

Corrosion of Inconel 800HT Alloy in Molten Fluoride Salts

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Molten salt reactors are one of the technologies developed under GEN IV nuclear research. The molten mix of LiF and BeF₂, LiF, NaF nad KF, or NaBF₄ and NaF act both as a reactor coolant both in primary and secondary loop. The combination of molten fluorides and high temperature creates highly corrosive environment. The aim of this work was to test corrosion resistance of Inconel 800HT alloy in molten FLiNaK, FLiBe and NaBF₄-NaF mix. The testing tube was filled with salt mixture and heated to 600-725 °C for a total of 1800 hours. The material exposed to NaBF₄-NaF mix showed mild corrosion attack on grain boundaries. Samples exposed to FLiNaK salt were more damaged, largely the part above the salt surface. The intergranular corrosion was also observed, more severe than in the case of NaBF₄-NaF environment. Corrosion in the FLiBe salt caused depletion of alloying metals from the material's surface. In all cases was dissolving of Cr into the melt identified as the main mechanism. The corrosion was accelerated by impurities in the salt mix, mostly water forming hydrogen fluoride gas in combination with insufficient seal of the testing tube.

Keywords: Corrosion, Molten Salt Reactor, Nickel alloy, GEN IV,

1 Introduction

Nuclear reactors using molten fluoride salts as a coolant are one of the directions of development of fourth generation reactors. There are two variants considered: liquid fuel in the form of fluoride (MSR - molten salt reactor) and solid fuel TRISO (FHR - Fluoride cooled High temperature Reactor). The system uses two circuits filled with molten alkali metal fluoride salt. The heat from the secondary circuit is used to produce steam to drive the turbine. There are several advantages of using molten salts to cool the reactor. The melt has a large heat capacity, good thermal conductivity and, due to the high boiling point, the reactor can reach high operating temperatures without needing to increase the pressure in the reactor vessel. Another advantage is the ability to clean and reprocess the fuel in the MSR design while the reactor is running and to separate unwanted fission products from the media [1,2]. The primary circuit is planned to be filled with a mixture of lithium and beryllium fluoride (FLiBe), in case of MSR together with dissolved fuel (U, Pu, Th salts). This mixture is used because of its low effective cross section. Operating temperatures are in the range 700-800 °C. For the secondary circuit, either FLiBe, a LiF-NaF-KF (FLiNaK) or a NaBF₄-NaF mixture can be used. Use of chlorides or other salts is also considered. The FLiNaK salt has properties close to FLiBe, so it is often used in experiments as a replacement for FLiBe salt to limit the need to use beryllium fluoride [3].

The molten fluoride salt environment makes high

demands on the durability of the material. Structural materials for high temperature applications usually resist corrosion by protective oxide layers formation. However, in the presence of fluorides, most oxides of these metals are not stable and by chemical reaction with the environment forming fluorides soluble in the melt and thus dissolving the oxide layer. Corrosion is induced by the selective dissolution of individual alloys into the environment, resulting in intergranular corrosion of the material. This phenomenon is most prominent in the case of chromium. The susceptibility of individual metals to corrosion increases in the following order: Ni, Co, Fe, Cr, Al. [4] The corrosion rate in fluoride salt melt environments is negatively affected by the presence of impurities, mainly water, oxygen, and dissolved metals. At operating temperatures, water reacts with the salt to form hydrogen fluoride. The subsequent reaction of HF and the material results in the dissolution described above. The formation of HF can sometimes cause corrosion on parts of the equipment above the melt level. The presence of impurities containing nickel and iron (NiF₂, FeF₂) also leads to the oxidation of chromium and the formation of CrF₃ [5].

Corrosion resistance in molten fluoride salts is secured by the choice of material and by the control of environment, focused on removing any impurities. Trace amounts of metallic impurities must also be removed from the melt. Since the salts used are hygroscopic, the reduction of moisture from the environment is very challenging, and rinsing the melt with a mixture of HF and H₂ is usually used [6].

As for the choice of material, nickel alloys are known for their temperature and corrosion resistance [7]. These mainly considered are Hastelloy-N, GH3535 and MONICR. The use of other materials (e.g. Inconel 800HT, Inconel 617 and steel 316) is also considered, but with increasing chromium content the corrosion resistance is getting worse. The reasons for using this alternative, less suitable, materials are their mechanical properties at high temperatures, well-known behaviour in irradiated conditions and long-term experience with practical use. Another reason is the better availability of these materials and the greater supply of semi-finished products for device construction. Steel 316 is particularly interesting from this point of view because of its widespread use in the power generation industry and the opportunities for additive manufacturing that have already been explored [8]. A solution to the lack of corrosion resistance can be the application of protective coatings. There is a wide range of nickel-based coatings available mostly for steel base, like the aforementioned steel 316. Nickel coatings or Hastelloy-N alloy cladding is mostly considered [9-12].

2 Materials and Methods

The aim of the experiment was to investigate the possibility of using Inconel 800 HT alloy in FLiNaK, FLiBe and NaBF₄-NaF molten salt environments. The reason for testing this material is mainly a consideration for a use in construction of a larger experimental apparatus. The described experiment was used to estimate the limits in the operation of such apparatus.

