fact, as iteration progresses, better potentials than the currently best set are produced. Even though POTFITML is successfully tested, the accuracy of the best potentials obtained so far (up to 2000 iterations) still needs improving. Further iterations are being performed to converge the POTFITML calculations. The convergence study would be useful to reveal the limitations of EAM formalism and parametrization to concurrently describe the elastic moduli and formation energies of point defects in Re.

PROGRESS AND STATUS

In this research, we explore the idea of using ML to improve fitting of an interatomic potential. The idea is based on the fact that a large number of fitted trial potentials is typically generated in developing a potential in which the best one is selected. The question is, how can we utilize the dataset of these trial potentials to improve the best set in the dataset? To answer this question, we use the dataset to train a ML model to solve an inverse problem. That is, rather than using ML to describe the typical parameter-to-property relationship, we use it to describe the property-to-parameter relationship, connecting the properties predicted by a potential to the parameters of the potential. Subsequently, a set of target properties is used to evaluate the trained model to produce a set of potential parameters (a new trial). Ideally, the new trial would represent the "target" potential. In practice, this is not the case. Therefore, if the new trial is better than the worst set, it can be used to replace the worst set in the dataset and the process can then be iterated. POTFITML is a computational framework that is developed in this research to automate the iteration. As a framework, it manages external codes to perform the following jobs, 1) a ML job to train and evaluate the ML model and to produce a trial parameter set, 2) a FORMAT job to format the trial parameter set into a potential file format that can be used directly in molecular dynamics (MD) codes, and 3) an MD job to perform MD simulations to obtain the properties of the trial potential.

In the previous report [1], the first version of POTFITML (previously called RFITML) has been completed. In that version, POTFITML is designed only as a daemon program. In this report, POTFITML is further developed to allow running it as a daemon (daemon mode) or as a regular program (interactive mode). In the daemon mode, POTFITML runs an external code by submitting a batch script to the job queue on a computer cluster. A different number of computer cores can be requested for each job within the job's respective batch script to optimize the utilization of cores for each job. However, if the computer cluster is over-subscribed, there be may a considerable wait-time in the job queue. Therefore, an interactive mode is added. In the interactive mode, the POTFITML runs the external codes directly. On a computer cluster, this means that POTFITML itself is run by submitting it to the job queue. Once POTFITML runs (after the wait-time), it interactively invokes the external programs directly. The disadvantage is that the maximum number of cores that is available to run the external programs is the number of cores may not be optimum since the scalability of each of the external programs may be different. Evidently, one may run POTFITML on a personal computer with the interactive mode directly without submitting it to a job queue.

In this report, POTFITML is tested to improve the fitting of an EAM potential for Re. The EAM potential is parametrized as follows. A Morse interaction with a smooth cutoff function is used for the pair interaction $\phi_{ij}(r)$ between atoms i and j separated by a distance r

$$\phi_{ij}(r) = D \left(e^{-2\alpha(r-r_0)} - 2e^{-\alpha(r-r_0)} \right) \Psi_{sc}(x)$$
(1)

$$\Psi_{sc}(x) = \frac{x^4}{1+x^4}$$

$$x = \frac{r-r_c}{h}$$
(2)

where r_c is the cutoff distance and is set to be 5.5 Å. The electron density $\rho(r)$ due to an atom at a distance r from the atom is taken from [2] where a consistent set of universal atomic electron density of all elements in the periodic table has been derived. The form of the atomic density is

$$\rho(r) = \rho_e \exp\left(-\beta(r/r_e - 1)\right) \tag{3}$$

where for Re, $\rho_e = 0.242$ Å⁻³, $\beta = 5.684$, and $r_e = 2.040$ Å. The embedding energy for inserting an atom in a location with a total electron density of ρ is

