6.1 COMPATIBILITY OF FeCrAl IN FLOWING Pb-LI AT 550°-600°C– B. A. Pint and S. J. Pawel (Oak Ridge National Laboratory, USA)

OBJECTIVE

This task is investigating the possibility of increasing the Pb-Li temperature in the dual coolant lead-lithium (DCLL) blanket concept in order to improve the overall system efficiency. Alloys based on FeCrAl are a potential candidate and monometallic thermal convection loops of a commercial FeCrAl alloy are being built and operated to establish a maximum operating temperature for operation in flowing eutectic Pb-Li.

SUMMARY

The second monometallic thermal convection loop (TCL) using Kathal FeCrAlMo alloy APMT tubing and specimens has completed 1000 h in commercial purity eutectic Pb-Li with a peak temperature of 600°C. After cleaning, the mass change data showed small mass losses for all of the specimens preoxidized at 1000°-1100°C to form an alumina scale prior to exposure in the loop. Larger mass losses were noted for specimens that were not pre-oxidized, especially at the lower temperatures. Characterization of the specimens is in progress.

PROGRESS AND STATUS

Introduction

The DCLL blanket concept (eutectic Pb-17 at.%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] With the addition of Al to Fe-Cr alloys, isothermal compatibility tests have shown low mass losses at up to 800°C [4-7]. Thermodynamic evaluations [8,9] indicated that Al_2O_3 should be stable in Pb-Li and inhibit dissolution by forming at the alloy surface, however, capsule studies found that a preformed α-Al_2O_3 surface layer transformed to $LiAlO_2$ during exposures at 600°-800°C [4,10]. To further evaluate the Pb-Li compatibility of FeCrAl-type, exposures in flowing Pb-Li are needed where changes in solubility with temperature can drive mass transfer [2,11]. In 2014, a monometallic thermal convection loop (TCL) was operated for 1000 h with a peak temperature of 550°C using commercial Fe-21Cr-5Al-3Mo alloy (Kanthal APMT) tubing and specimens in the hot and cold legs [12-15]. This was the first time that Pb-Li was flowed at 550°C without plugging flow and only small mass losses were noted after the exposure. The next step in the compatibility evaluation was to operate a similar TCL with a peak temperature of 600°C.

Experimental Procedure

The details of the TCL construction and operation have been previously described in detail [12-14]. The TCL was ~1 m tall and 0.5 m wide and heated on one side by resistively heated furnaces (i.e. the hot leg). The loop contained two specimen chains of 20 SS-3 type APMT specimens connected with APMT wire. One in the hot leg and one in the cold leg. Most of the specimens were heated treated for 8 h at 1050°C in air to form an α-Al_2O_3 surface layer or scale. Two specimens in each chain had no pre-oxidation treatment, preoxidation at 1000°C and preoxidation at 1100°C. Rectangular coupons of unalloyed tungsten were attached at the bottom of each specimen chain to act as a “sinker” to keep the relatively low density specimen chains from floating in the Pb-Li test fluid, and to act as “spacers” to keep the

specimen chain centered within the tubing and liquid metal flow path.

Commercial purity Pb-17Li was used with impurity levels of 1200ppmw O, 240 ppmw C and <10 ppmw N (average of 6 samples) and no metallic impurities above the detection limite of ~1 ppmw. The Pb-Li was melted in the fill tank above the hot leg and the loop was evacuated prior to filling. The TCL contained six thermowells that protruded about 0.3 cm into the flow path to monitor temperature at the top, bottom and middle of each leg. The flow rate within the loop was estimated by heating a section of the tubing using a gas torch for ~15 s and then tracking the temperature spike as that heated liquid move around the loop. Following 1000 h of operation at 600°C, the Pb-Li was drained into the dump tank.at the bottom of the cold leg. After removal, specimens were soaked in cleaning solution (1:1:1 mixture of ethanol, hydrogen peroxide, and acetic acid) while within the loop (as an assembled chain) and again upon removal from the loop (as individual specimens). The cleaning solution readily dissolved residual Pb-Li rendering weight change measurements more meaningful. Following exposure to the cleaning solution, the specimens were ultrasonically cleaned in acetone followed by air drying prior to remeasuring the mass using a Mettler Toledo model XP205 balance (~0.04 mg accuracy).

Results and Discussion

Figure 1 shows the temperatures measured at the six TCL thermowells after stabilization (minor adjustments to heaters and insulation over the first 24 h or so). After operation for ~440 h, one of the hot leg furnaces failed causing a drop in all temperatures. Unfortunately, during installation of a replacement furnace, it was damaged and could not be used. There was a ~215 h delay, before a new furnace could be purchased and installed where the loop operated at a lower temperature. The experiment was completed with a cumulative 1000 h at the peak temperature of 600°C.

Figure 1 also shows that the typical temperature gradient was ~85°C during this experiment. In the previous 550°C experiment, the temperature gradient was ~115°C. During the previous experiment, the velocity was measured at ~0.4 m/min twice. This value was lower than the 0.8-0.9 m/min observed in similar ORNL sodium loops. In the 600°C TCL experiment, the velocity was measured at 0.6-0.7 m/min, more consistent with the previous work. Both the increased velocity and decreased temperature gradient, support the possibility that the chain may not have been in “single file” orientation that may have inhibited flow in the previous experiment.