Static exposure of the material specimen to the molten fluoride salt was selected as the test method. A test vessel made of the investigated material was filled with a mixture of LiF-NaF-KF, NaBF₄-NaF or LiF-BeF₂ and heated in a laboratory furnace. In this configuration the whole vessel served as a specimen for the exposure test. The aforementioned test vessel, see Fig. 1, was manufactured by welding a tube to a metal plate forming a bottom. On the top of the vessel was a flange used to close the test tube with a lid equipped with a thermocouple shaft. For sealing the lid, a graphite ring plated with 304-steel was used.

The composition of Inconel 800 HT is given in the following table (Tab. 1).

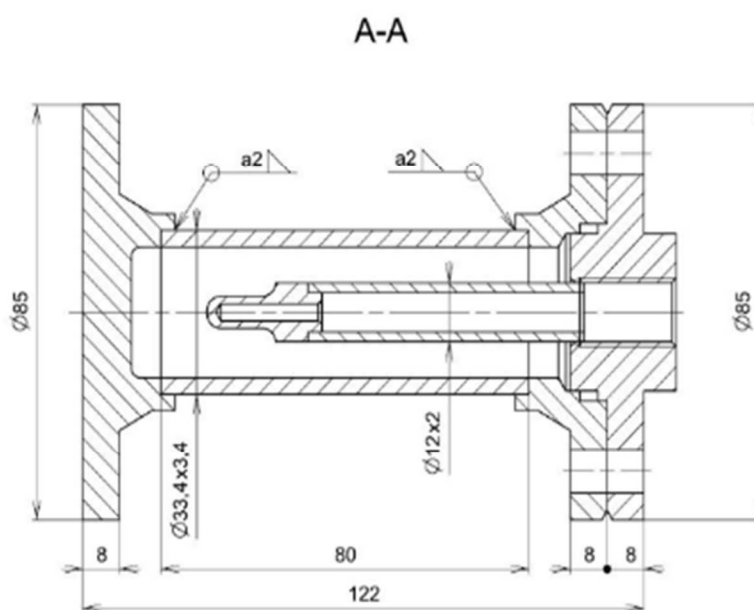


Fig. 1 Test vessel for molten salt corrosion experiment: as-received condition and dimensioned drawing

Tab. 1 Chemical composition of Inconel 800 TH

Ni	Fe	Cr	C	Al	Ti
30-35%	min. 39,5%	19-23%	0,05-0,1%	0,15-0,6%	0,15-0,6%

For the experiment, a total of five test vessels were prepared. 2 of them were filled with a $\text{NaBF}_4\text{-NaF}$ mixture, 2 with FLiNaK mixture and one with FLiBe mixture. The mixtures acting as a corrosive environ-

ment were prepared by mixing powders of pure substances followed by melting in a graphite crucible in protective atmosphere. The composition of used mixtures is given in the following table (Tab. 2).

Tab. 2 Chemical composition of used salt mixtures [13,14]

Salt mixture	FLiNaK	$\text{NaBF}_4\text{-NaF}$	FLiBe
Composition (% wt.)	29.2 % LiF 11.7 % NaF 59.1 % KF	96.8 % NaBF_4 3.2 % NaF	51.7 % LiF 48.3 % BeF_2

Since the corrosion rate in the molten salt environment is affected by the presence of oxygen and moisture, test vessels filling as well as the salt mixtures preparation was carried out in a glove box under a protective nitrogen atmosphere. The vessel was placed in the box, filled with the molten salt to three-quarters of its volume and closed with a flanged lid. After cooling to a room temperature, the specimens were removed from the glove box and placed in a laboratory furnace. The rest of the experiment was executed in air, without any additional environmental control.

The experiment was divided into three parts with gradually increasing temperature. The first part was carried out at 600 °C, the second at 650 °C and the third at 725 °C. The length of each part was 600 hours. At the end of the exposure, the test vessels were removed from the furnace and their condition was documented photographically. Subsequently, each specimen was cut: the flange was separated from the rest of the vessel and then cut vertically in half. Observing this cross-section by light microscopy was used to determine the durability of the gasket design. The test vessel filled with FLiBe salt was handled separately to minimise beryllium contamination in the lab. Because of different disassembly process, there are no available data regarding the gasket condition.

For metallographic analysis, a sample was cut from the part of the tube located below the melt surface. A light microscope Zeiss Observer Z1M and scanning electron microscope Tescan Mira 3 were used for observation. Additionally, an EDS (Energy-dispersive X-ray spectroscopy) analysis was performed to determine changes in chemical composition of the material. Due to the experimental setup where the entire container acts as the sample, corrosion loss by weight change or other similar method wasn't possible to measure. The evaluation of corrosion resistance was carried out only by visual inspection and metallography.

3 Results

After the experiment, each of the vessels was inspected for signs of corrosion. The microscopic examination of material samples was carried out afterwards.

