INVESTIGATION OF Pb-Li COMPATIBILITY FOR THE DUAL COOLANT TEST BLANKET MODULE—
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OBJECTIVE

The objective of this task is to assess the long-term, high-temperature compatibility of various materials with Pb-Li. One proposed fusion reactor concept uses SiC/SiC composites with a self-cooled Pb-17Li blanket. Another concept uses a SiC/SiC flow channel insert with a dual coolant of He and Pb-Li at ~800°C. These concepts also require piping to carry the Pb-Li between the first wall and the heat exchanger. To evaluate compatibility issues in these systems, monolithic SiC, SiC/SiC composites and potential piping and coating materials are being exposed to isothermal Pb-17Li in capsule tests at 700°-1200°C.

SUMMARY

Static capsule tests in Pb-17Li were performed on coated and uncoated type 316 stainless steel and Al-containing alloys at 800°C and the Pb-Li was analyzed after each capsule test. Chemical vapor deposited (CVD) aluminide coatings on type 316 substrates reduced dissolution by ~50X at 800°C compared with uncoated samples. Little effect of pre-oxidation was observed for the performance of the coating. These results indicate that aluminide coatings may be a viable option to allow conventional Fe- or Ni-base tubing alloys to carry PbLi from the first wall to the heat exchanger. Future work will need to include testing in a flowing system with a thermal gradient to fully determine the compatibility of these materials. In order to test the viability of using a thermal convection loop made of quartz, a quartz ampoule was filled with Pb-Li and exposed for 1000h at 800°C. No Si was detected in the Pb-Li after the test indicating that quartz may be a low cost construction option.

PROGRESS AND STATUS

Introduction

One proposed test blanket module (TBM) concept for ITER is a dual coolant (He and Pb-Li) system using advanced ferritic steels as the structural material and a silicon carbide composite as a flow channel insert [1,2]. Although the TBM design will operate at <500°C, thereby limiting compatibility problems, ultimately this blanket concept would be more attractive with a maximum operating temperature of 700-800°C. In this temperature range, critical compatibility issues need to be addressed. Recent effort has focused on the compatibility of ferritic-martensitic steels in Pb-Li at 400-600°C [3,4]. However, there has been less work examining corrosion-resistant coatings needed at higher temperatures.

Because of the low activity of Li in Pb-17Li, thermodynamic calculations have shown that Al_2O_3 should be stable [5], indicating that alloys or coatings that form an adherent external alumina scale should be resistant to dissolution in Pb-Li. Such coatings would be required for conventional piping materials to carry Pb-Li from the first wall to the heat exchanger at ~700°C. While a SiC flow channel insert could protect the steel walls from Pb-Li dissolution, it is unlikely this strategy could be used through the entire flow path. Contact of Fe- or Ni-base, alloys with flowing Pb-Li at this temperature would result in unacceptably high dissolution rates [3,4,6]. Although the use of refractory metals is one option [7], fabrication and durability of Nb or Mo tubing could be an issue. However, a protective coating could allow a conventional Fe- or Ni-base tubing alloy to be used.

Baseline compatibility data is being developed using static capsule tests and model materials in order to assess the performance of an aluminide coating or Al-containing alloys. Based on positive results at

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
316SS	65.1	8.9	19.9	0.02	490	3360	2380	68	1.94Si,1.67Mn, 1.38Mo,0.21Cu
ODS FeCrAl	67.8	0.02	20.0	10.6	7430	340	210	50	0.44Ti,0.23Y 0.04Si, 0.04Mn
Fe-28Al-2Cr+Zr	70.0	<	2.0	27.9	70	400	<	46	0.026Zr, 0.005Hf
Ni-42.5Al	<	57.3	<	42.6	40	380	<	<	<

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

700°C [8] CVD aluminide coatings on type 316 stainless steel substrates were tested at 800°C. Both sets of experiments show promising results, consistent with the thermodynamic assessment.

