

OVERVIEW OF MATERIALS AND FLUORIDE SALTS USED IN MOLTEN SALT REACTORS

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ABSTRACT

The purpose of this work is to explain the choice of materials and fluoride salts for Molten Salt Reactors. The article describes the problem of corrosion in these reactors and the possibilities of its mitigation. There are usable materials for MSR and their cover layer such as glass graphite. Further, there is described metal refining for the production of materials for MSR. Finally, fluoride salts, their purification, and the solubility of the fuel in them are described.

KEY WORDS

MSR reactors, fluoride salts, impurities, oxidation – reduction potencial, corrosion, Hastelloy, Monicr – Skoda JS a.s.

INTRODUCTION

The Generation IV International Forum (GIF) proposed six types of generation IV nuclear reactors. They are characterized by higher thermodynamic efficiency than current generation III reactors. One of these reactor types is the Molten Salt Reactor. This reactor is cooled with fluoride salts, which are very corrosive to most construction materials, so it is necessary to find a material that resists molten fluoride salts. The materials and the alloys used in Molten Salt Reactors must be very pure. The fluoride salts must also be very pure.

METAL MATERIALS SELECTED FOR MSR

The selection of pure materials for the alloys is in progress. Several materials have been chosen for using with fluoride salts, such as: Hastelloy, Monicr, Russian HN80M alloys. The French are considering a material usable up to 730 °C for high temperature hydrogen reactors. The chemical composition of this material is given in [1,2], but research has not been published about its composition. It is therefore a question of what stage of development this material is at. However, this material contains impurities such as phosphorus and sulphur in relatively large amounts. The chemical composition of this material is shown in Table 1, in mole%.

Ni	W	Cr	Mo	Fe	Ti	C	Mn	Si	Al	B	P	S
85.130	7.217	5.790	0.532	0.457	0.213	0.212	0.186	0.182	0.038	0.024	0.016	0.003

Tab. 1: Chemical composition of nickel alloy proposed by the French [1]

Haynes International is the author and manufacturer of Hastelloy N for MSRE (Molten Salt Reactor Experiment). Currently, this material is not available. The delivery time of this material is 3 to 4 years and the quantity which can be delivered is about 10 tons. That was the reason for the development of Monicr - Skoda JS a.s. for manufacturing an experimental loop for research on fluoride salts.

The materials and the fluoride salts used in molten salt reactors must be very pure. The metals were refined at the Kharkov Institute of Physics. Various refining technologies have been developed here. Two alloys, A and B, have been developed with high purity input components with a low level of interstitial impurities, see Table 2.

The alloy components (nickel, molybdenum, chromium, iron, aluminium, titanium, manganese) were previously stripped of foreign matter (impurities) by physical methods. Since these metals differ significantly, the same method cannot be used for all the elements. In order to refine nickel, molybdenum niobium, titanium and iron, an electron beam vacuum melting method (EBM) was used. High pressure annealing was used for refining chromium and aluminium. Manganese was refined by vacuum distillation.

Metal refining of the alloys resulted in the following: Nickel. Electrolytic nickel, twice subjected to EBM, was used as the starting material. Improvements reduced levels of iron, cobalt, phosphorus, aluminium, magnesium; the most significant reduction was the decrease in the content of arsenic, zinc, selenium and chromium. The double EBM gave nickel with a purity of 99.994 weight %. Molybdenum. After EBM, the content of metal impurities decreased 10 to 30 times. Silicon removal was not so significant. Tungsten impurities were not removed from the molybdenum at all. Gaseous impurities (oxygen, nitrogen and hydrogen) were removed. [5]

Alloy brand	Impurity content [weight %]						
	Ni	Mo	Cr	Fe	Ti	Al	Other elements
Monicr	Base stock	15,8	6,85	2,27	0,026	0,02	W 0,16; Co 0,03, C 0,014
Hastelloy N	72,2	16,1	6,31	0,03	4,03	0,01	W 0,06; Co 0,15, C 0,03
A	Base stock	11...12	6,5...7,5	≤ 1,5	≤ 0,5	≤ 0,8	Mn < 0,5; Si < 0,15
B	Base stock	11...12	6,2...7,2	≤ 1,5	≤ 0,5	≤ 0,8	Mn < 0,5; Si < 0,15; Nb – 0,5; Y – 0,05
HN80MTY	Base stock	13,2	6,81	0,15	0,93	1,12	Mn 0,13; W 0,072; C 0,025; Co 0,003; Si 0,04