3.1 Visual inspection

After the experiment, the outer surface of test vessels was covered with a layer of oxides. The layer was compact and did not peel off the surface. The two vessels containing the $\text{NaBF}_4\text{-NaF}$ mixture appeared undamaged, see Fig. 2 A, B.

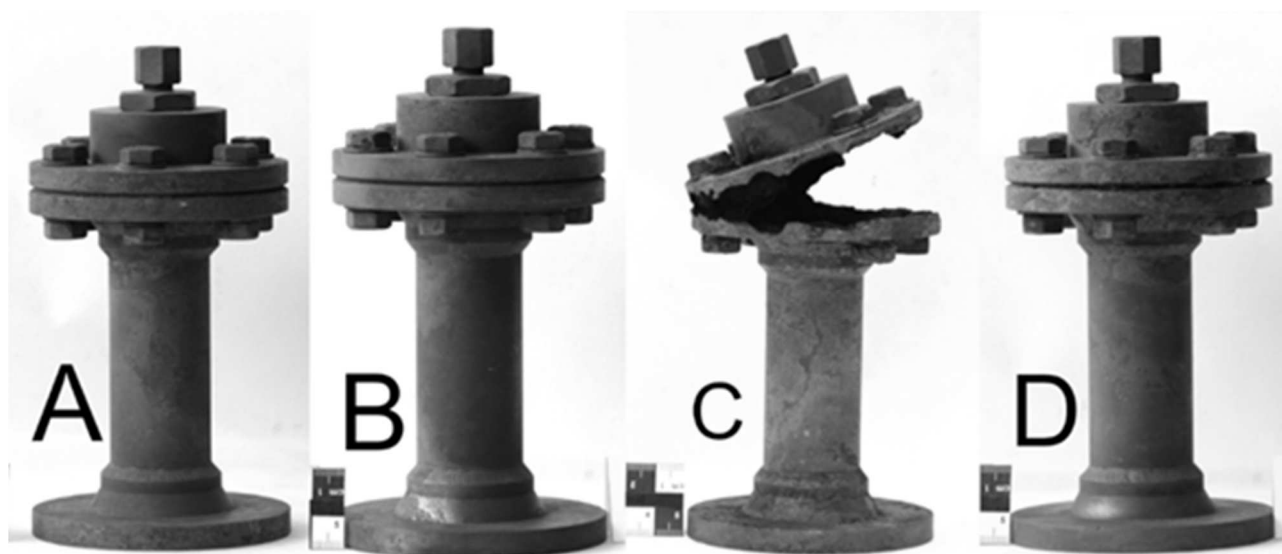


Fig. 2 Test vessels after the experiment with $\text{NaBF}_4\text{-NaF}$ (A, B) and FLiNaK (C, D)

In the case of test vessels filled with FLiNaK, damage was obvious. In one case, the lid was torn off (Fig. 2 C) and the interior was filled with corrosion products mixed with the remaining fluoride salt (Fig. 3).



Fig. 3 Detail of corrosion damage of Test vessels exposed to FLiNaK salt (specimen C on the left, specimen D on the right)

3.2 Performance of the gasket

The lid assembly of test vessels A, B and D were cut off and a cross-section of each was prepared. As shown in Fig. 4, exposure in $\text{NaBF}_4\text{-NaF}$ did not cause damage to the gasket. It would seem that some minor corrosion may have occurred in the seam on specimen B, but it may also be damage caused by cutting the specimen.

In contrast, exposure in FLiNaK salt, see Fig. 5, caused corrosion in the lid assembly, around the gas-

ket. On the cross-section, corrosion around the graphite ring is visible and the gap between the lid and the flange is widened on the outside. Similar gasket failure probably caused the extensive damage of the test vessel C. The increase in the volume of corrosion products then led to lifting the lid. This has completely broken the seal of the vial. Since the seal and caused the leak. Since the damaged gasket was located above the melt level, corrosion was probably initiated by the release of hydrogen fluoride from the molten salt. This indicates a presence of moisture in the salt mixture.

