2.1 DEVELOPMENT OF ODS FeCrAl FOR FUSION REACTOR APPLICATIONS – B. A. Pint, D. T. Hoelzer, D. Shin, J. O. Kiggans and K. A. Unocic (Oak Ridge National Laboratory, USA)

OBJECTIVE

The dual coolant lead-lithium (DCLL) blanket concept requires improved Pb-Li compatibility with ferritic steels in order to demonstrate viable blanket operation in a DEMO-type fusion reactor. The goal of this work is to develop an oxide dispersion strengthened (ODS) alloy with improved compatibility with Pb-Li and excellent mechanical properties. The current focus is determining the desired Cr and Al contents in the alloy and the minor additions to form a stable oxide dispersion for this application. The Cr and Al content effect are being investigated using model cast FeCrAl alloys and the oxide stability is being investigated using a combination of computation and diffusion couples.

SUMMARY

Capsule testing was completed on six cast FeCrAl alloys for 1,000h at 700°C. The alloys with 3%Al all showed mass losses similar to 9Cr steel under these conditions while those with 5%Al all showed little or no mass gain. In all cases, a $LiAlO_2$ reaction product formed but it was thinner for the 3%Al alloys and the surface was rougher. For the oxide stability, the thermodynamic model of HfO_2-Y_2O_3 was combined with the other two constituent pseudo-binaries of Al_2O_3-Y_2O_3 and Al_2O_3-HfO_2. Isothermal and isoplethal data from the experimentally assessed phase diagram of Al_2O_3-Y_2O_3-HfO_2 were used to adjust the ternary model parameters. Diffusion couple experiments at 1250° and 1650°C showed that Ti was very reactive with Al_2O_3 and Y_2O_3. Much less reaction occurred with HfO_2 and ZrO_2.

PROGRESS AND STATUS

Introduction

The DCLL blanket concept (Pb-Li and He coolants) is the leading U.S. design for a test blanket module (TBM) for ITER and for a DEMO-type fusion reactor.[1] With reduced activation ferritic-martensitic (FM) steel as the structural material, the DCLL is limited to ~475°C metal temperature because Fe and Cr readily dissolve in Pb-Li above 500°C and Eurofer 97 plugged a Pb-Li loop at 550°C.[2-3] For a higher temperature blanket for DEMO, structural materials with enhanced creep and compatibility are needed. ODS FeCrAl alloys are one possibility to meet this objective and considerable research on ODS FeCr alloys has shown an excellent combination of creep strength and radiation resistance.[4-7] Isothermal compatibility tests have shown low mass losses at up to 800°C.[8] Therefore, a materials development effort is underway, specific to this application. The first steps are to identify the Cr and Al contents needed for good Pb-Li compatibility and to identify stable oxide dispersions for a FeCrAl matrix.

Experimental Procedure

Static capsule tests were performed using Mo (inert to Pb-Li) inner capsules and type 304 stainless steel (SS) outer capsules to protect the inner capsule from oxidation. The uncoated cast FeCrAlY specimens were ~1.5 mm thick and 4-5 cm^2 in surface area with a 600 grit surface finish and were held with 1 mm diameter Mo wire. The capsules were loaded with 125g of Pb-Li in an Ar-filled glove box. The Pb-Li was melted and cast at ORNL and had Li contents of 15.6-16.5 at%. The Mo and SS capsules were welded shut to prevent the uptake of impurities during the isothermal exposure. After exposure, residual Pb-Li on the specimen surface was removed by soaking in a 1:1:1 mixture of acetic acid, hydrogen peroxide and ethanol for up to 72 h. Mass change was measured with a Mettler-Toledo balance with an accuracy of 0.01 mg/cm^2. Post-test specimen surfaces were examined using x-ray diffraction (XRD), a Veeco Wyko optical profilometer and secondary electron microscopy (SEM) equipped with energy dispersive x-ray (EDX) analysis. After surface characterization, the specimens were metallographically sectioned and

polished and examined by light microscopy.

