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Reliability of Molten Salt Static Corrosion Tests

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The presented paper shows the results of the reliability analysis process of static corrosion tests, from the preparation of the experiment and the experimental execution itself to the cleaning and analysis of the tested samples. Each part of the process is divided into individual steps, which are assessed in terms of safety, reliability, and repeatability of the obtained results. Based on this assessment, critical steps are identified and, depending on technical and financial possibilities, technical or organizational changes are recommended to maximize the safety, reliability, and repeatability of the results.

Keywords: Molten salts, coolant, corrosion, vulnerability of structural materials, risk, safety, reliability, static corrosion test, resistance of structural materials.

1. Introduction

Currently, molten salts are used in different industries for different types of applications, including, for example, the chemical industry (Misra 1988), metal extraction (Fray 2001), thermal energy storage technologies and solar tower technologies (Fereres et al. 2018), and they are also supposed in future Generation IV nuclear reactors. Some of those applications are well researched and better understood than others. For the nuclear application, which is of particular interest to this research group, there are still some questions left unanswered. Such as general behavior of the molten salts – especially around the melting point (Cantor et al. 1968; Janz 1967; Williams 2006), the interaction between molten salt and construction materials (Ignatiev and Surenkov 2013; Kondo et al. 2009; Muránsky et al. 2019), potential toxicity (Stefaniak 2011; Strupp 2011a; Strupp 2011b), neutronic properties (Cammi et al. 2011; Zhang et al. 2009), or the ways of how to measure its properties online (Gill, Kolb and Briggs 2014; Sabharwall et al. 2010).

The research group from CVŘ and ÚJV Řež, Czech Republic is trying to find answers to some of those questions (Uhlíř and Mareček 2005; Uhlíř 2008), where the main focus is put on measurement of neutronic behavior of FLiBe salt (Košťál et al. 2020; Losa et al. 2017), fuel reprocessing (Uhlíř and Mareček 2009; Uhlíř, Mareček and Škarohlíd 2012; Souček et al. 2005), and the impacts of corrosion of construction materials caused by molten salts (Cihlar 2020; Vlach 2019).

This paper focuses on specific molten liquid salts that have applications in nuclear reactors cooling, solar power plants, and energy storage systems. In the process of selecting the best possible liquid salt for the cooling of Generation IV nuclear reactors, these requirements are required: high boiling point; low vapor pressure; high specific heat; high thermal conductivity; high density at low pressure; low viscosity; low neutron absorption cross-section (for primary circuit media); and low corrosion aggressivity to structural materials (Juliana, Frutuoso e Melo and Rivero Oliva 2013). The high corrosion aggressivity of molten salts must be addressed before commercial

deployment is possible, because it severely reduces the safety of structural components, thus nuclear safety and the integral safety of the nuclear installation.

Research on the implementation of technological processes (Procházková et al. 2019) has shown that in all phases of technological processes, there are errors that cause poor quality results to the non-implementation of the process. These are errors caused by: selecting inappropriate technology; an inappropriate combination of material and the chosen procedure; poor preparation of the material for the measurement process; technical implementation; human error; poor organization of the measurement process; incorrect external conditions in which the measurement is carried out; and bad faith. For this reason, we focus on assessing the reliability of the experimental results.

The paper analyzes the whole process of static corrosion tests, from the preparation of the experiment and the experimental conduction itself to the cleaning and analysis of the tested samples. Each part of the process is divided into individual steps, which are assessed in terms of safety, reliability, and repeatability of the obtained results. Based on this assessment, critical steps are identified and, depending on technical and financial possibilities, technical or organizational changes are recommended to maximize the safety, reliability, and repeatability of the results obtained. This analysis is performed using the risk engineering technique "safety audit," consisting of the process model of test experiments and checklist for risk judgment (Procházková 2018).

2. Summary of Knowledge

From a practical aim, we focus on molten salt behavior and metal corrosion caused by the aggressivity of molten salts.

2.1 Molten Salts

Salts are chemical compounds of acids and alkalis. In nature, they occur as crystals with the following properties: powerful ionic bonds among the particles; high boiling and melting points; conduct an electric current in solution or a melt, but not in the solid-state; high values of dynamic viscosity, high thermal conductivity coefficient, and high density; and can be separated from the solution by crystallization (Janz 1967).

