

Facility to test the compatibility of materials in molten salts for MSBR applications

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ABSTRACT

Understanding the corrosion behavior of structural materials in molten fluoride salts like FLiNaK is crucial for the development of the Molten Salt Breeder Reactor (MSBR). However, handling of molten fluoride salts during long term experimentation is quite challenging due to their high reactivity with oxygen and moisture which is even greater than that of liquid metals and alloys. In this regard, a unique glove box coupled molten salt corrosion test facility (MSCTF) with automatic sample exchange system has been designed, developed and installed at Materials Group, BARC. All wetted parts of this facility were made up of pure nickel, and provision had been made to conduct molten salt corrosion experiments under controlled atmosphere at temperatures up to 1273 K (1000°C). Various Ni based alloys including the indigenously developed Ni-Cr-Ti-Mo alloy have been exposed to molten FLiNaK salt under static condition in MSCTF and few salient results are presented in this paper.

Keywords: Molten Salt, Corrosion, FLiNaK, Hastelloy, Ni-Cr-Mo-Ti

Introduction

In order to meet the demand for a sustainable source of carbon-free energy, various advanced nuclear reactors are being designed across the globe [1]. One of the most promising candidates in this fleet is Molten Salt Reactor (MSR) [2-3]. The major advantage of MSR comes from its liquid fuel characteristics such as (i) negative temperature coefficient of reactivity, (ii) facilitates online fuel reprocessing, and (iii) allows efficient utilization of fertile thorium [1-4]. MSR is associated with an inherent safety feature since it can passively regulate its own temperature [4]. In a scenario where the reactor gets overheated, the reactivity of the core automatically comes down thus making the temperature coefficient of reactivity negative [2-3, 5]. Keeping this in mind and with an aim to efficiently utilize its abundant thorium reserves, India has initiated MSR development program and proposed a conceptual design of Indian Molten Salt Breeder Reactor (IMSBR) [5-6].

The fuel salt for IMSBR is generally a mixture of fluorides of Uranium, Thorium and Lithium while the eutectic mixture of LiF, KF and NaF (46.5% LiF–11.5% NaF–42% KF also known as FLiNaK) has been selected as the

coolant salt for this system [3,5-6]. The major advantage of using molten fluoride salt is its better thermo-physical properties viz., high thermal conductivities, high specific heats, low viscosities, low vapour pressure (<1 atm), high boiling points and favourable neutronic properties viz. neutron absorption cross-section, irradiation stability etc [7]. However, corrosion of structural materials in molten fluoride salt environment is considered as a major issue in practical realisation of such MSBR system [8]. This is because of the fact that the reaction products formed by the fluorination of the alloying metals are soluble in the salt. Thus, the protective oxide film available at the material surface becomes chemically unstable in the presence of fluoride salt leading to thermodynamically driven dissolution of alloying elements in the molten salt and vice-versa [8-9].

In general, nickel based alloys, due to their satisfactory compatibility towards liquid fluoride salt combined with excellent high temperature mechanical properties, are proposed as structural materials for molten salt systems [9-10]. Researchers across the globe are working on fine tuning the composition of readily available Ni based alloys, like Hastelloy N and Inconel 800, to optimise the structural and mechanical properties which are best suited

for the operating temperature regime of molten salt reactors (923 K to 1273 K i.e. 650°C to 1000°C) [11-12]. In this context, a novel Ni based alloy with Mo, Cr and Ti as the principle alloying elements has been developed as the candidate (structural material) of the IMSBR [13,14]. The alloy (Ni-11Mo-7Cr-2.4Ti) was prepared using vacuum induction melting (VIM) route and its microstructural as well as welding characteristics have been investigated in detail [13-14]. Understanding the corrosion behavior of this alloy in molten fluoride salts like FLiNaK within the regime of reactor operating parameters is extremely crucial for the development of IMSBR. The biggest challenge, related to the handling of molten fluoride salts during experimentation and storage involves their high reactivity with oxygen and moisture which is even greater than that of liquid metals and alloys [7]. Keeping this in mind, a unique glove box coupled molten salt corrosion test facility has been designed, developed and installed at Materials Group, BARC. The facility provides an opportunity for long term corrosion testing of structural

and functional materials in molten salts (fluorides, chlorides, nitrates) under static conditions at elevated temperatures up to 1273 K (1000°C). The present report provides a detailed overview of the construction and working of this facility and presents the initial results from molten salt corrosion experiments carried on various imported Ni based alloys as well as the indigenously prepared Ni-Cr-Mo-Ti alloy.