A plate of high purity Al_2O_3 (99.8% purity) was obtained for preparation of samples to investigate the high temperature oxide phase reactions and stabilities. Slurries of TiO_2, ZrO_2, HfO_2 and Y_2O_3 were prepared for the diffusion couples. Samples were prepared by placing a row of the TiO_2, ZrO_2 and HfO_2 slurries on the Al_2O_3 plate and then dried. An additional row of Y_2O_3 slurry was placed next to the dried oxide slurries on each Al_2O_3 sample to produce samples of the three oxide ternary systems, Al_2O_3-Y_2O_3-HfO_2, Al_2O_3-Y_2O_3-ZrO_2, and Al_2O_3-Y_2O_3-TiO_2. Individual sections of each sample were annealed for 1h at 1250°C and 1650°C. After annealing, the microstructure was assessed using SEM and EDX analysis.

Results and Discussion

Figure 1a shows the mass change for the six cast FeCrAlY alloys after exposure to Pb-Li for 1,000h at 700°C. There was a distinct difference between the 3%Al and 5%Al alloys with minimal effect of the alloy Cr content. The results are shown in Figure 1b as a function of the alloy Cr and Al contents and the next series of experiments are shown to fill in the gap. Lower Cr contents are desired to avoid issues with embrittlement,[9,10] especially under irradiation. This is driving the focus for the next series of experiments. Figure 2 shows cross-sections from the exposed specimens. In each case, XRD indicated that the surface oxide was $LiAlO_2$, consistent with earlier results.[8,11] The oxide appeared to be slightly thinner on the 3%Al alloys. Based on the undulations in the surfaces, the roughness was measured on the specimens and was somewhat rougher on the 3%Al alloys compared to the 5%Al alloys. Concentration profiles in the substrate beneath the surface oxide will be performed in the future.

Compared to the highly stable Y-Ti-O nano-oxide dispersion in Fe-(9-14)Cr,[6] the Al addition presents serious challenges to the choice of oxide dispersion since Al_2O_3 is thermodynamically more stable than TiO_2 and readily reacts with Y_2O_3 to form a number of stable compounds. Ideally, another addition would react with Y_2O_3 to form a nano-dispersion in FeCrAl. Thus, understanding the high-temperature stability of the oxide dispersion in the presence of Al is important for developing ODS FeCrAl. A self-consistent model for Al_2O_3-Y_2O_3-ZrO_2 was obtained from the literature,[12] so the computational work focused on the