The uses of molten salts are vast. In thermal solar power plants, nitrate salts (e.g., NaNO3-KNO3) are used; for the high-temperature heat storage the carbonate salts such as Li2CO3-Na2CO3 are preferred (Tian and Zhao 2013); and fluoride salts FLiBe (LiF-BeF2), FLiNaK (LiF-NaF-KF), and NaF-NaBF4 are the preferred choice in the nuclear industry. The main advantages of molten salt reactors are these: high boiling temperature; high coolant output temperature; low working pressure; no fuel fabrication; potential continuous refueling; potential continuous fissile products removing; and a possibility to work in breading mode, according to (Serp et al. 2014).

However, the use of liquid salts, according to Serp et al. (2014), may also involve some unacceptable consequences caused by, e.g., high corrosion aggressivity towards structural materials; high melting temperature; toxicity of salt compounds; inappropriate neutron properties; and lack of operational experience. All the advantages and disadvantages are dependent on a particular design, and not all of them are included in every reactor design.

The underlying challenge of molten salts, which must be resolved in such a way as to allow the commercial deployment of molten salts in nuclear energy, is high corrosion aggressivity. A deeper understanding of the corrosion behavior, corrosion mechanics, and corrosion effects of molten salts on various construction materials is essential for subsequent application. Safety is a pillar of nuclear energy, which must be based on experiments and data derived from them.

Currently, the proposed molten salt reactors use various construction materials from specially developed molybdenum-nickel alloys through stainless steels and ceramics to graphite (IAEA 2020). Understanding the corrosion phenomena that occur when molten salts interact with these structural materials, either individually or in complex systems containing multiple structural materials, is an integral part of the development of new salt reactors.

The corrosion behavior of molten salts has been studied since the 1950s (Richardson, Vreeland and Manly 1953), and currently, it is done in several institutions worldwide using various experimental methods. One of the commonly used methods is a static corrosion experiment (Kondo et al. 2009; Muránsky et al. 2019). On the other hand, complex corrosion behavior can be studied using molten salt loops (Ignatiev and Surenkov 2013).

Another essential field for the potential industrial application of molten salts in nuclear energy is measuring molten salts' behavior. Precise and reliable sensors and methods developed for measuring many physical quantities of molten salts during the operation are critical prerequisites for their deployment. The focus is on pressure, pressure drop, temperature, and flow rate measurements (Gill, Kolb and Briggs 2014; Sabharwall et al. 2010).

The next field that needs to be thoroughly investigated is the verification of theoretical hypotheses on molten salts, their physical and chemical properties, for example, in such modes as melting and solidification. Although knowledge of some properties of some salts is already at a high level (Williams 2006), there are always many unknowns.

One of the issues discussed is the toxicity of some salts associated with the safety of operation. For example, in the case of FLiBe salt, it is the content of beryllium, which can have various unacceptable consequences on human health (Stefaniak 2011; Strupp 2011a; Strupp 2011b).

2.2. Corrosion

Corrosion of structural materials is a significant problem in many environments. It is the degradation mechanisms of materials and their joints. It is the spontaneous, gradual disruption of metals or non-metallic organic and inorganic materials due to chemical or electrochemical reactions with the surrounding environment. It can take place in gases, liquids but also soils, or various chemical substances in contact with the material. Often, the resistance to corrosion is achieved by a passivating oxide layer on the material's surface (Schweitzer 2010).

For molten salts, this method cannot be used because this layer is unstable (Young 2008). Therefore, we search for another way and for this reason, it is essential to investigate the effect of molten salts on structural materials, that is, the vulnerability of materials in molten salt environments; the impacts of corrosion on construction materials caused by molten salts; and the time changes of various structural materials in this highly aggressive environment. Commonly, this research is being conducted in several stages. The first stage is often static corrosion tests, followed by dynamic tests to simulate better the actual environment (Koger 1972; Raiman and Lee 2018). The preparation of the experimental molten salt loop for the dynamic tests is described in (Cihlář et al. 2021).

Impurities in molten salts, such as water and oxides, are usually the main driving force causing the corrosion of materials in molten salts. Other driving forces include temperature gradient and galvanic corrosion (DeVan 1969). To achieve high corrosion resistance in molten salts, it is necessary to carefully choose structural materials, use high-purity construction materials, and strictly control the salt composition.