Experimental Procedure

Capsule tests with static PbLi (detailed elsewhere [8,9]) were performed on alloy coupons with a 1.5mm thickness, a surface area of 4-5cm^2 and a 600grit SiC surface finish. The chemical composition of the alloys tested at 800°C are shown in Table 1. A Fe_3Al composition was selected as being similar to aluminide coatings formed on Fe-base alloys [10] and a Ni-42Al composition is similar to the composition of a CVD aluminide coating on a Ni-base alloy [11]. In addition, an oxide dispersion strengthened (ODS) FeCrAl (Plansee alloy PM2000) was tested as this class of alloy could be used without a coating. The alloy specimens were pre-oxidized for 2h at 1000°C in 1 atm dry O_2 before exposure to Pb-Li. Two specimens of type 316 stainless steel were CVD aluminized for 4h at 1050°C in a laboratory scale reactor and then immediately annealed for 2h at the same temperature [10]. These conditions produce a coating approximately 200µm thick with an Al-rich outer layer, ~$(Fe,Ni)_3Al$, about 20µm thick. One of the coated specimens was pre-oxidized for 2h at 800°C in air. All of the specimens were suspended in a welded Mo capsule using Mo wire. For these tests, the inner Mo capsule was loaded with high purity (99.9999%) Pb shot and Li in an argon-filled glove box. The Mo capsule was protected by an outer type 304 stainless steel capsule. Specimen mass was measured before and after exposure on a Mettler-Toledo balance with an accuracy of ±0.04 mg. The 1000h exposures were performed in resistively heated box furnaces. After exposure, the specimens were soaked in a mixture of acetic acid, hydrogen peroxide and ethanol for 24-72h to remove any residual Pb-Li. The composition of the Pb-Li after testing was determined by inductively coupled plasma analysis and combustion analysis. Post-test characterization of the specimens has not yet been performed.

Results and Discussion

Capsule results. Table 2 shows the mass changes for the specimens exposed at 800°C [12]. Compared

Table 2. Mass change of specimens after 1000h at 800°C in Pb-17Li with a Mo capsule.

Specimen	Pre-oxidation	Mass Change (mg)	Mass Change (mg/cm^2)
316SS	none	-79.51	-17.30
316SS + CVD Al	none	- 1.55	- 0.34
316SS + CVD Al	2h at 800°C	- 1.93	- 0.43
ODS FeCrAl	2h at 1000°C	+ 1.58	+ 0.24
Fe-28Al-2Cr+Zr	2h at 1000°C	- 1.55	- 0.37
Ni-42.5Al	2h at 1000°C	-12.12	- 2.72

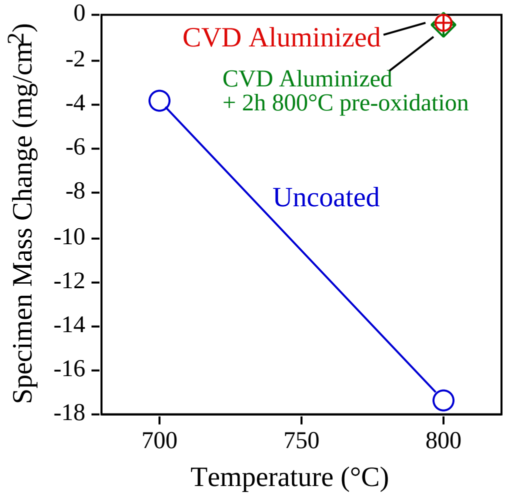

Figure 1. Specimen mass loss after 1,000h in Pb-17Li as a function of temperature for uncoated and coated type 316 stainless steel.

to the uncoated 316SS specimen, the aluminized 316SS specimens showed dramatically lower mass losses. Pre-oxidizing the coating for 2h at 800°C to form an alumina layer did not have a beneficial effect on dissolution. Figure 1 summarizes the 700° and 800°C results for uncoated and coated type 316 stainless steel in Pb-Li showing the ~50X reduction in dissolution at 800°C with the CVD aluminide coating.

The Al-containing alloys also showed lower amounts of dissolution. The mass gain for the ODS FeCrAl specimen may be due to entrapped metal, i.e., incomplete cleaning, however, the specimen appeared unaffected by the exposure. The Fe_3Al specimen had one ~3mm area where the oxide was removed causing some degree of mass loss. This selective attack area may be due to incomplete initial mixing of the Pb and Li. Undoped lithium would quickly attack alumina and any of these alloys at 800°C. The highest mass loss was for NiAl where ~90% of the oxide appeared to be spalled after exposure. One reason for the higher mass loss may be that Ni dissolves more readily than Fe in PbLi. This hypothesis was confirmed by the chemical analysis which showed a detectable Ni level in the PbLi after exposure of the NiAl sample, Table 3. Another factor is that this alloy did not contain a reactive element addition (Y, Zr, Hf, etc.) which improves the adhesion of the alumina layer formed during pre-oxidation, Table 1. Spallation of the alumina could have resulted in more attack for this specimen.

