COMPATIBILITY OF MATERIALS EXPOSED TO ISOTHERMAL Pb-Li – B. A. Pint and K. A. Unocic (Oak Ridge National Laboratory, USA)

OBJECTIVE

One proposed U.S. test blanket module (TBM) for ITER uses ferritic-martensitic alloys with both eutectic Pb-Li and He coolants at ~475°C. In order for this blanket concept to operate at higher temperatures (~700°C) for a DEMO-type reactor, several Pb-Li compatibility issues need to be addressed. Some of the issues currently being investigated are the behavior of dispersion strengthened Fe-Cr alloys compared to conventional wrought material, the performance of Al-rich coatings to inhibit corrosion and dissimilar material interaction between SiC and ferritic steel.

SUMMARY

Initial isothermal capsule experiments were conducted to compare the behavior of oxide dispersion strengthened (ODS) Fe-Cr alloys to prior results on wrought ferritic-martensitic (FM) alloys. Also, the performance of corrosion resistant, Al-rich diffusion coatings on these alloys was investigated. To further understand the performance of these coatings in Pb-Li, several experiments are in progress including a time series of experiments and a more detailed study of the unexpectedly high Al loss observed in prior experiments. New Pb-Li was cast to eliminate the prior issue of Li composition variability. Finally, to investigate any potential dissimilar material interaction between Fe and SiC, a set of capsules with SiC inner capsules is being assembled for exposures at 500°, 600° and 700°C.

PROGRESS AND STATUS

Introduction

A current focus of the U.S. fusion materials program is to address issues associated with the dual coolant Pb-Li (DCLL) blanket concept[1] for a test blanket module (TBM) for ITER and enhanced concepts for a DEMO-type fusion reactor. A DCLL blanket has both He and eutectic Pb-17 at.%Li coolants and uses reduced activation ferritic-martensitic (FM) steel as the structural material with a SiC/SiC composite flow channel insert (FCI). Thus, recent U.S. compatibility research has examined compatibility issues with Pb-Li.[2-7] Compared to Li,[8] a wider range of materials can be compatible with Pb-Li because of the low activity of Li.[9] In particular, SiC readily dissolves in Li, but not Pb-17Li.[2,4,10] However, like Pb, Pb-Li dissolves Fe, Cr and especially Ni from many conventional alloys above 500°C.[11,12] This is not a concern for a DCLL TBM operating at <500°C. However, a DCLL blanket for a commercial reactor would be more attractive with a higher maximum operating temperature, perhaps >600°C if ODS ferritic steels[13] were used. Even at 550°C, a recent study of Eurofer 97 (Fe-Cr-W) showed a very high dissolution rate in flowing Pb-Li.[12] Therefore, preliminary Pb-Li compatibility capsule experiments are being conducted at 500°-700°C in order to investigate several concepts before flowing Pb-Li compatibility tests are conducted. Recent capsule experiments have investigated (1) the effectiveness of Al-rich coatings to inhibit dissolution, (2) the effect of Fe and Ni impurities on the amount of dissolution and (3) potential dissimilar material effects between Fe and SiC. Additional capsule experiments to address the first topic are presented here and a second series of capsule experiments are being assembled to address the third topic now that chemical vapor deposited (CVD) SiC capsules have been machined.

Experimental Procedure

Static capsule tests were performed using Mo capsules and type 304 stainless steel (SS) outer capsules to protect the inner capsule from oxidation. The dissimilar materials capsules being assembled have an inner chemical vapor deposited (CVD) SiC capsule. Since this capsule cannot be sealed, it is placed

