ORNL/SPR-2021/2138

FY–21 MSR Milestone M3AT-21OR0704012
FLiBe Capsule Testing of 316H

B. A. Pint
C. G. Parker
D. Sulejmanovic
Y.-F. Su

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August 2021

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B. A. Pint, C. G. Parker, D. Sulejmanovic, Y.-F. Su
Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN, 37831

Summary
This milestone was originally envisioned for completion in FY20 and was delayed due to the COVID-19 response and lack of FLiBe salt. In March 2021, FLiBe salt was received from Kairos Power. The nine capsule experiments with type 316H stainless steel specimens and 316 capsules were completed at 550° and 650°C for 1000 h to bracket the conditions expected in the subsequent thermal convection loop (TCL) experiment. In three capsules, Be metal was added for redox control. The specimen mass losses were low, similar to those observed previously for FLiNaK salt and the addition of Be reduced the average mass loss. Initial characterization suggested that some reaction with Be may have occurred. Therefore, Be was not included in the TCL experiment.

Introduction
Compatibility of type 316H stainless steel with molten fluoride salts has been identified as a key research area for molten salt reactor development [1]. The 1960's molten salt reactor experiment (MSRE) used Ni-based Hastelloy N (Ni-7Cr-16Mo) as a structural alloy [2] but current developers favor 316H because of its ready availability and ASME code qualification. Numerous studies have been conducted of steel compatibility in FLiBe with general agreement that Cr is selectively removed during FLiBe exposure[3-7]. The current 1000 h capsule experiments were mainly conducted as a safety test prior to a flowing salt thermal convection loop (TCL) experiment [8-10] to determine if the salt showed good compatibility, which suggests that it has reasonable purity.

Procedure
As outlined in prior reports [8,11-13], isothermal capsule testing was conducted in a 316H capsules (25 mm outer diameter x 100 mm tall x 1.2mm wall) that are welded shut in the Ar-filled glovebox to prevent contamination during loading or testing. To achieve a salt volume (cm^3) to specimen surface area (cm^2) ratio >10, small 316H coupons (~6 x 12 x 1-5 mm) were used with one specimen per capsule. The 316H composition was measured by inductively coupled plasma and combustion analyses as 64.5wt.%Fe-17.2%Cr-13.3%Ni-2.3%Mo-1.9%Mn-0.54%Si-0.06%C. The FLiBe salt was purified using HF to reduce O and OH impurities [14,15]. Capsules were loaded with ~28 g of salt in an Ar-filled glove box with impurity levels ≤1 ppm O_2 and H_2O. For same capsules, ~1-2 mg of 5 mm diameter Be wire was added as a getter [3,4]. The specimens were attached to one end using Mo wire and the capsules were welded shut using gas tungsten arc welding. Each capsule was then electron beam welded in vacuum into a larger type 304 stainless steel (SS) capsule to provide secondary containment and prevent the primary capsule from oxidizing. The capsules were exposed in a box furnace in laboratory air for 1000 h at 550°C or 650°C After the isothermal exposure, the capsules were inverted to allow the salt to drain away from the specimen. After cooling, the capsules were opened in the same glove box. The specimens were cleaned after exposure by using deionized water at 40°C. Before and after exposure, the specimens were weighed using a Mettler Toledo XP205 balance with an accuracy of ~±0.04 mg. To examine the depth of attack, the 9 specimens were metallographically

sectioned and polished for imaging by light microscopy. As the mass changes were low and the depth of attack was minimal, no further characterization was performed.

Results

Figure 1 summarizes the mass change results for the specimens exposed in a total of nine capsules in three conditions and compared to prior FLiNaK capsule results. All of the specimens showed a small mass loss and the whiskers show one standard deviation. As expected, the addition of Be reduced the average mass loss for the 316H specimens.

Figure 2 shows polished cross-sections of representative specimens from each condition. The depth of attack was very small suggesting low impurities in the FLiBe salt and no concern for the TCL experiment. For the specimens with Be, an outer surface layer was observed. Based on prior work, this may be associated with a Be reaction with the steel to form a NiBe phase. Because the Ni is removed from the matrix to form this phase, the layer may be ferritic.

No further characterization was performed on these specimens because of limited time and budget. Also, in the previous FLiNaK test campaign, the results were quite different between the capsule and TCL experiments [10]. The current specimens may be further characterized before writing a journal publication about the FLiBe TCL results.

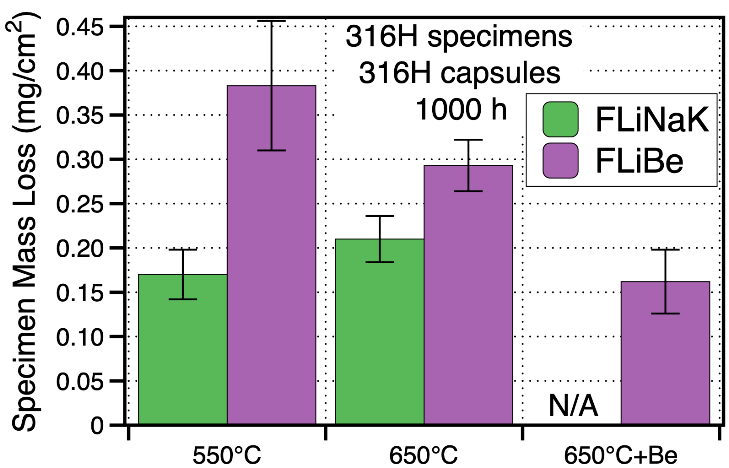

Figure 1. Specimen mass loss after exposure of 316H to FLiBe salt for 1,000h at 550°C and 650°C with and without Be additions. The results are comparable to prior results in FLiNaK salt.

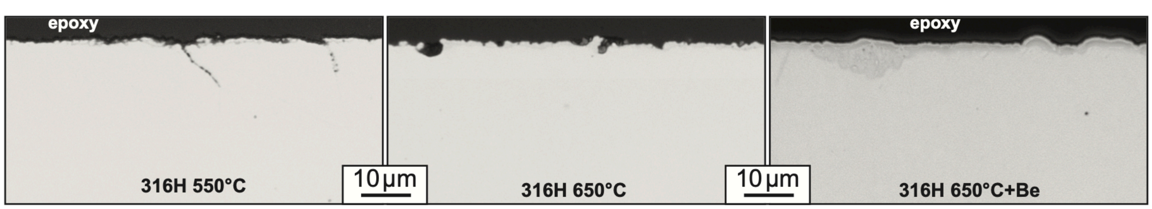

Figure 2. Light microscopy of polished cross-sections of one alloy 316 specimen after each exposure condition. The depth of attack was very small in each case.

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