Analysis of Corrosion of Hastelloy-N, Alloy X750, SS316 and SS304 in Molten Salt High-Temperature Environment

Ketan Kumar Sandhi and Jerzy Szpunar*

Department of Mechanical Engineering, College of Engineering, 57 Campus Drive, University of Saskatchewan, Saskatoon, SK S7N 5A9, Canada; ketan.sandhi@usask.ca
* Correspondence: jerzy.szpunar@usask.ca

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Abstract: Nickel superalloy Hastelloy-N, alloy X-750, stainless steel 316 (SS316), and stainless steel 304 (SS304) are among the alloys used in the construction of molten salt reactor (MSR). These alloys were analyzed for their corrosion resistance behavior in molten fluoride salt, a coolant used in MSR reactors with 46.5% LiF + 11.5% NaF + 42% KF. The corrosion tests were run at 700 °C for 100 h under the Ar cover gas. After corrosion, significant weight loss was observed in the alloy X750. Weight loss registered in SS316 and SS304 was also high. However, Hastelloy-N gained weight after exposure to molten salt corrosion. This could be attributed to electrochemical plating of corrosion products from other alloys on Hastelloy-N surface. SEM–energy-dispersive X-ray spectroscopy (EDXS) scans of cross-section of alloys revealed maximum corrosion damage to the depth of 250 µm in X750, in contrast to only 20 µm on Hastelloy-N. XPS wide survey scans revealed the presence of Fe, Cr, and Ni elements on the surface of all corroded alloys. In addition, Cr clusters were formed at the triple junctions of grains, as confirmed by SEM–EBSD (Electron Back Scattered Diffraction) analysis. The order of corrosion resistance in FLiNaK environment was X750 < SS316 < SS304 < Hastelloy-N.

Keywords: MSR; corrosion resistance; Hastelloy-N; alloy X-750; stainless steel 316; stainless steel 304; high temperature corrosion

1. Introduction

Molten salt reactors (MSRs) are among six of the Gen IV nuclear reactor concepts. These reactors use molten salt mixed with nuclear fuel as a heat transfer media. Since molten salt remains in liquid state during the operation of MSR, the reactor operates at near atmospheric pressure, which reduces the chances of leakages and explosions. Moreover, MSR operates at relatively higher temperatures (700–850 °C), which results in higher thermal efficiency [1]. These molten salt reactors have negative coefficient of reactivity, which makes MSR passively safe reactors [2,3]. Some MSR design concepts have been already proposed—Seaborg Technologies Wasteburner (SWaB) reactor burns the spent fuel from light water reactors (LWRs), and this allows for the minimization of nuclear waste [4]. Another design, advanced high temperature reactor (AHTR), has high thermal efficiency, large power output (3400 MW [th]), and uses thorium as fuel [5].

The chloride and fluoride salt mixtures have been proposed for use in the MSR [6]. The high temperature stability, high heat capacity, low vapor pressure, high boiling point, and stability in nuclear environment are characteristics that make these salts an excellent coolant for MSR application [7]. Although MSR reactors have many advantages, the salts are extremely corrosive to materials used in construction of the reactor [8]. The fluoride salts with eutectic mixture of LiF–NaF–KF (FLiNaK) with mol % of 46.5-11.5-42 are one of the candidates to be used in MSR.

Many studies have been performed to develop corrosion resistance material for use in MSR [9–11]. All alloys when exposed to this highly corrosive salt (FLiNaK) at elevated temperatures (700–850 °C) tend to corrode. Some of these alloys are usually protected from...
oxidation in air by their protective oxide layer. However, in the MSR, these protective layers tend to dissolve, and corrosion occurs by removing the oxide forming elements. Alloys containing Al, Cr, Si, and Mo form fluorides when exposed to fluoride salts. The necessary condition for the formation of fluorides (e.g., CrF₂, FeF₂, NiF₂) is that the Gibbs free energy of formation of fluoride must be negative [9]. Figure 1 shows the Gibbs free energy of formation of fluoride for different metals. The candidate metals from this graph for use in molten salt environment are Ni, Mo, and W. The rate of corrosion, however, also depends on other factors such as galvanic coupling of different metals in the alloy, temperature, and concentration of elements. Considering this, an alloy with 70% Ni, 16% Mo, 7% Cr, and 5% Fe was developed in Oak Ridge National Laboratory [12].

Figure 1. Gibbs free energy of formation (kJ/mol) of fluoride for different metals at 850 °C [10].