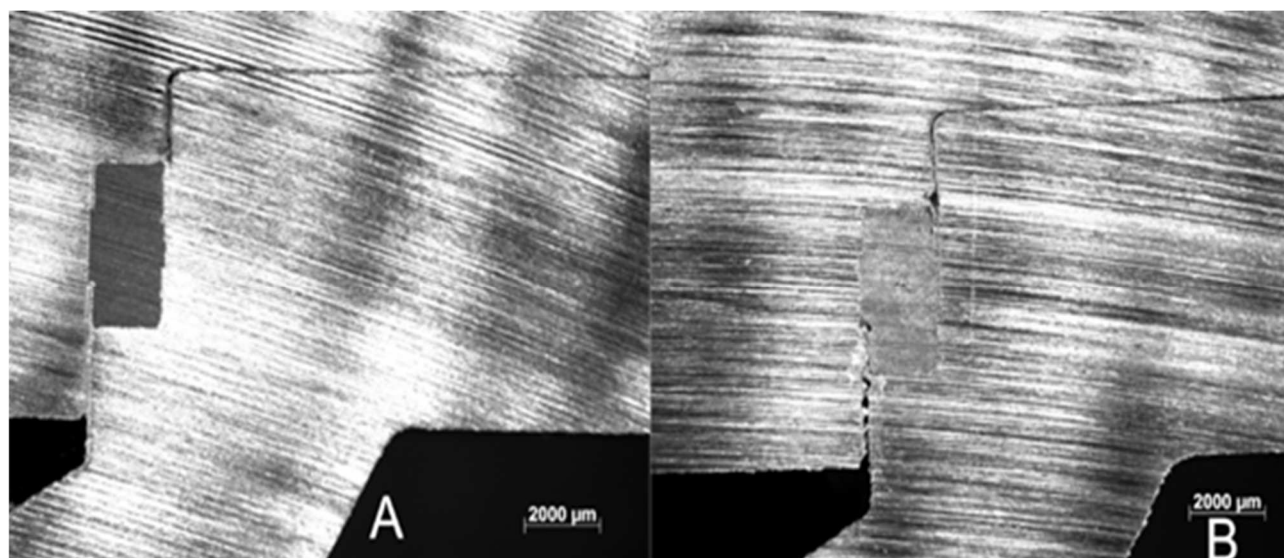


Fig. 4 The lid assembly cross-section after exposure in $\text{NaBF}_4\text{-NaF}$

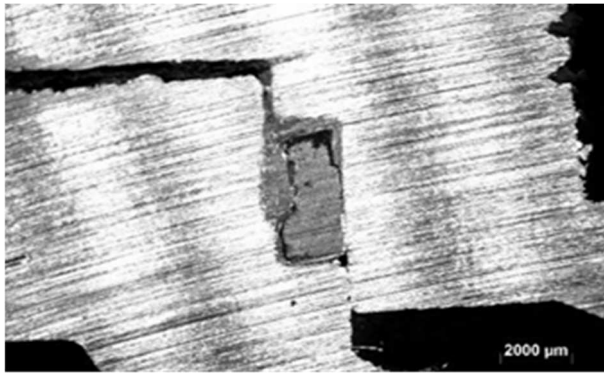


Fig. 5 The lid assembly cross-section after exposure in FLiNaK

3.3 Metallographic analysis

To obtain data on the corrosion of the material in molten salt, a metallographic analysis of a cross-section of the inner wall of the test vessels was performed. The results showed varying levels of corrosion damage in all environments used. In all cases, the grain boundaries were most affected.

The inner surface of the samples exposed to the NaBF₄-NaF salt was covered with green crystals (see Fig. 6). The solidified salt was also green coloured. The surface does not appear damaged in visual inspection (except for the aforementioned crystals), but the green colour of the salt indicates a presence of chromium dissolved from the material. EDS analysis of the particles confirmed high Cr content (13 % wt.) together with Na and F. Fe and Ni weren't detected in the particles.

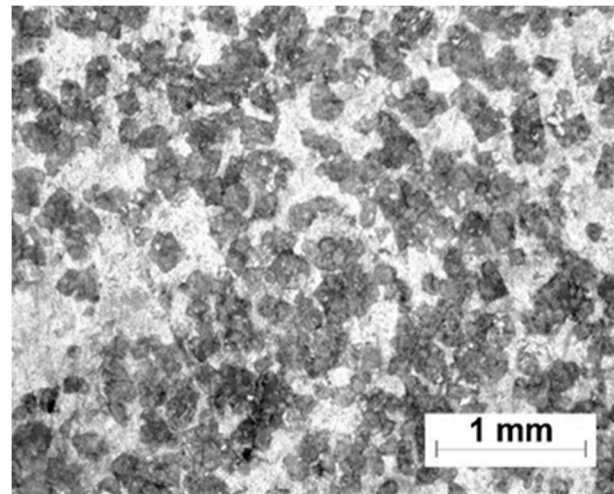


Fig. 6 The inner wall of test vessel exposed to NaBF₄-NaF

Microscopic examination of the wall cross-section revealed dissolution of the material along grain boundaries (see Fig. 7). The depth of the observed intergranular corrosion varies a bit depending on the location but didn't exceed 150 μm. The appearance of corrosion damage coincides with selective dissolution of chromium along grain boundaries revealed in exposure tests on similar materials [11]. The EDS analysis of the damaged area confirmed the selective corrosion. It has also been observed by EDS that fluorine penetrates along grain boundaries into the material. In parts closer to the surface there are cavities where the material has been completely dissolved. It is in these areas that the presence of fluorine was detected.

