6.1 LIQUID METAL COMPATIBILITY - S. J. Pawel (Oak Ridge National Laboratory, USA)

OBJECTIVE

The objective of this task is to identify potential structural materials having sufficient compatibility with flowing Pb-Li eutectic that the maximum metal temperature for operation can be increased to improve overall system efficiency.

SUMMARY

Operation of the first thermal convection loop (TCL) using dispersion strengthened FeCrAl (Kanthal APMT) tubing and specimens was completed in the previous reporting period. The working fluid in the TCL was eutectic Pb-17at%Li, and both the peak temperature (550˚C) and temperature gradient (116˚C) were maintained without interruption for 1000 h. When TCL operation was terminated, a problem with complete draining of the Pb-Li from the loop was encountered, which delayed retrieval of specimens and related examinations; however, post-exposure analysis of all specimens is now nearing completion.

PROGRESS AND STATUS

Introduction

Currently, the maximum allowable wall temperature for the dual coolant lead-lithium (DCLL) blanket concept is set at 475˚C based primarily on corrosion limitations of the structural containment materials. To increase overall system efficiency, potential structural materials are being sought with a combination of high strength and creep resistance with simultaneous resistance to dissolution in eutectic Pb-Li at temperatures > 500˚C. Preliminary research using static capsule exposures has indicated that dispersion strengthened FeCrAl (Kanthal APMT) may be resistant to dissolution in eutectic Pb-Li at temperatures in the range of 600-800˚C, at least in part due to the stability of an Al-rich oxide film. However, corrosion data in a flowing system must be generated to analyze the potential for issues associated with thermal gradient mass transfer – that is, relatively high dissolution in hotter portions of the flow system with concomitant deposition in the colder portions – which has been known to disrupt heat transfer and even plug flow paths completely in some temperature gradient/material combinations.

Thus, thermal convection flow loops (TCLs) are being incorporated as the follow-on step to capsule testing for evaluation of liquid metal compatibility. The initial testing associated with this effort utilized a mono-metallic TCL fabricated of Kanthal APMT tubing (26.7 mm OD, 3.1 mm wall) with a “chain” of APMT specimens (miniature tensile specimens termed “SS3s”) inserted within each of the hot leg and cold leg of the TCL for use in post-exposure evaluation. Examination and analysis of specimens exposed within the loop was advanced substantially in the reporting period.

Results

Figure 1 reports the weight change of the APMT specimens – after cleaning in a solution of 1:1:1 mixture of ethanol, hydrogen peroxide, and acetic acid – removed from the loop as a function of exposure temperature. The cleaning solution readily dissolves residual Pb-Li rendering weight change measurements more meaningful. Following exposure to the cleaning solution, the specimens were further cleaned ultrasonically in acetone followed by air drying. 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.

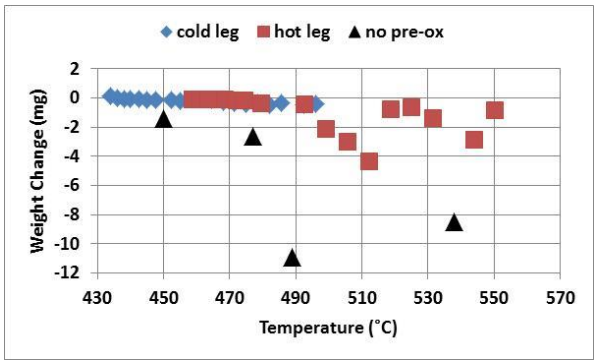

Figure 1. Weight change of specimens as a function of exposure temperature in the Pb-Li thermal convection loop. Black triangular data points correspond to specimens that were not given the pre-oxidation heat treatment.