$$F(\rho) = a_1 \sqrt{\rho} + a_2 \rho + a_3 \rho^2 \tag{4}$$

To generate the initial dataset to train the ML model, FM fits are performed using the POTFIT code [3]. The parameters to fit are D, α , r_0 , h, a_1 , a_2 , and a_3 . Six liquid structures under various strain tensors are used as reference structures. Note that these are the only reference structures used in the FM fit. Density functional theory (DFT) calculations are performed to obtain the forces, energies, and stresses in the reference structures. Each structure contains 64 atoms, therefore a total of 6*64*3 forces, 6 energies, and 6*6 stresses is used to fit the potential. The factor 3 in the number of forces is because there are 3 components for every force vector. Likewise, the factor 6 in the number of stresses is because there are 6 components for energy stress tensor. The FM fit is performed using a force:energy:stress weight ratio of 1:1920:320. This ratio corresponds to an effective weight ratio of 1:10:10, i.e. an energy and a stress component are intended to be 10 times more important than a force component. In a force-matching fit, simulated annealing is employed to better explore the parametric space. For each parameter, a new value is randomly sampled from within the allowed range. In POTFIT, we found that even though a random sampling is used, the parameters are iterated sequentially, i.e. the random sampling is performed for parameter 1, followed by parameter 2, and so on. This procedure introduces a bias which favors parameters located earlier in the parameter array. Hence, we modified POTFIT to also select the parameters randomly.

From the FM fit, we obtain 2000 fitted potential sets. These sets are divided into 4 datasets, i.e. dataset1 to dataset4, with 500 sets each. To test POTFITML, we employ the TensorFlow, POTFIT, and LAMMPS codes to perform the ML, FORMAT, and MD jobs, respectively. Four independent tests are performed using the four datasets. The error of a set is calculated as a weighted sum of property errors with respect to target properties. The list of 19 target properties with their target values and weights is presented in Table 1. Mean-absolute-error (*mae*) and root-mean-squared-error (*rmse*) modes are supported in POTFITML

$$e_j = (p_j - q_j)/q_j \tag{5}$$

$$mae = \frac{\sum_{j=1}^{L} w_j |e_j|}{\sum_{j=1}^{L} w_j}$$
(6)

$$rmse = \sqrt{\frac{\sum_{j=1}^{L} w_j e_j^2}{\sum_{j=1}^{L} w_j}}$$
(7)

where p_i is property-j, q_j is target property-j, e_j is relative error of property-j, w_j is the error weight of property-j, and *L* is the number of properties. The following results are obtained using the *mae* error mode.

Table 1. List of target value and weight of properties of Re EAM potential to test POTFITML. Target values labelled with (Exp.) are from experiments, the rest are from our DFT calculations. E_c is cohesive energy, E_f is point defect formation energy. Interstitial positions C to BC are depicted in [4]. Units are Å, eV, and GPa. EAM_{FM} is the best potential from the FM fit, while EAM1 to EAM4 are the best potentials produced by POTFITML (so far up to 2000 iterations) using dataset1 to dataset4, respectively. The *mae* is the error of the potentials calculated using Equation 6.

Property	Target	Weight	EAM _{FM} set1937	EAM1 iter1675	EAM2 iter572	EAM3 iter725	EAM4 iter277
а	2.761 (Exp.)	1	2.782	2.818	2.752	2.822	2.759
c/a	1.614 (Exp.)	1	1.605	1.604	1.613	1.613	1.605
E _c [hcp]	8.03 (Exp.)	10	7.69	8.143	8.50	8.11	8.26
<i>E</i> c [hcp-fcc]	0.06	10	0.05	0.06	0.05	0.05	0.06
<i>E</i> _c [hcp-bcc]	0.31	10	-0.01	0.01	0.001	0.02	-0.002
E _f [Vac]	3.08	1	2.77	3.47	3.12	3.36	3.36
E_f [C]	6.52	1	5.83	7.74	6.49	7.99	6.99
<i>E</i> _f [O]	8.13	1	5.86	7.86	6.66	8.11	7.08
$E_f[S]$	6.53	1	6.14	8.09	6.69	8.39	7.31
$E_f[T]$	6.52	1	6.07	8.08	6.86	8.38	7.31
<i>E</i> _{<i>f</i>} [BO]	7.41	1	5.89	7.83	6.59	8.21	7.02
E _f [BS]	8.09	1	5.64	7.50	6.33	7.82	6.78
<i>E</i> _f [BT]	Relaxed to BO	1	6.42	8.40	6.28	8.66	6.76
<i>E</i> _f [BC]	Relaxed to BO	1	5.64	7.47	6.33	7.83	6.76
C ₁₁	613 (Exp.)	1	414	450	475	458	459
C33	683 (Exp.)	1	591	764	605	685	667
C ₁₂	270 (Exp.)	1	300	304	328	307	312
C ₁₃	206 (Exp.)	1	286	302	288	285	287
C44	163 (Exp.)	1	82	123	86	106	103
mae			0.325	0.267	0.313	0.296	0.282

