

# The protective performance of a molten salt frozen wall in the process of fluoride volatility of uranium

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Abstract The fluoride volatility method (FVM) is a technique tailored to separate uranium from fuel salt of molten salt reactors. A key challenge in R&D of the FVM is corrosion due to the presence of molten salt and corrosive gases at high temperature. In this work, a frozen-wall technique was proposed to produce a physical barrier between construction materials and corrosive reactants. The protective performance of the frozen wall against molten salt was assessed using FLiNaK molten salt with introduced fluorine gas, which was regarded as a simulation of the FVM process. SS304, SS316L, Inconel 600 and graphite were chosen as the test samples. The extent of corrosion was characterized by an analysis of weight loss and scanning electron microscope studies. All four test samples suffered severe corrosion in the molten salt phase with the corrosion resistance as: Inconel 600 > SS316L > graphite > SS304. The presence of the frozen wall could protect materials against corrosion by molten salt and corrosive gases, and compared with materials exposed to molten salt, the corrosion rates of materials protected by the frozen wall were decreased by at least one order of magnitude.

Qiang Dou douqiang@sinap.ac.cn **Keywords** Molten salt reactors · Fluoride volatility method · Corrosion · Frozen wall · Protective performance

## **1** Introduction

Molten salt reactors (MSR) are a unique type of reactor in the six candidate Gen-IV reactors [1, 2], in which the liquid fuel consists of fluoride fuel and fluoride coolant salt circulates in the primary loop of the reactor. Due to the properties of MSR fuel, pyroprocessing technologies, which have the advantages of irradiation resistance, low critical risk and less radioactive waste, have been considered as the most suitable option for the treatment of MSR fuel [3, 4]. Pyroprocessing technologies, which have been developed or are under development for treatment of MSR fuel, include the fluoride volatility method (FVM), vacuum distillation and electrochemical separation. However, construction materials of equipment exposed to fluoride melt and corrosive gases at high temperature will suffer from severe corrosion, especially in the process of fluoride volatility of uranium. The reported corrosion rate of Hastelloy N alloy exceeded 25 µm/h when exposed to fuel salt of the molten salt reactor experiment (MSRE) [5]. In 1970s, the China Institute of Atomic Energy (CIAE) invested much effort in developing the FVM to treat used oxide fuel, but in the end they terminated this research because of serious corrosion of equipment and engineering problems for scale-up [6]. In the fuel salt processing flowsheet [7] proposed by the Shanghai Institute of Applied Physics (SINAP) for the thorium molten salt reactor nuclear energy system (TMSR), FVM was also adopted to separate uranium from used fuel salt, and research showed that corrosion-induced weight loss of the fluorinator was

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significant, and the content of Cr, Fe, Ni and Mo in the molten salt also increased significantly [6] after the fluoride volatility experiment was conducted, even when Hastelloy C-276 was used in the construction of the fluorinator. Corrosion will not only shorten the lifetime of a facility and increase the volume of the secondary radioactive waste, but also pollute the fuel salts.

Corrosion of the fluorinator during the process is a key issue that has limited the smooth development of the FVM. The existing method for corrosion prevention is to form a protective coating or a dense layer of oxide on the inner surface of the reactor [8]. However, corrosion in fluorides is characterized by diffusion of the active element alloy and the dissolution on the alloy/molten salt interface and the protective films mentioned above are unstable in hightemperature fluoride salt [6]. Until now, there have been no better ways for corrosion prevention in fluoride salts. The development of a new corrosion-resistant material is expensive, difficult and time-consuming, so the most suitable choice available is to optimize the existing technology and make use of the characteristics of molten salt. A frozen wall of molten salt with the same composition of the molten medium acting as protective layer might solve the issue.

