

**DOE-ER-0313/52
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UC-423, -424**

**FUSION MATERIALS
SEMIANNUAL PROGRESS REPORT
FOR THE PERIOD ENDING**

June 30, 2012

**Prepared for
DOE Office of Fusion Energy Sciences
(AT 60 20 10 0)**

DATE PUBLISHED: September 2012

**Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
Managed by
UT-Battelle, LLC
For the
U.S. DEPARTMENT OF ENERGY**

FOREWORD

This is the fifty-second 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, 2012. 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 Betty Waddell, Oak Ridge National Laboratory. Their efforts, and the efforts of the many persons who made technical contributions, are gratefully acknowledged.

Peter J. Pappano
Research Division
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1. FERRITIC/MARTENSITIC STEEL DEVELOPMENT

1.1 Stability of Strengthening Precipitates in Model Ferritic Steels — 1 L. Tan (Oak Ridge National Laboratory)

The effect of thermal aging, creep, and irradiation on the stability of strengthening precipitates including VN, TaN, and TaC are being systematically evaluated. Three model alloys, Fe-1WVN, Fe-1WTaN, and Fe-1WTaC, have been designed and fabricated. Nano-precipitates of VN, TaN, and TaC with sizes ~9 – ~19 nm and densities in the orders of 10^{20} – 10^{22} m⁻³ were developed in these alloys. These nano-precipitates will serve as good initial point for investigating their stabilities under different tests. Free dislocation densities were characterized in the order of 10^{13} – 10^{14} m⁻². Tensile tests of the control samples have been completed, which results will be used as benchmarks for the aging and irradiation experiments. Thermal aging of the alloys at 600°C and 700°C for 100 h has been completed. Creep testing of a Fe-1WTaC sample at 600°C and 170 MPa (~70% yield strength) has been completed. The microstructures of the tested samples are being characterized. Longer time thermal aging and creep tests of the other samples are in progress. Self-ion (Fe²⁺) irradiation of the samples has been planned at 500°C and 700°C for 20 dpa and 200 dpa. Tensile samples of the alloys have been prepared for neutron irradiation at 300°C, 500°C, and 650°C for up to ~20 dpa.

1.2 Dual Ion Beam Irradiation Studies of Cavity Evolution in a Tempered Martensitic Steel and Nanostructure Ferritic Alloys — 8 T. Yamamoto, Y. Wu, and G. R. Odette (University of California, Santa Barbara), K. Yabuuchi and A. Kimura (Kyoto University)

Dual ion (Fe³⁺ and He⁺) irradiations (DII) were performed at 500°C and 650°C, to nominal dpa and He levels of ≈ 10 to 47 dpa and ≈ 400 to 2000 appm, respectively. The irradiations were performed at dual ion beam facility, DuET, located at Kyoto University in Japan. The actual dpa, He and He/dpa vary with depth. The alloys studied include a normalized and tempered martensitic steel, (TMS) F82H mod.3 (at 500°C), and two nanostructured ferritic alloys (NFAs), MA957 (at 500 and 650°C) and 14YWT-PM2 (at 650°C). The dual ion results are compared to in situ He injection (ISHI) irradiations in HFIR at a much lower dpa rate. The cavity microstructures were characterized by TEM. Cavities in the DII were observed at depths up to ≈ 1600 nm. The TMS F82H irradiated at 500°C contains a moderate density of non-uniformly distributed cavities with sizes ranging from ≈ 1 nm up to ≈ 20 nm, including large faceted voids along with small bubbles. In contrast, the MA957 contains a uniform distribution of small ≈ 1.3 nm diameter bubbles. Notably a similar bubble distribution is observed up to ≈ 80 dpa and ≈ 3900 appm He at 650°C, demonstrating the outstanding He management capability of nm-scale features in MA957 and 14YWT. The net swelling in F82H at 500°C reaches ≈ 0.29% at 45 dpa, with a well-established post-incubation population of growing voids. Swelling is also observed in the ISHI irradiations, which manifest even more pronounced bimodal cavity size distributions compared to those for DII conditions. Notably, the incubation dose (He and dpa) for

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swelling appears to be shifted too much lower dpa in the ISHI case, and the swelling rate appears to be higher. While the absolute swelling and swelling rates are low in both cases, it is important to emphasize that it is likely that the cavity volume fraction will continue to increase, and even accelerate, at higher dpa, perhaps reaching the nominal 0.2%/dpa proposed by Garner. In contrast the bubbles in the NFA are very similar in all cases, even for the DII at 650°C.

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B. Yao, D. J. Edwards, and R. J. Kurtz (Pacific Northwest National Laboratory), G. R. Odette and T. Yamamoto (University of California, Santa Barbara)

Two ODS alloys and one RAFM steel (i.e., PM2000, 14YW, and modified F82H, respectively) with an adjacent 4.7 μm thick NiAl layer bond were neutron irradiated to a dose of 21.2 dpa at 500°C. An *in situ* $^{59}\text{Ni}(n, \alpha)$ reaction in the NiAl layer produces high-energy He ions, which are implanted into the alloys. This report summarizes initial TEM characterization results. The PM2000 contains a high density of small (< 2 nm) He bubbles in the matrix, while at the interface of Y-rich particles large voids are found. Both 14YW and modified F82H (F82H-mod) exhibit a large number of voids. The voids in the 14YW sample coexist with small Y_2O_3 particles typically less than 10 nm. Dislocation loops in all three samples were also quantified. A higher density of $\langle 100 \rangle \{100\}$ than that of $\frac{1}{2} \langle 111 \rangle \{111\}$ loops was observed. The loop size in PM2000 is bigger than those in the other samples.

