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FY23 Milestone M3RD-23OR0603032
The Dissolution of Cr and Fe at 850°C in FLiNaK and FLiBe

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Introduction

Understanding the solubility of common alloying elements, such as Cr and Fe, in fluoride salts, can provide valuable insights into the degradation process of alloys in these salts and assist in modeling the attack observed in static and flowing conditions [1-2]. Previous isothermal dissolution experiments at 550°-750°C in FLiNaK showed little effect of time or temperature on the amount of dissolution, which was not expected. Therefore, the purpose of this milestone was to extend those observations to 850°C where Ni-based alloys have been developed [3] with 100+X better creep strength than the historic Hastelloy N alloy developed for molten salt reactors more than 50 years ago. Exposures were conducted in the same commercial FLiNaK and FLiBe salts used in the lower temperature exposures.

Procedure

An established static salt testing methodology was used for these experiments [2,4-6]. To facilitate dissolution, large Cr and Fe specimens (nominally 99.99% purity) measuring approximately 2 cm x 1 cm x 0.2 cm were cut and polished to a 600-grit finish and were weighed using a Mettler Toledo XP205 balance with an accuracy of ~±0.04 mg. The Cr specimens were mounted in low carbon arc cast Mo capsules (25 mm outer diameter x 100 mm tall x 1.2mm wall) using Mo wire. The Fe specimens were mounted using Ni-200 wire in 4340 steel capsules of the same size. The purified, commercial FLiNaK salt contained 300ppm Zr and 590 ppm O [2] as determined by inductively coupled plasma, optical emission spectroscopy (ICP-OES) and combustion analyses [7]. Characterization of the commercial FLiBe salt is in progress. Capsules were loaded with ~30g of salt in an Ar-filled glove box with impurity levels ≤1 ppm O_2 and H_2O. These primary capsules were shut inside the glovebox and then placed inside type 304 stainless steel outer capsules that were welded shut to protect the inner capsule and provide containment in the event of failure. The capsules were exposed in resistively heated box furnaces in laboratory air at 850°C. After the 100-2000 h isothermal exposures, the capsules were inverted to allow the salt to drain away from the specimen before the capsules were cooled and opened in the same glove box. The specimens were cleaned after exposure by using sonication in deionized water at 40°C. After cleaning, the specimens were weighed again to obtain specimen mass change.

Results

The mass change results for Cr specimens exposed to commercial FLiNaK salt in Mo capsules are shown in Figure 1 including prior experiments conducted at 550°-750°C. The previous experiments were conducted at 500-2000 h. However, since little effect of time was observed, the 850°C experiments included a 100 h exposure instead of 1000 h. At 850°C, increasing mass loss with time was observed but the amount of

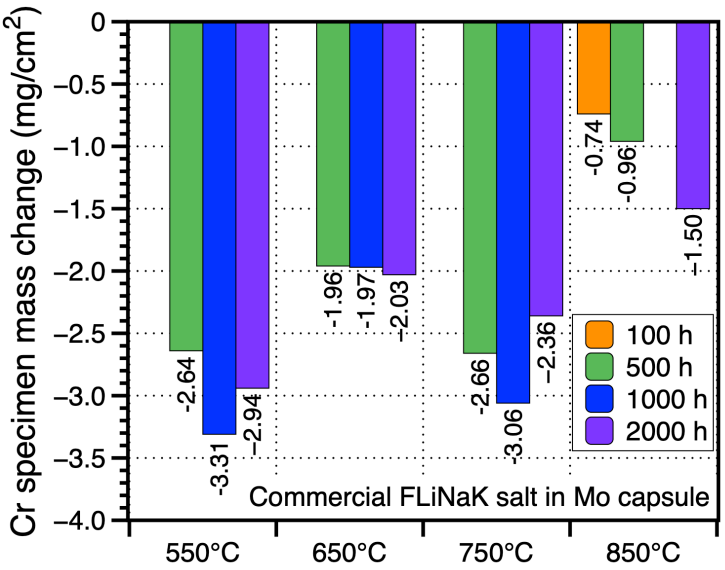

Figure 1. Specimen mass change of Cr specimens exposed at several times at each temperature to commercial FLiNaK salt in Mo capsules.

mass loss was lower than at the lower temperatures.

The ICP-OES analysis of the salt after exposure is shown in Figure 2. Consistent with the mass change data in Figure 1, much less Cr was detected in the salt after similar exposures at 850°C (the 100 h specimen was not analyzed). The Fe contents in the salt also were low at 850°C with similar values measured after testing at 750°C in the previous study. While there was an increase in the Cr content with time at 850°C, that was not observed for Fe in the steel capsules.

