6.1 LIQUID METAL COMPATIBILITY — S. J. Pawel (Oak Ridge National Laboratory)

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 utilized commercially pure eutectic Pb-17at%Li, and both the peak temperature (550˚C) and temperature gradient (116˚C) were maintained without interruption for 1000 h. The previous report documented limited but somewhat irregular weight change of loop specimens as a function of exposure temperature, with the observation that a pre-oxidation process substantially improved compatibility with the Pb-Li over the exposure period. Further, post-exposure mechanical property data indicated a large increase in strength with concomitant loss of ductility for APMT specimens exposed at or below about 475-500˚C in the TCL. In this reporting period, analysis of TCL specimen cross sections for composition gradients and follow-up tests to examine mechanical properties of APMT exposed for 1000 h in argon (rather than Pb-Li) is documented.

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.

Kanthal APMT is an iron-based alloy produced by a powder metallurgy process and generically contains about 21% Cr, 3% Mo, 0.2% Cu, and 5% Al. This alloy does <u>not</u> possess the physical/mechanical properties required for fusion materials service and is <u>not</u> being considered as a candidate alloy, but it is available in tubing and several other forms necessary to fabricate a thermal convection loop (TCL) and was evaluated in the experiment described here as a surrogate for more appropriate materials under development but not yet readily available. While previous compatibility data for APMT in static capsules containing Pb-Li are encouraging, 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, TCLs are being incorporated as the follow-on step to capsule testing for evaluation of liquid metal compatibility.

Results

Cross-section element maps developed via energy-dispersive x-ray (EDX) analysis for two specimens exposed in the hot leg are shown in Figure 1. The maps reveal the qualitative concentration of the indicated element corresponding to the analysis area, which is shown in the upper left in each set of six photos (denoted “SE” for secondary electron image). The relative concentration of each element is suggested by the relative black/white intensity in the photograph, with relatively bright white indicating higher concentrations and lesser amounts indicated by darker shading.

One specimen – exposed at 474.5˚C near the bottom of the hot leg – represents the results for all the specimens that received the pre-exposure oxidation treatment (8 h at 1050˚C in air) intended to form a continuous alumina film. The weight loss for this particular specimen was very low which, with only rare exception for specimens exposed up to the peak temperature of 550˚C, was the trend observed for all specimens receiving the pre-oxidation treatment. As suggested by Figure 1, this specimen retains an aluminum oxide film over most of the surface, which is shown by the very high concentration of Al and O on the surface in a band (bright white) about 1-2 µm thick. Note that Cr – a typical component of passive films on many grades of stainless steels – is not present in the oxide film in significant amounts, and that there is no apparent composition gradient in the oxide film (indicating Pb or Li penetration or loss of Al) or in the parent metal below the oxide film (which would indicate selective leaching of primary alloy elements into the Pb-Li). Specimens exposed at other positions (temperatures) around the TCL exhibited identical patterns, although the continuity of the alumina film became more irregular as the exposure temperature increased, with the result that at the highest exposure temperatures, very little if any alumina film remained on the specimen surface.

The other specimen in Figure 1 – exposed at 538˚C with no oxidation treatment prior to exposure – exhibited a weight loss ~ 50 times that of the previously mentioned pre-oxidized specimen. The element maps (grey scale detection sensitivity not identical to that for the other specimen) indicate essentially a surface free of Cr- or Al-oxides and considerably more roughness than the pre-oxidized counterparts. The latter observation is consistent with irregular initiation of the dissolution process common to many alloys exposed to liquid metal environments. Even in the absence of a protective alumina film and with considerably higher weight loss than the pre-oxidized specimen, the element maps do not indicate composition gradients in the base metal at/near the specimen surface, which suggests uniform dissolution in Pb-Li.

Taken together, these results – in particular, the relative absence of the alumina film for pre-oxidized specimens exposed at the highest TCL temperatures – suggest that the alumina film on the APMT specimens is not completely stable in Pb-Li and perhaps dissolves slowly in Pb-Li with extended exposure time. That the film dissolves slowly is indicated by the large difference in weight losses for specimens with and without the pre-exposure oxidation treatment and the relative lack of surface roughness on originally pre-filmed specimens for which the film has apparently dissolved. No trace of another compound (for example, lithium aluminate) was found on the post-exposure specimens, suggesting either that any such compound formation is soluble or that the post-test cleaning procedure confounds detection of such a compound. That even the highest weight loss among these pre-oxidized specimens was only 2.2 mg/cm^2 (for the pre-oxidized specimen exposed at 512˚C; not at the highest loop temperature of 550˚C) corresponds to an average wastage rate of only ~ 25 µm/y suggests that the oxide film is relatively protective for extended periods and perhaps could be rendered even more protective via composition changes or alternate heat treatments.

Previous analysis of the APMT specimens exposed within the TCL revealed a substantial increase in tensile strength with a concomitant decrease in ductility for specimens exposed at or below about 490˚C; specimens exposed at higher temperatures revealed mechanical properties unchanged from the as-received material (with or without a pre-oxidation treatment). Post-exposure weight change data and metallographic examination of selected samples indicated very little interaction between APMT and Pb-Li at these temperatures, suggesting the cause of the change in mechanical properties was related to time-at-temperature effects as opposed to compatibility effects (for example, penetration of Pb or Li contributing to a precipitate within the APMT). To further consider this possibility, an experiment was conducted to examine potential mechanical property changes as a result of time-at-temperature in argon for comparison with the Pb-Li exposures. The results are summarized in Figure 2. Clearly, the change in mechanical properties with extended time-at-temperature below about 490˚C is independent of compatibility with Pb-Li, and appears consistent with something akin to “475˚C embrittlement” observed within some grades of stainless steels, which involves precipitation of brittle Cr- and/or Mo-rich phases throughout the structure. Selected specimens are being prepared for TEM examination to identify the constituent(s) responsible for this mechanical property anomaly.