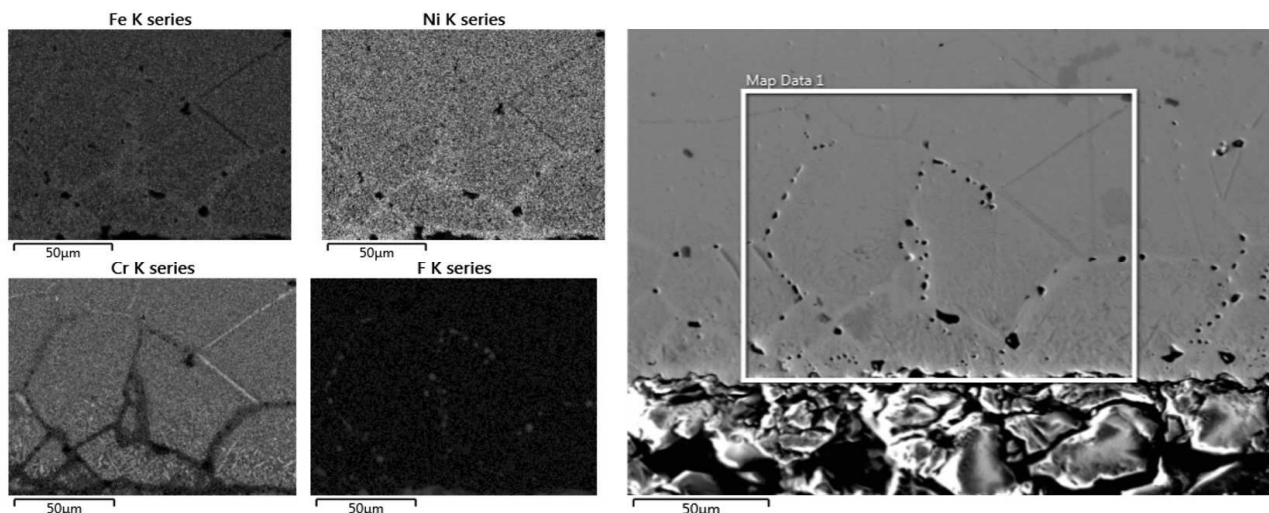


Fig. 7 Cross-section of material exposed to NaBF₄-NaF: EDS analysis (left) and cross-section (right)

The material exposed in FLiNaK was not, unlike the previous case, covered by the green chromium fluoride crystals. The solidified salt inside the vessel was coloured green, so dissolution of chromium was also taking place. Again, corrosion along the grain boundaries is visible on the cross-section of the inner wall (see Fig. 8), and as in the previous cases, the depth of

damage varies in different places. The intergranular corrosion was detected to greater distance from the surface, up to 500 μm. The affected areas along grain boundaries are also much more connected and the cavities of dissolved material are more prominent. Unlike the case of NaBF₄-NaF salt, there was no fluorine detected in the material.

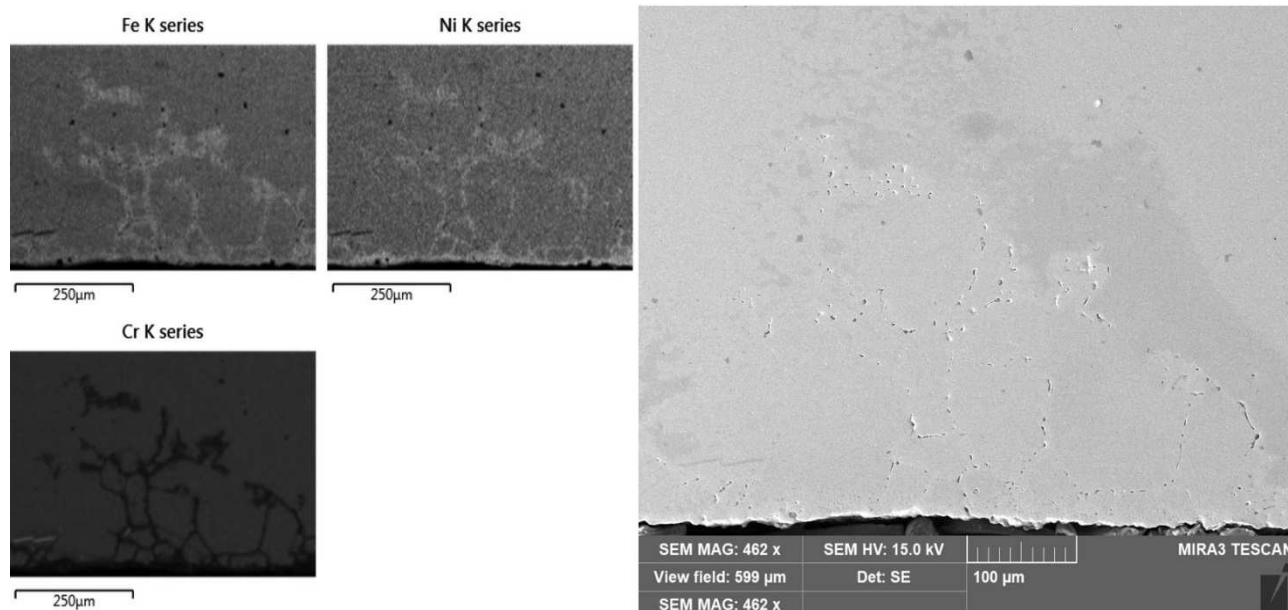


Fig. 8 Cross-section of material exposed to FLiNaK: EDS analysis (left) and cross-section (right)

Unlike the other salt mixtures, the exposure to FLiBe salt caused a different form of corrosion damage. The dissolution of Cr on grain boundaries is still visible, but there is also a layer on the surface where both Cr and Fe are completely missing. This part is porous and contains only Ni (see Fig. 9). Thickness of

this layer is 20 to 50 μm. The intergranular corrosion of Cr reaches depth up to 150 μm. From this pattern, it can be concluded that intergranular corrosion with selective chromium dissolution occurred first, followed by formation of the porous Ni layer.

