6.4 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

A series of Pb-Li capsule experiments were conducted to study the dissimilar material interaction between Fe and SiC using both materials as specimens and capsules. Because of uncertainties associated with the mass change data, metallographic cross-sections were completed to study the reactions observed at 500°-700°C after 1,000 h in Pb-Li.

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 issues with Pb-Li.[2-8] Compared to Li,[9] a wider range of materials can be compatible with Pb-Li because of the low activity of Li.[10] In particular, SiC readily dissolves in Li, but not Pb-17Li.[2,4,11] However, like Pb, Pb-Li dissolves Fe, Cr and especially Ni from many conventional alloys above 500°C.[12,13] 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[14] 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.[13] 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. One concern is the potential dissimilar material effects between Fe and SiC because Fe forms more thermodynamically stable carbides than Si.[15] A series of capsule experiments have been completed [7,8] and characterization of all of the specimens is in progress. A large mass loss for Fe in a steel capsule could not be repeated in a second experiment and the large mass loss is not consistent with a smaller loss observed in a Mo capsule.

Experimental Procedure

Static capsule tests were performed using Fe (mild steel) or SiC inner capsules and type 304 stainless steel (SS) outer capsules to protect the inner capsule from oxidation. Since the SiC 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. For the Fe 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 chemical vapor deposition (CVD) SiC spacer is used to hold a specimen that is ~1.5 mm thick and 2-3 cm^2 in surface area. Unalloyed Fe

specimens were used (rather than FM steel) to simplify the experiment and the CVD SiC specimen was high-purity material from Rohm & Haas. For the Fe capsules, the capsules were loaded with 125g of Pb-Li in an Ar-filled glove box. For the smaller CVD SiC capsules, only 20g of Pb-Li was used. The Pb-Li for the Fe capsules was commercially manufactured. The Pb-Li for the SiC capsules was melted and cast at ORNL and had Li values of 15.6-16.5 at%. The, Fe, 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. The specimens were metallographically mounted in epoxy to study the surface reaction product, which is in progress.

Results and Discussion

Figure 1 shows the mass change for the unalloyed Fe and SiC specimens in steel and SiC capsules at 500°-700°C.[7,8] The first Fe specimen exposed at 700°C in a steel capsule had a mass loss of -16.4 mg/cm^2. Since this was 3-4X higher than losses measured for 9Cr steel, this experiment was repeated, and a small mass gain was measured for the second specimen, Figure 1. An Fe specimen in a Mo capsule at 700°C had a mass loss of -4.5mg/cm^2 which is consistent with mass losses for 9Cr steels. The SiC specimens in SiC capsules were damaged during loading and unloading, thus chipping dominated the mass changes. The scatter in the results suggests that characterization rather than just mass change is needed.

Figure 2 shows polished cross-sections of the specimens in Figure 1 (except 2i is the Fe specimen with the large mass loss.) While C transport cannot be ruled out by these results, it appears that O may be a more important factor. For the SiC specimen in the steel capsule, the SiC specimen may getter all of the O in the Pb-Li resulting in a thick oxide (Figure 2k) and a mass gain. With a SiC capsule, O may react with the capsule wall as well as the specimen, resulting in a thinner oxide layer (Figure 2l). A similar