Table 3. Chemical composition using inductively coupled plasma and combustion analysis of the starting Pb, commercial Pb-Li ingot and the Pb-Li after capsule exposures at 800°C for 1000h (in ppma except for Li in atomic%).

Test	Li	Fe	Cr	Ni	Mn	Si	Al	Mo	C	O	N	S
Starting Pb	n.d.	<4	<4	<4	<4	<40	<8	<2	<170	1270	<40	<50
Comm. PbLi	14.3%	<30	<70	<30	<30	<120	<60	<40	750	4820	180	<50
316SS	16.5%	<30	<30	**270**	<30	<120	<60	<20	480	2040	<40	<50
316SS+Al	17.6%	<30	<30	<30	<30	<120	<60	<20	590	1370	<40	<50
316SS+Al/O	17.5%	<30	<30	<30	<30	<120	<60	<20	730	2100	<40	<50
FeCrAl	17.3%	<30	<30	<30	<30	<120	<60	**90**	460	5280	<40	<50
Fe_3Al	16.3%	<30	<30	<30	<30	<120	<60	<20	540	1230	<40	<50
NiAl	16.7%	<30	<30	**150**	<30	<120	<60	<20	520	2640	<40	<50
Quartz	14.5%	<30	<30	<30	<30	<120	<60	<20	430	4550	<40	<50

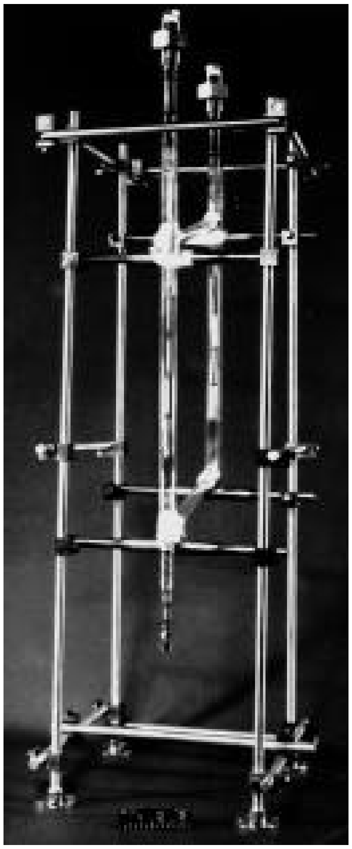

Figure 2. Photograph of a quartz thermal convection loop at ORNL [14].

Loop experiment planning. Future work will eventually include flowing liquid metal experiments with a temperature gradient. Static capsule experiments can only be expected to produce a limited assessment of the compatibility issue because saturation of the liquid metal with the element or elements dissolving eventually inhibits further dissolution [6,8]. Since low-cost quartz loops (Figure 2) were previously used for testing Pb [13] and Bi-Li [14], the possibility of constructing Pb-Li loops out of quartz was investigated by testing Pb-17Li in a quartz ampoule at 800°C. A mass change could not be determined in this test but the post-test PbLi chemistry showed no detectable Si dissolved in the liquid metal, Table 3. Thus, a quartz loop appears to be a viable, low-cost option, at least for an initial assessment of compatibility of SiC composites and Al-containing alloys and coatings, for tests in flowing Pb-Li.

The current procedure of in-capsule melting of Pb and Li to form the Pb-17Li composition would not be viable for a loop testing program. Therefore, purchase of pre-alloyed Pb-Li has been investigated and commercial Pb-Li was obtained from N. Morley at UCLA. The chemical composition of the as-received commercial PbLi is shown in Table 3. In particular, O and N are much higher than in the starting Pb used in the capsule experiments. Based on the high Li-O affinity and prior results, the high O content likely suppressed the measured Li in the commercial material. The commercial PbLi is being used in two recently started capsule experiments examining monolithic SiC at 1000°C.

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