A frozen wall can form and be maintained on the inner wall of the fluorinator by controlling the temperature difference between its outer and inner walls with forced cooling. Similar technologies, including a cold crucible, have been used in other industries [9, 10], but the study of frozen-wall technology in molten fluoride salt systems has rarely been reported. Takeuchi et al. [11] reported corrosion tests of several kinds of alloys in 2CsCl-NaCl molten salt with the introduction of chlorine gas in which alloys were covered with a frozen wall of molten salt and showed that promising materials such as Hastellov C-22 exhibited good corrosion resistance under pyrochemical conditions. Because of the much more severe corrosive environment during fluoride volatility of the MSR fuel salt, it is necessary to study the feasibility of the use of frozen-wall technology. In this paper, the concept of the frozen wall and our demonstration test results are presented and the protective performance of a molten salt frozen wall was determined in FLiNaK (LiF-NaF-KF: 46.5-11.5-42 mol.%) molten salt coupled with the introduction of fluorine, which was regarded as a simulation of the FVM process. In addition, the influence of additional factors affecting the protective performance of the frozen wall is discussed.

# 2 The concept of frozen wall and demonstration test

The frozen wall can form and adhere to the inner surface of a fluorinator by controlling the temperature difference between its outer and inner walls. The key factor is to control the balance of internal heat and outer wall cooling. To predict the characteristic behavior of frozen walls, a single vessel with finite radius R was considered for analysis [12, 13].

As shown in Fig. 1, during the formation of the frozen wall, the molten salt is at a state with an initial temperature  $(T_i)$  greater than the melting point or fusion temperature  $(T_f)$ . With the cooling medium flowing through the outer surface of the metallic wall, molten salt closest to the inter wall begins to solidify and the solid–liquid interface moves away from the heat-transfer surface, and the surface heat



Fig. 1 (Color online) Physical analysis model for cylindrical vessels with a molten salt frozen wall: a sectional model and b temperature distribution

flux decreases due to the increasing thermal resistance of the frozen salt. When the rate of heat transfer reaches an equilibrium, the temperature gradient within the frozen wall ranges between  $T_1$  and  $T_f$ . The heat flux can be calculated by from the following equation:

$$q = 2\pi\lambda \frac{T_{\rm f} - T_1}{\ln \frac{R}{R_{\rm f}}},\tag{1}$$

where q is heat flux (W/m),  $\lambda$  is the thermal conductivity coefficient of solid salt (W/m °C),  $T_{\rm f}$  is the fusion temperature of molten salt (°C),  $T_{\rm 1}$  is the temperature between the frozen wall and the metallic wall (°C), R is the inside radius of cylindrical vessel (m) and  $R_{\rm f}$  is the inside radius of the vessel with frozen wall (m).

The temperature of the outer surface of the metallic wall  $(T_0)$  is related to convective heat transfer between the cooling medium and the metallic wall.  $T_1$  is approximately equal to  $T_0$  because the thermal conductivity coefficient of metal is much higher than that of solid salt. The relationship between the thickness of the frozen wall and heat-transfer flux of cooling medium can be established from Eq. (1).

The experimental equipment to demonstrate frozen-wall technology was constructed in SINAP [14–16]. Figure 2 shows the schematic of the test container, which is up-flow tapered with a heat exchange jacket. Thirty-three thermocouples positioned at three different heights in the test container were used to measure the radial temperature, and the measurements were used to monitor the formation of a frozen wall and its thickness.

During the experiment, molten salt was pumped into the test container from a storage tank and then kept stationary or continuously flowing according to the experimental purpose. With control of the heat exchange, a certain thickness of frozen wall can form and adhere to the inner surface of the test container. In order to master the behavior of different molten salt systems, a molten nitrate salt (NaNO<sub>2</sub>-NaNO<sub>3</sub>-KNO<sub>3</sub>: 40.0-7.0-53.0 wt.%) and a molten fluoride salt (LiF-NaF-KF: 46.5-11.5-42 mol.%, FLiNaK) were used successively. The weight of molten salt loaded in the device was about 200 kg.