Description of Experimental facility

Figure 1 shows the schematic of a fully automated Molten Salt Corrosion Test Facility (MSCTF) which facilitates static compatibility testing under inert gas atmosphere as well as in vacuum ($\sim 10^{-6}$ mbar). It also incorporates the feature of automatic sample immersion/exchange thereby overcoming the challenge of sample insertion/removal at high temperature keeping the controlled atmosphere intact.

As shown in Figure 1, a double chamber retort (144 mm outer diameter (OD) x 450 mm long x 12 mm thick) made

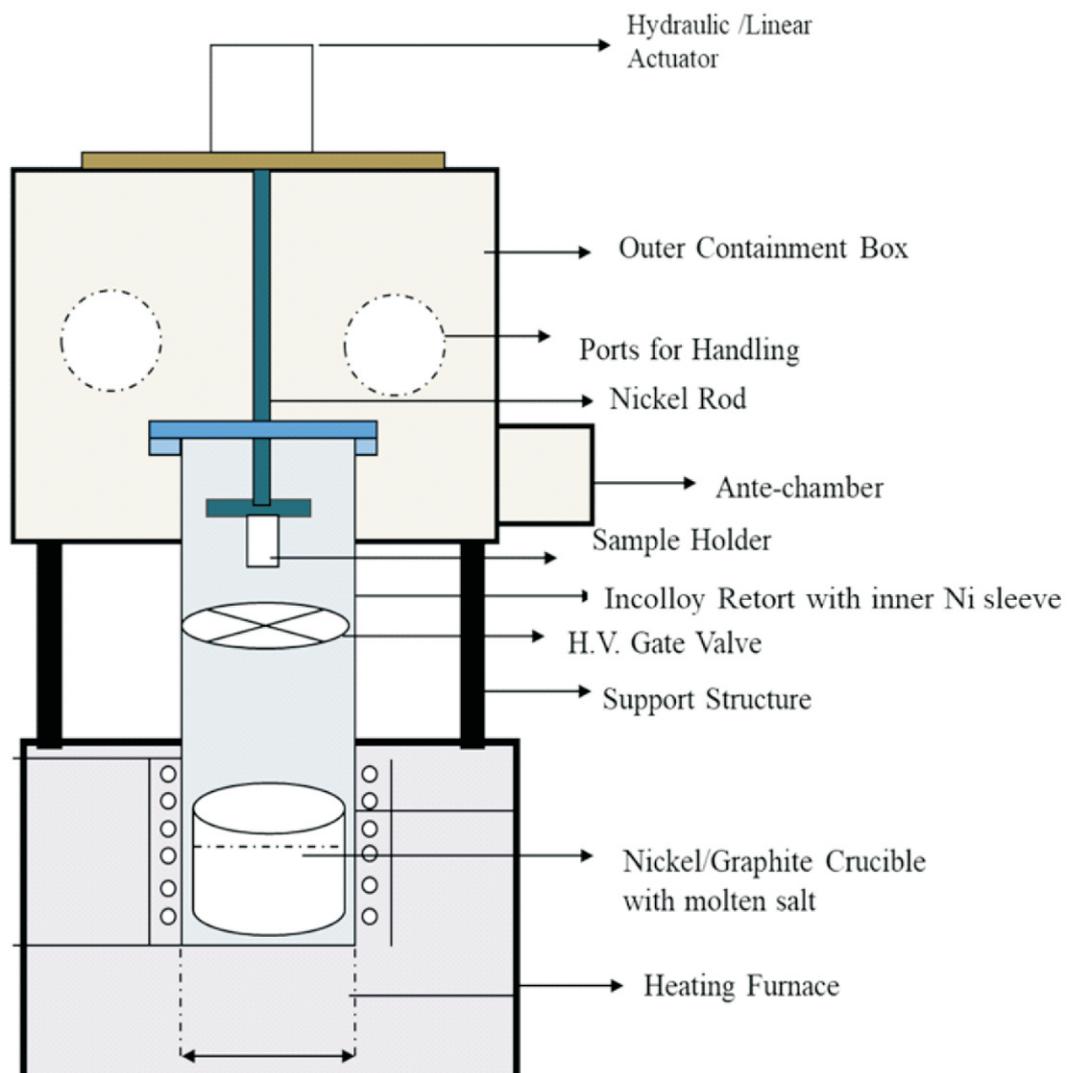


Fig. 1: Schematic of molten salt corrosion test facility (MSCTF)