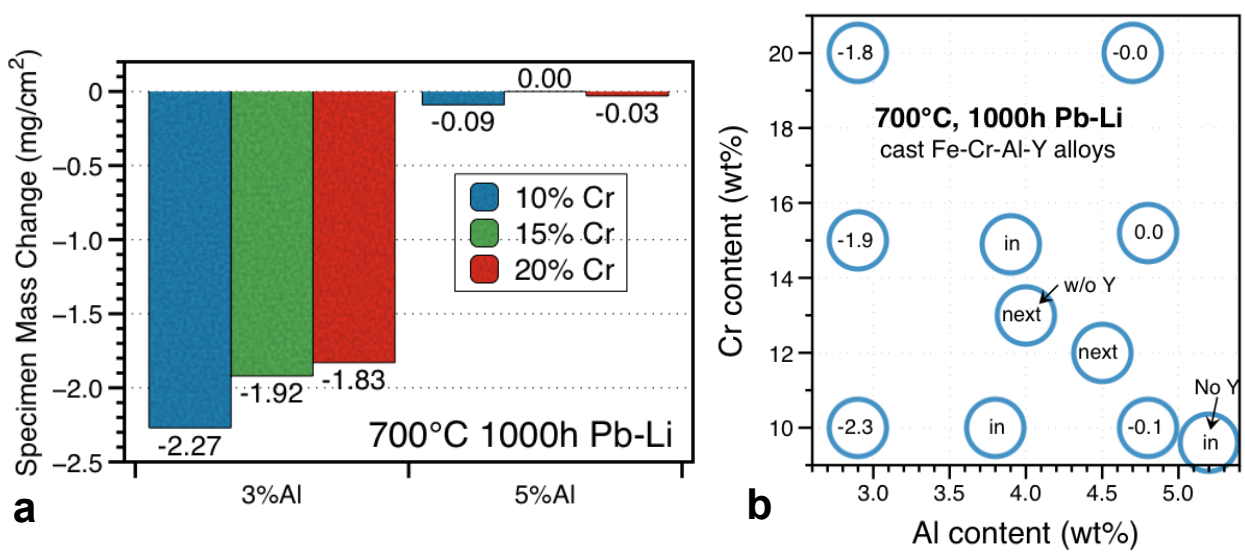

Figure 1. Specimen mass change is shown (a) for the cast FeCrAlY alloys after exposure to Pb-Li for 1,000h at 700°C and (b) as a function of the Cr and Al contents with the next experiments noted.

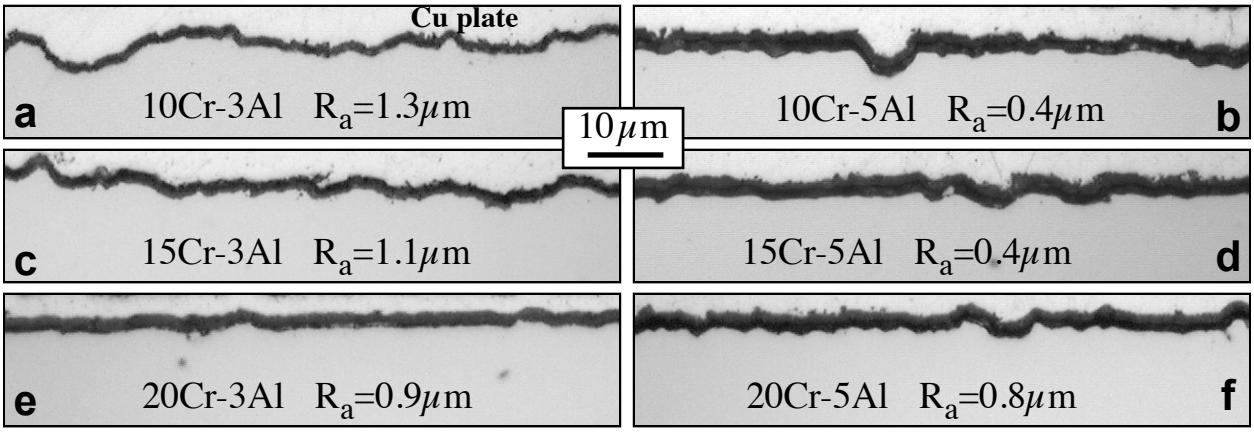

Figure 2. Polished cross-sections of cast FeCrAl alloys after 1000h at 700°C in Pb-Li.

Al_2O_3-Y_2O_3-HfO_2 system where there was disagreement between the experimental and predicted phase diagrams.[13,14] A model for a constituent pseudo-binary HfO_2-Y_2O_3 in Al_2O_3-Y_2O_3-HfO_2 was updated to compare with reported ternary isothermal phase equilibrium data and phase fractions at 1650°C from the Al_2O_3-Y_2O_3 equi-concentration to pure ZrO_2 and HfO_2 were obtained. Figure 3 shows ternary and psuedo-binary diagrams at 1650°C for the Al_2O_3-Y_2O_3-HfO_2 system. The binary diagram is from the dashed line in Figure 3a.