A corrosion study was also performed by Ignatiev et al. on nickel-based alloys Kh80MTYu, KhN80M-VI, and MONIKR, exposed to a fluoride salt environment (<700 °C) for 1200 h in a naturally circulated molten salt loop consisting of hot and cold regions to simulate the actual service condition in MSR [13]. It was found that the Cr depletion occurred in the FLiNaK environment; however, this process can be slowed down by removing impurities from the molten salt. Moreover, the accumulation of depleted Cr was observed on the cold region of the salt loop. This shows the importance of temperature in controlling the mechanism of corrosion in FLiNaK environment. Sellers et al. [14] studied corrosion behavior of multiple alloys in nine sealed stainless steel 316 crucibles. However, one crucible leaked and reacted with furnace refractory material, producing harsh corrosive vapors that corroded all the nine crucibles, leading to catastrophic damage of all specimens. This study demonstrated the highly corrosive nature of experimental setup required for such experiment [14]. Slama et al. studied MoNiCr, an alloy developed by COMTES FHT Inc., at 700 °C in LiF–BeF₂ environment for a duration of up to 3 months. The tests revealed the Cr and Mo elements were depleted from the alloy and were accumulated in the molten salt. Cr depletion was seen primarily on grain boundaries [15]. Another study with Ni–Mo super alloy GH3535 concluded that the corrosion occurs by dissolution of Cr and Mo from the grain boundaries [16].

In the present study, Hastelloy-N, X750, SS316, and SS304 were tested in a FLiNaK environment at 700 °C for 100 h. This study aimed at understanding the corrosion mechanism of these alloys and their behavior when they are used in the MSR at the same time. For the first time, EBSD and XPS examination was performed on corroded alloy samples to understand the nature of corrosion products and a role of the grain boundaries and triple junctions in chromium depletion in an MSR environment.
2. Materials and Methods

Hastelloy-N and X-750 were procured from Haynes International in the form of small sheets of size 2” × 2” (0.063–0.125”). Stainless steel 316 and 304 of size 5” × 5” × 1.5 mm was obtained from YIEH United Steel Corporation. The nominal chemical composition of these alloys is given in Table 1.

| Materials | C | Si | Mn | Ni | Cr | Mo | Cu | Fe | Nb + Ta |
|-----------|---|----|----|----|----|----|----|----|---------|
| X750      | 0.08 | 0.5 | 1 | Balance | 17 | - | 0.5 | 5 | 1.2 |
| Hastelloy-N | 0.06 | 1 | 0.8 | Balance | 7 | 16 | 4 | - |
| SS316 | 0.024 | 0.60 | 0.98 | 10.13 | 16.77 | 2.02 | 0.10 | Balance | - |
| SS304 | 0.03 | 0.75 | 2 | 10 | 18 | - | - | Balance | - |

Small coupons of the size 15 × 10 × 1.5 mm were cut from original sheets. All samples were abraded progressively using SiC grinding paper of grit size 320, 500, and 800 to obtain the same surface finish on all samples. The samples were then washed in 1M NaOH solution, cleaned using acetone, and dried in warm air. The samples were stored in vacuum desiccator to avoid oxidation/corrosion before testing. The weight of the samples was recorded before corrosion test.

Eutectic salt mixture with 46.5LiF–11.5NaF–42KF mol % was prepared. Pure nickel crucible obtained from Delta-Scientific Laboratory was filled with this salt mixture. All 4 alloy samples were submerged in the salt. The crucible was then placed in MTI Corp GSL 1500× tubular furnace (see Figure 2 for detailed set-up). Argon gas was filled in the tubular furnace and corrosion test was set to run at 700 °C for 100 h. After this test, the samples were left in furnace to cool down. Crucible was removed from the furnace and samples were taken out. For cleaning salts from sample surfaces, 1M Al(NO3)3 solution was prepared and samples were ultrasonically cleaned in this solution. Samples were then cleaned using deionized water and dried in warm air. The weight of all samples was recorded after corrosion tests.