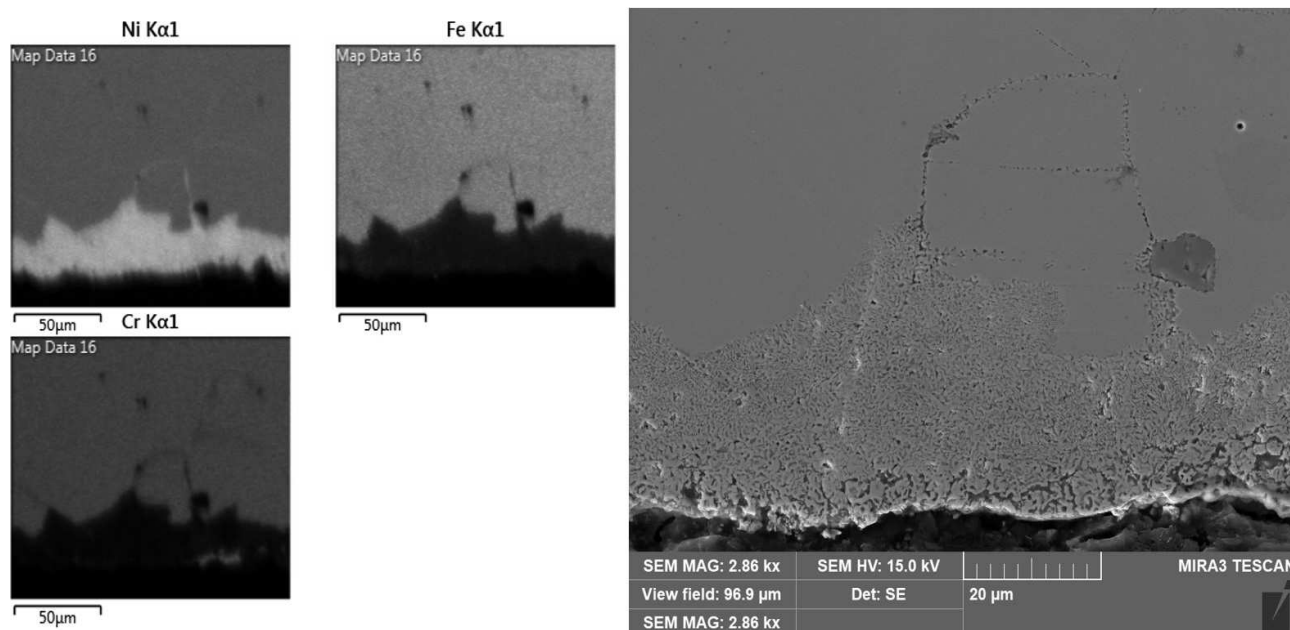


Fig. 9 Cross-section of material exposed to FLiBe: EDS analysis (left) and cross-section (right)

As mentioned above in the “Visual inspection” section, one FLiNaK salt test vessel experienced a seal failure. Although this case can be considered a failed experiment, the material was still examined to see what effect does have exposure to molten salt without the protective atmosphere. As can be expected in such conditions, the corrosion rate was high, about half of the vessel wall thickness was corroded. The surface

was covered with a non-compact layer of corrosion products, composed of a mixture of fluorides and oxides. There was no selective corrosion found, even in greater distance from the surface. It is obvious that in the event of loss of the protective atmosphere, the material is rapidly degraded and even for limited time, it can't be expected any resistance to corrosion.