Some demonstration test results [17–19] indicated that the formed frozen wall was a uniform layer, attaching to the metallic surface tightly. In our research, the thickness of the frozen wall changed from several millimeters to centimeters according to the rate of heat exchange, which could be calculated from radial gradient temperatures measured by thermocouples. The temperature gradient and the thickness of the frozen wall remained stable for dozens of hours. Analytical solutions to determine the interface location at various time intervals were established and validated. This test demonstrated that the frozen wall can form and be maintained by controlling the temperature gradient, and it should have a protective effect on the reactor wall by preventing direct contact between it and the corrosive gases and molten salt.

#### **3** Protective performance experiments

A series of specific experiments was conducted to confirm the protective performance of the frozen wall in a simulated pyroprocessing environment. The equipment for the protective performance tests was designed such that it could be conducted in molten salt and included the introduction of fluorine gas. The schematic of the protective performance test equipment is shown in Fig. 3. The equipment was installed in a glove box covered with an argon atmosphere. It mainly consisted of an electric heating furnace (3 kW), a nickel crucible ( $\Phi$ 150 mm × H200 mm), a cooling rod, a set of thermocouples and various accessories for operation. The cooling rod was



Fig. 2 Schematic of the container used for a demonstration test



Fig. 3 Schematic of the protective performance test device for molten salt frozen wall

made of Inconel 600 with a concentric jacket structure. There were two electric lifting devices, one for the cooling rod and the other for the furnace cover. The maximum stroke of the lift for the cooling rod was 100 mm. The test samples and cooling rod could be synchronously lifted. There were six thermocouples arranged in a radial direction, and the distances from the outside surface of the cooling rod were 0 mm, 3 mm, 5 mm, 10 mm, 20 mm, and 30 mm successively.

As shown in Fig. 3, before the experiments, test samples were attached to the outside surface of the cooling rod and wrapped with nickel wire. A control group of test samples was placed relatively far away from the cooling rod to ensure full exposure to the molten salt phase during the entirety of the experiment.

During the experiment, the salt was heated to 510 °C and then kept at 510 °C for 6 h to ensure the salt was melted completely. After that, the test samples and cooling rod were lowered into the molten salt with air cooling. The temperature of salt near the cooling rod began to decrease. When the surface temperature of the test samples reached a temperature lower than the melting point (454 °C) of the salt, a layer of solidified salt should be formed and deposited on the samples. The thickness of the frozen wall was determined by measuring the temperature gradient using the thermocouples [13] and confirmed by the measured result with caliper after the experiment. When the thickness of the frozen wall reached 7-10 mm and remained stable, the fluorine gas (20-80 Vol.% F<sub>2</sub>/Ar mixture) was bubbled through the bottom probe tube for 12 h. During this process, the thickness of the frozen wall was controlled by adjusting the cooling air flow rate. The test conditions are summarized in Table 1.

SS304, SS316L, Inconel 600 and graphite were chosen as the test samples. The length, width and thickness of the samples were 20 mm, 10 mm and 3 mm, respectively. All the samples were ultrasonically cleaned in acetone and distilled water, followed by hot air drying before the experiment. The dimensions and weight of all samples were measured. FLiNaK salt with a melting point of 454 °C was selected for this experiment. The weight of the salt prepared for the corrosion test was approximately 3.5 kg (the liquid level of the molten salt was approximately 100 mm), and the moisture in the salt was thoroughly removed by preheating. Impurities present in the FLiNaK salt dramatically influence the extent of corrosion [8]. For the present study, since the main purpose was to compare the relative corrosion performance of materials protected by a frozen wall with unprotected materials, the influence of trace impurities on corrosion was not specifically investigated.

After the corrosion test, the test samples were removed with the cooling rod from the molten salt. The unprotected samples were easily taken out, while the protected samples were covered with solid salt and their extraction required removal of the solid salt. All the samples were thoroughly washed with 1 mol/L KNO<sub>3</sub> solution, distilled water and then dried. The samples were evaluated by measuring weight change and scanning electron microscopy (SEM, LEO1530VP, ZEISS, Germany).

