# OVERVIEW OF RECENT COMPATIBILITY ASSESSMENTS IN FLOWING EXPERIMENTS: Sn, Li, Pb-Li AND MOLTEN SALTS

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## ABSTRACT

The compatibility of several liquids is being considered for fission and fusion reactor applications. Following a 70-year-old methodology, static capsule testing is followed by testing in a flowing thermal convection loop (TCL) with a ~100°C temperature gradient to assess mass transfer and dissimilar material interactions. Recent TCL examples include Sn, Pb-Li and FLiBe. With a peak temperature of 400°C, Sn heavily attacked pre-oxidized FeCrAl specimens. A series of Pb-Li TCLs showed that dissolution significantly increased with a peak temperature of 700°C and a reaction occurred between SiC and FeCrAl. Lowering the peak temperature to 650°C reduced the reaction. Minimal reaction was observed with type 316H stainless steel in flowing FLiBe at a peak temperature of 650°C, similar to results in FLiNaK. Current Li work has focused on liquid metal embrittlement of F82H steel.

## INTRODUCTION

Several liquids are being considered for fission and fusion reactors including blanket and plasma-facing component (PFC) applications [1,2]. A full compatibility assessment requires testing in flowing liquid with a temperature gradient [3] in order to assess mass transfer and dissimilar materials effects where two materials can affect their compatibility [4]. The following reviews recent work at Oak Ridge National Laboratory supporting fusion and fission programs and illustrating the need for flowing experiments.

## EXPERIMENTAL PROCEDURE

The TCL experiments followed similar procedures as detailed previously [5-14]. Measured alloy compositions are shown in Table 1. Chains of 20 specimens hung in the hot leg (HL) and cold leg (CL) of the TCL for 1000 h. Hot spot tests [6] typically show TCL velocities of 1-2 cm/s. After exposure, the liquid was drained and the chains were removed. For static capsule testing, typically low carbon arc cast Mo capsules are used with the specimen held on one end using Mo wire. Capsules are loaded and tungsten inert gas welded in an argon-filled glove box. The primary capsules were welded inside a secondary type 316L stainless steel capsule prior to exposures in a resistively heated box furnace. At the end of the experiment, the capsule was inverted to allow the liquid to drain away [3]. In all cases, residual liquid was cleaned from the specimens using specific techniques to determine the specimen mass change using a Mettler Toledo XP205 balance with an accuracy of ±0.04 mg. Selected specimens were metallographically mounted and polished for imaging using light and electron microscopy to characterize the reaction and any remaining products.

TABLE 1. ALLOY COMPOSITIONS MEASURED USING INDUCTIVELY COUPLED PLASMA AND COMBUSTION ANALYSES

| Alloy | Fe | Cr | Al | Ni | Si | C | Other |
|---|---|---|---|---|---|---|---|
| F82H | 88.9 | 8.1 | 0.02 | 0.06 | 0.08 | 0.10 | 1.8W,0.5Mn,0.2V,0.09Ta,0.02Cu |
| APMT | | 69.0 | 21.6 | 4.9 | 0.12 | 0.53 | 0.03 2.8Mo,0.1Mn,0.2Hf,0.1Y,0.1Zr |
| ODS FeCrAl† | | 83.6 | 9.8 | 6.0 | | | 0.06 0.22Y,0.27Zr,0.10O,0.04N |
| ODS FeCrAl | | 80.8 | 11.6 | 6.2 | 0.03 | 0.02 | 0.03 0.39Zr, 0.38Y, 0.49Ti, 0.02Mn |
| 316H | | | | | | | |

† Heat 4H795C

## RESULTS & DISCUSSION

For PFC's, Sn is attractive because of its lower vapor pressure compared to Li [1]. In initial static capsule testing [15], high purity (99.995%) Sn showed very poor compatibility with F82H specimens in static tests at 400°C. However, more promising results were obtained with pre-oxidized APMT at 400°C-500°C. The specimens were pre-oxidized for 2 h at 1000°C in air to form a dense, adherent and stable α-$Al_2O_3$ surface layer [7]. Based on these results, a TCL was built from APMT tubing (pre-oxidized 8 h at 1050°C) and operated with flowing, high-purity Sn for 1000 h with a peak temperature of 400°C. Fig. 1a shows the large mass losses observed for 3 different pre-oxidized FeCrAl alloys exposed: APMT and two ODS FeCrAl alloys (Table 1), a 10Cr alloy from ORNL [16] and a 12Cr alloy from Japan. The temperature for each specimen was estimated based on a linear gradient between thermocouples at the top, middle and bottom of each leg. In this case, the total gradient was ~55°C. Only small mass changes were observed on the CL. Especially above 380°C, large mass losses were observed. Examples of mass loss are shown in Fig. 1b. While pre-oxidation appeared effective in static capsule testing, it was not sufficient to prevent significant dissolution in flowing Sn. These results suggest that Sn will be challenging to use with conventional alloys.