For validation, diffusion couples were annealed at 1250° and 1650°C. Figure 4 shows the couples annealed for 1h at 1650°C. At both temperatures, Al_2O_3 and Y_2O_3 reacted to form a 50-60μm layer. By far, TiO_2 was the most reactive with Al_2O_3, diffusing millimeters into the substrate, Figure 4. Both ZrO_2 and HfO_2 were more stable, with limited solubility in Al_2O_3 and <1μm thick reaction layer at 1650°C.

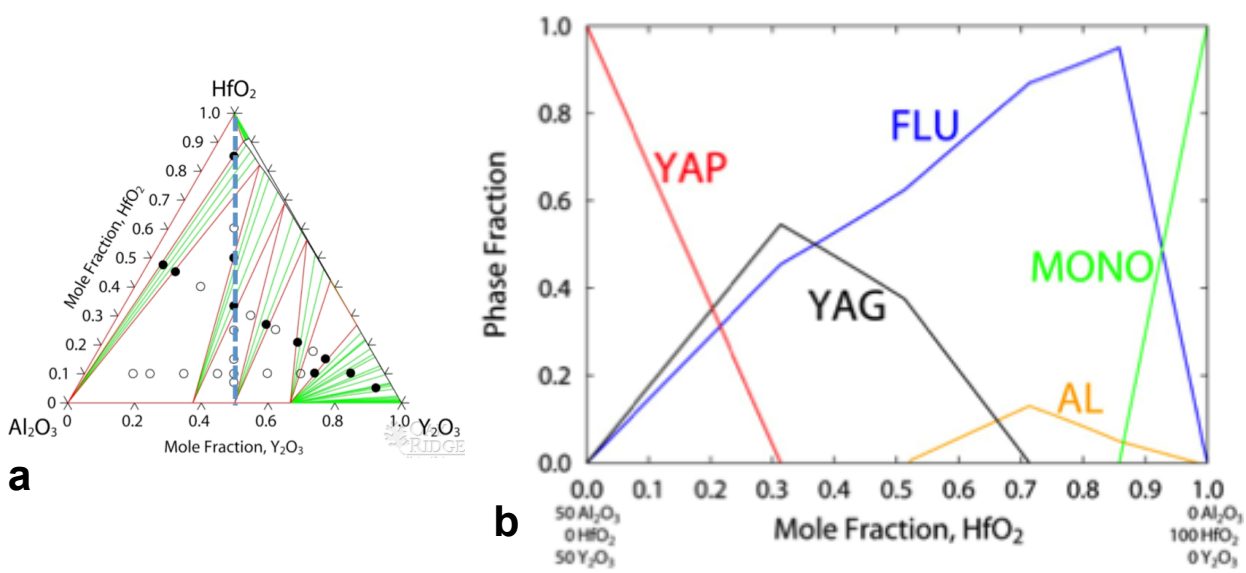

Figure 3. (a) Ternary and (b) pseudo-binary diagrams at 1650°C calculated for the Al_2O_3-Y_2O_3-HfO_2 showing YAP ($YAlO_3$), YAG ($Y_3Al_5O_{12}$), FLU (flourite), AL (Al_2O_3) and MONO (monoclinic) phases.