A fully connected deep neural network with 8 hidden layers (32 nodes per layer) is used as the ML model. The *tanh* activation function is employed in each node. Several other activation functions have been explored as well, e.g. *sigmoid* and *ReLU* functions, however *tanh* appears to produce reliable potentials more consistently than the others. The ML model is trained using the Adam optimizer with a learning rate of 0.001 and with a mean squared error as the loss function. The training is performed with a batch size = 32, number of epochs = 50, and 5-fold validation.

Figure 1 shows the evolution of the error of the potential that is produced with POTFITML at each iteration. The best, the worst, and the average error of sets in the dataset at each iteration are also shown in the figure. In all the tests, POTFITML can consistently produce potentials that are more accurate than the

initially best set from the FM fit. In fact, as the iteration progresses, better potentials than the currently best set are produced. This demonstrates that employing ML to improve the fitting of interatomic potential is feasible. The best sets within the 2000 iterations so far correspond to iterations 1675 (dataset1), 572 (dataset2), 725 (dataset3), and 277 (dataset4). The properties of these sets are summarized in Table 1. In Table 1, these sets are labelled as EAM1, EAM2, EAM3, and EAM4, respectively. The properties of the initially best set among the 2000 sets from the FM fit (set 1937) are also shown in Table 1 and the potential is labelled as EAMFM. Even though POTFITML is successfully tested, the properties of EAM1 to EAM4 are still unsatisfactory. Nevertheless, as evident from Figure 1, the average error still decreases steadily as the iteration progresses. Further iterations are being performed to converge the calculations. If the potential remains unsatisfactory after convergence, this would indicate that the accuracy of the potential is likely limited by the EAM parametrization (Equations 1 to 4) or even by the EAM formalism itself.



Figure 1. Error of Re EAM potential set obtained with POTFITML at each iteration (blue curve) calculated with Equation 6. The shaded area spans the worst and the best errors in the dataset, while the average error is plotted as a black curve. Blue circles and asterisks are iterations that produce potentials that are better than the initially best set and currently best set, respectively. The iteration numbers of the currently best sets are shown. Panels a) and b) are results obtained with dataset1 and dataset2, respectively. Panels c) and d) are plotted in the continuation of Figure 1.





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11. FUSION SYSTEM DESIGN

No contributions this reporting period.

12. IRRADIATION METHODS, EXPERIMENTS, TESTING AND SCHEDULES

12.1 HIGH TEMPERATURE VACUUM TENSILE FRAME EQUIPPED WITH NON-CONTACT VIDEO EXTENSOMETER—X. Chen, C. Stevens, K. Smith, J.W. Geringer, Y. Katoh (Oak Ridge National Laboratory), H. Sakasegawa (National Institutes for Quantum and Radiological Science and Technology)

OBJECTIVE

The aim of this task is to install a high temperature vacuum tensile test system in the hot cell to provide the cutting-edge mechanical testing capability for the Fusion Materials Science Program.

SUMMARY

The installation of a high temperature vacuum tensile test system was completed in March 2019. The tensile frame is stationed in cell #1 of the Irradiated Materials Examination and Testing facility (IMET) at Oak Ridge National Laboratory (ORNL). The frame is capable of both room temperature and elevated temperature mechanical testing in vacuum with a non-contact video extensometer for strain measurements.