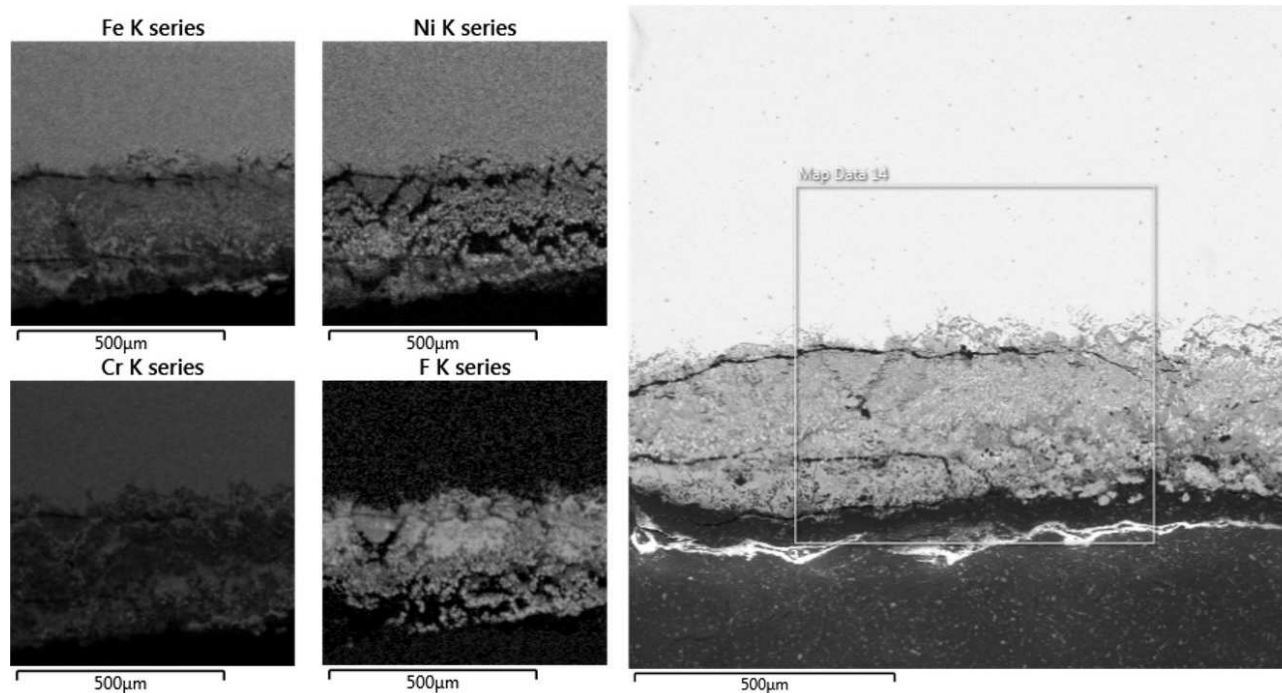


Fig. 10 Cross-section of material exposed to FLiNaK after the loss of protective atmosphere (specimen C): EDS analysis (left) and surface layer (right)

In FLiNaK and NaBF₄-NaF salt, corrosion was induced by chromium dissolution along the grain boundaries. The observed condition is the result of a total of 1800 hours of exposure at temperatures of 600-725 °C. Inconel 800 HT contains significantly more chromium than other materials intended for fluoride salt melt environments, so some degree of corrosion attack is to be expected. In both these salt mixtures, corrosion proceeded by a similar mechanism, i.e., dissolution of the material along grain boundaries. The dissolved chromium is contaminating the salt melt, which could pose a problem in case this material is used for construction of experiment equipment [5]. In FLiNaK salt is the corrosion spreads to a greater distance from the surface, about 3 times more than in NaBF₄-NaF mixture. Unlike the NaBF₄-NaF environment, in FLiNaK there was also found corrosion above the melt level (on the gasket assembly) indicating HF release from the molten salt. This was caused by incomplete removal of moisture during the preparation. Without active control of the protective atmosphere during the experiment the application of Inconel 800 HT in FLiNaK is limited.

The corrosion found on specimen immersed in FLiBe mixture is different to other two tested salts. There is still selective corrosion on grain boundaries present, but near the surface, there is a layer where both Cr and Fe are dissolved completely in bulk material, not only around grain boundaries. This layer is about 50 µm thick and composed only of porous Ni. The reason for this layer formation isn't clear. While the intergranular corrosion reached about the same

distance from the surface as in the case of NaBF₄-NaF salt, this bulk dissolution of alloying elements seems more serious. Because of this together with missing data about the state of the gasket assembly, more experiments are needed to find a conclusion about the resistance of Inconel 800 HT to FLiBe salt.

4 Discussion

For all the fluoride salt mixtures used in this study, the corrosion of Inconel 800 HT happened through a similar process. In all cases the main cause was dissolution of Cr from the matrix, predominantly along grain boundaries. This process has been observed in a number of other studies and is considered the main corrosion mechanism of Cr containing alloys in molten fluoride salts. Although experimental data are available mainly for the FLiNaK mixture, the corrosion mechanism is the same for all salt mixtures used in this experiment [15]. This is further supported by published tests in FLiBe salt on other materials containing Cr, namely 316 stainless steel [17].

The surface condition of the specimen exposed to the FLiBe salt in this experiment was different to the other. The surface layer was depleted of Fe in addition to Cr. According to Quilong et al. [11], Fe can be affected by dealloying like Cr. While their study did not observe such significant dealloying, there were cavities present in comparable manner, located away from grain boundaries. There are other studies both on 316 steel and Inconel 800 HT where the Fe dealloying was observed. It is reported [19], that less stable metal fluorides like FeF₂, FeF₃ and NiF₂ are causing significant

increase in corrosion rate in 316 steel. That study also claims that these observations are similar to Inconel 800 H in other sources. Thus, it is possible to compare these different alloys.

