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Abstract

Full immersion corrosion experiments were conducted on GH3535 alloys with and without Al₂O₃ coating in FLiNaK molten salt. The corrosion behavior of GH3535 alloys with and without Al₂O₃ coating in the molten salt was investigated using SEM, EDS, XRD and other characterization techniques. The results indicate that the corrosion behavior of both coated and uncoated GH3535 alloys in FLiNaK molten salt at 700 °C exhibited selective leaching characteristics of Cr and Mo, with the uncoated alloy showing significant intergranular corrosion features. The Al₂O₃ coating did not enhance the corrosion resistance of the alloy; instead, it dissolved in the FLiNaK molten salt and formed a corrosion cell with the alloy surface exposed by the dissolution and spalling of the coating, thereby accelerating the corrosion of the GH3535 alloy.

Full Text

Corrosion Behavior of GH3535 Superalloy in FLiNaK Molten Salt

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ABSTRACT

As one of the most promising next generation reactors, the molten salt breeder reactor (MSBR) with excellent inherent security has attracted increasing attention in recent years due to energy shortages and safety concerns with traditional nuclear reactors. The most significant service characteristic of structural materials used in MSBRs is exposure to FLiNaK molten salt, which is highly corrosive to reactor structural materials and affects the safe operation of nuclear power plants. A polycrystalline Ni-Mo-Cr-Fe superalloy was developed and used as an important structural material in MSBRs at Oak Ridge National Laboratory (ORNL), but the corrosion mechanism of this alloy in FLiNaK molten salt has not been fully determined since the study was terminated in the 1970s for political reasons. Alloys serving in harsh environments often employ protective coatings to improve corrosion resistance, yet few studies on coating corrosion resistance in FLiNaK molten salt have been reported. Al_2O_3 and Cr_2O_3 coatings typically exhibit excellent corrosion resistance in molten salts such as sulfates, nitrates, and halides, but whether oxide films provide corrosion resistance in FLiNaK molten salt remains undetermined.

In this work, immersion corrosion experiments were conducted on GH3535 alloy specimens with and without Al_2O_3 coatings in FLiNaK molten salt. The corrosion mechanism of the alloy in FLiNaK was investigated using SEM, EDS, and XRD. The influence of Al_2O_3 coating on corrosion resistance in FLiNaK molten salt was also examined. The results show that both coated and uncoated GH3535 alloy exhibit exsolution corrosion characteristics of Cr and Mo elements in FLiNaK molten salt at 700 °C for 400 h. The difference is that the uncoated alloy shows intergranular corrosion characteristics, while the alloy with Al_2O_3 coating exhibits pitting corrosion characteristics. The Al_2O_3 coating cannot improve the corrosion resistance of the alloy in FLiNaK molten salt. The Al_2O_3 film dissolves in molten salt and results in exposure of the alloy surface. The corrosion rate increases due to formation of a corrosion cell between the oxide film and the exposed alloy surface.

KEY WORDS GH3535 alloy, FLiNaK molten salt, Al_2O_3 coating, corrosion

Introduction

Due to energy shortages and safety concerns with traditional nuclear reactors, molten salt reactors with inherent safety attributes have become one of the most promising fourth-generation reactors [1-4]. Ni-Mo-Cr-Fe alloys have been used for key structural components such as reactor vessels and loop piping in molten salt reactors because of their excellent corrosion resistance, irradiation performance, and high-temperature mechanical properties [5]. Compared with

structural materials in other nuclear reactors, the most significant characteristic of the service environment for molten salt reactor structural materials is exposure to molten FLiNaK salt, which is extremely corrosive to Ni-Mo-Cr-Fe alloys [6]. Therefore, the corrosion behavior of Ni-Mo-Cr-Fe alloys in molten salt environments is critical to the safe operation of molten salt reactors.