The crystallographic characterization of corroded samples was performed using X-ray diffractometer (Rigaku Ultima IV X-ray Diffraction (XRD)) with Cu Kα radiation at 44 mA and 40 kV. Measurements were taken in the 2θ range of 10° to 80° with 0.02° step size. The chemical composition of surface of corroded samples was determined using an X-ray photoelectron spectroscopy (XPS) Kratos AXIS Supra system equipped with 500 mm Rowland circle mono-chromated aluminum Kα 1486.6 eV source and combined with hemi-spherical analyzer (HSA) and spherical mirror analyzer. Multiple spots of size of 300 × 700 μm were analyzed at different positions on the surface of samples. All survey
scans were collected in the binding energy range $-5$ to 1200 eV with 1 eV step size and 15 keV accelerating voltage and 15 mA emission current. For analyses of surface morphology, a Hitachi SU6600 Scanning Electron Microscope (SEM) was used. The distribution of elements on corroded surface were analyzed using Oxford Instruments X-Max 80 mm$^2$ Large Area SDD Silicon Drift Detector Energy-Dispersive X-ray Spectroscopy (EDXS). To analyze the depth of corrosion and cross-sectional analyses, we hot mounted the samples using Poyfast-Struers and polished. The EDXS maps and line scans were collected from the cross-section of the corroded samples.

3. Results and Discussion

3.1. Weight Loss Measurements

The weight of the corroded samples changed because of removal of the certain elements from the alloy. The weight loss per unit surface area ($w$) of the sample was calculated. This weight change per unit surface area ($w$) was compared for tested alloys. The maximum $w = 9.98$ mg/cm$^2$ was registered for alloy X750, which showed the maximum depletion of Cr from the surface. SSS316 and SS304 also lost weight after corrosion, and the weight losses per unit surface area ($w$) were 1.99 mg/cm$^2$ and 2.13 mg/cm$^2$, respectively. The Hastelloy-N, on the other hand, interestingly gained weight, and the weight loss per unit surface area ($w$) was $-3.10$ mg/cm$^2$. This gain in weight could have been because of deposition of corrosion products from the other samples on the surface of Hastelloy-N. From the weight loss/gain trend, it seems the corrosion resistance of alloys tested was X750 < SS304 < SS316 < Hastelloy-N.

3.2. Analysis of Surface Morphology after Molten Salt Corrosion Tests

Figure 3 shows the scanning electron microscope (SEM) secondary electron images (SEI) of alloys X750 (a), Hastelloy-N (b), SS304 (c), and SS304 (d) after corrosion test of 100 h at 700 °C in molten salt environment. The surface morphology of all samples showed interesting features; the sample of X750 alloy was heavily corroded and formation of chromium oxide can be seen in form of layered structure in the inset image in the figure at higher resolution. This was later confirmed to be chromium and oxide by SEM EDXS. The morphology of alloy Hastelloy-N showed granular surface and grains with micro-holes, which were observed in the high-resolution image. Both stainless steel samples were also corroded, and the remnants of chromium oxide layer was seen on the surface of sample SS304 (Figure 3c). At high-resolution (inset image), grains of nickel were observed. The surface of SS316 showed (Figure 3d) two distinct surfaces, the granular surface in the middle and the smooth outer surface. From these images, all the samples showed corrosion on the surfaces; cross-sectional images, discussed later, will show the depth of corrosion.

For analyses of distribution of selected elements (Ni, Fe, Cr, O) on the surface of corroded alloys, we collected SEM-EDXS maps at multiple regions on the samples. Figure 4 shows the elemental maps of top surfaces. The alloy X750 showed the strong presence of Cr and O on the surface. The Cr was distributed evenly on the surface showing uniform corrosion in this alloy. The layered-like structure observed earlier was confirmed to be chromium oxide. This Cr present on the surface came from the base alloy and diffused to the surface for the duration of the corrosion test. On the surface of Hastelloy-N, similar uniform corrosion was seen with comparatively less Cr. However, both stainless steel samples showed non-uniform corrosion. In SS316, the middle region of the scanned map showed a Cr oxide patch and Fe in the outer region of patches. Similarly, SS304 had remnant protective chromium oxide layer. Fe and Ni were seen around these remnants. From these top surface EDXS scans, it can be concluded that the uniform corrosion occurred in Ni base alloys X750 and Hastelloy-N, and non-uniform corrosion took place in steel samples.
Figure 3. Scanning electron microscope images of (a) X750, (b) Hastelloy-N, (c) stainless steel 304 (SS304), and (d) stainless steel 316 (SS316) after exposure to molten LiF–NaF–KF (FLiNaK) salt at 700 °C for 100 h.