out of Incolloy 800 is located at the heart of the MSCTF. Incolloy 800 has been chosen as the material of construction for this retort as it can withstand high temperatures during continuous operation for prolonged duration (~ 5000 h). The retort is completely covered from inside with a 6 mm thick pure Nickel sleeve which is known to be resistant to fluoride attack and to minimise galvanic corrosion due to dissimilar metal contact with the molten salt [15]. For example, a study by Olson et al. showed that the corrosion rate in Hastelloy N with graphite crucible at 850°C is $45.5 \pm 4.0 \mu\text{m}/\text{year}$ while with Ni crucible, the rate is $15.8 \mu\text{m}/\text{year}$ [16]. A crucible (100 mm OD x 100mm long x 3mm thick) containing the molten salt is kept at the bottom of this retort where its temperature could be raised up to 1000°C by a heating furnace. At present, the crucibles for holding molten fluoride salts are made of pure nickel, although there is a provision to customise the material of construction as per the operating conditions. The nickel crucible is held in a crucible cage which increases the ease of lifting it out of the retort during replacement of salt. Dedicated holders made out of pure nickel are used for holding the corrosion coupons. As shown in Figure 3, various geometries of holders are available for holding single or multiple samples at a time.

The sample holder is fixed to a pure nickel rod which is connected to a linear actuator system responsible for the insertion and removal of samples inside molten salt. In order to prevent moisture ingress into the salt during preparation and handling, the entire top portion of the retort is enclosed in a tailor-made glove box which facilitates sample exchange and salt preparation under controlled atmosphere. The inert gas is passed through silica gel based moisture purifier and catalyst based

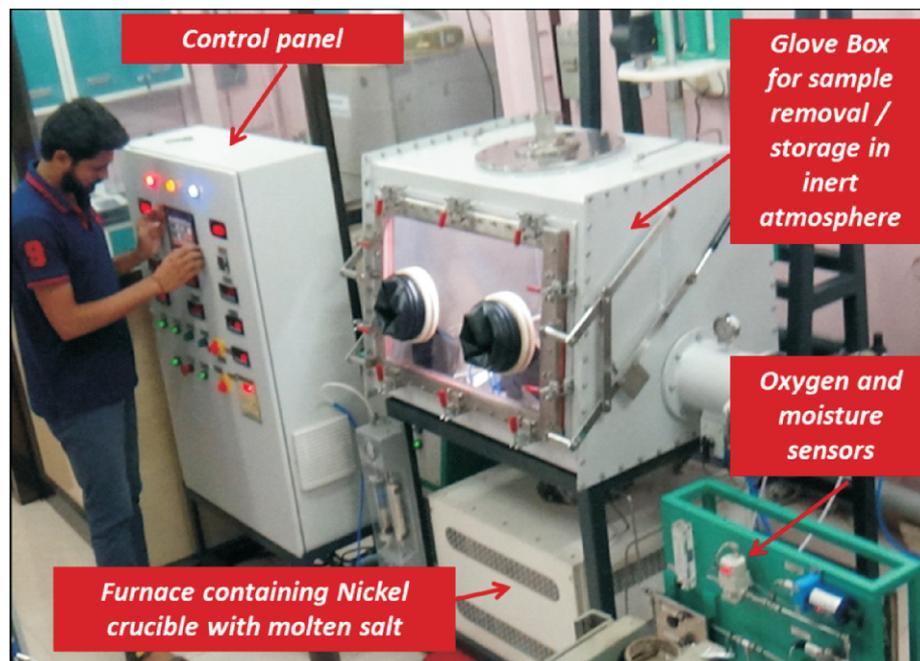


Fig. 2: The molten salt corrosion test facility developed in Materials Science Division, BARC