Tab. 2: Chemical composition of alloys for fluoride salts [3,4]

Niobium. Niobium NB-1 serves as a starting material for melting. Two consecutive EBMs contained the following impurities: Al - 0.004, Fe - 0.0001, Cr < 0.001, Ni < 0.0004, Si - 0.005, Cu - 0.0006, Ca < 0.003 weight percent.

Titanium. TG-90 titanium sponge was used as the starting material for melting. The EBM method provided a titanium ingot of 99.99 weight %.

Iron. Armco iron bars were used as starting material for melting. Iron was transformed into drops during EBM. A Brinell bar hardness test was performed prior to melting, which was 830 MPa, and refining reduced it to 624 MPa. The level of iron purity was essentially determined by the nickel and cobalt content.

Chromium. Iron, silicon, aluminium, nickel and also interstitial impurities such as nitrogen, oxygen and carbon are the major foreign substances in chromium. High vacuum annealing of chromium samples at 1200 °C for 5 hours reduced the content of interstitial impurities by approximately 10 times. [5]

The above description is a guide to how impurities can be reduced in metallic materials.

The cobalt content can be reduced to 0.2 % by selecting suitable ores for nickel and chromium production. The purest ore is from Cuba.

MATERIALS COMPATIBILITY

Fluoride salts are compatible with graphite and graphite fuels [7]. In the MSRE the reactor core contained bare graphite as the neutron moderator, which was in direct contact with the fuel salt. The post-experiment study showed absolutely no erosion or corrosion of the graphite [8]. This reactor experiment showed that the molten fluoride salt does not react with the graphite under operating reactor conditions or decompose in radiation fields. In addition to the MSRE exposure at 650 °C and capsule irradiation exposure up to 900 °C, a special experiment was conducted to test very high-temperature compatibility at 1 400 °C [9]. Graphite did not react with the salt even at this extremely high temperature.

STRUCTURAL MATERIALS

When considering the performance of materials in an AHTR (Advanced High-Temperature Reactor), the materials were classified into four broad categories:

- 1) Graphite and C/C composites;
- 2) Reactor vessel materials;
- 3) High-temperature metallic components;
- 4) High-temperature, melt – infiltrated – composite components.

GRAPHITE AND CARBON-CARBON COMPOSITES

The graphite core, reflector, vessel insulation and C/C composite core supports and control rods will operate in a molten salt environment over a range of temperatures from 500 °C up to 1 100 °C or higher. Extensive prior work has demonstrated that graphite is compatible with molten fluoride. Fine-grained isotropic, moulded, or isostatically pressed high-strength graphite suitable for core support structures is available.

REACTOR VESSEL MATERIALS

The reactor vessel must be capable of operating at 500 °C and may need to withstand temperatures up to 800 °C for 100 hours under emergency conditions. The vessel must demonstrate adequate strength and creep resistance, good thermal-aging properties, low irradiation degradation, workability, good corrosion resistance, and ability to develop and maintain a high-emissive surface in air. Past experience has demonstrated that nickel-based alloys demonstrated good resistance to molten salts. Therefore, it is proposed that stable, high-strength, high-temperature materials, such as 9Cr-1MoV, be coated with a high nickel coat for the reactor vessel application. Should the vessel be required to withstand excessive off-normal temperatures, base materials such as 304L, 316L, 347, Alloy 800H, or HT may be appropriate. In addition, monolithic materials with adequate corrosion resistance to molten fluoride salts and high-temperature strength may include Alloy 800H or HT, Hastelloy N, and Haynes 242. [10]