Figure 4. SEM–energy dispersive X-ray spectroscopy (EDXS) elemental maps of Ni, Cr, O, and Fe on the surface of alloy X750, Hastelloy-N, SS316, and SS304 after 100 h exposure to molten FLiNaK salt at 700 °C.
For better understanding of corrosion, the polished cross-sections of the samples were scanned using SEM–EDXS, and the elemental maps of different elements were collected for all alloys tested. Figure 5 shows these collected elemental maps for X750 alloy’s cross-section—these scans show the presence of possibly TiAl gamma prime ($\gamma'$) particles in the base alloy. The region beneath the outer surface showed mostly Ni and very little Cr, which indicates that Cr had been depleted from this region. In Hastelloy-N (Figure 5), the cross-section showed similar depletion of Cr. Moreover, characteristic Si–Mo clusters were observed in the base of Hastelloy-N. In addition, a very thin layer of nickel was detected on the outer surface and a thick layer of Fe and Cr was also found close to surface. This may be attributed to plating of corrosion products on the surface from other alloys tested. A similar Cr- and Fe-depleted band was also observed in the SS304 sample. A layer of oxide was present close to the surface of this alloy. In the SS316 alloy, depletion of Fe, Cr, and Mn was also noticed. All these elemental maps of cross-sections showed the extent of corrosion in the cross section of alloy. It is possible to conclude from these maps that corrosion resistance was the lowest in alloy X750 and that Hastelloy-N had the highest corrosion resistance.

Figure 5. Cross-sections SEM–EDXS elemental maps of alloy X750, Hastelloy-N, SS304, and SS316 after 100 h corrosion at 700 °C in molten FLiNaK environment.

To measure the exact depth of corrosion in all alloys the line scans were collected from the cross-sections of the corroded samples at multiple regions. The depth of corrosion was measured as change in composition of Cr and Fe compared to the base alloy. Figure 6 depicts the SEM–EDXS line scans of selected elements (Ni, Fe, Cr) collected from cross-section of alloys after exposure to molten FLiNaK salt for 100 h at 700 °C. From these elements, the corrosion depth can be measured as the changes in the Cr, Fe, and Ni intensity along the line scan. The maximum depth of corrosion of 247 µm was registered for alloy X750 and minimum depth of corrosion of 19 µm was recorded for Hastelloy-N. However, for steel samples SS304 and SS316, similar depths of corrosion of 30 µm and 40 µm, respectively, were recorded. Therefore, from these EDXS line scans, the increasing trend of corrosion was Hastelloy-N < SS304 < SS316 < X750.
3.3. XRD and XPS Analyses of Alloys after Corrosion

Characterization of the corrosion products on the surface of all alloys was performed using XRD and XPS. Figure 7a shows the XRD spectra of alloys after corrosion in FLiNaK environment. For Hastelloy-N, only Ni and Cr peaks were observed. However, for X750, additional NiO and Fe$_2$O$_3$ peaks were identified. Both steel samples SS304 and SS316 showed iron oxide and nickel peaks. In XPS survey scans, shown in Figure 7b, Ni 2p was identified at binding energy 856.5 eV, Fe 2p at 712.2 eV, and Cr 2p at 577.2 eV, and other peaks of F 1s at 685 eV (metallic fluoride), K 2p at 293 eV, and Cu 2p at 932 eV (doublet separated by 19.75 eV) were also noted. The sources of this Cu were alloys X750 and SS304 with 0.5 wt % and 0.1 wt % of Cu, respectively. The atomic concentration of different elements can be calculated from the XPS survey scans using the following Equation (1):

$$C_x = \frac{I_x/S_x}{\sum I_i/S_i}$$  

(1)

where $C_x$ is the atomic concentration of the x element, $I_x$ the peak area of this element $x$, and $S$ is the relative sensitivity factor (RSF) of this element. CasaXPS software was used for the calculation of atomic concentration, and the results were plotted as bar charts, as shown in Figure 7c. The atomic concentration of Cr on surface of alloy X750 was 23.6%, while on Hastelloy-N, only 2.9% was detected. Furthermore, SS304 and SS316 showed 7.0% and 4.1% of Cr, respectively. The atomic concentration of nickel, on the other hand, was minimal in SS304, with only 5.1%, and maximum in Hastelloy-N at 12.2%. Moreover, nickel atomic concentrations for X750 and SS316 were 7.0% and 6.0%, respectively. From these XRD and XPS results, we can conclude that Hastelloy-N was the least corroded and X750 was the most corroded alloy. This was mainly supported by the presence of Cr diffused to the surface from the base alloy. However, the atomic concentration on the surface may not always represent the actual corrosion, since the corrosion products from one alloy can become deposited on another alloy. Moreover, because of plating of the corrosion products from one alloy to another, the weight loss or gain will also not represent the true corrosion comparison between the alloys. However, the EDXS cross-section line scans presented in
Figure 6 are the best way to compare the corrosion damage in the investigated alloys in molten salt environment.

