2.2 DEVELOPMENT OF ODS FeCrAl FOR FUSION REACTOR APPLICATIONS – B. A. Pint, D. T. Hoelzer 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 by evaluating the Pb-Li compatibility of model cast FeCrAl alloys.

SUMMARY

Several additional isothermal Pb-Li capsule tests of cast FeCrAlY specimens were completed at 700°C in order to further guide alloy selection. Characterization of specimens after exposure indicated that the Cr and Al contents remain relatively constant near the exposed surface and that $LiAlO_2$ formed on the surface of all of the exposed alloys. In general, the results suggest that the Al content needs to be near 5 wt.% for excellent Pb-Li compatibility. Based on this information, several powder compositions were procured and have now been received and analyzed. These powders are now being milled to produce the first batches of ODS FeCrAl for evaluation.

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. Previously, initial results were provided on the Pb-Li compatibility of six FeCrAl alloys with 10-20%Cr and 3-5%Al and the stability of various potential oxide dispersions in a FeCrAl matrix [9].

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 15.4-15.6 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) 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 and electron probe microanalysis (EPMA) using wavelength dispersive x-ray analysis.

Results and Discussion

Figure 1 shows the mass change data for the first 6 FeCrAlY experiments (3 and 5%Al) and 5 of the 6 new experiments as a function of the actual Cr and Al contents of the cast alloys. In general, the goal is to develop an ODS FeCrAl with the lowest necessary Al and Cr levels as lower Al contents make the alloy easier to process and weld and lower Cr contents are desired to avoid issues with embrittlement,[10,11] especially under irradiation. The new results for alloys with 4-5%Al are mostly consistent with the previous results. However, the alloys with 3 and 5%Al showed little effect of Cr on the mass change, the 4%Al alloys showed less mass loss with higher Cr contents between 10 and 15%. For the Fe-12Cr-4.5Al+Y specimen, the 0.3 mg/cm^2 mass loss was almost as low as the alloys with 5%Al. One of the capsules leaked with a specimen with ~13Cr-4Al and no Y (marked "in progress" in Figure 1). A more extended procedure is being used to remove this specimen from the capsules and remove residual Pb-Li. The mass change for this alloy will be provided in the next report. This specimen was exposed to identify the effect of Y on compatibility. Historically, Y and other rare earth elements or oxides are added to alumina-forming high temperature alloys like FeCrAl to improve the adhesion of the thermally grown alumina scale, especially at ≥1000°C [12,13]. For this application in Pb-Li at ≤700°C, it is not certain if Y provides any benefit. An additional specimen with ~10Cr-5Al but no Y was also exposed to study the effect of Y. Compared to the specimen with Y and similar Cr and Al contents, the mass change was very similar but slightly less without Y. Since Y has very low solubility in FeCrAl, one benefit of Y may be in stabilizing a finer grain size in these cast alloys by the formation of FeY_x precipitates. Grain size of ODS alloys is determined by the processing and heat treatment and will be less of an issue.

In order to better understand the mass change data in Figure 1, the reaction products were analyzed and the composition profile of sectioned specimens were determined. Consistent with prior observations [8,14], the reaction products on the most recently exposed specimens were identified as $LiAlO_2$ by XRD.