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D. T. Hoelzer, K. A. Unocic, E. T. Manneschildt and M. A. Sokolov (Oak Ridge National Laboratory)

Plate samples of ODS 14YWT-SM12 were produced from material extruded at 850, 1000 or 1150°C followed by rolling parallel to the extrusion direction at 1000°C to 50% reduction in thickness. Results from tensile tests conducted at 25°C to 800°C showed slightly lower strengths, but significantly better ductility in all the 14YWT-SM12 heats compared to results from previous 14YWT heats. The microstructural characteristics observed by SEM and TEM analyses showed that slightly larger grains with less uniformity in grain size had formed in the SM12 heats compared to previous 14YWT heats. The observed differences in the mechanical properties and grain structures of the SM12 heats compared to previous 14YWT heats were most likely due to the lower O, C and N levels achieved in producing the 14YWT-SM12 heats.

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- The smallest 2-3 nm features in NFA are Y₂Ti₂O₇ complex oxide cubic pyrochlore phase. The interface between the bcc Fe-Cr ferrite matrix and the fcc Y₂Ti₂O₇ plays a critical role in the stability, strength and damage tolerance of NFA. To complement other characterization studies of the actual embedded nano-features, a mesoscopic interface was created by electron beam deposition of a thin Fe layer on a {111} Y₂Ti₂O₇ bulk single crystal surface. We recognize that the mesoscopic interfaces may differ from those of the embedded NFs, but the former will facilitate characterization and investigations of the functionality of controlled interfaces, such as interactions with point defects and helium. The Fe-Y₂Ti₂O₇ interface was characterized using scanning electron microscopy (SEM), including electron back scattering diffraction (EBSD), atomic force microscopy (AFM), X-ray diffraction (XRD), and transmission electron microscopy (TEM). The polycrystalline Fe layer has two general orientation relationships (OR) that are close to: a) (110)_{Fe}||((111)_{Y₂Ti₂O₇} and [001]_{Fe}||[1-10]_{Y₂Ti₂O₇} [notably, this is a Nishiyama-Wasserman (NY) OR]; b) (001)_{Fe}||((111)_{Y₂Ti₂O₇} and [100]_{Fe}||[1-10]_{Y₂Ti₂O₇}. High resolution TEM was used to characterize details of the interfaces, which ranged from being atomically sharp, with interface defects, to those with more diffuse interface zones that, in some cases, included a thin FeO_x layer.
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 C. Shih, Y. Katoh, K. Ozawa, and L. Snead (Oak Ridge National Laboratory)
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A. S. Sabau, E. Ohriner, Y. Katoh, and L. Snead (Oak Ridge National Laboratory)

The high-heat flux testing facility using Plasma Arc Lamps was demonstrated at ORNL for W samples. The test sections were designed by taking into account safety and materials compatibility requirements in order to handle the testing of low level irradiated tungsten articles. An enclosure was designed and fabricated. Test sections for the high-heat flux testing using PAL of low-level radioactive sample were designed and fabricated. The test sections were assembled and proof-of-principle testing was conducted, demonstrating the readiness of the new facility for irradiated samples.

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W. Setyawan and R. J. Kurtz (Pacific Northwest National Laboratory)

Density functional theory was employed to investigate grain boundary (GB) properties of W alloys. A range of substitutional solutes across the Periodic Table was investigated to understand the behavior of different electronic orbitals in changing the GB cleavage energy in the $\Sigma 27$ [110]{525} GB. A number of transition metals were predicted to enhance the GB cohesion. This includes Ru, Re, Os, Ir, V, Cr, Mn, Fe, Co, Ti, Hf, Ta and Nb. While lanthanides, s and p elements were tended to cause GB embrittlement.

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G. R. Odette, E. Stergar, D. Gragg, K. Fields, and J. Heathcote (University of California, Santa Barbara), C. H. Henager and R. J. Kurtz (Pacific Northwest National Laboratory)

There are many claims in the literature regarding various approaches to “ductilizing” W that are difficult to assess, in part because different tests have been used to “characterize” the various alloys. Thus one objective of this work is to demonstrate the application of accepted fracture mechanics test methods, so as to provide a common toughness-based metric for comparing different W alloys and composites. A major challenge to testing monolithic W is pre-cracking a very brittle material. A variety of approaches to pre-cracking an elemental W plate were attempted. The most successful method was compression fatigue applied in the axial direction of a small, initially notched bend bar, with the crack in the (T-L) orientation. The linear elastic toughness was measured in 3-point bend tests and averaged $K_{Ic} = 8.34 \pm 0.43 \text{ MPa}\sqrt{\text{m}}$.