PROGRESS AND STATUS

A high temperature vacuum tensile frame was installed in cell #1 of the IMET facility at ORNL in March 2019. Figure 1 shows an overview picture of the frame inside the hot cell. The entire test system consists of:

- 1. Instron[®] 5967 electromechanical universal testing system (Figure 2(a)).
- 2. Oxy-Gon[®] FR210 customized tensile test vacuum furnace system (Figure 2b)).
- 3. Pfeiffer® ACP 40 dry roughing pump and TPH 2301 turbomolecular pumps (Figure 2(c)).
- 4. ThermoFish Scientific® ThermoFlex15000 air cooled recirculating chiller (Figure 2(d)).

The tensile frame has a load capacity of 30 kN with a 5 kN load cell currently installed. The frame crosshead speed covers the range of 0.001 to 1000 mm/min. Both tensile and compression tests can be performed using the frame. The ultimate vacuum level achievable for this frame is approximately 7-8 x 10^{-7} torr at 800°C. The heating system utilizes tungsten mesh heating elements with the maximum temperature rated at 1200°C. The usable working zone within the vacuum furnace has dimensions of 7.6 cm diameter x 10 cm height.



Figure 1. Overview of the high temperature vacuum tensile frame installed in the IMET hot cell.







(c)

ughing pump

191

10



Figure 2. Major components of the high temperature vacuum tensile frame. (a) Tensile frame and its controller, (b) customized tensile test vacuum furnace system, (c) vacuum pumps, (d) chiller

The system also allows three type N thermocouple measurements with one used for temperature control and one type C thermocouple probe for overtemperature protection. The locations of these thermocouples are shown in Figure 3.



Figure 3. Inside view of the vacuum chamber.

Currently, a SS-J3 tensile fixture shown in Figure 4 has been installed in the tensile frame. Temperature gradient across the specimen gauge is fully captured by measurements from two thermocouples. The specimen retaining pin design minimizes potential loss of broken tensile halves after testing. In the initial room temperature testing of ten Eurofer97 tensile specimens, both the fixture design and tensile frame exhibited outstanding performance.



Figure 4. SS-J3 tensile specimen fixture.

To facilitate non-contact strain measurements, the ORNL team collaborated with the QST team in designing a non-contact video extensometer system with the QST team leading the camera, lens, light source, and image acquisition system and ORNL leading the fixture design and system installation. The system is capable of global strain measurements with the potential for digital image correlation related measurements. The system is also designed for both room temperature and elevated temperature measurements. The major components (shown in Figure 5) for this system consists of:

- 1. Keyence CA-H2100M camera.
- 2. VS Technology telecentric lens VS-TC1-300CO.
- 3. 165-SHU UV curing ultra-high-pressure mercury vapor lamp light source.
- 4. CV-H1XA Simulation-Software.
- 5. Newport DS65-XYZ linear stage.
- 6. Dovetail with a stopper design.
- 7. Quick release locking knob.



Figure 5. Component view of non-contact video extensometer system.

12.2 PREPARATION OF CREEP TUBE SPECIMENS FOR CREEP RABBIT CAPSULES—M. Ando, H. Sakasegawa, T. Kato, T. Nozawa and H. Tanigawa (National Institutes for Quantum and Radiological Science and Technology), J. W. Geringer, P. L. Mulligan, S. M. Curlin, J. Schmidlin, Y. Kato (Oak Ridge National Laboratory)

OBJECTIVE

The objectives of this work were to prepare creep tubes (helium gas pressurized tubes) for High Flux Isotope Reactor (HFIR) creep rabbit capsules and measure their diameter by a laser profilometer before HFIR irradiation. This work is part of the U.S. Department of Energy (U.S. DOE) – National Institutes for Quantum and Radiological Science and Technology (QST), fusion materials collaboration.

SUMMARY

Four creep tube specimens were prepared for creep rabbit experiments. The creep tube specimen has many fabrication steps and needs much time for preparation. This report summarizes the series of fabrication records for the creep tube specimens.