The corrosion rates of the test samples were calculated from their weight loss according to the following equation:

Corrosion rate 
$$(\mu m/h) = \Delta M \times 10^6/(\rho \times A \times t),$$
 (2)

where  $\Delta M$  is the weight loss (g), t is the immersion time (h), A is the surface area (mm<sup>2</sup>) and  $\rho$  is the density of the sample (g/cm<sup>3</sup>).

In addition, the surface and cross section of the samples were observed and compared by SEM.

#### 4 Results and discussion

Figure 4 shows representative variation of the temperature gradient during the corrosion test of Inconel 600; the different curves represent the change of temperature at different positions. At the beginning of the formation of the frozen wall, the cooling rod attached with samples was immersed into molten salt, the air produced by the compressor passed into the cooling rod. As shown in Fig. 4, the temperature of molten salt close to the cooling rod decreased and reached the melting point in 50 min. At about 100 min, the temperature near the surface of the cooling rod (0 mm, 3 mm and 5 mm) was much lower than

**Table 1** The experimentalconditions during the protectiveperformance test

| Test materials                          | SS304, SS316L, Inconel 600, graphite    |
|---|---|
| Salt                                    | FLiNaK (46.5-11.5-42 mol.%)             |
| Temperature of molten salt              | 510 °C                                  |
| Flow rate of F <sub>2</sub> /Ar mixture | 200 mL/min                              |
| Cooling medium                          | Air, 25 °C, 2.7–3.4 m <sup>3</sup> /min |
| Position of samples in crucible         | Frozen-wall phase, molten salt phase    |
| Thickness of frozen wall                | 7–10 mm                                 |
| Immersion or exposure time              | 14 h                                    |



Fig. 4 (Color online) The temperature gradient during the corrosion test (Inconel 600)

the melting point, the thickness of the frozen wall was considered to have reached the set point and the thickness of the frozen wall was maintained at the set point by finetuning the cooling air flow rate. Fluorine gas was introduced into the bottom of molten salt with a flow rate of 200 mL/min at 120 min and gas flow was maintained for 12 h, similar to the duration used in the fluoride volatility process. During corrosion tests, the temperature of the frozen wall was stable at approximately 425 °C, while the temperature of the molten salt phase was about 480 °C. Figure 5 shows photographs of samples and cooling rod covered with a frozen wall after the corrosion test. The sample attached to the central cooling rod was uniformly covered by a layer of solidified salt; the thickness of the frozen wall was about 7 mm.

Figure 6 shows photographs of the samples in the frozen wall and molten salt phases after the corrosion test. The samples were thoroughly cleaned to make sure all the adhered salt was removed. Visual inspection revealed the corrosion of the samples. All samples protected by the frozen wall were visually almost entirely intact after the corrosion test, while the samples immersed in molten salt



Fig. 5 (Color online) Photographs of samples after the corrosion test

were extensively corroded to various degrees. Samples of SS304 and graphite immersed in molten salt had suffered severe corrosion. The degree of corrosion of SS316L and Inconel 600 in molten salt appeared slightly smaller (Fig. 6). The corresponding weight loss of different sample materials is listed in Table 2. For all samples, the weight loss ratio in the frozen-wall phase was much less than that in the molten salt phase. This demonstrated that the frozen wall was effective in corrosion protection. The sequence of corrosion resistance for different samples materials in molten salt was: Inconel 600 > SS316L > graphite >SS304. Metals with high Ni content have good corrosion resistance [18]. For the three alloys tested in the present experiment, the Ni content in Inconel 600 was the highest  $(\sim 72\%)$  and demonstrated the best corrosion resistance. The Ni contents of SS316L and SS304 were similar  $(\sim 10\%)$ , but SS316L also contains Mo elements ( $\sim 2\%$ ), which enhance corrosion resistance. Consequently, the corrosion resistance of SS316L was better than SS304. The corrosion of graphite mainly arose because of the reaction between graphite and F<sub>2</sub>.