HIGH-TEMPERATURE METALS

High-temperature metallic or composite materials are needed for use at 1 000 °C in the presence of molten fluoride salts on one side and an insulation system in contact with air on the other side. Piping and heat exchangers are examples of applications in the latter conditions. Pumps and other components submerged below the primary salt pool will need to survive higher temperatures for short times or be replaceable at reasonable expense. The metallic materials used in these environments must demonstrate adequate strength, good thermal-aging properties, low-irradiation degradation, workability, and good corrosion resistance. Again, based on advanced level material and the need for high nickel for fluoride corrosion resistance, stable high-strength, high-temperature metallic materials such as Inconel 617, Haynes 230, Alloy 800H, Hastelloy Xor XR, VDM 602CA and HP modified with high nickel coatings need to be evaluated. Should higher-temperature alloys be required, Haynes 214, cast Ni-based super alloys, and ODS MA 754 are possible candidates.[11]

HIGH-TEMPERATURE MELT-INFILTRATED COMPOSITES

LSI C/C (Liquid Silicon Infiltrated) composites are a potentially attractive construction material for high-temperature heat exchangers, piping, pumps, and vessels for the AHTR because of their ability to maintain nearly full mechanical strength at high temperatures, the simplicity of their fabrication, their low residual porosity, and their moderate cost [13,14]. Intermediate heat exchangers fabricated from these materials could potentially be located inside the AHTR-VT vessel, eliminating the need for external primary piping and heat exchangers.

Chopped carbon fibre can provide a particularly attractive material that can be readily formed by pressing with dies and machined using standard milling tools and then assembled into complex parts. In the United States, centrifugal pump impellers and casings are now routinely machined from carbon-fibre reinforced phenol resin preforms, a machining process that could be readily extended to the machining of C/C perform materials prior to LSI processing for use at high temperatures. [14]

LSI C/C-SiC composite heat exchangers, and other components, capable of operating with high-pressure helium, molten fluoride salts, and sulphuric acid, could have great value both for thermo-chemical production of nuclear hydrogen with the sulphur-iodine or hybrid process. [6]

C/SiSiC COMPOSITES

C/SiSiC composites were made at the Institute of Structures and Design, Germany Aerospace Centre (DHL), utilizing an experimental process that has been described in separate papers [8,9]. The C/SiSiC composites were formed by a Si melt infiltration process into a base carbon fibre material to convert the matrix carbon into SiC and to fill residual porosity. The Si melt infiltration was performed in a furnace at 16 500 °C at a pressure of 1 mbar with a holding time of 45 min. The C/SiSiC composite coupons were fabricated and cut from flat plates. Wet chemical analysis found the elemental composition of the ceramic to be: SiC = 69 mol % (59 % vol.), Si = 19 mol % (22 % vol.) and C = 12 mol % (19 % vol.). The carbon fraction remained mostly in the form of carbon fibres that were retained in the microstructure. Chemical vapour deposits (CVD), pyrolytic C (PyC) and SiC coatings were applied by Hyper-Therm High-Temperature Composites, Inc., on several coupons. The C/SiSiC composites were first coated with SiC, followed by an over layer of PyC. [12] The following figures show the strength of these materials at high temperatures.

	Hours to Rupture		
	760 °C 103 MPa	871 °C 31 MPa	982 °C 14 MPa
Inconel-617	20 000	30 000	7 000
Haynes-230	8 200	6 500	5 000
Hastelloy-X	900	5 000	2 100
Incoloy-800H	130	1 200	920
304 Stainless Steel	10	100	72

Tab. 3: Stress rupture data of several relevant alloys. [20, 21, 22]