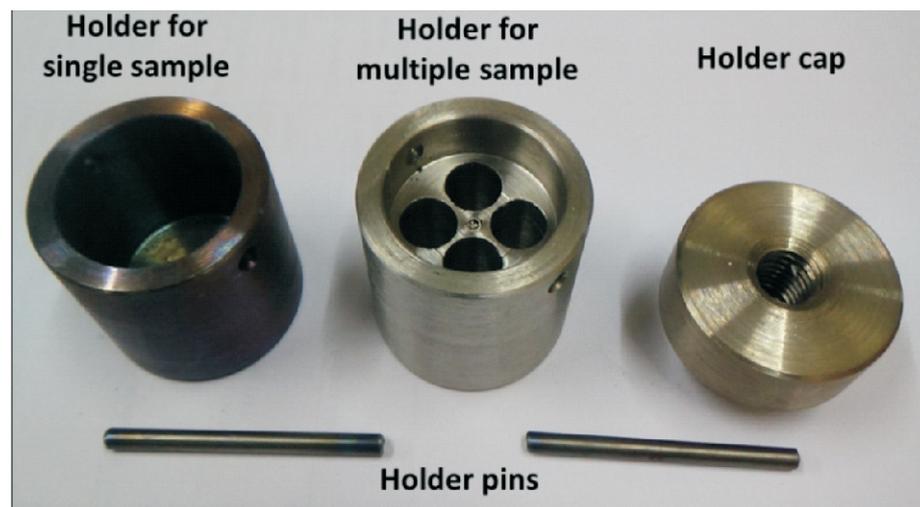


Fig. 3 : Different sample holders of molten salt compatibility testing (MOC: Nickel)

oxygen purifiers before introducing into the retort or glove box. Dedicated moisture and oxygen sensors are placed in the inlet and outlet gas lines of the retort to monitor the experimental conditions. Intermittent purging by dry argon gas helps to maintain an inert environment during molten salt corrosion experiment as well as to carry possible gaseous HF away from the autoclave as outlined by Ouyang *et al.* [17].

A special feature of the MSCTF system is a pneumatically operated high vacuum gate valve (MOC: Incolloy 800H)

which can separate the Incolloy retort into two halves when the samples are held in the upper chamber. During the start of an experiment, the valve is kept closed and the samples holder with samples fixed to the nickel rod is kept in the upper half of the retort (i.e. above the HV gate valve). Thereafter, the salt in the nickel crucible is allowed to melt under inert atmosphere. During melting, the salt is heated in a stepwise manner to the operating temperature to ensure only minimal oxide impurities get generated by the accelerated reaction of residual moisture with the salt during the heating process. The temperature of the salt is measured by a K-type thermocouple connected to the inner surface of the Incolloy Retort. After melting, both parts of the retorts are brought under similar controlled atmospheric conditions before opening of the valve and lowering the sample holder to the bottom half to dip it inside molten salt for the required duration of exposure. Conductance based level indicators have been installed at various locations to monitor and control the movement of the sample holding rod. Once the experiment is completed, samples are brought to the upper part and the valve is closed back. Under the closed condition of the valve, top cover flange of the retort could be opened and the samples can be removed or fresh samples could be loaded. This arrangement allows to keep the salt in molten state without exposing it to outside atmosphere during sample exchange. The presence of glove box containment provides an additional layer of safety allowing removal of the samples and handling of the salt under inert atmosphere for sampling/refilling as and when required.

Special kind of seals are designed for this system which allow movement of the sample holders through the top

flange covering the retort as well as the glove box without breaking the controlled atmosphere. The major flanges of the retort including the valve flange as well the retort cover flange are cooled by a recirculating chilled water to maintain the ambient temperature. The entire operation of the system is automatically controlled through PLC based controllers which operates through a compact LCD type touch screen interface. All required interlocks and indicators have been incorporated to ensure operational safety. The above discussed arrangement helps to make the sample exchange process fast, simple and efficient in addition to repeated usage of the set-up for conducting multiple experiments.

Experiments Conducted

The MSCTF system has been successfully commissioned and several experiments have been conducted in FLiNaK salt. In the initial stage of commissioning, FLiNaK salt has been prepared in 500 g scale as shown in Figure 2. In order to reduce the contamination of moisture to a minimum, the powders of individual salts (LiF, KF and NaF) have been introduced inside the glove box of MSCTF and dehydrated in high purity argon atmosphere. The salt was later prepared by mixing the powders in eutectic composition (46.5 mol% LiF–11.5 mol.% NaF–42 mol% KF) and heating the mixture at 873 K (600 °C). The melting point of the resultant salt mixture was found to be 729 K (456 °C) which was very close to the melting pint of FLiNaK (727 K or 454 °C). The impurity content of the prepared salt was analysed through ICP-MS and is listed in Table 1. Fig. 4 gives a photograph of the FLiNaK salt held in the nickel crucible inside MSCTF.