Figure 7 shows the corrosion rates of samples after the corrosion test, and the corrosion rates of each material represent the average of at least three samples. The corrosion rates of SS304 and graphite exceeded 60  $\mu$ m/h. The corrosion resistance of Inconel 600 was slightly better than the other three materials when exposed to molten salt. The frozen wall imparted good corrosion resistance, and the corrosion rates of samples covered with frozen wall were all less than 1.4  $\mu$ m/h. For Inconel 600, the corrosion rate was reduced from 8.1  $\mu$ m/h in the molten salt phase to 0.17  $\mu$ m/h in the frozen wall. The decreased rate of corrosion of the four materials spanned more than one order of magnitude.

Theoretically, the samples protected by the frozen wall should not have been corroded. However, as shown in Table 2 and Fig. 7, all the samples in the frozen-wall phase also suffered various degrees of corrosion, and the sequence of corrosion resistance was the same as that observed in the molten salt phase. We speculated that corrosion was occurring during the formation of the frozen wall. In order to confirm this, a series of comparative experiments was undertaken. All the experimental conditions were the same as described above in the corrosion test except the samples were taken out for analysis as soon as the thickness of frozen wall reached the set point. The total time of this formation process was 120 min. Figure 8 shows the corrosion depth of samples in the frozen-wall phase in these comparative experiments. The corrosion depths during formation of the frozen-wall phase were very close to those going through the whole process of protective performance test. Once the frozen wall had formed and the thickness remained at the set point, the test samples Fig. 6 (Color online) Photographs of samples in the frozen-wall phase (left) and molten salt phase (right) after the corrosion test: **a** SS304, **b** SS316L, **c** Inconel 600 and **d** graphite



Table 2 The weight loss of different materials measured immediately after formation of the frozen wall

|                                   | SS304             |                   | SS316L               |                      | Inconel 600          |                      | Graphite          |                      |
|-----------------------------------|-------------------|-------------------|----------------------|----------------------|----------------------|----------------------|-------------------|----------------------|
|                                   | Frozen-wall phase | Molten salt phase | Frozen-wall<br>phase | Molten salt<br>phase | Frozen-wall<br>phase | Molten salt<br>phase | Frozen-wall phase | Molten salt<br>phase |
| Original weight (g)               | 4.230             | 4.216             | 4.578                | 4.556                | 4.625                | 4.614                | 1.055             | 1.050                |
| Weight after<br>corrosion test(g) | 4.141             | 0.501             | 4.550                | 3.018                | 4.616                | 4.120                | 1.049             | 0.347                |
| Weight loss(g)                    | 0.089             | 3.715             | 0.028                | 1.538                | 0.009                | 0.494                | 0.006             | 0.703                |
| Weight loss ratio (%)             | 2.1               | 88.1              | 0.6                  | 33.8                 | 0.2                  | 10.7                 | 0.6               | 67.0                 |





covered with frozen wall were well protected and little affected by molten salt and oxidizing gases.



Fig. 8 Corrosion depth of several materials in the frozen-wall phase

The surface and cross section of the Inconel 600 sample was viewed using an SEM after the corrosion test (Figs. 9 and 10). For the sample protected by the frozen wall (Fig. 9a), the surface was smooth and without obvious



Fig. 9 Surface view SEM images of Inconel 600 after corrosion test: a sample protected by the frozen wall and b sample exposed to molten salt

voids and damage. The surface of the sample exposed to molten salt (Fig. 9b) was more porous and exhibited visible destruction. Corrosion was observed on the surface with pit sizes ranging between 2 and 10  $\mu$ m.

The cross-sectional image of the sample exposed to molten salt (Fig. 10b) shows grain boundary corrosion to a depth of 15.2–19.5  $\mu$ m below the surface. For the corresponding sample protected by the frozen wall, the depth of corrosion was less than half of this (Fig. 10a).