MATERIAL/SALT SYSTEM DESCRIPTION

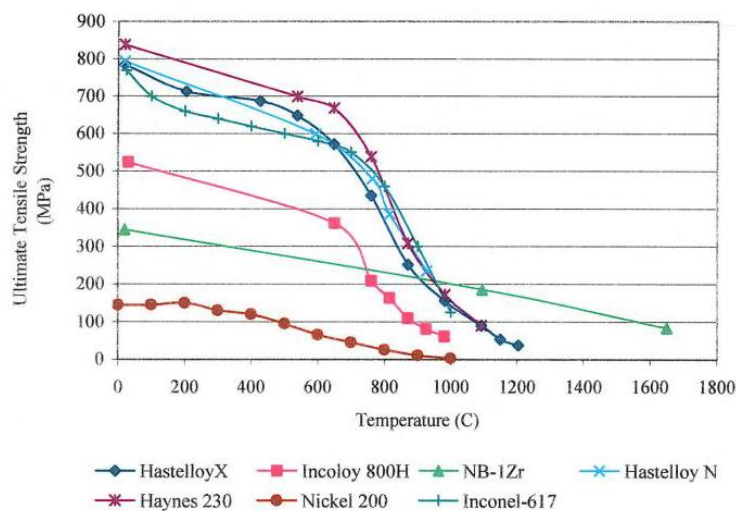


Fig. 10: Ultimate tensile strength as a function of temperature of the alloys selected for corrosion tests [15, 16, 17, 18, 19, 20, 21, 22]

Several molten fluoride salts have been investigated for nuclear applications:

(a) LiF-BeF₂ (66-33 mol%) (FLiBe) was extensively studied in the 1960s at the Oak Ridge National Laboratory (USA) for the development of molten salt reactor systems with a thermal neutron spectrum (MSRE for the experimental molten salt reactor) and MSBR for a molten salt reactor with a thermal neutron spectrum). In these systems, the core material was graphite and the Ni-based alloy (Hastelloy-N) was used for the external structures. The problems associated with the use of graphite are its low irradiation life (5 years), the lack of recycling processes, and waste management.

(b) LiF-BeF₂ salt is also suitable as a coolant in nuclear fusion power plants. Indeed, tritium can be produced by neutron reaction with lithium, and the use of LiF salt is safer. In addition, the FLiBe salt has a low tritium solubility and therefore has a low tritium content. Several corrosion studies of structural materials have been conducted for this application.

(c) The Advanced High-Temperature Reactor (AHTR) concept is currently under development. The term includes solid fuel and a solid core associated with the molten salt as a coolant. The best developed project is with spherical fuel particles. The original AHTR project was cooled by He. The new design is cooled with fluoride salts. Due to their high thermal stability (reactor operating temperature above 900 °C), selected salts for primary and secondary loop LiF-BeF₂ (66-34 mol%) and LiF-NaF-KF (46.5-11.5- 42 mol %) (FLiNaK). From the neutron point of view, it is recommended to use LiF-BeF₂ primary coolant. There is a more detailed description in work [23].

(d) Since 2000, the CNRS-France study has explored the innovative design of the MSR, the Molten Salt Fast Reactor (MSFR). The liquid fuel considered for it is LiF-ThF₄-UF₄ (77-20-3 mol%). Compared to the American concepts MSRE and MSBR, MSFR works with a fast neutron spectrum. Under these conditions, the reactor

operating temperature is between 650 and 850 ° C, which is higher than in the older US MSR systems. In this system, the graphite is replaced by a metal alloy.

In all of these nuclear applications, high temperature fluoride salt chemical resistance is a key element in the selection of the structural material. For applications (b) and (d), neutron irradiation resistance must also be considered. For the AHTR reactor system, several alloys (metal samples) immersed in FLiNaK static salt at 850 ° C for 500 hours were studied: Haynes-230, Inconel-617, Hastelloy-N, Hastelloy-X, Nb-1Zr, Incoloy-800H , Ni-201. For four aluminium alloys based on Ni, Hastelloy-N, Hastelloy-X, Inconel-617 and Haynes-230, corrosion loss was found to increase with Cr content in the alloy. For Haynes-230, significant loss of chromium caused the dissolution/precipitation phenomenon, especially the formation of W-rich precipitates at the grain boundaries. The Nb-1Zr refractory alloy exhibited strong corrosion and brittleness. Due to the remarkably high corrosion resistance of Ni in molten fluoride salts, Ni-201, predominantly Cr-free, was virtually immune to attack. There is no proven metal material for temperatures above 800 ° C. The French have the idea of replacing the additive molybdenum tungsten in their material. It is important to monitor the purity of the salts. For salts, the contents of Fe, Ni, Cr, etc. are often high. These impurities are the source of the corrosion of the structural materials. In addition to the purity of the salts, the solubility of the fuel in the salts must also be monitored. [24] For example: for plutonium see Fig. 2.