Fig. 4: FLiNaK salt prepared in MSCT Facility

Composition of FLiNaK salt prepared in MSCTF obtained through ICP-MS (Table 1)

Al (%)	Cr (ppm)	Cu (ppm)	Fe (ppm)	Mn (ppm)	Ni (ppm)	Li (%)	Na (%)	K (%)	F (%)
0.33	14.1	11.1	332.5	6.4	<7	9.49	12.76	13.82	38.6

List of materials exposed to FLiNaK in MSCTF till date (Table 2)

S.No	Materials	Temperature (K)	Duration (h)	Atmosphere
1.	Hastelloy N	973	72	High Purity Argon
2.	Hastelloy X	973	72	High Purity Argon
3.	Ni-Cr-Mo-Ti alloy	973	50	High Purity Argon
4.	Ni-Cr-Mo-Ti alloy (welded)	973	50	High Purity Argon

Composition of various materials exposed in FLiNaK inside MSTCF (Table 3)

	Ni	Mo	Cr	Ti	Fe	Si	Mn	V	C	Cu	W	Al	Nb
Hastelloy N (Imp)	bal	16.11	6.31	<0.01	4.03	0.38	0.53	0.02	0.03	0.01	0.06	<0.01	NA
Hastelloy X	bal	8.76	21.34	0.01	19.29	0.58	0.54	-	0.59	-	0.4	0.08	NA
Ni-Cr-Mo-Ti alloy	bal	11.07	7.16	2.42	0.073	0.13	0.23	NA	0.03	NA	NA	0.25	0.25

Various samples of commercially available Hastelloy along with the indigenously produced Ni-Cr-Mo-Ti alloy have been exposed to FLiNaK in this facility. The effect of welding on the molten salt compatibility of Ni-Cr-Mo-Ti alloy has also been studied by exposing samples having a bead on plate by Tungsten Inert Gas (TIG) welding on them. The TIG welding was carried out at a voltage of 15 V with a current of 70 A using a filler of the same composition as the base material. The temperature of exposure has been chosen to be 973 K (700°C) for the present experiments since this falls within the operating regime of IMSBR and a large corrosion database of Ni based alloys in fluoride salts is available at this temperature [5,9-10, 18]. Table 2 gives the list of experiments carried out in this facility till date along with the experimental parameters while Table 3 gives the composition of each material listed in Table 2.

The exposed samples were cleaned off to remove the adherent molten salt by repeatedly exposing them to a 1 M solution of aluminium nitrate (Al(NO₃)₃) and washing with deionized water. Afterwards, the weight loss due to molten salt exposure was measured in order to find the corrosion rate as per equation 1.

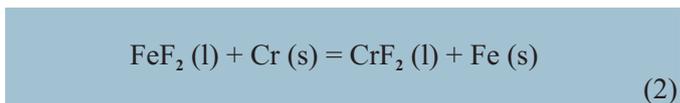
$$\text{Corrosion rate} \left(\frac{\mu\text{g}}{\text{cm}^2 \cdot \text{h}} \right) = \frac{\text{Weight Loss (g)} \times 10^{-6}}{\text{Surface Area exposed (cm}^2\text{)} \times \text{Duration of exposure (h)}} \quad (1)$$

Afterwards, the surface of the samples before and after exposure to molten salt was analysed with the help of Scanning Electron Microscope (SEM) and Electron Back Scattered Diffraction (EBSD).

Results and Discussions

Table 4 enlists the weight loss and corrosion rate of various materials after exposure to FLiNaK in MSCTF at 973 K while figure 5 depicts the variation in corrosion rate in the

form of bar graphs for better comparison. It could be clearly observed from Figure 5 that the molten salt corrosion rate of indigenously produced Ni-Cr-Mo-Ti alloy was comparable to that of imported Hastelloy N and much lesser than that of Hastelloy X, which has the highest chromium content among all the exposed alloys. It should be noted that the corrosion rate in fluoride salt is generally higher in the initial periods of exposure due to presence of impurities as well as due to the presence of a higher fraction of fresh active surface of exposure [8, 9, 18]. The metallic impurities such as Fe, if present in fluoride salt, can selectively leach out Cr via the reaction shown in Equation 2.



Where “l” and “s” stands for liquid and solid phases respectively.