During the 1960s-1970s, Oak Ridge National Laboratory in the United States conducted research on the corrosion behavior of Hastelloy N alloy in molten salts [5,7]. The results indicated that corrosion products of the alloy in fluoride salts dissolve in the molten salt, and material corrosion resistance cannot rely on passive films but depends on the thermodynamic equilibrium of corrosion reactions between the material and molten salt. This requires that the material composition be close to thermodynamic equilibrium with the molten salt composition. The studies also concluded that corrosion of the alloy in molten fluoride salts was primarily depletion corrosion of Cr. In the early 1980s, research [7,8] pointed out that the depletion corrosion behavior of high-Mo nickel-based alloys in LiF-NaF-KF molten salt involved simultaneous depletion of both Mo and Cr, not just Cr. When Mo content was low, intergranular corrosion occurred, but when Mo content increased above 15% (mass fraction), uniform corrosion was observed. At the beginning of this century, studies [9] found that low-activation ferritic steel and JLF-1 (8.92Cr-2W) exhibited Cr depletion corrosion in FLiNaK molten salt, along with Fe depletion corrosion. Olson et al. [10] investigated the corrosion behavior of various alloys in FLiNaK molten salt at 850 °C for 500 h, showing that weight loss increased with increasing Cr content.

Alloys in harsh service environments often employ protective coatings to improve performance. For example, in hot corrosion environments such as sulfate and halide molten salts, continuous and dense Al_2O_3 and Cr_2O_3 films can be formed during corrosion, or protective coatings such as Al_2O_3 or Cr_2O_3 can be directly applied to alloy surfaces to improve corrosion resistance [11-16]. Phahle et al. [17] reported that AlF_3 has strong adhesion to alloy surfaces, unlike Cr fluorides which have weak bonding with the alloy substrate. Studies on the corrosion behavior of Al-containing oxide dispersion strengthened (ODS) alloys in fluoride salts indicated that Al is beneficial for corrosion resistance in fluoride salts [18].

Currently, no studies have been reported on the effect of coatings on the corrosion resistance of superalloys in FLiNaK molten salt. While Al_2O_3 provides excellent protection in sulfate, nitrate, and halide molten salt hot corrosion environments, whether it possesses corrosion resistance in molten fluoride salt environments remains unclear.

This work investigates full immersion corrosion experiments on GH3535 superalloy with and without Al_2O_3 coating in FLiNaK molten salt. The corrosion behavior and mechanism of GH3535 alloy in molten salt are discussed, and the influence of Al_2O_3 coating on the corrosion resistance of GH3535 alloy and its mechanism are comparatively analyzed.

Experimental

The experimental alloy was independently developed GH3535 superalloy based on Hastelloy N, prepared using an advanced dual-smelting process. The main chemical composition of the alloy (mass fraction, %) was: Mo 16, Cr 7, Fe 4, C 0.04, Si 0.4, Mn 0.5, Ti+Al+Ta<2, Ni balance. The alloy was melted by vacuum induction melting and vacuum consumable melting, cast into 85 mm diameter ingots, and then hot-rolled into 16 mm diameter rods. After solution treatment at 1180 °C for 1 h followed by water quenching, corrosion specimens with dimensions of 30 mm × 10 mm × 2 mm were cut from the forged blanks. Some alloy specimens were ground with sandpaper up to 1200 grit, degreased, dehydrated, and dried with cold air for uncoated alloy corrosion experiments. The remaining specimens were coated with Al₂O₃ by plasma spraying with a coating thickness of 100 μm to investigate the effect of Al₂O₃ coating on molten salt corrosion resistance. All specimens were weighed using an electronic balance with 0.01 mg precision before the experiments.

The molten salt used in the experiments was eutectic FLiNaK (mole fractions of LiF, NaF, and KF were 46.5%, 11.5%, and 42%, respectively). Impurity content was tested to be: acid radical ions (SO₄²⁻ + PO₄³⁻ + NO₃⁻) less than 20% × 10⁻⁶; total oxygen content (including acid radicals, oxides, and water) less than 750 × 10⁻⁶; metal ions approximately 100 × 10⁻⁶%. The experimental temperature was 700 °C and the duration was 400 h. Uncoated and coated specimens were tested in two separate containers under identical conditions to prevent mutual influence.

The graphite crucibles were made of isostatic graphite (CDI-1A). The inner walls and components (hanging rods, lids, bolts, and nuts) of the graphite crucibles were ultrasonically cleaned in ethanol, vacuum-dried at 700 °C for 24 h, cooled to room temperature, and quickly transferred to a glove box for standby.