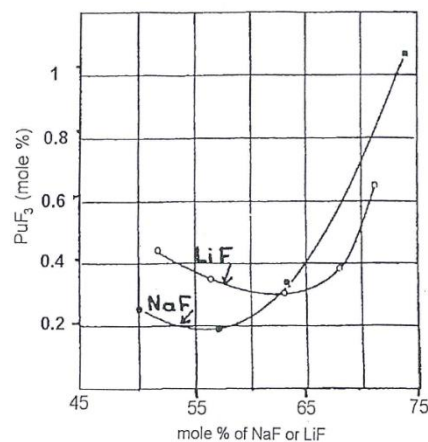


Fig. 2: Effect of solvent system composition and temperature on the solubility of PuF₃. PuF₃ solubility in LiF – BeF₂ and NaF – BeF₂ at 565 °C [25]

COMPOSITION OF IMPURITIES

Impurities can strongly influence the properties of the mixture by chemical reactions. Changes cause impurities such as Cr, Fe, Ni, which come into the salt mixture in the production of individual fluorides. Accurate measurements have been made in the Ampul programme in Ekaterinburg to reduce the content of corrosion products by means of zonal remelting, i.e. in a manner similar to that of semiconductor cleaning. *Another problem is the presence of H₂O in the salt mixture. Many salts are highly hygroscopic, and therefore the handling of the salts should be carried out after drying in an inert atmosphere of Ar or He. Also gases such as hydrogen and hydrogen fluoride need to be inspected for impurities and cleaned.*

In addition to corrosion products involved in the further dissolution of atoms from the material, some fission products also have a major impact on the fuel mixture. This is particularly the case for tellurium, which forms a large number of different compounds with Cr and elutes it intensively from the material matrix. [26]

SOLUBILITY

Double solubility should be considered:

- a) *Solubility of PuF₃ in the salt mixture. The results reported are limited to mixtures of LiF and BeF₂ and LiF + BeF₃ + ThF₄ salts for an expected temperature range of 600 °C - 700 °C. This solubility can be seen in the next table.*

Brand of mixture	LiF+NaF+BeF ₂ [mo.l %]	T _{melt} [°C]	Solubility XF ₃ [mol. %]			
			T = 550 °C	T = 600 °C	T = 650 °C	T = 700 °C
D	15 + 58 + 27	479 – 480	1.2	2.0	2.9	4.1
E	17 + 58 + 25	494 – 496	1.9	3.0	4.5	6.1
F	18 + 58 + 24	515	2.3	3.95	6.0	9.0

Pu was considered as the nuclear fuel in MSR reactors. Therefore, the solubility of *Pu* in these salts is important.

b) Complexes are formed in the salt phase diagram which are insoluble, e.g. LiF + BeF₂ (80-20 mol%). Pure LiF is an insoluble component up to 710 °C. For a mixture of 60-40 mol% of the Li₂BeF₄ phase it is insoluble below 440 °C. It is highly desirable to manage criticality and security so that the primary phase (the first solid precipitating upon cooling) does not contain a fuel component. This was accomplished with the MSRE reactor for UF₄. For PuF₃ fuel it is less certain. The lowest melting point (360 °C) is obtained for a given mixture at the so-called eutectic point, where LiF - BeF₂ (48 - 52 mol%) is present (see Fig. 3). This would be advantageous, but in melting the viscosity of the system is considerably higher for the formation of Li₂BeF₄. Therefore, the optimal ratio of LiF: BeF₂ = 2: 1, i.e. BeF₂ (34.0 mol%) was chosen - see point 1 in Fig. 3 due to the single phase of heating or cooling the mixture. However, this mixture results in a low solubility of PuF₃. [26]

