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. Two of the issues currently being investigated are the potential for dissimilar material interactions in Pb-Li and the effect of impurities in the Pb-Li on the amount of alloy dissolution.

SUMMARY

Initial isothermal capsule experiments were conducted to address two Pb-Li compatibility issues. First, the effect of Fe and Ni impurities in the Pb-Li on the amount of mass loss of representative austenitic (316SS) and ferritic (P92) steels was investigated at 700°C by adding controlled amounts of these elements to the Pb-Li in the capsule. Unexpectedly, the specimens tested with Fe and Ni additions showed higher mass losses. The second set of experiments investigated the potential dissimilar material interaction between Fe and SiC in Pb-Li. Using carbon steel capsules, the mass loss of Fe and SiC samples was measured after isothermal exposures at 500°, 600° and 700°C. For the SiC specimens, larger mass changes were measured than in prior studies at higher temperatures. These tests will be repeated with SiC capsules. For all of these recent experiments, variations in the composition of the Pb-Li source material has confounded the results.

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.[2-6] Compared to Li,[7] a wider range of materials can be compatible with Pb-Li because of the low activity of Li.[8] In particular, SiC readily dissolves in Li, but not Pb-17Li.[2,4,9] However, like Pb, Pb-Li dissolves Fe, Cr and especially Ni from many conventional alloys above 500°C.[10,11] 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 oxide dispersion strengthened (ODS) ferritic steels[12] 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.[11] 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 second and third topics are presented here to follow up on results presented in the previous report.[6] The planned dissimilar capsule experiments with SiC capsules have been delayed due to problems machining the chemical vapor deposited (CVD) SiC.

Experimental Procedure

Static capsule tests were performed using Mo or carbon steel inner capsules and type 304 stainless steel

outer capsules to protect the inner capsule from oxidation. 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.) The representative FM steel was P92 (Fe-9Cr-2W) and the austenitic steel was type 316 (Fe-19Cr-10Ni-2Mo). 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. The Mo and Fe capsules were loaded with 125 g of commercial purity Pb-Li in an argon-filled glove box. Commercial Pb-Li was earlier obtained from UCLA and is designated Pb-Li (1). The Li content of this material typically measured <15 at.% after exposure. Because of the higher Li contents noted in the initial experiments with a new batch of commercial Pb-Li from Atlantic Metals, denoted Pb-Li (2), the experiments with carbon steel capsules used 100g of Pb-Li and 25g of high purity Pb. Additions of Fe or Ni powder (1000 ppma) were made during capsule loading. The capsules were welded shut in a glove box to prevent the uptake of impurities during 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.

Results and Discussion

Figure 1 shows the mass change data from the study of Fe and Ni impurities on the mass loss of P92 and 316SS. All of the exposures were performed in Mo capsules for 1,000 h at 700°C. Because of the differences in the two commercial Pb-Li sources, the exposures without additions were repeated with the second Pb-Li source. However, the mass losses were not significantly different despite the large differences in the Li content between the two batches, Table 1. It was expected that the addition of Fe and Ni impurities to the Pb-Li would reduce the mass loss because the isothermal rate of dissolution of an element *i* is governed by:

$$J_i = k\,(C_i^S - C_i) \qquad [1]$$

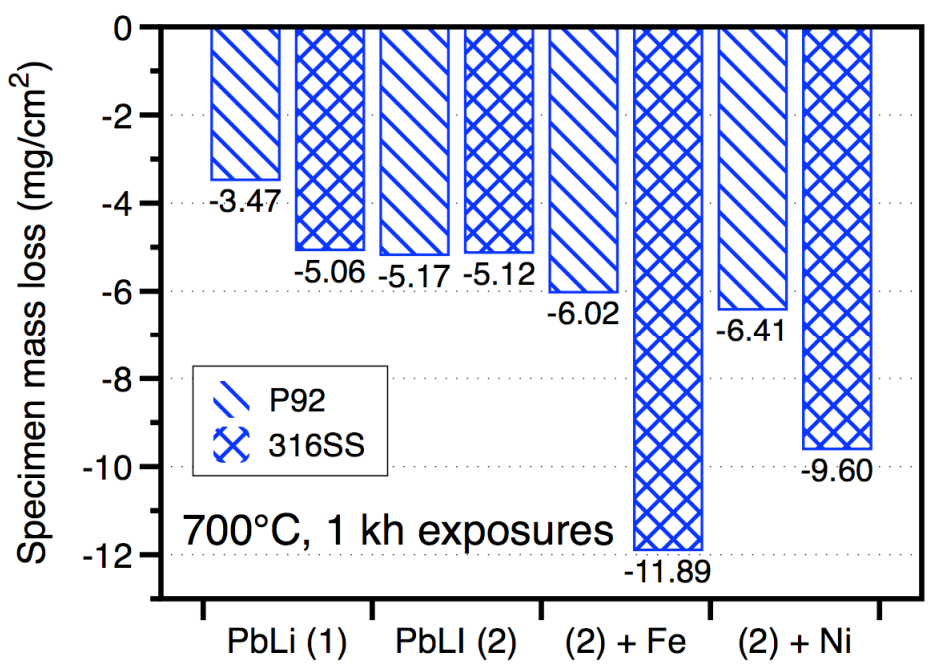

Figure 1. Mass loss results from P92 and 316SS exposed for 1,000 h at 700°C with two different Pb-Li sources and with either 1000 ppma Fe or Ni additions to Pb-Li (2).

Table 1. Chemical composition measured using inductively coupled plasma and combustion analysis of the Pb-Li after the 1,000 h capsule test.