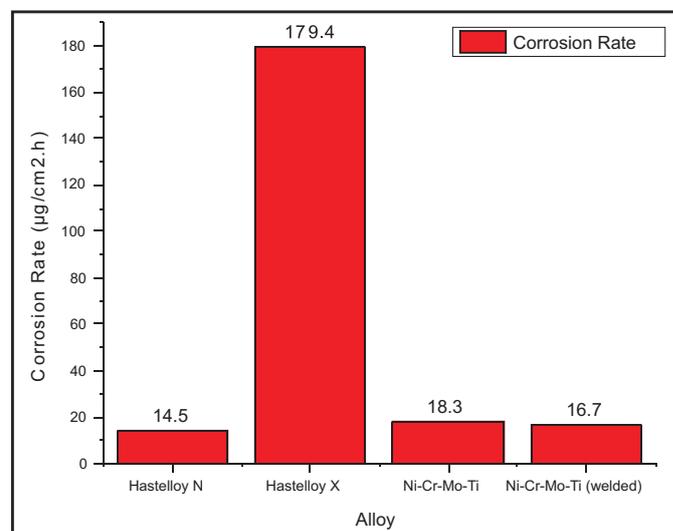


Fig. 5: Corrosion rate of various materials exposed in FLiNaK at 973 K in MSCTF

	Duration (h)	Initial wt. (g)	Final wt. (g)	Wt. loss (g)	Area (cm ²)	Corrosion rate (μg/cm ² .h)
Hastelloy N	72	6.3584	6.3533	0.0051	4.8828	14.50
Hastelloy X	72	7.6577	7.5838	0.0739	5.7132	179.65
Ni-Cr-Mo-Ti	50	3.6701	3.6666	0.0035	3.8521	18.17
Ni-Cr-Mo-Ti (welded)	50	1.4562	1.4543	0.0019	2.2269	17.06

Weight Loss, Exposed Surface Area & Corrosion Rate of materials exposed in FLiNaK at 973 K in MSCTF (Table 4)

However, once the impurities are exhausted, a thermodynamic equilibrium is reached and the corrosion rate decreases (generally for exposure at extended periods of time). Therefore, the corrosion rate of Ni-Cr-Mo-Ti alloy after prolonged duration of exposure (> 500 h) in FLiNaK is expected to be significantly lower than what has been obtained for 50 h exposure in the present case.

Nevertheless, the present experiment has revealed that the presence of TIG welding on Ni-Cr-Mo-Ti alloy did not adversely affect its corrosion resistance. The corrosion rate was found to be rather lower in the case of welded samples as shown in Figure 5. As molten salts are ionic in nature and good electrical conductors, oxidation-reduction reactions will readily occur when these salts act as a medium [9,18-20]. In the presence of an inherent oxidant (ox), fluoride salts generally oxidize the alloying elements (M) in the structural materials like Cr, Ni, Mo, etc, thereby forming their respective fluorides [18-20]. In general, such formation of metal fluorides and their subsequent dissolution in FLiNaK is an irreversible electrochemical process which occurs via Equations 3,4 and 5 [19-20].

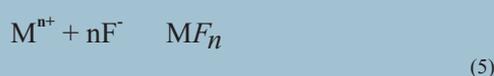
Anodic dissolution of metal:



Cathodic evolution of the oxidant in the salt:



Formation of metal fluorides:

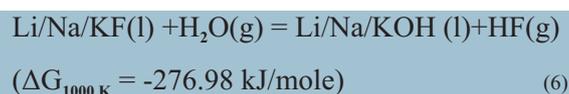


where “Ox” and “Red” represent the oxidant present in the salt (oxidizing impurities like moisture or HF which are inherently present in fluoride salts) and its corresponding reductant respectively while F⁻ in equation 5 is generated from HF or from the salt itself.

The fluoride of the alloying element (M) having higher stability (i.e. lowest free energy, ΔG° in Equation 5) will have a greater tendency for the formation of fluorides in the presence of fluoride salts like FLiNaK [18-20]. Such elements will therefore, be preferentially leached out in the molten salt. The fluoride forming tendencies of the major alloying elements in Ni-based alloys like Hastelloy N are as follows [18]:-

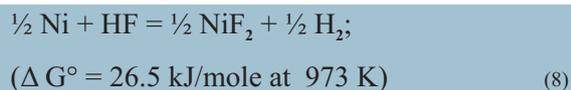
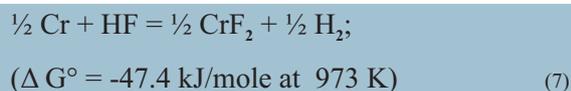


Therefore, it is evident that chromium will have the highest probability of leaching out from structural materials and thus the corrosion rates in FLiNaK as observed in Figure 5 could be directly related to the chromium content of the respective materials. Nevertheless, the corrosive nature of halide salts like fluorides and chlorides are aggravated in the presence of oxidising contaminants like H₂O and HF [18-20]. In fact, the inherent presence of a small quantity of such contaminants in fluoride salts is generally the cause of the initiation of corrosion attack over structural materials as shown in equation 4 [19-20]. Due to the inherent hygroscopic nature of the fluoride salts, they tend to absorb moisture during the production and subsequent storage. As per Equation 6, the presence of moisture in fluoride salts leads to the formation of a highly deleterious corrosive agent, HF [20-21].