After corrosion, the specimens were cleaned, dried, and examined for surface morphology and corrosion product phase analysis. Following surface analysis, the specimens were subjected to electroless Ni plating, cold-mounted, mechanically ground, and polished for cross-sectional microstructure observation and elemental composition analysis. A D/MAX-RA X-ray diffractometer (XRD, CuKα) was used to analyze the phase composition of corrosion products on specimen surfaces at 50 kV tube voltage and 100 mA tube current, with a scanning range of 15°-85°, step size of 0.02°, and scanning speed of 4°/min. An INSPECT-F50 scanning electron microscope (SEM) was used to observe and analyze the surface morphology and measure the corrosion layer thickness, and an ISIS energy dispersive spectrometer (EDS) attached to the SEM was used for qualitative analysis of corrosion layer composition.

Results

The weight losses of GH3535 alloy specimens with and without Al₂O₃ coating after 400 h corrosion in FLiNaK molten salt at 700 °C were 9.29 and 1.86

mg/cm², respectively. Compared with the uncoated alloy, the coated alloy exhibited much more severe corrosion weight loss. The weight loss of uncoated GH3535 alloy was calculated by subtracting the post-corrosion weight from the pre-corrosion weight. For the coated GH3535 alloy, since the coating basically completely peeled off after corrosion, the weight loss was calculated by subtracting the post-corrosion weight from the weight of the specimen before coating application. If considering the residual Al₂O₃ coating remaining on the specimen surface, the corrosion weight loss of the coated alloy would be greater than 9.29 mg/cm². Evidently, the Al₂O₃ protective coating on GH3535 alloy surface not only failed to provide protection but actually aggravated alloy corrosion.

2.1 Surface Corrosion Morphology and Products

Figure 1 [Figure 1: see original paper] shows SEM images of the surface corrosion morphology of GH3535 alloy with and without Al₂O₃ coating after 400 h corrosion in FLiNaK molten salt at 700 °C. As seen in Fig. 1a, the surface of the uncoated alloy showed no significant corrosion morphology features, and no large corrosion products were observed. In contrast, large corrosion products were present on the surface of the coated alloy (Fig. 1b). EDS analysis indicated that the chemical composition of the corrosion products (mass fraction, %, the same below) was: O 57.62, Al 33.76, Cr 7.95, Fe 0.13, Ni 0.54. This suggests that the corrosion products on the alloy surface were Al-rich oxides, and it can be inferred that the large corrosion products on the alloy surface may have formed from residual Al₂O₃ coating after molten salt corrosion. Figure 1c shows an enlarged view of the corrosion surface morphology of uncoated GH3535 alloy. Blocky corrosion products several micrometers in size were observed on the specimen surface. EDS analysis showed the chemical composition of these corrosion products to be: O 56.14, Al 0.11, Cr 40.76, Fe 0.62, Ni 2.37, indicating that the corrosion products were Cr-rich oxides. In addition to large corrosion products, the coated GH3535 alloy surface also exhibited corrosion products similar in morphology and size to those on the uncoated alloy surface (Fig. 1d). The small corrosion products had a chemical composition of: O 35.36, Al 8.36, Cr 51.63, Fe 1.58, Ni 3.07. Thus, the small corrosion products were also Cr-rich oxides but contained Al. Further enlarged morphology of the uncoated alloy corrosion surface is shown in Fig. 1e, where clear intergranular corrosion features and grain detachment were observed. In contrast, the coated specimen surface exhibited numerous corrosion pits, showing obvious pitting corrosion characteristics. Compared with the uncoated alloy, no significant intergranular corrosion features were observed (Fig. 1f).

Figure 2 [Figure 2: see original paper] shows XRD patterns of the corrosion surfaces of GH3535 alloy with and without Al₂O₃ coating after 400 h corrosion in FLiNaK molten salt at 700 °C. As seen in Fig. 2a, the XRD pattern of the uncoated alloy surface only showed γ matrix peaks and LiCrO₂ peaks, suggesting that the corrosion product was LiCrO₂. In addition to γ matrix peaks and LiCrO₂ peaks, the XRD pattern of the coated specimen surface also

showed LiAlO_2 peaks. Therefore, the corrosion products of the coated alloy may be LiAlO_2 and LiCrO_2 . Combined with EDS analysis results, it can be determined that the corrosion product of the uncoated alloy was LiCrO_2 , while the large oxide product on the coated specimen surface was LiAlO_2 and the small oxide product was LiCrO_2 . It should be noted that Li did not appear in EDS but appeared in XRD patterns because Li is a light element that cannot be detected by EDS.