PROGRESS AND STATUS

Introduction

Irradiation creep of structural materials is one of the important irradiation properties. However, it is not easy to obtain the irradiation creep data because irradiation creep is caused by an applied stress on a specimen under neutron irradiation [1]. For this reason, creep tube type specimens, which are pressurized by helium gas, are usually used to obtain the irradiation creep data [2]. For the RAFM steel F82H, irradiation creep experiments were performed using pressurized creep tube specimens in FFTF/MOTA, HFIR-RB11J, 12J and 15J capsules [3-4]. In these experiments, the hoop stresses and diameter changes were obtained. However, irradiation creep data has not yet been obtained for highdose (>20dpa) irradiated F82H at 300°C. Therefore, if a creep rabbit capsule can be developed, it becomes easy to obtain the irradiation creep data for 300°C irradiation because the rabbit capsule is not expensive and can be irradiated in the HFIR target rod positions. This report summarizes the fabrication of creep tubes for creep rabbit irradiation and the results of diameter measurements of each tube before HFIR irradiation.

Preparation of creep tube specimens

Material and Fabrication of parts

The material used is F82H IEA heat (Plate No. 2-20, which was a base metal area of TIG welding plate from Heat No. 9741.) [5]. The tubing and two types of end caps (top and bottom) were machined by the KMTL (Kobe Material Testing Laboratory Co., Ltd. In Japan) from this material. Figure 1 shows a drawing of these parts a)-c) and a sampling position for original plate (d). The direction of tube length was chosen to be parallel to the rolling direction of the plate. ID was also engraved with each bottom end cap. These tube parts were machined and then polished on the surface side. The surface roughness after polishing was about 4 nano-inches. These parts were transferred to Oak Ridge National Laboratory (ORNL).

Assembly of the creep tube (EB weld)

Assembly of the creep tube specimens was performed by the ORNL Materials Joining Group. The creep tube specimens were fabricated by electro-beam welding a tube section and each end cap (upper and lower) in a vacuum chamber. SiC thermometry pieces were inserted in each tube [6]. Thus, the irradiation temperature can be determined after irradiation, and moreover this reduces the amount of pressurized helium gas needed compared to previous tube experiments.

Post weldment heat treatment

Post weldment heat treatment (PWHT) of each tube was performed in a vacuum furnace of the Materials Joining Group. Heat treatment was at 720°C for 1 hr. Also, very thin tantalum foil wrap was used to prevent oxidization of tube surfaces during this PWHT. After the PWHT, the diameter of each unpressurized specimen was measured using a non-contacting laser profilometer system (Beta Lasermike Model 162).

Pressurizing the creep tube specimens

Each tube was pressurized by the Material Joining Group using helium gas. The pressurizing conditions are shows in Table 1. The small protrusion on the bottom end cap was seal welded by the electron beam after stabilization of the tube specimen at the design gas pressure in the weld chamber. The diameter of the sealed specimens was then measured by the laser profilometer. The diameter change of each tube was checked before/after pressurizing with the helium gas.





Table 1. The pressurizing conditions foreach PT tube

10	- Hoop		Fill Pressure	Actual Fill pressure	Temp.
ID	Engraved ID	Stress	(psig)	(psig)	°°
Tube1	B-10	0	0.0	0	21
Tube2	B-02	150	1280.6	1280	21
Tube3	B-03	300	2521.4	2525	22
Tube4	B-04	380	3325.7	3326	22

Results

These creep tubes were measured by a laser profilometer in a similar manner to that used with previous irradiated tubes. The measurement method is shown in Figure 2. In this case, the average diameter for a tube means the average value of tube diameters at positions from -3/10 to +3/10 from the center of the tube length. Moreover, a final average diameter was obtained from results of four measurements. Tables 2a)-b) show the average diameters of each tube before and after pressurization with helium gas and the gas pressure.



Figure 2. The measurement method of a creep tube using a laser profilometer.