inside a Mo capsule and held shut by a Mo wire welded into the Mo capsule lid that is welded shut. A similar SS capsule was used to protect the Mo capsule in these experiments. For the Mo capsules, the 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. (Mo can be considered to be essentially inert under these conditions.) For the SiC capsules, a CVD SiC spacer is used to hold a specimen that is ~1.5 mm thick and 2-3 cm^2 in surface area. The representative FM steel used in prior work was P92 (Fe-9Cr-2W) and two ODS alloys were evaluated, one from ORNL (14YWT) and the other (ODM401) from Dour Metal sro. All of the alloy compositions are shown in Table 1. Prior to aluminizing, specimens were polished to a 0.3 μm alumina finish and cleaned ultrasonically in acetone and alcohol. Aluminizing was performed in a laboratory-scale CVD reactor consisting of an inductively heated alumina tube with flowing H_2 carrying the $AlCl_x$ vapor. The reactor has been described in detail elsewhere.[14] For the dissimilar metal experiments, unalloyed Fe specimens were used to simplify the experiment and the CVD SiC specimen was high-purity material from Rohm & Haas. For the Mo capsule results reported here, the capsules were loaded with 125g of Pb-Li in an Ar-filled glove box. To avoid recent issues with variable Li composition in the current commercial batch of Pb-Li,[7] high purity Pb and Li were placed in the capsule. For the SiC capsules, 20g of Pb-Li were loaded into the much smaller capsules. This material was cast at ORNL and chemical analysis showed a Li content of 16.5at.%Li in one stick and 15.9% in a second stick that is being used for the next series of coated specimens in Mo capsules. The Mo and SS capsules were welded shut in a glove box 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. Characterization included x-ray diffraction (XRD), x-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM) equipped with energy dispersive x-ray (EDX) analysis and electron microprobe analysis (EPMA) equipped with wavelength dispersive x-ray analysis.

Results and Discussion

Figure 1 shows the mass change data for both ODS alloys and aluminized alloy 401. A prior report outlined that it was not possible to aluminize the 14YWT alloy,[15] likely due to the high N content and small grain size in this batch (the prior report contained composition from a similar alloy batch whereas Table 1 contains data for the same batch as was used in these experiments). Compared to P92, the higher Cr content ODS alloys showed a similar mass loss after 1,000h at 700°C in Pb-Li and a similar improvement when aluminized. The similar mass changes are not particularly surprising since Fe and Cr are thought to dissolve in Pb-Li at a similar rate with no enrichment observed at the dissolution front of P92.

One issue that has arisen with these coatings is the unexpectedly high Al loss observed after 1,000h at 700°C.[5] The as-coated Al composition profiles reported previously has always been from representative

Table 1. Alloy chemical compositions (atomic% or ppma) determined by inductively coupled plasma analysis and combustion analysis.

Material	Fe	Ni	Cr	Al	O	C	N	S	Other
14YWT	83.3	0.03	14.1	0.04	6000	3000	**5170**	69	0.59W,0.12Y 0.28Ti,0.08Si
401	82.6	0.12	14.8	0.12	6560	610	1770	89	0.17Mo,0.13Y 1.2Ti,0.08Si
P92 (9Cr-2W)	87.2	0.1	9.9	0.02	80	5120	**2330**	87	0.55W, 0.46Mn 0.30Mo,0.32Si

