



## Compatibility of container materials with Cr in molten FLiNaK salt



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### ARTICLE INFO

#### Article history:

Received 6 June 2014

Received in revised form 26 August 2014

Accepted 6 September 2014

Available online 16 September 2014

#### Keywords:

Molten salt

FLiNaK

Corrosion

Cr-rich film

### ABSTRACT

Corrosion tests of pure metal Cr were performed in molten FLiNaK (46.5 mol% LiF–11.5 mol% NaF–42 mol% KF) salt at 700 °C for 500 h using graphite crucible and Ni crucible, in order to investigate the interaction effect of Cr and the container materials in molten salt environments. Results indicate that the container materials accelerated the corrosion process owing to different electromotive potentials between metal Cr and the crucibles, and promoted formation of Cr-rich films on the surface of crucibles. The Cr-rich film on the graphite crucible was identified to be Cr<sub>7</sub>C<sub>3</sub> with a little amount of Cr<sub>23</sub>C<sub>6</sub>. Whereas the Cr deposited on the Ni crucible surface and formed Ni–Cr alloy by thermal diffusion mechanism.

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### 1. Introduction

Molten salt reactor (MSR), one of the Generation-IV nuclear reactors, has attracted much more attention in recent years. Using molten fluoride salt mixtures as the primary coolant or even the fuel itself, the MSR has many desirable properties, such as simplified design, high thermodynamic efficiency, inherent safety, etc. [1–3]. However, the molten fluoride salts are highly corrosive, especially at high temperatures. Therefore, corrosion of the structural materials in molten fluoride salt environments is one of the main problems that prevent the MSR from long time operation [3,4].

As molten salts have many advantages (high thermal conductivities, low viscosities, high boiling points, etc.) for using as reactor coolants or energy storage fluids in industry, lots of work has been done to study the corrosion behaviors and the corrosion mechanism of materials in molten salt environments [5–9]. Corrosion of various kinds of materials in molten FLiNaK salt showed that the Cr composition of these alloys was selectively attacked and the weight loss correlated directly with the initial Cr and C contents of the alloys [9]. Although Cr is one of the most vulnerable elements in molten salt environments, it is widely used in high

temperature alloys because it could improve the high temperature oxidation resistance of materials. Therefore, much attention has been paid to investigate of Cr corrosion and movement in molten salt environments.

During the tests, some research results show that the corrosion of the alloys was affected not only by the salt components but also by the container materials in static molten salt corrosion tests [10]. Meanwhile, the elements (e.g. Cr) dissolved into the salts would react with the container materials during the corrosion tests [9], which might affect the material properties. In the static corrosion tests of several high temperature alloys exposed to NaF–27CaF<sub>2</sub>–36MgF<sub>2</sub> molten salt, Misra et al. [7] found that the corrosion depth of the sample in much greater in quartz crucible than that in alumina crucibles. Kondo et al. [11] also revealed that the corrosion rate of reduced activation ferritic steel, JLF-1 (8.92Cr–2W) in molten FLiNaK salt was larger by 3 times in Ni crucible compared to the JLF-1 crucible. Additionally, the elements dissolved into the molten salts could react with container materials. Ozeryanaya [10] has found that, in molten alkali and alkaline-earth chlorides heat treatment baths, the Zr dissolved into the molten salt could transfer to the experimental crucibles and formed 0.02 cm thick Zr–Mo layer and ZrC particles on the surface of the molybdenum crucible and glass–carbon crucible, respectively. In the static corrosion tests of Incoloy 800H in graphite crucible, Olson et al. [9] found that a Cr-rich grey film was formed on the graphite crucible. The film was identified to be Cr<sub>7</sub>C<sub>3</sub> using XRD analysis. It was said that the Cr dissolved into the FLiNaK salt would deposit onto the graphite crucible surface due to the activity

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gradient, which increased the Cr dissolution rate during the tests. Because the MSR reactor designs contain large quantities of graphite and nickel-based alloys, and a similar Cr transfer behavior, Cr was selectively removed in the hot section and deposited in the cold section, was also found in the thermal convection and pump loops experiments conducted in Oak Ridge National Laboratory [12], so the interaction effect of Cr with graphite and alloys should be detailed investigated in the molten salt environments. However, to our knowledge, the interaction effect of Cr and other materials in molten fluoride salt environments are scarcely studied and are therefore highly desirable.

With this background, static corrosion tests were undertaken to determine the corrosion behaviors of metal Cr in molten FLiNaK salt at 700 °C for 500 h using graphite crucible and Ni crucible. The purpose of this research is to investigate the mutual effect mechanism of Cr with the container materials in molten salt environments. Results show that Cr-rich films were detected on the walls of both crucibles after the tests. The corrosion behaviors of metal Cr in graphite crucible and Ni crucible are compared and discussed.