2.2 Cross-Sectional Morphology and Element Distribution

The secondary electron and backscattered electron images of the cross-section of the uncoated GH3535 alloy specimen after corrosion are shown in Figs. 3a [Figure 3: see original paper] and 3b, respectively. The alloy surface corrosion layer was shallow, not exceeding 10 μm , and contained a small amount of LiCrO_2 oxide particles.

EDS line scan analysis results from the corrosion layer surface toward the substrate of the uncoated GH3535 alloy are shown in Fig. 4 [Figure 4: see original paper]. Both Cr and Mo contents showed a decreasing trend near the specimen surface, indicating that Cr and Mo underwent depletion corrosion (Figs. 4a and 4b). Fe showed the opposite trend, with enrichment occurring in the Mo and Cr depletion layer (Fig. 4c). This is actually a relative enrichment of Fe caused by depletion of Mo and Cr. O and Al showed little change, only exhibiting weak baseline peaks (Figs. 4d and 4e), confirming their absence. Ni also did not undergo depletion corrosion (Fig. 4f).

Figure 5 [Figure 5: see original paper] shows the secondary electron and backscattered electron images of the cross-sectional morphology of the coated GH3535 alloy specimen after corrosion. Compared with the uncoated alloy, the coated alloy showed a significantly increased corrosion layer thickness, exceeding 20 μm , with a substantially increased number of oxide particles (LiCrO_2 and LiAlO_2) in the corrosion layer.

EDS line scan analysis results from the GH3535 alloy corrosion layer surface toward the substrate are shown in Fig. 6 [Figure 6: see original paper]. Within the range of 0-20 μm from the alloy surface, significant enrichment of Cr, O, and Al was observed (Figs. 6a, 6d, and 6e), which was found to be due to scanning across oxide particles (LiAlO_2 , LiCrO_2). Within the range of 20-50 μm from the alloy surface, a significant Cr depletion zone existed, with Mo also showing depletion at the same location, while Fe showed relative enrichment and Ni showed no depletion (Figs. 6b, 6c, and 6f). The cross-sectional morphology and EDS line scan results of the corroded specimens show that compared with the uncoated alloy, the coated alloy exhibited increased corrosion layer thickness, greater depletion depth of Mo and Cr, and more severe alloy corrosion.

Discussion

3.1 Corrosion Reaction of Uncoated Alloy in FLiNaK Molten Salt

Based on experimental results, the uncoated alloy primarily underwent depletion corrosion of Cr and Mo in FLiNaK molten salt. Corrosion pits of varying degrees appeared around alloy grain boundaries and carbides. Grain detachment was also observed from the corrosion surface morphology, with obvious intergranular corrosion features. Corrosion product LiCrO_2 formed but in small quantities and sizes.

Figure 7 [Figure 7: see original paper] shows a schematic diagram of the corrosion reaction of uncoated GH3535 alloy in FLiNaK molten salt. The solution-treated GH3535 alloy is a C-supersaturated solid solution, which is unstable. Therefore, during long-term aging at 700 °C for 400 h, supersaturated C atoms precipitate from the austenite. C atoms diffuse to grain boundaries and form carbides with Mo and Cr elements that diffuse there, precipitating M_{12}C -type carbides along grain boundaries [19]. In molten fluoride salts, since the electrode potential of carbides in the alloy is much higher than that of the alloy matrix, carbides and the surrounding matrix metal form a corrosion cell, with carbides as the cathode and the alloy matrix as the anode.

Carbide precipitation consumes large amounts of alloying elements such as Mo and Cr in grain boundary regions. The diffusion rates of Mo and Cr in grain boundaries are much greater than in grain interiors, causing the Mo and Cr consumed at grain boundaries to not be replenished in time from grain interiors, thereby forming depletion zones at grain boundaries. Since the passive state of grain boundaries is destroyed, the electrode potential of grain interior regions becomes higher than that of grain boundary regions, making grain interiors the cathodic region and grain boundaries the anodic region. In the corrosive medium, grain boundaries and grains form an active-passive cell with a large cathode-small anode area ratio, leading to significant intergranular corrosion. In the two corrosion cells formed between grain boundaries and carbides and between grain boundaries and grains, grain boundaries serve as the anode in both cases, meaning both corrosion cells cause corrosion of grain boundary regions. When carbides are located on grain boundaries (such as secondary carbides precipitated on grain boundaries), they further accelerate corrosion of grain boundary regions.