Measure-	Hoop		0			
ment ID	Stress	1	2	3	4	Avg.
Tube1	0	0.18090	0.18090	0.18091	0.18091	0.18090
Tube2	150	0.18083	0.18085	0.18087	0.18087	0.18086
Tube3	300	0.18081	0.18080	0.18080	0.18081	0.18080
Tube4	380	0.18082	0.18082	0.18082	0.18082	0.18082

 Table 2a)
 Average diameter of each tube before pressurizing with helium gas

Table 2b) Average diameter of each tube after pressurizing with helium gas

	Measure-	Hoop					
Rabbit ID	ment ID	Stress	1	2	3	4	Avg.
FHC01	Tube1	0	0.18090	0.18090	0.18090	0.18089	0.18090
FHC02	Tube2	150	0.18090	0.18090	0.18089	0.18089	0.18089
FHC03	Tube3	300	0.18089	0.18089	0.18088	0.18088	0.18089
FHC04	Tube4	380	0.18094	0.18094	0.18094	0.18094	0.18094

Figures 3a)-d). show results of the diameter measurements for each tube after pressurizing with helium gas. Each red line shows the average value of central 3/5 zone for four measurements. This value will be compared with that of irradiated tubes.



Figure3. a)-d). Results of diameter measurement by a laser profilometer.

Figure 4 shows the difference of diameter of each tube before and after pressurizing with helium gas. It shows that as the helium gas pressure increases, the tube diameter also very slightly increases. The hoop stress level of tube4 at RT is about 200 MPa. The effective stress is estimated to be about 175 MPa. At this time, the elastic strain can be estimated 0.08 % using 217 GPa as Young's modulus of F82H. To compare, the measured diameter change of tube4 is 0.00011 inch, giving an effective strain obtained of about 0.08 %. From these results, it is considered that the diameter change of tube is as expected due to the pressurized helium gas.



Figure 4. Difference of diameter of each tube before and after pressurizing with He gas.
These specimens were assembled in the rabbit capsules after checking with a helium leak detector and then irradiated at 300°C up to 3.7dpa in HFIR [6]. Post irradiation evaluation will be performed soon.

Acknowledgement

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12.3 MINIATURE MECHANICAL TEST DEVELOPMENT FOR TUNGSTEN-BASED MATERIALS—L. M. Garrison, S. K. Wonner (Oak Ridge National Laboratory)

OBJECTIVE

The aim of this work is to develop miniature mechanical test methods that can be used to evaluate neutronirradiated tungsten and tungsten composite samples.

SUMMARY

A literature search of different miniature mechanical test methods is being conducted to evaluate tungstenbased samples with the transmission electron microscope (TEM) disk geometry, where the diameter is 3 mm and the thickness is 0.5 mm. Shear punch testing method has been investigated for these discs and detailed in this update. Another test of importance is 3-point bend testing, which is still being evaluated in literature.

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Background

Because of activation concerns after irradiation, miniaturized, non-standard tungsten sample geometries are often used. Additionally, many tungsten-based composites employ tungsten foils or tungsten fibers which cannot be tested in standard size mechanical test fixtures. A common sample geometry of irradiated small specimens is TEM disks, 3 mm in diameter and 0.5 mm thick. Due to its small size and unique geometry, a literature search is being conducted to evaluate viable mechanical test methods that could be used to analyze these samples. One test of interest is shear punch testing, which is summarized below.

Shear Punch Testing

Shear punch testing (SPT) is a technique specialized for when the amount of test material is limited. The following mechanical properties can be found from SPT data: yield strength, ultimate strength, and strain hardening exponent values. This test is comprised of a flat punch, which is brought down to punch through a thin sample, shearing the material at a constant speed through a die (Figure 1).



Figure 1: Schematic of the Shear Punch Apparatus.

The shear stress, τ , can be found using equation 1, where P is the applied load, r is the average of the die radius and punch radius, and t is the sample thickness [1].

$$\tau = \frac{P}{2\pi r t}$$
(1)

It was found in Reference [1] that the creation of a SPT master curve allows for the results to be independent of thickness, within a certain range, which is defined differently for every material. This master curve can be created by plotting shear stress versus the normalized displacement of the punch, which is the resultant punch displacement divided by the thickness of the sample. Utilizing a 1% offset on the master curve, the yield shear strength ($\sigma_{shear ys}$) and ultimate shear strength (σ_{USS}) are correlated with the tensile yield strength ($\sigma_{tensile ys}$) and ultimate strength (σ_{UTS}) using equations 2 and 3 [1]. Thus, this methodology allows estimation of the tensile and ultimate strength of the bulk material from small, 3 mm wide disks.