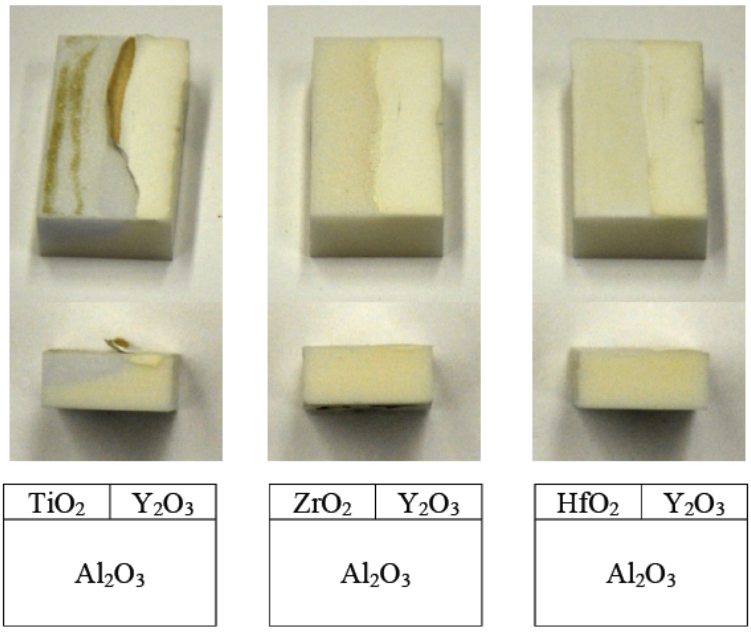

Figure 4. Three diffusion couple annealed for 1h at 1650°C.

Figure 5 shows EDS analysis of the Al_2O_3-Y_2O_3-HfO_2 and Al_2O_3-Y_2O_3-ZrO_2 couples annealed at 1650°C. In the reaction zone in each case, there appears to be less Hf present in the Y_2O_3 phase while Zr appeared to form a complex $(Al,Y,Zr)O_x$ phase, Figure 5b. However, both Hf and Zr are more soluble in

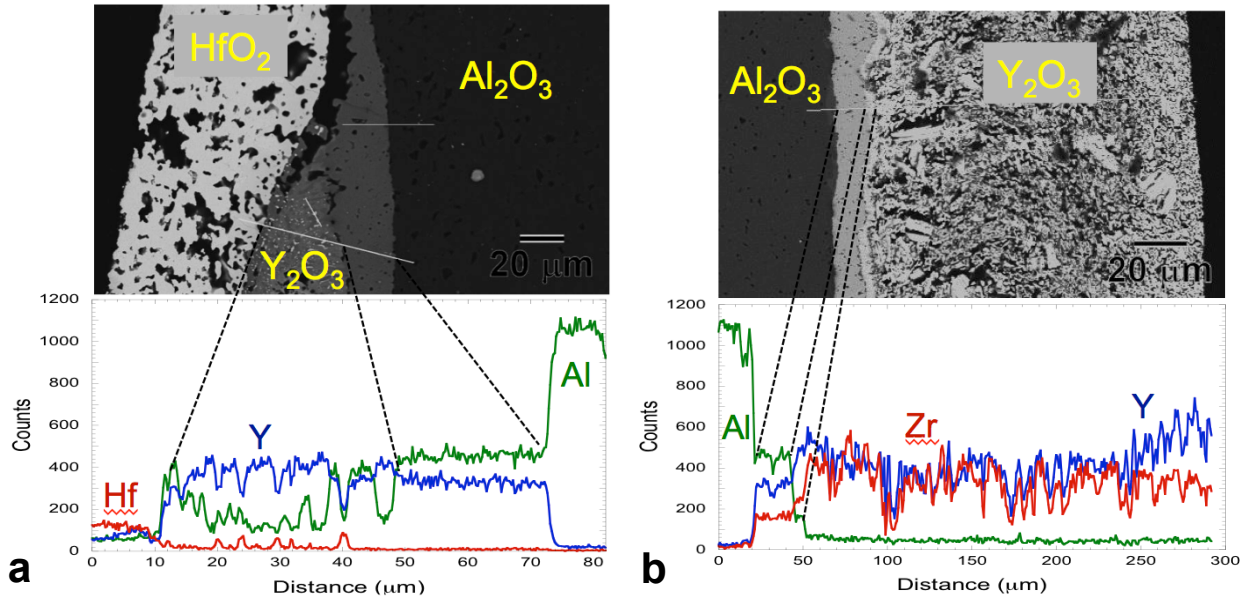

Figure 4. SEM images and EDS profiles for diffusion couples after 1h at 1650°C (a) Al_2O_3-Y_2O_3-HfO_2 and (b) Al_2O_3-Y_2O_3-ZrO_2.

FeCrAl than Y [15] and therefore may coarsen more readily during processing or high temperature exposure.

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