< indicates below the detectability limit of <0.01% or <0.001% for interstitials

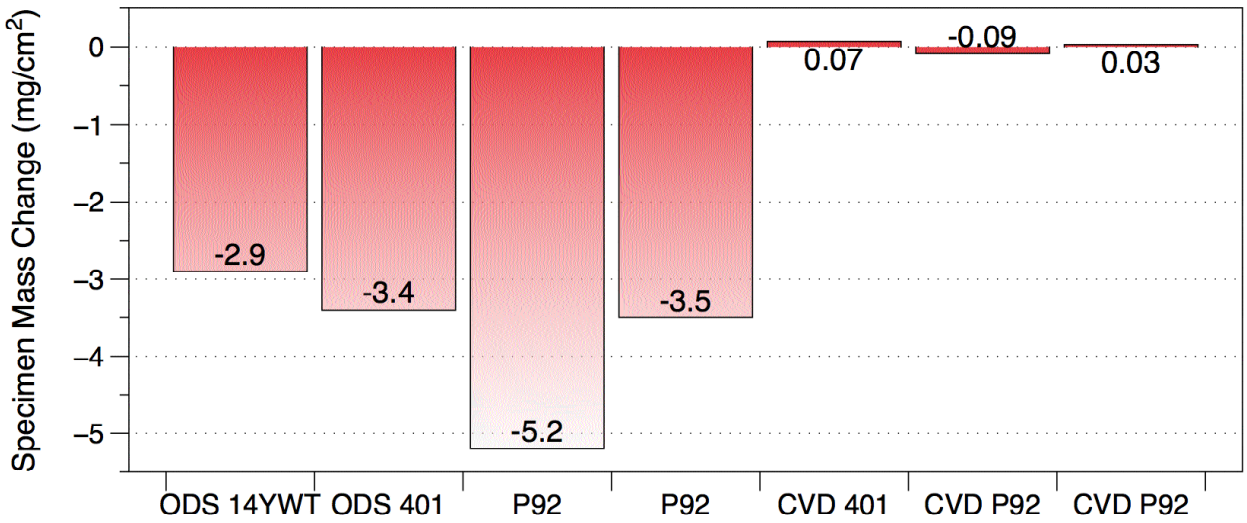

Figure 1. Mass change results after exposure for 1,000h at 700°C in Pb-Li for both wrought and ODS FeCr alloys and with and without CVD aluminide coatings.

coated specimens as coating composition could not be measured before and after exposure without cutting the specimen. In order to provide more quantitative information about the Al loss, a coated P92 specimen has been cut into three pieces. The first piece was mounted and analyzed by EPMA, Figure 2. The other pieces were pre-oxidized for 2h at 800°C in dry O_2, similar to the prior study. One piece was

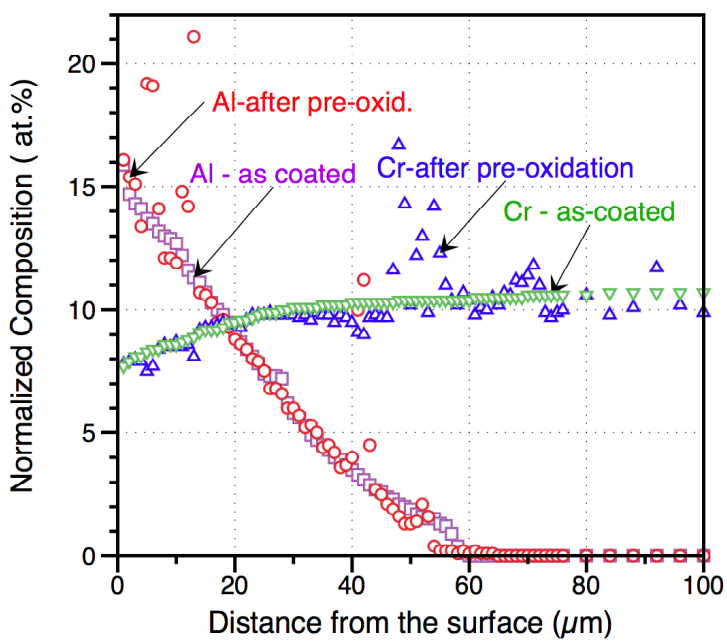

Figure 2. Specimen mass change from a series of capsule experiments with carbon steel capsules and Fe and SiC specimens exposed to Pb-Li for 1,000 h at each temperature.

mounted for analysis and the last piece is being exposed to Pb-Li for 1,000h at 700°C so that EPMA profiles can be obtained all from the same coated P92 specimen. Figure 2 shows clearly that very little Al was lost during pre-oxidation due to the formation of an Al-rich oxide on the surface or interdiffusion with the substrate. After the last piece is exposed to Pb-Li, a similar Al profile will be measured from that specimen to quantify the Al loss.

In addition to that coating experiment, three P92 specimens have been coated and will be exposed to Pb-Li for 500, 2,000 and 5,000h to measure the kinetics of Al loss and determine the effectiveness of these coatings at longer times Those experiments all should begin in the next period and be completed in 2011.

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