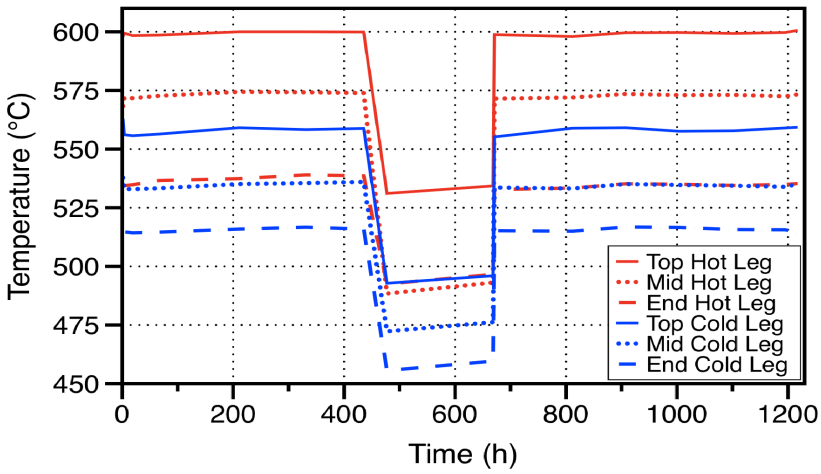

Figure 1. Thermowell temperatures measured during the experiment.

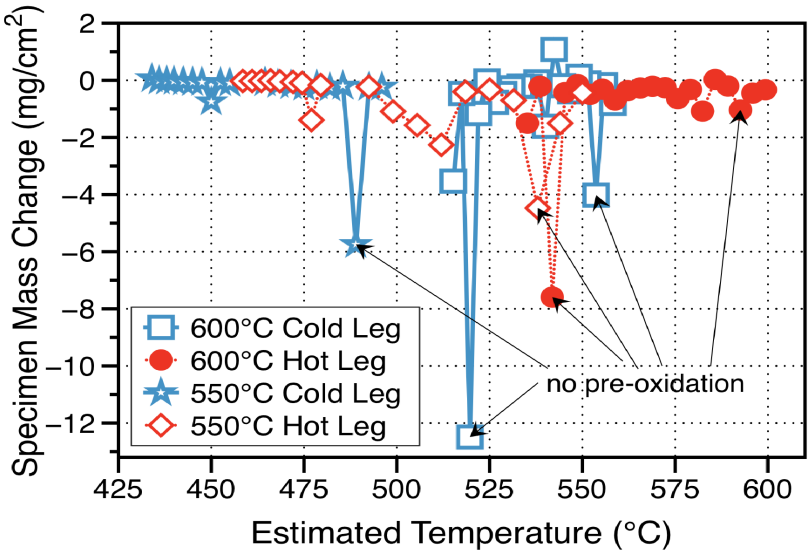

Figure 2. Specimen mass change in the hot and cold legs of the 550° and 600°C TCLs. Specimens that were not pre-oxidized are noted from both experiments.

Figure 2 shows the mass change data after cleaning for the hot and cold leg specimens from both TCLs as a function of estimated temperature in each chain of specimens. The approximate temperature of each specimen location was estimated by linear extrapolation as a function of position between thermowell locations at which the temperature was known. In the recent experiment, the mass losses were modest for the pre-oxidized specimens and comparable to the previous experiment. Varying the pre-oxidation temperature from 1000°-1100°C showed little effect on the mass loss. As observed previously, the mass losses can partially be attributed to spallation of the oxide layer, which transforms from α-Al_2O_3 to $LiAlO_2$ during the exposure. The largest mass losses were observed for the specimens that were not pre-oxidized prior to exposure. Presumably, faster dissolution occurred prior to the establishment of a protective Al-rich oxide. However, the bare specimen at the top of the hot leg did not show a large mass loss. At the higher temperature (~592°C), the oxide layer may form more quickly as Al is relatively immobile at <600°C. Figure 3 shows plan-view scanning electron microscopy images of the surface oxide morphology. Characterization of the other specimens is in progress followed by room temperature tensile testing.

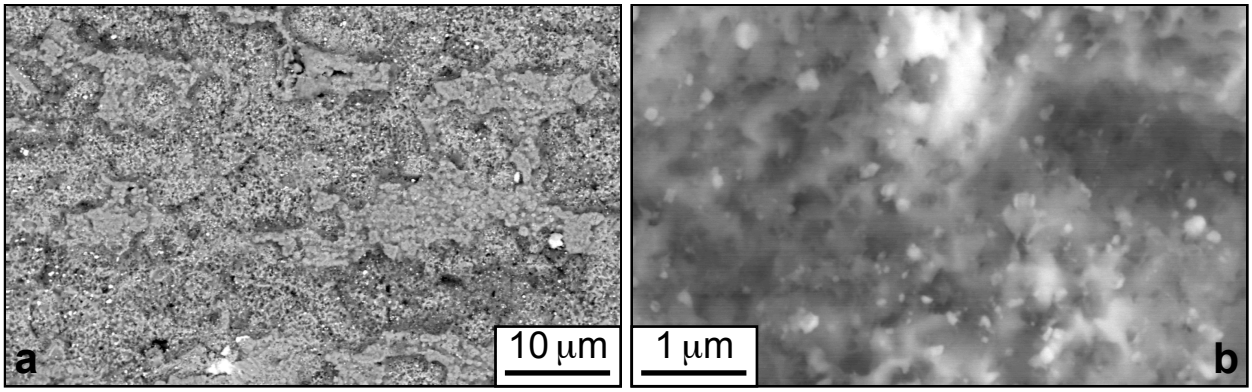

Figure 3. Scanning electron microscopy of the scale formed on an APMT specimen without pre-oxidation after exposure at ~593°C for 1000 h.

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