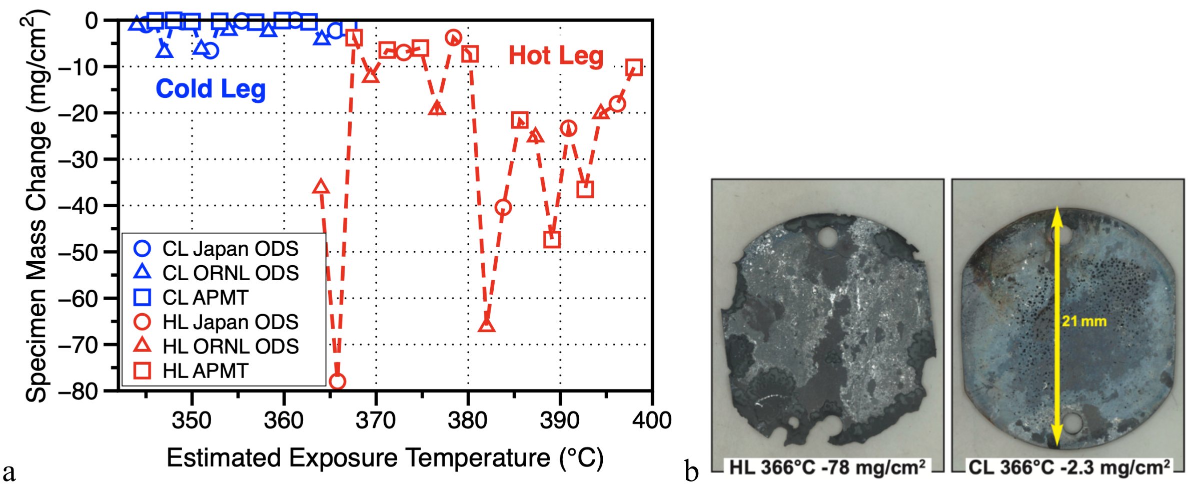


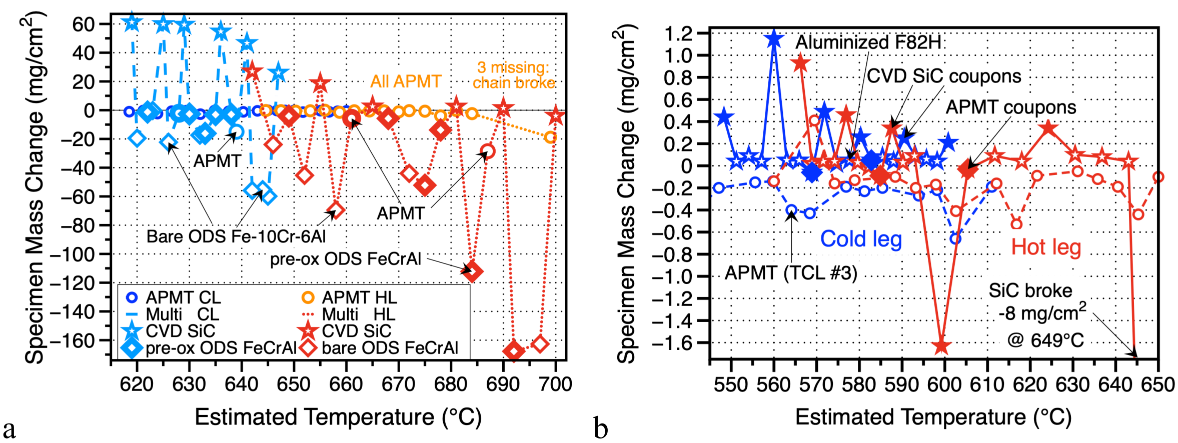

*FIG. 1. (a) specimen mass change after 1000 h in flowing Sn as a function of estimated exposure temperature and (b) images of two ODS FeCrAl specimens after exposure from the hot leg (HL) and cold leg (CL).*