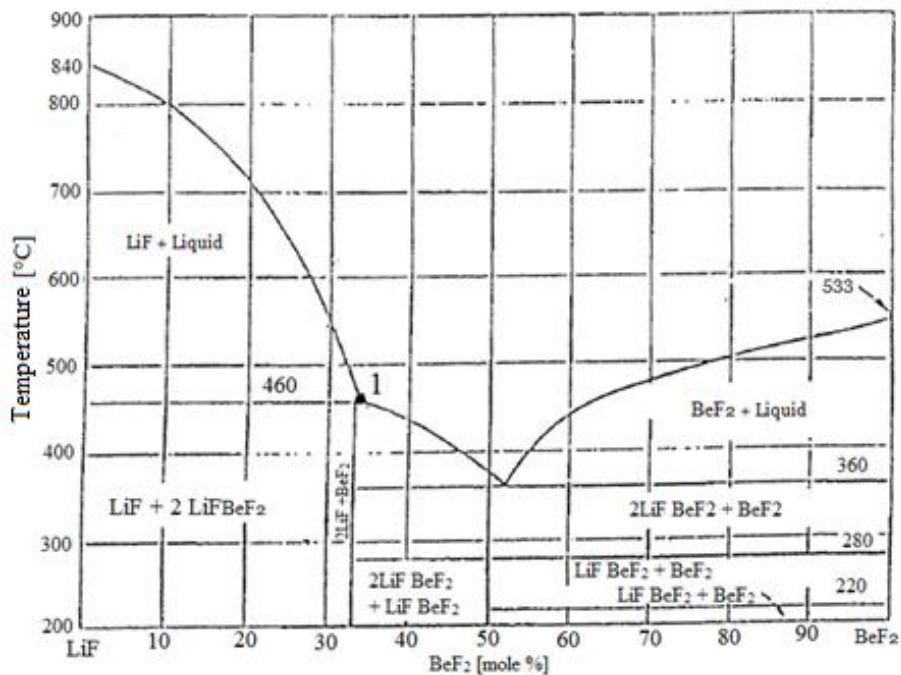
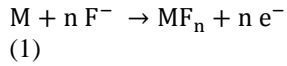


Fig. 3: LiF –BeF₂ phase diagram showing melting points of the salt mixture and phase [26]

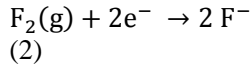
In [27], the experimental material boron nitride was used for tubes as a replacement for glass in the US in the 1950s and 1960s. This material is usable.

Another thing to watch for is the corrosion between the structural material and the fluoride salts.

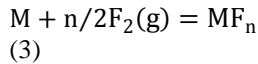
Corrosion in molten salt is essentially an electrochemical process, originating from an electronic exchange. This reduction-oxidation reaction involves one (or some) metallic element(s) of the alloy and dissolved oxidizing species. The oxidation electrode reaction for a metal M corresponds to:



where M is a metallic element (i.e.: Fe, Ni, Cr, etc.), and MF_n is the corresponding metallic ion whatever its exact speciation in the fluoride melt (with $0 < n < 6$). The reduction electrode reaction considering the reference electrochemical species $F^-/F_2(g)$ is:



The generalized oxidation reaction is:



Olander (2002) [29] discussed the fundamentals of this redox state in fluoride salts. He stated that the redox condition is properly termed the ‘fluorine potential’ based on

$$\Delta \bar{G}_{F_2} \equiv RT \ln p_{F_2} \quad (4)$$

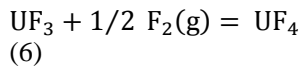
where $\Delta \bar{G}_{F_2}$ is the partial molar Gibbs free energy of the fluorine gas, p_{F_2} is the fluorine potential.

The fluorine potential theoretically controls the equilibrium concentrations of structural metals dissolved in the fluoride-based salts (see Eqs. (3) and (4)).