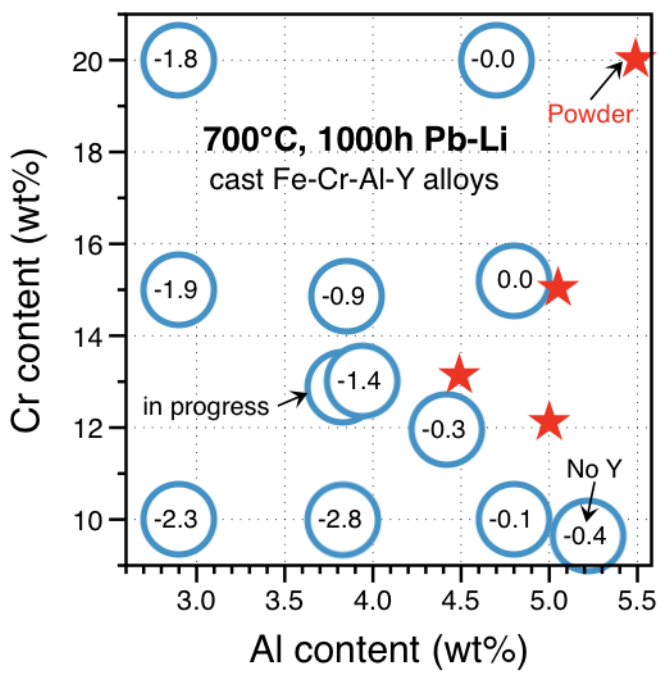

Figure 1. Specimen mass change of case FeCrAlY alloy coupons after exposure to Pb-Li for 1,000h at 700°C as a function of the Cr and Al contents. The stars mark the composition of four powder batches.

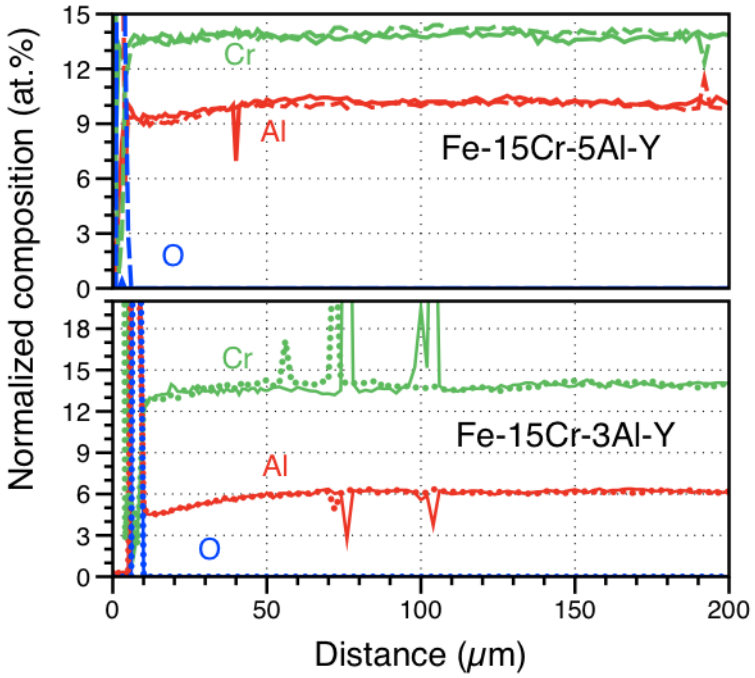

Figure 2. EPMA line profiles from polished sections of two FeCrAl specimens after exposure in Pb-Li for 1,000h at 700°C. In both cases, two line profiles were taken in different locations.

Figure 2 shows normalized composition profiles from two specimens analyzed by EPMA. Two profiles are shown for each specimen one with a dashed line and the other solid. In at.%, the 5 and 3 wt.% Al alloys contain ~10 and 6 at.%Al. The O profiles are shown to mark the substrate surface. Some Al depletion was apparent to a depth of ~40μm from the surface. For the alloy with 6at%Al in the matrix, the surface content has dropped to ~4.6%Al, while the other alloy had dropped to ~9%. Some amount of Cr depletion also was observed but not as significant. The disruptions in the Cr and Al profiles are where the line scan intersected a Y-rich precipitate, which is depleted in Al. The Cr and Al profiles do not fit the inductively coupled plasma (ICP) chemical analysis of these two alloys. That difference is currently being investigated.

Based on the results in Figure 1, several FeCrAl powder compositions were selected to fabricate ODS alloys. The compositions of the as-received powders are shown as stars in Figure 1. These powders will be ball milled with Y_2O_3 and then extruded to consolidate. The resulting ODS FeCrAl will then be evaluated in mechanical and compatibility experiments.

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