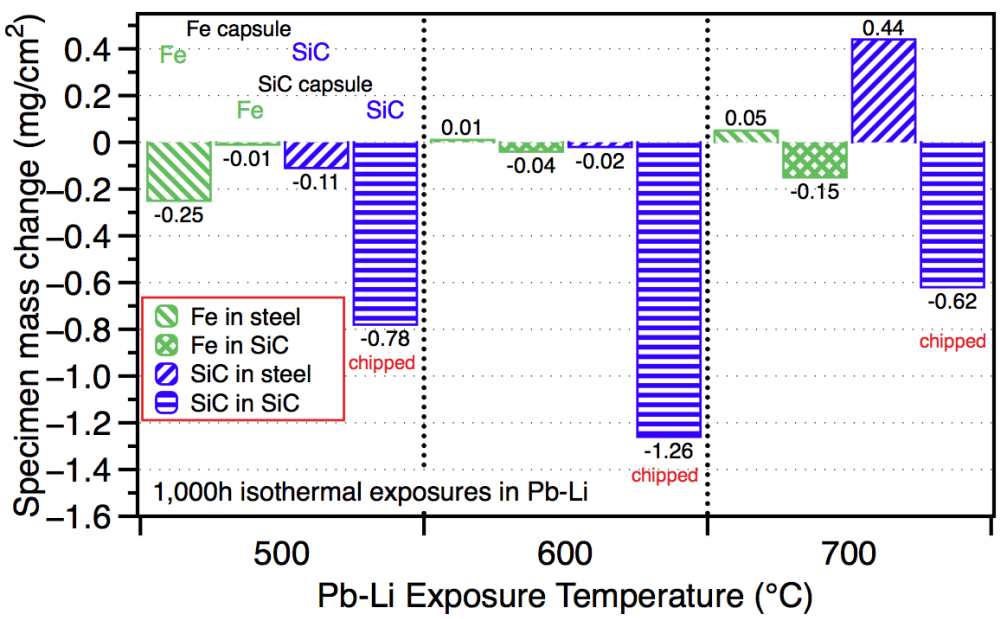

Figure 1. Specimen mass change from a series of capsule experiments with carbon steel or CVD SiC capsules and Fe and CVD SiC specimens exposed to Pb-Li for 1,000 h at each temperature.