The weight changes for most specimens are based on the original weight of the pre-oxidized specimen – that is, treated 8 h at 1050˚C in air to form a chromium oxide film rich in alumina prior to exposure – but data for a few specimens that did not receive the pre-oxidation treatment are included in Fig. 1. The data indicate the oxide film is at least partially protective of the substrate in Pb-Li because all four of the specimens exposed in the TCL without the oxide film – independent of exposure temperature – exhibit significantly greater weight loss than adjacent specimens that were pre-oxidized. However, with the exception of the specimen exposed at the lowest temperature within the loop (bottom of the cold leg), all specimens exposed within the TCL experienced at least some amount of weight loss suggesting that the oxide film is only partially stable in the Pb-Li environment, perhaps being converted to a lithium aluminate constituent and/or becoming somewhat soluble with extended time at temperature. Such a reaction seems more aggressive at higher temperature – specimen weight loss is at least sporadically greater at exposure temperatures above 500˚C in the hot leg – but visual assessments and metallographic cross sections of specimens reveal at least small regions that retain some of the original oxide film even at the highest exposure temperature. Using specimen density (~ 7.2 g/cm^3), specimen area (2.0 cm^2), and total exposure time (1000 h) to convert weight change to average uniform dissolution rate, note that even the highest weight loss among the pre-filmed specimens, approximately 4.4 mg, corresponds to only about 25 µm/y (1.0 mil/y) of annual wastage. While this is not a fixed rate, likely accelerating somewhat as more and more of the oxide film is removed, it is threatening primarily from the perspective of potential deposition at cold spots (plugging flow or disrupting heat transfer) as opposed to gross structural thinning considerations. Metallographic cross-sections revealed only very modest surface roughening in locations where the oxide film was removed, and element mapping revealed no chemical gradients in the base metal at exposed (no oxide film) locations, suggesting general dissolution as opposed to selective leaching in Pb-Li. Further, the element maps indicated no surface deposits on any specimens, suggesting all dissolution products are relatively soluble for the total extent of mass loss in this experiment.

Figure 2 presents the yield strength as a function of TCL exposure temperature for each APMT specimen. For comparison, data are also included for APMT specimens that did not receive the pre-oxidation heat treatment (8 h in air at 1050˚C) but were placed at a few positions around the loop for comparison exposure. While there appears to be some distinction between hot leg and cold leg specimens, it is clear that the yield strength of APMT increased substantially as a result of exposure at the relatively lower temperatures around the TCL. Note that the specimens with the largest weight loss – those not receiving the pre-oxidation treatment – did not deviate from the pattern of yield strength established by the pre-oxidized specimens. The most dramatic increase in yield strength results for specimens exposed below about 475-500˚C, although the temperature at which the strength increase occurs appears to modestly depend on whether specimens were exposed within the cold leg or hot leg (in which there is larger potential for dissolution rather than deposition). Interestingly, the yield strength of as-received APMT specimens (mill-annealed), pre-oxidized specimens, and pre-oxidized specimens heat treated for 1000 h

at 550˚C in argon was consistently 647 ± 10 MPa for all conditions. These observations suggest that precipitation reactions within the APMT structure, rather than interaction with the Pb-Li eutectic, are responsible for the change in yield strength. It seems likely that a precipitation reaction similar to that termed "475˚C embrittlement" is responsible for the observed increase in yield strength. In this reaction, an embrittling phase rich in chromium – often termed α-prime in many susceptible stainless alloys – forms on grain boundaries and within the bulk alloy to cause a significant increase in strength and concomitant loss of ductility. Advanced techniques, such as transmission electron microscopy and/or focused ion-bean studies, are necessary to confirm the occurrence of 475˚C embrittlement, and such work will be initiated soon. It is important to note that the data for the ultimate tensile strength (UTS) of the APMT specimens followed the same pattern as that for the yield strength, with nominal UTS values near 862 MPa increasing to about 1207 MPa as the exposure temperature was decreased.

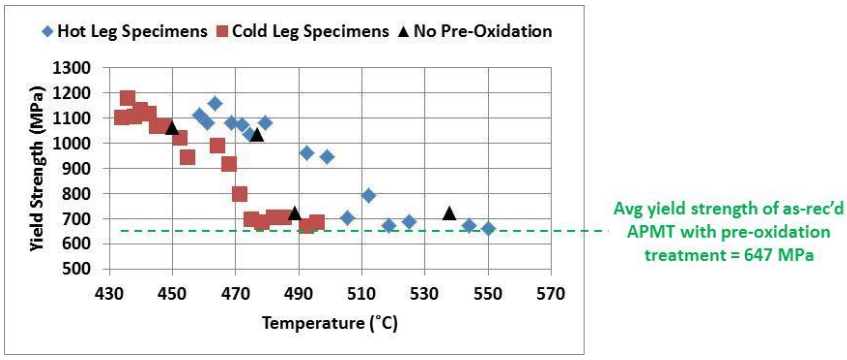

Figure 2. Room temperature yield strength of APMT specimens exposed to flowing Pb-Li eutectic for 1000 h at the indicated temperature. Black triangles represent data for specimens that did not receive the pre-oxidation heat treatment.