Where “l” and “g” stands for liquid and gaseous phases respectively.

The gaseous HF generated is dissolved and strongly retained in the molten fluoride salt which lead to further corrosive attack on the alloying elements of structural materials. Two such reactions with Cr and Ni are shown in equation 7 and 8 [18, 20-21]



Thus, maintaining purity of the salts in terms of oxygen and moisture through a suitable control system is of utmost importance during a molten salt corrosion experiment.

The attack by molten fluoride salt has been found to mainly confine to the grain boundaries during the first 50 h of exposure in the present study. This phenomenon, in fact,

etches the samples so well that all grain boundaries in the weld and base metal could be clearly revealed by the attack [22]. Figure 6 shows SEM micrograph of the exposed surface of welded sample of Ni-Cr-Mo-Ti alloy after exposure to molten FLiNaK at 973 K for 50 h. Even in un-etched condition, the elongated columnar grains of the welded region could be clearly distinguished from the large equiaxed grains of the base material which could be attributed to the preferential attack at the grain boundaries. In addition, pits due to molten salt corrosion were observed in the base metal only [18]. This complemented our result of lower corrosion rate of the welded samples (Figure 5) although such a reduction in the presence of welding is counterintuitive. In order to understand this fact, a detailed grain boundary analysis of the weld and base metal regions of the as-received Ni-Cr-Mo-Ti alloy was conducted through EBSD. Figure 7 shows the fraction of grain boundaries with various mis-orientation angles in weld and base metal regions of Ni-Cr-Mo-Ti alloy. It was clearly