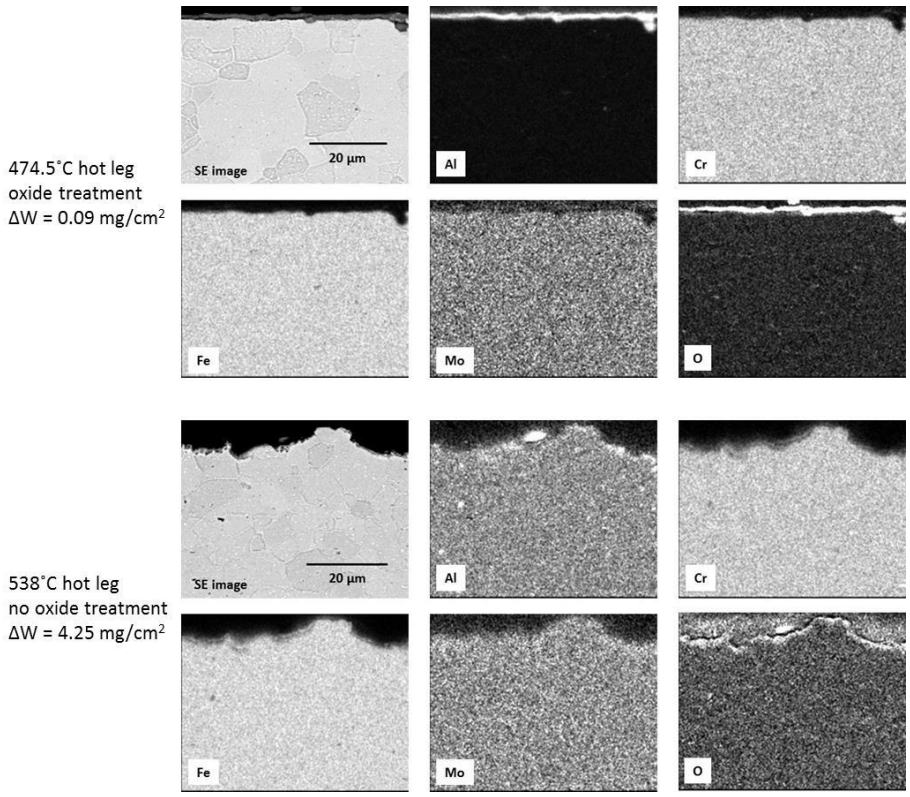

Figure 1. Element maps for major constituents of APMT made from specimens exposed in the hot leg of the TCL. Top series of photos (two rows of three) is for a specimen receiving the pre-oxidation treatment and subsequently exposed at 474.5˚C within the TCL; bottom series of photos (corresponding two rows of three) is for a specimen without the pre-exposure oxidation treatment exposed at 538˚C. Relative abundance of the specified element is indicated by relative light coloration; relative absence of the element indicated by relative darkness on the map.

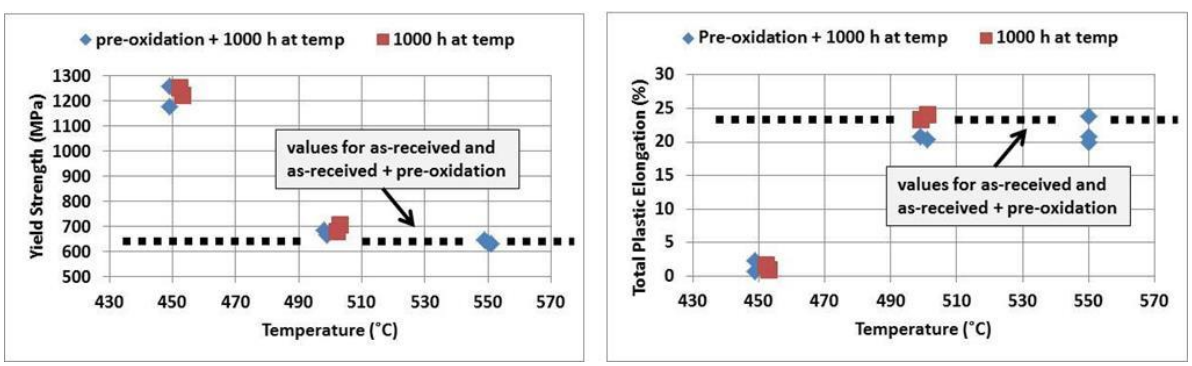

Figure 2. Room temperature yield strength (left) and total plastic elongation (right) for APMT specimens heat treated in argon for 1000 h at the indicated temperatures. [Actual treatment temperatures were 450, 500, and 550˚C; data staggered slightly on plot for ease of visualization.] The pre-oxidation treatment indicated was 8 h in air at 1050˚C, and in selected cases this treatment was performed prior to the subsequent heat treatment for 1000 h in argon. Tensile tests performed at room temperature following exposure in the TCL.

Future plans include completion of the fabrication process for two additional APMT thermal convection loops with attendant specimens and components. It is anticipated that the next TCL will be operated similarly to the prior experiment (1000 h duration, ~ 116˚C temperature gradient) except that all temperatures around the loop will be raised by 50˚C.