As the anodic region, grain boundary areas lose electrons to form cations such as Cr^{3+} , Mo^{6+} , Fe^{3+} , and Ni^{2+} according to the reaction $\text{M}(\text{Cr}, \text{Mo}) \rightarrow \text{M}^{n+} + \text{ne}$. The lost electrons flow to the cathode (carbides and grains). Due to impurities such as H_2O and FeF_3 in the corrosive medium [20], H_2O reacts with FLiNaK molten salt at high temperature to form H^+ and O^{2-} according to $2\text{LiF} + \text{H}_2\text{O} \rightarrow 2\text{Li}^+ + \text{O}^{2-} + 2\text{HF}$. Cations Fe^{3+} and H^+ in the solution flow to the cathodic region (carbide and grain surfaces), where they gain electrons and are reduced to Fe and H_2 according to $\text{Fe}^{3+} + 3\text{e} \rightarrow \text{Fe}$ and $2\text{H}^+ + 2\text{e} \rightarrow \text{H}_2$. Anions O^{2-} and F^- in the molten salt flow to the anodic region (grain

boundary areas), forming Cr_2O_3 and CrF_2 with metal cations that have lost electrons according to $2\text{Cr}^{3+} + 3\text{O}^{2-} \rightarrow \text{Cr}_2\text{O}_3$ and $\text{Cr}^{3+} + 3\text{F}^- \rightarrow \text{CrF}_3$. The oxidation product Cr_2O_3 further reacts with O^{2-} and Li^+ in the molten salt to form LiCrO_2 according to $\text{Cr}_2\text{O}_3 + \text{Li}_2\text{O} \rightarrow 2\text{LiCrO}_2$. When the H_2O content in the corrosive medium is high, Mo is more easily oxidized to form MoO_3 according to $\text{Mo}^{6+} + 3\text{O}^{2-} \rightarrow \text{MoO}_3$, and MoO_3 dissolves into the fluoride salt, causing significant Mo corrosion [21]. Cr and Mo on the alloy surface are corroded and dissolve into the fluoride salt. Metal elements in grain boundary regions continuously lose electrons, are oxidized to cations, and detach from the metal matrix. The driving force generated by element concentration differences between grain boundary regions and grain interiors, as well as between the alloy surface and substrate interior, causes continuous diffusion of elements from the alloy matrix to the specimen surface and grain boundaries. Cr and Mo form concentration gradients between the specimen surface and substrate, exhibiting depletion corrosion phenomena.

It should be noted that during the depletion corrosion process of Cr and Mo, Fe did not undergo depletion but remained in the Ni matrix at the alloy surface. Since the Cr and Mo contents in the surface layer of the alloy decreased sharply, this caused a relative increase in Fe content, creating a false appearance of Fe enrichment at the surface. In reality, this is a relative enrichment phenomenon.

3.2 Corrosion Behavior of Al_2O_3 Coating in FLiNaK Molten Salt

Experimental results show that the Al_2O_3 coating dissolved and peeled off in FLiNaK molten salt and therefore did not provide effective corrosion protection. Figure 8 [Figure 8: see original paper] shows a schematic diagram of the reaction of Al_2O_3 coating in FLiNaK molten salt. While Al_2O_3 coatings typically have good molten salt corrosion resistance, they lack corrosion resistance in FLiNaK molten salt because fluoride salts are highly hygroscopic. Impurity H_2O reacts with FLiNaK molten salt to form H^+ , F^- , and O^{2-} ions according to $2\text{LiF} + \text{H}_2\text{O} \rightarrow 2\text{Li}^+ + \text{O}^{2-} + 2\text{HF}$ [22]. HF readily reacts with Al_2O_3 according to $\text{Al}_2\text{O}_3 + 6\text{HF} \rightarrow 2\text{AlF}_3 + 3\text{H}_2\text{O}$ [23,24]. Additionally, the molten salt contains impurities such as NiF_2 , FeF_2 , CrF_2 , and HF [7,9,10], which promote accelerated dissolution corrosion of Al_2O_3 . Furthermore, Li^+ and O^{2-} in the molten salt react with the corroded and loosened Al_2O_3 to form LiAlO_2 according to $\text{Al}_2\text{O}_3 + \text{Li}_2\text{O} \rightarrow 2\text{LiAlO}_2$.