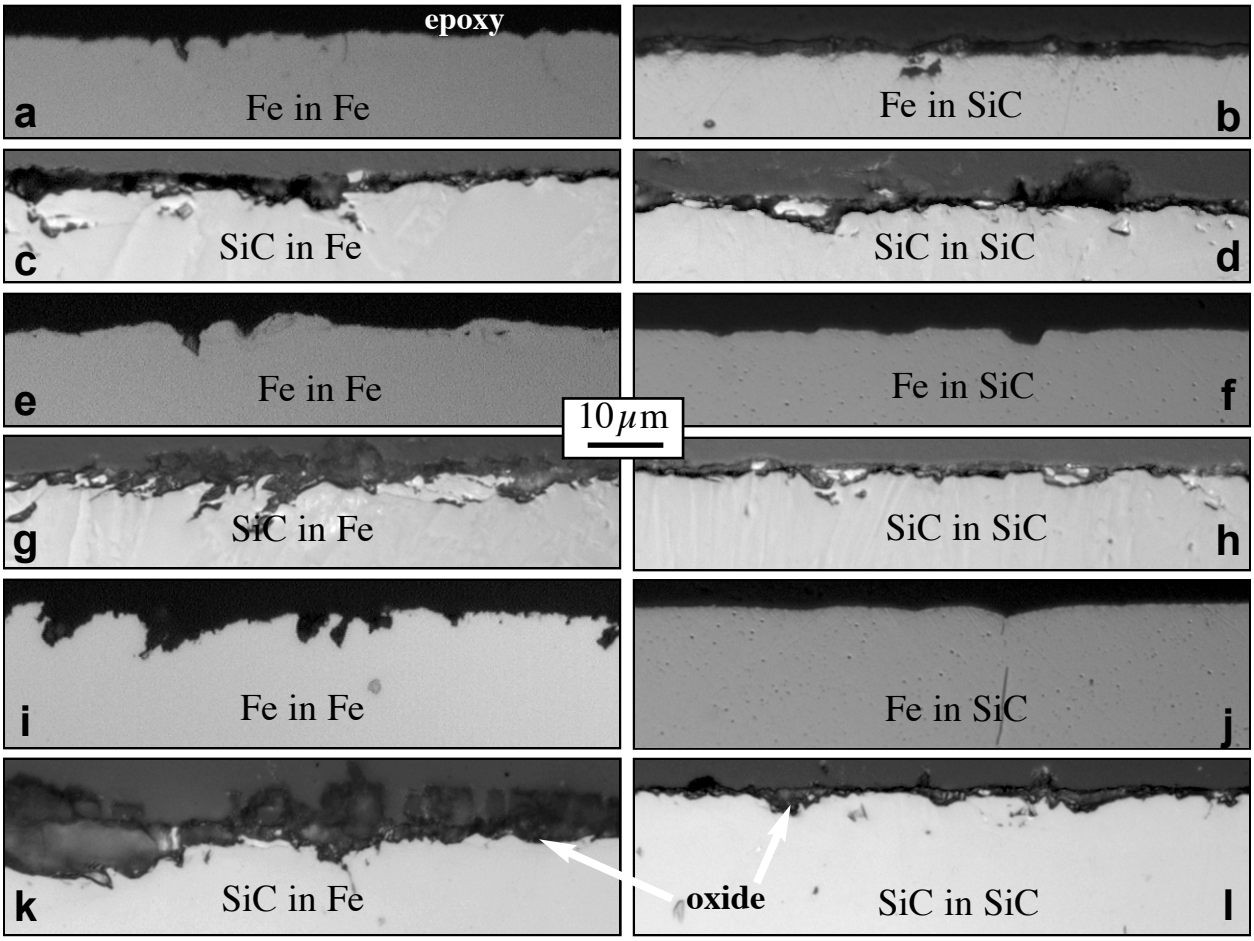

Figure 2. Polished cross-sections of Fe (a,b,e,f,i,j) and SiC (c,d,g,h,k,l) specimens after 1000h exposures in Pb-Li at (a-d) 500°C, (e-h) 600°C and (i-l) 700°C exposed in steel (a,c,e,g,i,k) or SiC capsules (b,d,f,h,j,l). Oxides were noted on the SiC specimens but not on the Fe specimens.

difference was observed at 600*C (Figures 2g and 2h). At 500°C, it was difficult to observe a difference. For the Fe specimens, a progressive roughening of the specimens surface was observed with increasing temperature in the steel capsules (Figures 2a, 2e and 2i). In these experiments, the Pb-Li could become saturated with Fe as both the specimen and walls dissolved. This could reduce the mass loss of the specimen. For the Fe specimen in a SiC capsule, all of the Fe dissolution would be expected from the specimen. However, relatively little dissolution was observed, even at 700°C, Figure 1.

One difference between the two types of capsule experiments is the specimen surface area to Pb-Li volume ratio. In the SiC capsule the ratio is 1.5 (2.7 cm^2 vs. 1.8 cm^3) while in the Fe capsule it is 0.5 for just the specimen (5 cm^2 vs. 11 cm^3). However, if the capsule wall also is considered (as the Fe capsule also may dissolve) the ratio is 2.5 because of the large surface area of the capsule in contact with ~11cm^3 of Pb-Li. While this difference needs to be considered in the results, it does not appear sufficient to explain the range of results observed. Further characterization is needed of these specimens including more characterization of the oxide on the SiC and hardness measurements of the Fe specimens as a possible indicator of C pickup.

In addition to exposing Fe specimens in Fe and Mo capsules, two additional capsule tests were performed. A coated and pre-oxidized 316 specimen was exposed at 700°C to study coating behavior on an austenitic steel at this temperature. A small mass gain of 0.2mg/cm^2 was observed. Also a pre-oxidized PM2000 (ODS FeCrAl) specimen [3] was again exposed at 600°C for 1000h to determine if the α-Al_2O_3 thermally grown oxide would transform to $LiAlO_2$. Curiously, the oxide appeared to be removed at this temperature after cleaning, similar to the previous result. The oxide was retained after Pb-Li exposures at 500°, 700° and 800°C.

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