Molten fuel salts (i.e., molten fluoride salts containing UF_4/UF_3) have been fairly well studied (Toth *et al.* 1994 [30]). In uranium-containing fluoride mixtures, the fluoride potential p_{F_2} can be expressed as the concentration ratio of quadrivalent to trivalent uranium ions:

$$\ln p_{F_2} = 2 * \ln [UF_4]/[UF_3] + 2/RT * \Delta G^\circ \quad (5)$$

where ΔG° is the standard free energy change of the reaction:



The ratio UF_4/UF_3 generally considered in MSR ranges from about 10 to 150.

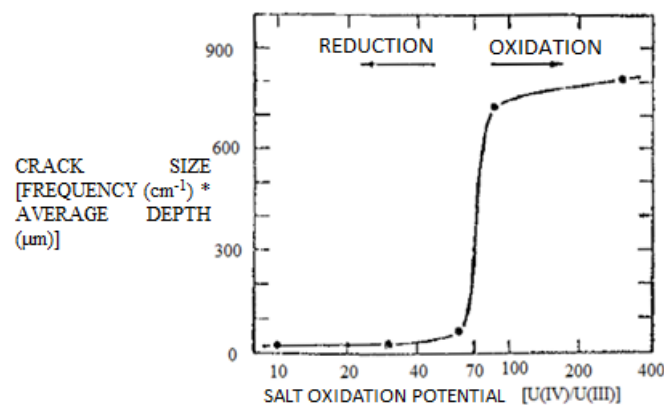
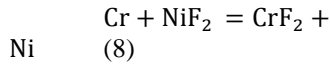
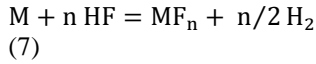


Fig. 4: Salt oxidation potential [26]

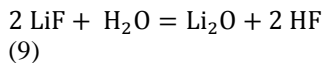
The corrosion rate depending on potential has the same curve.

CORROSION BY OXIDANT IMPURITIES

Addition of oxidants can have a strong effect on the fluorine potential because F_2 , HF, metallic cations are strong oxidants and may oxidize a metal M as follows:



Water is one of the main pollutants and dissolves with production of HF



Toth *et al.* (1994) [5] assessed that fission of uranium or plutonium based fuel yields different changes in the molten salt redox. Besides, transmutation of 7Li and 6Li produces tritium fluoride, analogous to hydrogen fluoride, and transmutation of beryllium produces free fluorine. [28] Similarly, the corrosion of the material increases when the oxidation zone is moved.

	Temp (K)	Medium	Na	Cr	Fe	Ni	W	Mo	Ag	Au	F ⁻
Jenkins <i>et al.</i> (1970)	773	LiF-NaF-KF		-0.70	-0.41	0					
Jenkins <i>et al.</i> (1970)	773	LiF-BeF ₂ -ZrF ₄			-0.39	0					
DeVan (1969)	873	LiF-BeF ₂		-0.83	0.46	0					
Grjothheim (1957)	1123	NaF-KF		-0.70	-0.12	0					
Cabet, experimental	1173	LiF-NaF	-1.86	-0.68	-0.42	0	+0.44	+0.19	+0.52	+1.57	
Cabet, computed	1173	Hypothetical (pure substances)	-2.23	-0.80	-0.41	0	+0.05	+0.23	+0.99	+2.02	+2.46
Williams <i>et al.</i> (2006)	1273	NaF-AlF ₃			-0.55	0	+0.20				

Tab. 4.: Oxidation potentials of metals referenced to Ni/NiF₂ [V] [28]

Céline Cabet's calculations and measurements are particularly interesting in this table. The calculation is made for hypothetically pure material without impurities and admixtures.

Real measurements made on a real LiF-NaF mixture with traces of impurities and admixtures contain different values from the theoretical calculation.

The calculation values compared to the measured values would be useful for determining the contamination of the salts used.

CONCLUSIONS AND RECOMMENDATIONS

According to GIF, the only licensed material is used up to 800 °C, but it is not corrosion resistant to fluoride salts. It is possible to remedy this by applying surface layers to the material which are corrosion resistant to fluoride salts. It follows from the above that there are materials that can withstand temperatures above 700 °C but which do not fully meet corrosion resistance. This is the reason why these materials need to be coated with glass graphite.

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