More scatter was observed in the relative ductility data, which is represented by total plastic elongation plotted in Fig. 3. Note that the lowest ductility values occurred at temperatures consistent with the concomitant increase in strength, but it is also apparent that very large variations in ductility were observed for adjacent specimens within the TCL (at an exposure temperature difference of only ~ 2˚C). Again, the elongation of as-received APMT specimens (mill annealed), pre-oxidized specimens, and pre-oxidized specimens heat treated for 1000 h at 550˚C in argon was consistently 23 ± 2% for all conditions. It is not clear that 23% elongation for sheet material can be expected for all forms and heats of material, because the as-received tubing used to fabricate the TCL was relatively brittle when strained at room temperature (order of 2% elongation or less, but not analytically assessed in specimens of the same size/shape as those exposed within the TCL).

Consistent with the above observations of wide variations in ductility even for similar exposure temperature, Fig. 4 shows representative views of the fracture surface of post exposure tensile specimens from the TCL hot leg. In this case, the specimen exposed at lower temperature exhibited increased yield strength and a largely planar fracture surface, indicating a predominantly cleavage-style fracture with some amount of cracking evident. In contrast, the specimen exposed at a temperature almost 50˚C higher generated a nominal yield strength and a very ductile fracture surface indicative of micro-void coalescence.

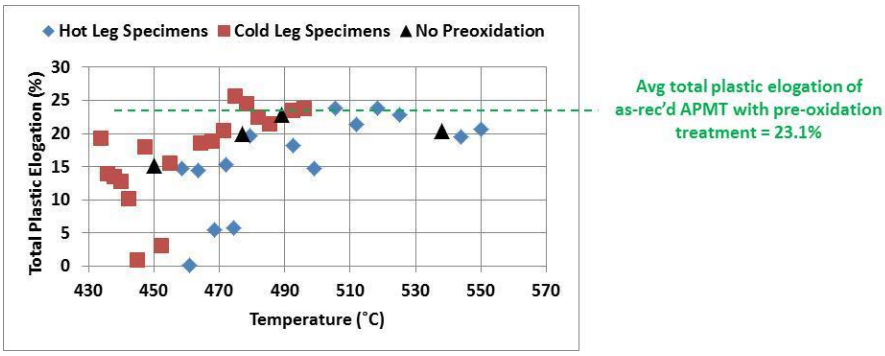

Figure 3. Room temperature ductility data (represented by total plastic elongation) of APMT specimens exposed to flowing Pb-Li eutectic for 1000 h at the indicated temperature. Black triangles represent data for specimens that did not receive the pre-oxidation heat treatment.

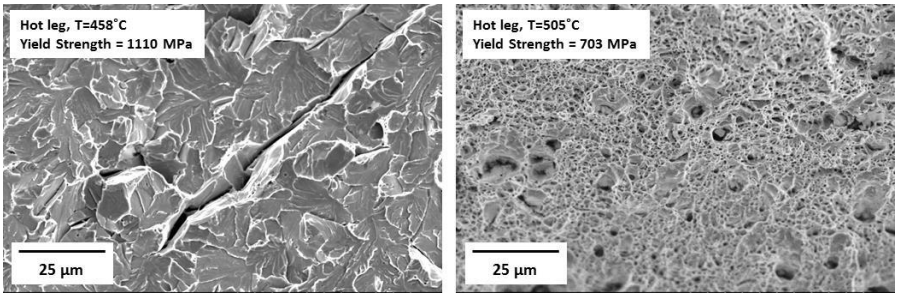

Figure 4. Post-exposure fracture surface of APMT miniature tensile specimens. The specimen exposed at relatively lower temperature (at left) reveals a high yield strength and a brittle fracture while the specimen exposed at relatively higher temperature (at right) reveals a lower strength and is much more ductile.

Future plans include continued analysis of specimens removed from the TCL (primarily, to confirm the strengthening mechanism observed at relatively low exposure temperatures) and initiation of the fabrication process for two additional APMT thermal convection loops with attendant specimens and components.