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$$C_x = \left( \frac{I_x}{S_x} \right) / \left( \frac{\sum I_i}{\sum S_i} \right) \quad (1)$$

where $C_x$ is the atomic concentration of the x element, $I_x$ the peak area of this element x, and $S$ is the relative sensitivity factor (RSF) of this element. CasaXPS software was used for the calculation of atomic concentration, and the results were plotted as bar charts, as shown in Figure 7c. The atomic concentration of Cr on surface of alloy X750 was 23.6%, while on Hastelloy-N, only 2.9% was detected. Furthermore, SS304 and SS316 showed 7.0% and 4.1% of Cr, respectively. The atomic concentration of nickel, on the other hand, was minimal in SS304, with only 5.1%, and maximum in Hastelloy-N at 12.2%. Moreover, nickel atomic concentrations for X750 and SS316 were 7.0% and 6.0%, respectively. From these XRD and XPS results, we can conclude that Hastelloy-N was the least corroded and X750 was the most corroded alloy. This was mainly supported by the presence of Cr diffused to the surface from the base alloy. However, the atomic concentration on the surface may not always represent the actual corrosion, since the corrosion products from one alloy can become deposited on another alloy. Moreover, because of plating of the corrosion products from one alloy to another, the weight loss or gain will also not represent the true corrosion comparison between the alloys. However, the EDXS cross-section line scans presented in Figure 6 are the best way to compare the corrosion damage in the investigated alloys in molten salt environment.

![Figure 7](image.png)

**Figure 7.** (a) XRD spectra of samples after corrosion in FLiNaK environment, (b) XPS wide survey scans from the surface of alloys after corrosion test, and (c) bar chart comparing the atomic concentrations calculated from XPS wide survey scans for Ni 2p, Fe 2p, and Cr 2p.

### 3.4. Microstructural Analysis of Corroded Alloys Using EBSD

For better understanding the corrosion in molten fluoride salts at elevated temperature, we collected EDXS and EBSD scans from the cross-sections of the alloys. Figure 8a shows the Cr elemental map for alloy X750 and corresponding EBSD scan. In the EBSD graph, red arrows are pointing at the corrosion initiation sites in the vicinity of outer surface. Similarly, Figure 8b depicts the Mo elemental map with the red arrows pointing at the corrosion initiation sites. The white arrow is pointing at the Mo–Si compound in the Hastelloy-N. Figure 8c,d both show similar corrosion sites. Interestingly, these corrosion initiation sites were located at the triple junction of the grains. This may be attributed to high elastic energy accumulated at these junctions, which provide sites for Cr transport to the surface and corrosion initiation. The white arrow in Figure 8d points at the grain boundary where the corrosion was not started yet. It is clear from these results that the corrosion started first at the triple junction, then at the grain boundaries, followed by general corrosion.
Figure 8. EDXS elemental map along with EBSD maps showing different grains in random colors for (a) X750, (b) Hastelloy-N, (c) SS316, and (d) SS304 after corrosion in molten FLiNaK salt at 700 °C for 100 h.

4. Conclusions

The corrosion tests of 100 h were conducted in molten FLiNaK environment at 700 °C for alloy X750, Hastelloy-N, stainless steel 316, and stainless 304 in argon cover gas. Elemental analysis of the distribution of corrosion products after corrosion and analysis of EDXS, XRD, and XPS results demonstrated that Hastelloy-N performed better than any other alloy during corrosion tests, with the corrosion depth in this alloy being about 10 times lower than for alloy X750. The corrosion process changed the elemental composition of the surface layer of all investigated alloys, and poor corrosion performance was correlated with accelerated diffusion of Cr to the surface of the alloy. The amount of Cr on the surface of X750 was about 10 times higher than on the surface of Hastelloy-N. Analysis of corrosion nucleation sites allowed us to identify most frequent corrosion sites at the triple junction of grains, followed by grain boundaries and general corrosion. From the weight loss measurements, the corrosion resistance of alloys tested can be classified as X750 < SS304 < SS316 < Hastelloy-N.

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