The behaviour of the studied material in $\text{NaBF}_4 + \text{NaF}$ mixture differed from the other two salts through the precipitation of large particles on the surface. According to a technical report from ORNL [18], the presence of such particles was observed on Hastelloy N alloy after exposure in CrF_3 -contaminated fluoroborate salt. According to that report, the precipitates are Na_3CrF_6 , which is consistent with the estimated composition of these particles based on our EDS analysis. Since the salt volume in the experiment was quite small compared to the material surface area, a Cr-related contamination was possible. The significantly higher Cr content in Inconel 800 HT compared to Hastelloy H is certainly a contributing factor to the contamination.

The assessment of the corrosion rate in this work wasn't precise. Corrosion rate is usually determined by weight loss, which was not possible in this experiment due to the specimen design. The corrosion resistance was estimated only by the metallographic analysis. Together with most sources focusing on FLiNaK salt, this makes it hard to compare the results with other studies. In this work, it was determined that Inconel 800 HT is most corrosion resistant to $\text{NaBF}_4 + \text{NaF}$ mixture. But there are studies claiming higher corrosion rates in fluoroborate compared to fluoride salts [14,17]. It should be considered that these studies were conducted with different material (Hastelloy N) and at higher temperature. Another key factor is the effect of impurities in the melt on corrosion resistance. Corrosion rate in fluoride salts mixtures is prone to increase when impurities are present. In addition to the metal fluorides discussed above, these are primarily: moisture, hydrogen, and hydrated salts [5,20]. Thus, the presence of inadequately removed contaminants in the fluorides may compromise corrosion resistance compared to fluoroborate.

Part of the experiment dealt with the lid gasket performance and corrosion above the melt level. According to some sources, $\text{NaBF}_4\text{-NaF}$ mixture is not stable enough at high temperatures and decomposes producing BF_3 [15]. This is a potential concern when considering potential use of $\text{NaBF}_4\text{-NaF}$. However, decomposition of $\text{NaBF}_4\text{-NaF}$ was not observed during this experiment. Although it was not possible to analyse the salt after experiment, the decomposition would cause pressurization of the vessel and corrosion above the melt level. As stated in the results chapter, no corrosion was detected above the melt level. The absence of significant decomposition can be explained by suitable purity of the mixture and by the lower temperature in the experiment, as studies on thermal stability focus on 800°C and above. Compared to this,

FLiNaK salt caused a considerable damage to the lid assembly. In this case it was HF produced by insufficiently removed moisture causing the issue. This observation supports the above conclusions on the presence of impurities in the FLiNaK salt.

5 Conclusion

During this project, corrosion resistance of Inconel 800 HT was tested in 3 different molten salt mixtures: FLiNaK, FLiBe and $\text{NaBF}_4\text{-NaF}$. Small test tubes were filled with the salt mixture and heated up to 725 °C for 1800 hours. The test vessels were sealed by a flange fitted with a 304-steel plated graphite gasket. After the test, the material was metallographically examined for signs of corrosion. EDS analysis was performed to identify cases of selective dissolving of alloying elements. In case of test vessels filled with FLiNaK and $\text{NaBF}_4\text{-NaF}$, the lid assembly and gasket were studied for corrosion to investigate its effectivity.

- The chosen sealing design was effective for $\text{NaBF}_4 + \text{NaF}$ mixture. On the other hand, in the case of FLiNaK salt, the corrosion on the gasket was evident and would lead to a leak if the exposure was longer. On one of the test vessels, the gasket failed causing destruction of the sample. Since the gasket was located above the melt level, the main cause of the corrosion is the presence of HF released from the melt.
- Inconel 800 HT can resist molten $\text{NaBF}_4 + \text{NaF}$ mixture for a limited time. Intergranular corrosion and selective Cr dissolving at grain boundaries occurs to some extent. Contamination of the salt by corrosion products was evident and could be a larger problem during the future experimental device operation than the material damage itself. The lack of signs of corrosion damage above the melt level indicates that no significant decomposition of the molten salt releasing corrosive gasses happened.
- Exposure to a molten FLiNaK mixture caused more severe damage to the material. There is a significant dissolution of Cr around the grain boundaries as well as intergranular corrosion present. The corrosion reached about 3 times greater distance from the surface than in case of $\text{NaBF}_4 + \text{NaF}$ mixture and the cavities are much more connected.

The use of Inconel 800HT in FLiNaK is not suitable without active environment control.

- For FLiBe salt, a different type of corrosion was observed. Both Cr and Fe was completely dissolved in the surface layer, to about 50 μm depth. This layer was composed only of porous Ni. In greater distance from the surface, the corrosion progressed along the grain boundaries, like in other tested salt mixtures. Maximum depth was similar to results observed in $\text{NaBF}_4 + \text{NaF}$ salt. By comparing this observation with the published studies, it was found that the probable cause was contamination of the salt with Ni and Fe fluorides.

Results of this study show that Inconel 800 HT can be used in contact with molten $\text{NaBF}_4 + \text{NaF}$ mixture for limited time and if the risk of melt contamination is considered. In case of FLiNaK salt, the intergranular corrosion is much more prominent, and the material can't withstand this environment for significant time. Corrosion above the melt level was also present. Similar case is molten FLiBe salt, where a different corrosion process was identified, but the material is also affected to a greater extent.

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