Fig. 10 Cross-sectional SEM images of Inconel 600 after corrosion test: **a** the sample protected by the frozen wall and **b** the sample exposed to molten salt

Figure 11 shows the SEM–EDS spectra of the phases labeled as 1 in Fig. 9b and shows the relatively high F content on the surface of Inconel 600 exposed to molten salt. The main constituents of Inconel 600 are Fe, Cr and Ni (1:1.7:7.2, respectively). After the corrosion test, the proportion on the surface of Inconel 600 sample changed to 1:1.6:7.7, respectively, which indicated that Fe and Cr were more reactive than Ni in the experimental environment and fluorides formed on the surface of the sample.



Fig. 11 (Color online) EDS pattern of Inconel 600 exposed to molten salt

These results indicated that the corrosion rates of all materials were significantly decreased when they were under the protection of the frozen wall. In the gas–liquid bubbling reaction environment, metals or carbon in the sample can react with fluorine gas or free fluorine ions [20–22], as shown in Eqs. (3) and (4):

$$2\mathbf{M} + x\mathbf{F}_2 \to 2\mathbf{M}\mathbf{F}_x,\tag{3}$$

$$\mathbf{M} + n\mathbf{F}^{-} \to \mathbf{M}\mathbf{F}_{n} + n\mathbf{e}^{-},\tag{4}$$

where M is a metallic element or carbon (M = Cr, Fe, Ni, Mo, C) and Cr is the most reactive metal [18] in common alloying elements, and  $MF_x$  or  $MF_n$  is the corresponding metallic ion, whatever its exact speciation in the fluoride melt (with 0 < n < 6).

Some fluorides produced by corrosion can dissolve in the molten salt, while some are insoluble and will deposit at the bottom or float on the surface of the salt. Volatile substances, such as CF<sub>4</sub>, may also form by the reaction between carbon and fluorine gas. This conclusion was verified by the color change of the molten salt and the presence of powdered material in the furnace observed after the corrosion test. In this experiment, fluorine gas and free fluorine ions in molten salt are the main sources of corrosion reactants. Impurity ions could also contribute to corrosion [6]. When samples were exposed to molten salt, fluorides on the surface of samples that were being constantly produced by corrosion were continuously dissolved by molten salt, which deepened the corrosion. In this experiment, under identical experimental conditions, corrosion rates depended on the sample's intrinsic corrosion resistance.

These experiments indicated that the frozen wall can act as a physical barrier between construction materials and corrosive reactants, and these materials can therefore withstand severe condition as long as the frozen wall is well controlled. Although corrosion cannot be avoided during the formation of the frozen wall, because the period of formation of the frozen wall is relatively short, corrosion is strictly limited and will not affect the safe operation of the equipment. To reduce corrosion during the formation of frozen wall, it is better to choose an alloy with relatively good intrinsic corrosion resistance as the container material and use high-purity salt without nuclide to form the initial frozen wall when applied in the pyroprocessing of spent fuel.

### 5 Conclusion

The protective performance of frozen walls on corrosion of construction materials was assessed using FLiNaK molten salt with introduced fluorine gas, which was considered to simulate the FVM process. SS304, SS316L, Inconel 600 and graphite were chosen as the test samples. During the whole corrosion test period, the thickness of the frozen wall was kept within the range of 7-10 mm to ensure the samples well covered. The extent of corrosion was characterized by weight loss and SEM. All four test samples suffered severe corrosion when exposed to molten salt. The ranking of corrosion resistance was: Inconel 600 > SS316L > graphite > SS304. The frozen wall could protect these materials against corrosion induced by molten salt and corrosive gases. Although corrosion could not be avoided during the formation of the frozen wall, compared with materials exposed to molten salt, the corrosion rates of these four materials protected by the frozen wall were still decreased by at least one order of magnitude. These results indicated that the frozen wall can act as a physical barrier between construction materials and corrosive reactants, and these materials can stand severe condition as long as the frozen wall is well controlled.

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