A second testing challenge is associated with ductile phase toughened composites. In this case it is necessary to measure the toughness resistance curve associated with extensive stable crack growth. The resistance curve is expressed in terms of J_r/K_r -da curves. Ductile phase toughening (DPT) is largely due to the formation an intact bridging zone behind the tip of crack, which results in a increase in the remote load stress intensity needed for continued crack growth with increasing crack length, da. The main challenge in this case was measuring da as a function of the load (P) and load point displacement (d). We successfully characterized a K_r -da curve for a W-25%Cu heavy metal composite by

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combining load P-d curves with digital image correlation (DIC) measurements of crack growth on the specimen surfaces that could not be observed by normal optical methods. The W-Cu system also served as a model system for an initial exploration of DPT of W composites. The results showed that DPT increased the maximum load toughness of the composite to an average of 24.9 ± 3.5 MPa \sqrt{m} , or a maximum load capacity of ≈ 3 times higher than for monolithic W. The detectable initiation toughness of the composite, marking persistent crack growth, was also increased to ≈ 45 MPa \sqrt{m} compared to 8.34 MPa \sqrt{m} for the monolithic W. More importantly, extensive stable crack growth in the composite provided a major increment of effective post-peak load plastic ductility that is completely absent in linear elastic, monolithic W.

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 K. J. Leonard, T. Aytug, and L. L. Snead (Oak Ridge National Laboratory), A. Gapud (University of South Alabama), W. J. Weber (University of Tennessee)

A program is in place to examine the effects of room temperature ion irradiation on the superconducting properties of several YBCO HTS materials that utilize different flux pinning strategies. This will provide a first investigation into both the radiation-induced flux pinning and changes to pre-existing pinning centers. The work will then be expanded to low temperature irradiation testing that will include *in situ* measurement of self-field superconducting properties as a function of dose, with a further analysis into defect annihilation and effects of temperature excursions on conductor properties.

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 B. A. Pint and K. A. Unocic (Oak Ridge National Laboratory)

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L. Yang, F. Gao, H. L. Heinisch, and R. J. Kurtz (Pacific Northwest National Laboratory)

In a fusion reactor environment He is produced at high rates in steels by nuclear (n, α) transmutation reactions. Understanding the deleterious effects of He, especially the nucleation of He bubbles at GBs in steels, is one of the most important issues in nuclear fusion technology. The accumulation of He atoms and nucleation of He bubbles in the $\Sigma 3$ $\langle 110 \rangle \{112\}$ GB in α -Fe have been previously studied [1] using molecular dynamics with our newly developed Fe-He potential [2]. It was found that the accumulation of He atoms, the formation of He bubbles, and the evolution of the GB structure all depend on the local He concentration and temperature. In order to broaden understanding of the effects of GB structure on the accumulation of He atoms and the nucleation of He bubbles in α -Fe the interaction of He with the $\Sigma 73b \langle 110 \rangle \{661\}$ GB in α -Fe is currently being investigated using the same methodology as that used for our earlier studies of He in the $\Sigma 3$ GB. It is found that in the $\Sigma 73b$ GB at low He concentrations most He atoms migrate to the GB dislocations in a very short time, and they can move along the GB dislocation lines at high temperatures. He atoms seldom congregate to form clusters in the $\Sigma 73b$ GB, even at 800 K, compared to clustering in the $\Sigma 3$ GB. Emission of an Fe self-interstitial atom (SIA) caused by a single He is observed at 300 K, while it occurs for the clusters containing at least four He atoms at 600 K in the $\Sigma 3$ GB and higher temperatures. At a local He concentration of 5 % (as defined within 20 Å from the GB), a large number of He clusters are formed. The nucleation of He bubbles is more significant at higher temperatures in the $\Sigma 73b$ GB, while it depends only slightly on the temperature in the $\Sigma 3$ GB. Most of the He clusters are distributed along the GB dislocation lines in the $\Sigma 73b$ GB at low temperatures, forming platelet like configurations. At a 10 % local He concentration, a large number of SIAs are created, which results in the propagation of GB dislocations along the $\langle 112-1 \rangle$ direction.

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R. E. Stoller and Y. N. Osetskiy (Oak Ridge National Laboratory)

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J. Marian (Lawrence Livermore National Laboratory) and T. Hoang (University of California, Berkeley)

Due to its advantageous physical properties, tungsten (W) is being considered as a candidate structural material in fusion applications. In this paper, we perform stochastic cluster dynamics calculations of irradiation damage accumulation in pure W under fast neutron spectra up to doses of 1.5 dpa in the 400–600°C interval. Our calculations suggest that He bubbles and dislocation loops accumulate under fusion conditions, but

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not under fast fission spectra. We study the temperature dependence of swelling and find that it is maximum in the 550–590°C temperature range, falling precipitously above 600°C. Swelling levels are very low, never surpassing a fraction of a percentage point. We also provide hardening estimates based on the accumulation of sessile dislocation loops under fusion conditions and show that they are moderate, ranging between 70 and 137 MPa at 400°C.

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Y. Katoh, J. McDuffee (Oak Ridge National Laboratory)

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