	Pb-Li source	Add	Li at.%	O at.%	Fe ppma	Ni ppma	Cr ppma
316SS	Comm.1	No	13.7	0.21	19	52	7
	Comm 2	No	37.3	1.95	481	305	1436
		+Fe	23.5	4.52	**422**	283	304
		+Ni	21.0	1.37	520	**874**	210
P92	Comm 1	No	14.7	0.29	28	3	<3
	Comm 2	No	33.3	9.90	751	6	570
		+Fe	20.4	2.41	**182**	8	31
		+Ni	29.7	1.17	171	**717**	97

where J_i is the flux of species *i* into (positive) or from (negative) the liquid metal, C_i^S is the solubility of *i* in the Pb-Li and C_i is the instantaneous concentration of i in the liquid. In a capsule experiment, dissolution continues until the liquid metal is saturated, i.e. $C_i^S = C_i$.[13] Thus, by adding Ni to the capsule, this should have increased C_{Ni}, thereby decreasing the magnitude of J_{Ni}. The largest decreases were expected for the dissolution of P92 with the Fe addition and 316SS with Ni. However, the expected decrease was not observed, the amount of dissolution was higher with the additions. The variation in Li content in the capsules may have confounded the results, Table 1. Table 1 also includes the Fe, Ni and Cr contents in the Pb-Li in units of ppma. Higher Ni levels were evident after Ni was added to the capsule but the Fe addition was not as evident compared to the other experiments. The Fe powder may not have been incorporated into the Pb-Li as readily as the Ni powder which has a higher solubility in Pb.[10] Another possibility is that the dissolution of other elements compensated for the addition. For example, when Ni was added, the 316SS specimen lost more Fe and Cr. Or, when Fe was added, the P92 specimen lost more Cr. Cross-sections of all of the specimens are being prepared for chemical analysis. The Pb-Li chemical analysis suggested that the higher Li levels resulted in more Cr in the liquid, perhaps due to the formation of the Li-Cr-N compound.[14,15] Table 1 also includes the O content measured. It is not clear if this variation is due to the source material or variability in contamination during handling and measurement. A higher O content could have increased Cr dissolution by first forming Cr_2O_3 or a Cr-rich oxide, which then dissolved.

Based on the initial dissimilar materials experiments performed at 600°C,[6] a plan for a more complete data set was developed with carbon steel and SiC capsules at 500°-700°C. The SiC capsule experiments have been delayed due to problems machining the capsules. After addressing a problem with the CVD SiC feedstock, capsules are expected in the next quarter. Figure 2 shows the mass change data for the Fe and SiC specimens in a carbon steel capsule. Since iron carbides are more stable than SiC, it is possible that mass transfer could occur[16]. However, very small mass changes were observed at 500° and 600°C for both samples. The small mass losses for SiC are higher than those observed at 800°-1200°C.[2,4] The previous SiC specimen exposed in a steel capsule at 600°C showed a 0.04 mg/cm^2 mass loss but surface analysis of the specimen revealed only residual Li and C and no clear degradation.[6] The 700°C SiC specimen will be characterized to determine the source of the mass gain. The high mass loss for the Fe specimen at 700°C suggests increasing Fe dissolution between 600° and 700°C, as expected. The Fe in the Pb-Li after the experiment increased from 1380 ppma after the 600°C experiment to 17,150 ppma after 700°C. The addition of Pb to these capsules reduced the Li contents to 14-27% from the higher range seen in Table 1. However, the Pb addition did not address the apparent Li variability in the commercial source that will have to be addressed either by remelting or acquiring a

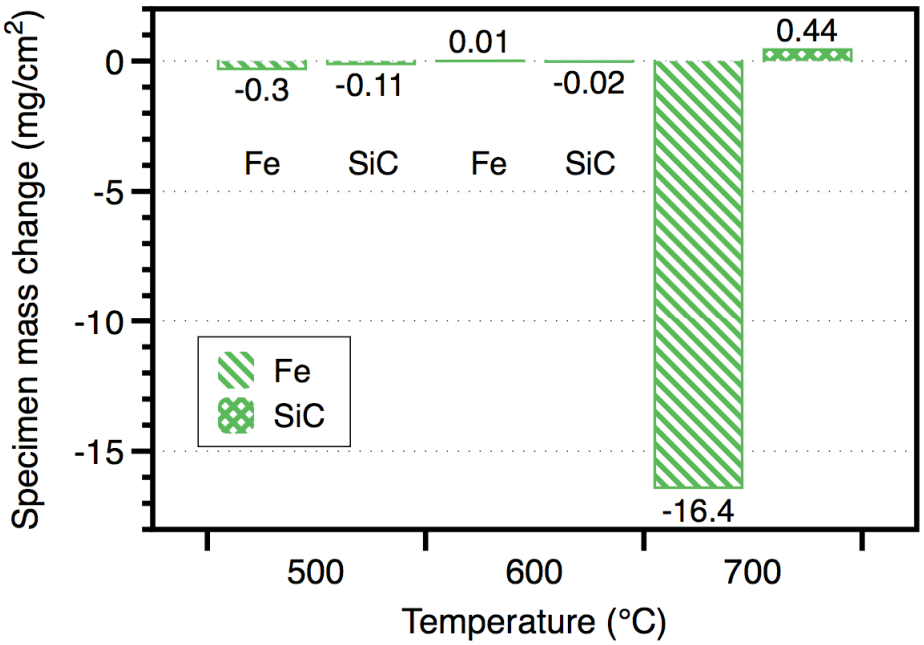

Figure 2. Specimen mass change from a series of capsule experiments with carbon steel capsules and Fe and SiC specimens exposed to Pb-Li for 1,000 h at each temperature.

different supply before additional capsule experiments are conducted.

Further characterization is needed on both groups of specimens. Also, the dissimilar materials results will have more context for evaluation when the results for the SiC capsules are available for comparison.

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