3.3 Corrosion of Alloy After Al_2O_3 Coating Failure

Since Al_2O_3 can chemically react with FLiNaK molten salt, defects in the Al_2O_3 coating such as cracks and pores preferentially undergo dissolution. When the coating becomes very thin at these defect locations, F^- in the molten salt penetrates the coating and chemically reacts with the substrate metal. When the coating completely fails, O^{2-} also reacts with the metal, forming corrosion products such as Cr_2O_3 and CrF_2 through reactions like $2\text{Cr}^{3+} + 3\text{O}^{2-} \rightarrow \text{Cr}_2\text{O}_3$, $\text{Mo}^{6+} + 3\text{O}^{2-} \rightarrow \text{MoO}_3$, and $\text{Cr}^{3+} + 3\text{F}^- \rightarrow \text{CrF}_3$, as shown in Fig. 9a [Fig-

ure 9: see original paper]. As corrosion progresses, the alloy surface eventually becomes exposed due to coating dissolution and peeling. Since the initially corroded alloy surface (corrosion pits) contains numerous defects such as composition inhomogeneity and porosity, a corrosion cell forms between this region and the finally exposed intact alloy surface, with the newly exposed intact alloy surface as the cathode and the defective corrosion pit as the anode [25]. Metal in the corrosion pit continuously loses electrons and is oxidized to cations such as Cr^{3+} , Mo^{6+} , Fe^{3+} , and Ni^{2+} , with the lost electrons flowing to the intact alloy surface. Cations such as Fe^{3+} and H^+ in the molten fluoride salt flow to the cathodic region, where they gain electrons and are reduced to Fe and H_2 . Anions such as O^{2-} and F^- flow to the anodic region (corrosion pit), forming Cr_2O_3 and CrF_2 with metal cations that have lost electrons, as shown in Fig. 9b. For the exposed alloy surface, the same corrosion reactions as the uncoated alloy (intergranular corrosion) occur, but the influence of the Al_2O_3 coating makes the corrosion morphology features caused by intergranular corrosion and carbides less significant. After Al_2O_3 coating dissolution and peeling, the alloy specimen eventually exhibits the same corrosion behavior as the uncoated alloy, showing depletion corrosion of Cr and Mo and forming a surface layer depleted in Cr and Mo.

Based on the above experimental results, oxide coatings do not provide corrosion resistance in fluoride salts. Since Ni and Fe in the alloy did not undergo depletion corrosion, protective coatings of pure Ni or pure Fe could be considered. Additionally, alloy corrosion resistance could be improved by adjusting the alloy substrate composition, such as reducing the content of Mo and Cr which are prone to depletion corrosion. The content and distribution of carbides in the alloy should also be controlled to reduce corrosion caused by corrosion cells formed between carbides and the alloy substrate, particularly the amount of carbides distributed along grain boundaries. Whether these approaches can effectively improve alloy corrosion resistance in molten salt requires further verification.

Conclusions

Both uncoated and Al_2O_3 -coated GH3535 alloys exhibited depletion corrosion characteristics of Cr and Mo after 400 h corrosion in FLiNaK molten salt at 700 °C. The uncoated alloy showed intergranular corrosion features, while the Al_2O_3 -coated alloy showed significant pitting corrosion features. The Al_2O_3 coating dissolved in molten FLiNaK salt, forming pitting pits at the earliest coating failure locations and creating corrosion cells with the finally exposed intact alloy surface due to oxide film dissolution and peeling, thereby accelerating alloy corrosion. The corrosion layer depth, size, and number of corrosion products all increased significantly. The Al_2O_3 coating did not improve the corrosion resistance of GH3535 alloy in FLiNaK molten salt at 700 °C.

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