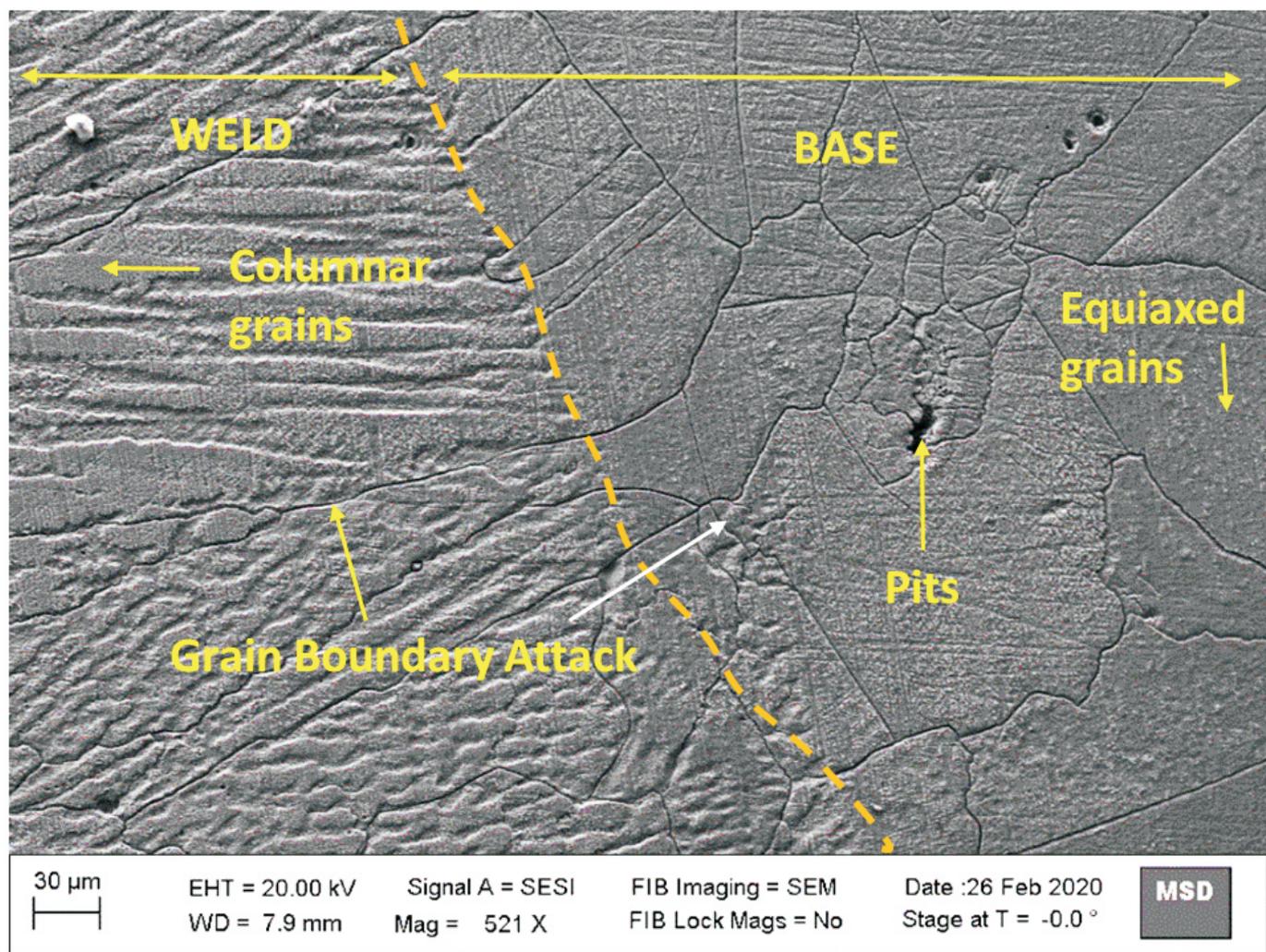


Fig. 6: SEM image of exposed surface of indigenously developed Ni-Cr-Mo-Ti alloy showing grain boundary attack in the weld and base metal

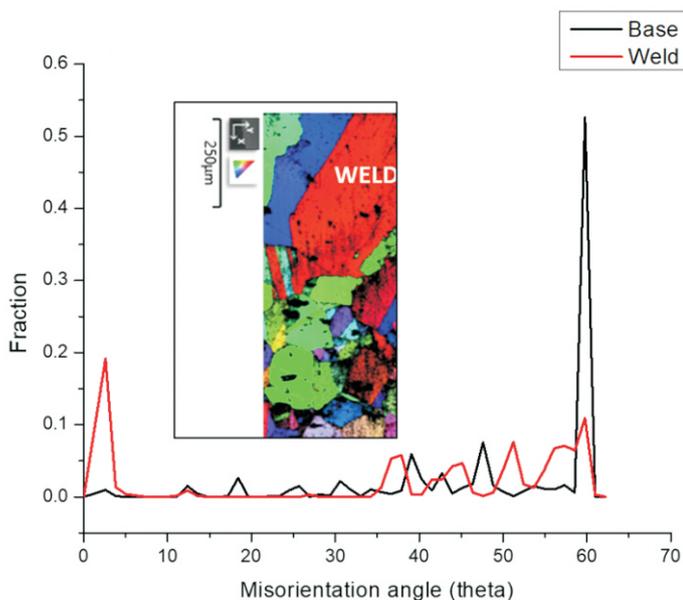


Fig. 7: Fraction of high and low angle grain boundaries in the base metal and weld zone of as-received Ni-Cr-Mo-Ti alloy. Inset shows EBSD map of Ni-Cr-Mo-Ti alloy used for analysis

observed that the fraction of high angle grain boundaries (i.e. with higher mis-orientation angle and higher grain boundary energy) was distinctly lower in the case of the weld region. Since the probability of grain boundary attack is directly related to the energy of the grain boundaries, it is highly possible that reduction in the number of high angle grain boundaries is responsible for decrease in corrosion rate at the welded region [23]. The results from the present study indicate that the alloy microstructure and the grain boundary distribution play a prominent role in the corrosive attack by molten fluoride salt.

Summary

A fully automated Molten salt corrosion test facility (MSCTF) has been developed at Materials Group, BARC. The set up facilitates corrosion testing in molten fluoride and other salts up to 1273 K under inert atmosphere or vacuum. The facility has been successfully commissioned and several corrosion tests in FLiNaK salt have been carried out. The molten salt corrosion behavior of indigenously developed Ni-Cr-Ti-Mo alloy has been tested at 973 K in FLiNaK and found satisfactory. The effect of welding of the molten salt corrosion behavior have also been explored where results showed no reduction in corrosion resistance in the case of welded samples.

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