$$\sigma_{tensile ys} = 1.77 \sigma_{shear ys}$$
(2)
$$\sigma_{UTS} = 1.8 \sigma_{USS}$$
(3)

Three-point disc bend test

Inspiration for another method, the three-point disc bend test, is taken from literature and is shown in Figure 2 [2]. This method involves bending the 3 mm diameter disk with a known force and measuring the deflection. In addition, the specimen failure mode can be evaluated via Scanning Electron Microscopy [2]. This technique is useful to measure irradiation embrittlement; thus, current work is being done to design a fixture to use in these tests. It should be noted that there is a typo in Figure 2 from the original publication, where the span between the two bottom supports should not be 3 mm, but rather it should be 2 mm. Also not labeled on the diagram is the radius of curvature of the three supports, which is 0.25 mm [3]. Both these notes were clarified by a later paper written by the same group running this test in Reference [2].



Figure 2: Three-point bend test schematic. Note, there was a typo in the original publication and the distance between the two support pins should be 2 mm. The total width of the sample is 3 mm, and the radius of curvature for the supports is 0.25 mm [2].

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SUMMARY

Neutron irradiation experiments were performed in support of the research and development of fusion reactor materials using various materials irradiation facilities in the High Flux Isotope Reactor (HFIR).

The HFIR Cycle 483 began on November 13, 2018 but abnormal radiation readings resulted in shutdown the same day (6.52 MWD). The abnormal readings were caused by deformations in the reactor's curved fuel plates attributed to changes in the manufacturing process of the fuel plates. Corrective actions have been developed to address the problem and prevent recurrence. Efforts are underway to implement those corrective actions. Reactor restart is expected before the end of calendar year 2019.

Twenty-two target zone rabbit capsules remain in the reactor to complete the scheduled irradiations. These capsules are listed in Table 1 along with condensed information on material, specimen type, temperature, fluence, and period of irradiation.

Experiment Designation	Primary Materials	Specimen Types	Irradiation Temperature (°C)	Max Exposure (dpa)	Number of Reactor Cycles
F13A5	FeCrAlY Steel	Bend bar	300	28	16
F13B4	FeCrAlY Steel	tensile	300	50	29
JCR11-05	SiC/SiC	bend bars	950	200	115
JCR11-07	SiC/SiC	Mini bend bars	950	100	47
JCR11-08	SiC/SiC	Mini bend bars	950	200	115
SCF8	SiC/SiC	Bend bars	600	100	45
SCF9	SiC/SiC	Bend bars	600	200	90
SCF11	SiC/SiC	Bend bars	950	100	57
ES01	EUROFER reference alloy	Tensile/MPC**	220	20	12
ES02	EUROFER reference alloy	Tensile/MPC**	240	20	12
ES03	EUROFER reference alloy	Tensile/MPC**	275	20	12
ES04	EUROFER reference alloy	Tensile/MPC**	300	20	12
ES05	EUROFER reference alloy	Tensile/MPC**	325	20	12
ES06	EUROFER reference alloy	Tensile/MPC**	350	20	12
ES07	EUROFER reference alloy	Tensile/MPC**	375	20	12
ES11	EUROFER reference alloy	Bend Bar	220	20	12
ES12	EUROFER reference alloy	Bend Bar	240	20	12
ES13	EUROFER reference alloy	Bend Bar	275	20	12

Table 1. The HFIR fusion materials program rabbit capsules to continue irradiation with reactor startup

Experiment Designation	Primary Materials	Specimen Types	Irradiation Temperature (°C)	Max Exposure (dpa)	Number of Reactor Cycles
ES14	EUROFER reference alloy	Bend Bar	300	20	12
ES15	EUROFER reference alloy	Bend Bar	325	20	12
ES16	EUROFER reference alloy	Bend Bar	350	20	12
ES17	EUROFER reference alloy	Bend Bar	375	20	12

*completed irradiation this reporting period **MPC = Multi-Purpose Coupon