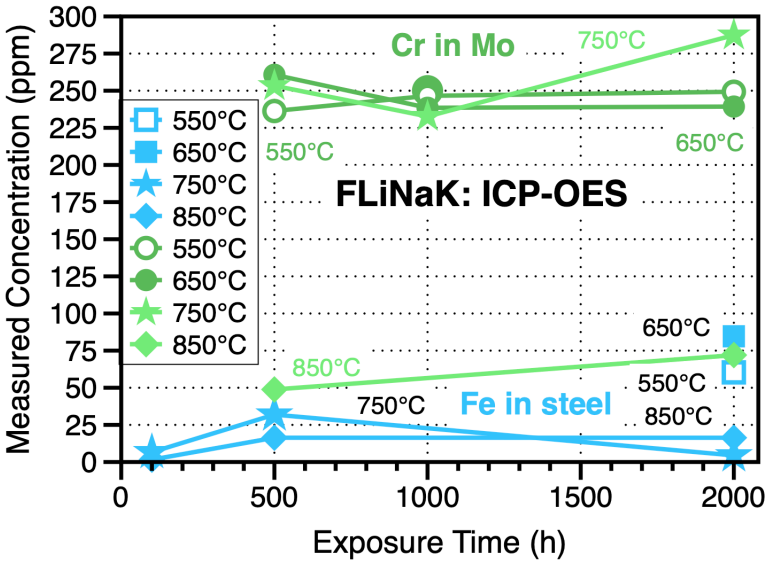

Figure 2. Measured Cr and Fe contents in commercial FLiNaK after exposures in Mo and steel capsules, respectively.

The black squares in Figure 3 show the mass change results of Cr specimens after 850°C exposures in FLiBe salt. A clear increase in mass loss with time was observed. Compared to the results at lower temperatures, the mass losses were higher at 850°C and showed more effect of exposure time. Figure 4 shows the Fe specimen mass change (black circles) after 850°C exposures in FLiBe salt. After a mass loss after 100 h, longer times at 850°C resulted in specimen mass gains. At lower temperatures, the mass losses decreased with increasing temperature, the opposite of what was observed for the Cr specimens. Compared to the Cr specimen mass losses, the mass losses were smaller for the Fe specimens. However, the 4340 steel capsule also was in contact with the salt which could have resulted in faster salt saturation with Fe. The mass gain of the Fe specimens may be due to other elements in the 4340 capsule dissolving and reacting with the Fe specimen. This reaction may increase with exposure time and temperature resulting in specimen mass gains. The Fe specimen surfaces need to be characterized to understand these results.

Because the commercial FLiBe salt was received in the middle of the year, the specimen and FLiBe characterization could not be completed during FY23. Salt chemistry measurements using ICP-OES are needed to compare the FLiNaK and FLiBe results at 850°C and fully understand the effect of time and temperature on Cr and Fe dissolution in these two salts.

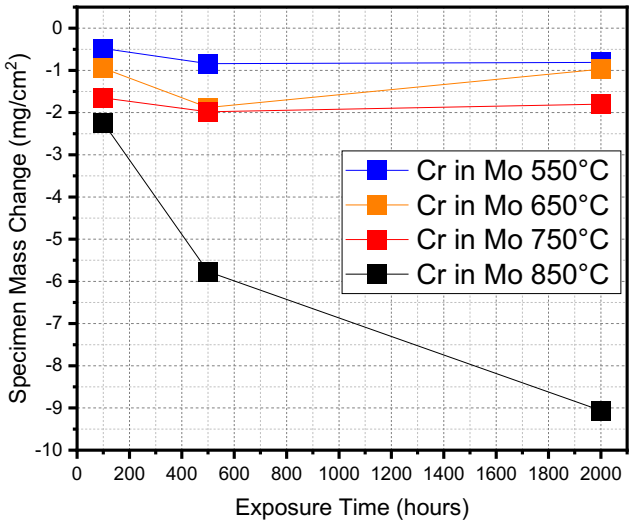

Figure 3. Specimen mass change of Cr specimens as a function of exposure time at 550°-850°C in commercial FLiBe salt in Mo capsules.

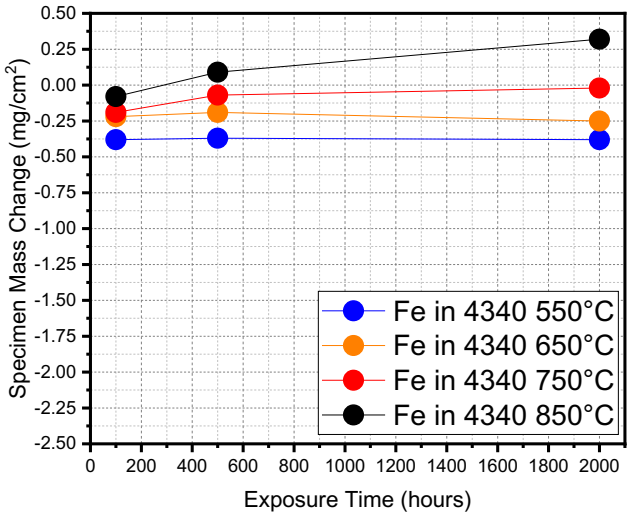

Figure 4. Specimen mass change of Fe specimens as a function of exposure time at 550°-850°C in commercial FLiBe salt in steel capsules.

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