## 2. Results and discussion

### 2.1. Mass changes and surface morphology of metal Cr

The weight losses of metal Cr tested in molten FLiNaK salt at 700 °C for 500 h are presented in Fig. 1, showing that the weight loss of the samples tested in graphite crucible are more serious. The average weight loss of the sample tested in the graphite crucible is about 24 mg/cm<sup>2</sup> larger than that in Ni crucible after the corrosion tests. Since the salt was purified by a hydrofluorination purification process and all the salt handling operations were done in the glove box, the effects of impurities on corrosion were not specifically investigated. The solubility of Cr is 2700 ppm in molten FLiNaK containing 2.5 mol% UF<sub>4</sub> at 800 °C [12], combining the solubility of Cr and the quantity of the salt used in the tests, the weight loss of Cr should be lower than 1.16 g after the corrosion test in molten FLiNaK salt at 700 °C, because high temperature and UF<sub>4</sub> have a high effect on the extent of material corrosion [4]. However, the total weight loss of samples tested in graphite crucible was larger than it. The above results suggest that the container materials play a role on corrosion of the alloys in molten salts corrosion tests. As in the previous study [9,13], the graphite accelerated the corrosion process when graphite was presented in molten salt corrosion tests. It was said that the graphite is more electropositive than Cr and galvanic corrosion occurred when graphite and alloys are in electrical contact, which is discussed in the later section.

Fig. 2 shows the plan-view SEM images of the metal Cr after the corrosion tests. According to the morphology, the plan-view image of the sample tested in graphite crucible (Fig. 2a) is almost the same to that in Ni crucible (Fig. 2b). It is noted that, in addition to intergranular corrosion, some pits are also observed on the

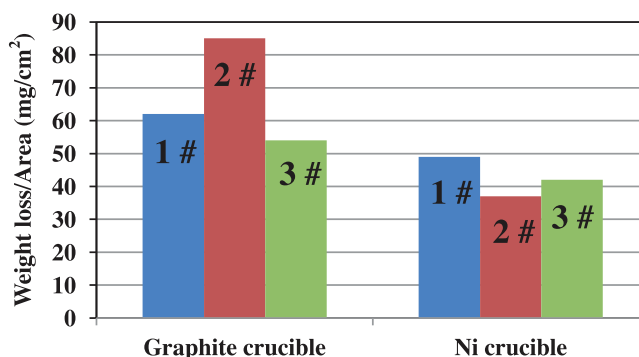


Fig. 1. Weight loss of metal Cr after exposure to molten FLiNaK salt at 700 °C for 500 h in graphite crucible and Ni crucible.

samples, which is due to dislocation or structural defects in the metal Cr [14]. Additionally, the SEM/EDS analysis did not reveal any positive signs of carburization of the Cr tested in graphite crucible, which is consistent with the results that no carburization occurred in Inconel, Hastelloy-N and Hastelloy-B when they were tested in molten fluoride salts with graphite [15,16]. Results suggested that container materials did not alter the corrosion pattern, although the weight losses of the samples tested in graphite crucible are more serious.

### 2.2. Surface morphology of Cr-rich film

A compact gray film was formed on the graphite crucible and holding rod after the corrosion test in molten FLiNaK salt at 700 °C for 500 h. As shown in Fig. 3, the graphite holding rod was covered by the compact gray film, with some sparsely distributed flower-like grains on the surface of the film (Fig. 3b). According to the morphology and the EDS analysis, the main composition of the gray film was Cr and C, while Cr was enriched at the flower-like grains compared to the other part of the film (Fig. 3b and Table 1). According to Fig. 3c, the thickness of the compact Cr-rich gray layer was about 5 μm.

Fig. 4 shows the morphological SEM images of the surface and cross-section of Ni crucible holding rod after the corrosion test in molten FLiNaK salt at 700 °C for 500 h. The surface of the Ni holding rod was rough and covered with irregular polyhedral grains (Fig. 4a) and the EDS analysis revealed an enrichment of Cr on the surface. Though the surface was coarse, EDS point analysis showed a reasonably uniform Cr content on the surface of the crucible. According to the EDS cross-sectional line scan, the Cr composition depth profile was about 60 μm (Fig. 4b).

According to the SEM/EDS analysis, there was Cr deposited on the surface of the containers for both of the corrosion tests. Combining with the weight loss of metal Cr samples and Cr concentration of the salts after the corrosion tests (Table 2), it was calculated that 1.28 g and 0.86 g Cr deposited on the graphite

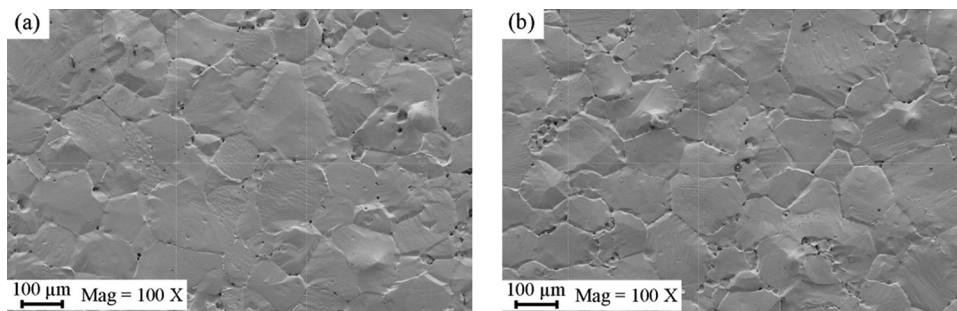


Fig. 2. Plan-view SEM images of the metal Cr after corrosion tests in molten FLiNaK salt at 700 °C for 500 h. (a) Graphite crucible, and (b) Ni crucible.

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