For Pb-Li, six TCL experiments have been conducted for 1000 h each with commercial Pb-17at. %Li and APMT tubing [6]. The first four monometallic TCLs had increasing peak temperatures from 550° to 700°C and generally small mass losses below 675°C when the APMT specimens were pre-oxidized for 2 h at 1000°C in air [6-8]. In the fifth TCL with ODS Fe-10Cr-6Al and high-purity chemical vapor deposited (CVD) SiC specimens with a peak temperature of 700°C, severe attack was observed with large dissolution of FeCrAl and the formation of a thick (>100 µm) carbide-silicide reaction product on SiC specimens [9]. Fig. 2a compares the relatively small mass changes in the monometallic APMT TCL to those in the multi-material TCL where the SiC specimens showed large mass gains in the CL. The sixth TCL lowered the peak temperature to 650°C and pack aluminized and pre-oxidized F82H showed only small mass changes, Fig. 2b [10]. Previous capsule testing showed no mass change of CVD SiC in PbLi, but the testing was conducted in a SiC capsule [17].

For Li, excellent compatibility was demonstrated with a V-4Cr-4Ti TCL with a peak temperature of 700°C [5]. For steels in Li, liquid metal embrittlement (LME) [18] of 4340 and F82H steels was evaluated using hollow tensile specimens at 200°C. Results for 4340 steel showed LME but similar testing for F82H did not show similar evidence of LME [19].

For molten salts, several recent TCL experiments have been conducted with KCl-$MgCl_2$-NaCl and Ni-based alloys showing low mass changes with dried or purified salt [11,12,20]. Monometallic type 316H TCLs have been conducted with a peak temperature of 650°C with FLiNaK and FLiBe with only small mass changes, Fig. 3 [13,14]. Characterization indicated only minor dissolution of Cr, Fe and Mn and an indication of mass transfer of Fe with specimen mass gains in the CL [13]. Fig. 3 also shows the mass change data from 1000 h 316H monometallic capsule tests at 550° and 650°C. In contrast to the TCL data, small mass losses were observed in both static experiments whereas mass gains were observed in the CL of the FLiNaK TCL.

Similar to Pb-Li, dissimilar TCL experiments are needed with graphite and beryllides (e.g., $Be_{12}Ti$) to better understand compatibility for fission and fusion applications before proceeding with forced convection flowing experiments where prototypical conditions can be achieved.




a b

*FIG. 2. Specimen mass changes after 1000 h in flowing Pb-Li as a function of estimated exposure temperature in a monometallic APMT TCL and a multimaterial TCL with SiC and a peak temperature of (a) 700°C with ODS Fe-10Cr-6Al [9] and (b) 650°C with pack aluminized F82H specimens [10] in the hot leg (HL) and cold leg (CL).*

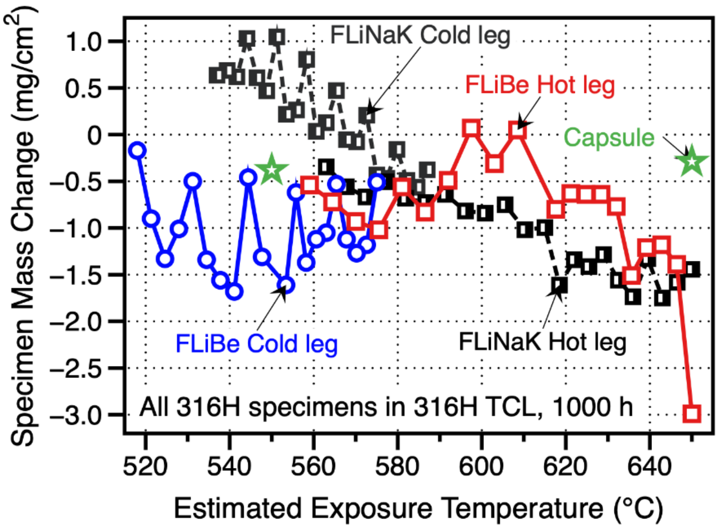



*FIG. 3. Specimen mass change of 316H specimens as a function of estimated exposure temperature in flowing FLiBe salt compared to values [13] for FLiNaK salt after 1,000 h. Static capsule mass changes in FLiBe at 550° and 650°C are shown as stars.*

## SUMMARY

Recent compatibility results were reviewed for fission and fusion applications. Flowing experiments allow more complete compatibility assessments including mass transfer and dissimilar material effects. Results obtained in static experiments often provide an incomplete assessment of compatibility. Recent results have confirmed the value of thermal convection loops in the progression towards evaluations in prototypical flowing conditions.

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