As stated above, molten salt impurities provoke unacceptable behavior called thermodynamically-driven corrosion. It severely impacts the construction materials due to the difference of Gibbs free energy of the alloying materials and salt constituents. Commonly the thermodynamically-driven corrosion has the highest significance at the beginning of the operation until an equilibrium between molten salt and structural materials is achieved.

The most damaging impurities are moisture, oxides, and metallic impurities. Water reacts with molten salts constituents forming a hydrofluoric acid, which consequently attacks alloying elements of construction materials. The metallic impurities deplete the commonly used alloying element chromium from the construction material (Koger 1972). A common strategy to reduce impurity-driven corrosion is an extensive purification process. Usually, the purification process consists of the following steps: vacuum drying, hydrofluorination, active metal treatment and more (Anderson et al. 2015).

In the later stages of any molten salt coolant system, thermal gradient-driven corrosion becomes prominent. Because in higher temperatures, solubility is more significant than in lower temperatures, the corrosion products dissolve in hotter sections of the system, they are transported with the salt flow, and consequently, deposited in the cooler sections of the system (Britsch et al. 2019; Koger 1972).

The galvanic corrosion, sometimes also referred to as activity-driven corrosion, is caused by the presence of two or more different construction materials with the different activity of dissolved species. In this situation, dissolved species migrate from the material with higher activity to the material with lower activity. The most prone combinations of structural materials to this corrosion are those, at which the low-activity material has a chemical affinity to dissolved species and can form thermodynamically favorable compounds. However, in most molten salt nuclear reactor designs, it is necessary to combine multiple construction materials. Therefore, the effect of galvanic corrosion is being studied (Lantelme and Groult 2013; Sellers 2012).

The radiolytic stability of molten salts and the expected effect of radiolysis on corrosion should not be a considerable issue (Ignatiev and Surenkov 2013; Sohal et al. 2013). The only exception is the transmutation of lithium and fluorine, which can possibly produce hydrogen and oxygen, which would significantly increase the oxidation potential of the salt (Lantelme and Groult 2013).

3. Corrosion Tests and Measurement Procedure

Corrosion tests of structural materials in liquid salts are significantly challenging on finance, time, and organization. Therefore, it is not feasible to perform a large number of experiments with hundreds or thousands of samples to be analyzed. These limits place high demands on the repeatability and reliability of experimental results. Each experiment starts with sample preparation, continues with the test itself, and finishes with sample analysis. The description of the whole process is in the following paragraphs.

Each specimen is obtained by cutting bigger pieces of the chosen construction material with dimensions of approximately 30 x 15 x 1 mm. Afterward, the specimen is ground using the silicon carbide foil with a roughness of 500 particles per square millimeter. Before the beginning of an experiment, each specimen dimension is measured and weighted.

In order to perform a test, firstly, the NaF-$NaBF_4$ salt is prepared by combining the exact amount of crystalline NaF (3 wt%) and crystalline $NaBF_4$ (97 wt%) to obtain the eutectic mixture. Approximately 250 g of the eutectic mixture is contained in a graphite crucible and put into CLARE 4.0 electric furnace at a temperature of 500 °C. After four hours, the NaF-$NaBF_4$ eutectic mixture is melted and ready to be poured into ampules with specimens.

Approximately 30 g of molten salt is poured into each graphite ampule containing one specimen to cover the specimen completely. Ampules are let to cool down to room temperature and inspected for salt leakage, the level of salt, and any irregularities. Inspected ampules are closed and put into the furnace.

The whole test is performed in a CLARE 4.0 electric furnace which is part of an experimental glove box with a nitrogen atmosphere (Fig.1.). Individual specimens are contained separately, each in its own small graphite ampule. Four ampules with specimens are put into a larger safety graphite crucible. During each experiment, two crucibles with eight individual specimens in total are in the electric furnace. The electric furnace has an automatic control system that keeps temperature 2 °C around the target temperature. The nitrogen overpressure atmosphere is set manually to slight overpressure.

Fig.1. The experimental glove box with CLARE 4.0 electric furnace for molten salt static corrosion experiments.

When the experiment is finished, the electric furnace is turned down, and all the ampules with salt and specimens are let to cool down to room temperature at the place. This cooling process takes approximately 48 hours. After cooling down, the salt is carefully separated from the specimen by hand or using brushes and scalps. The following cleaning steps include ultrasound cleaning in demineralized water for approximately twenty-four hours, cleaning with alcohol, and drying.

As well as before the experiment, all the specimens are weighted. Visual inspection and comparison of specimens are performed, and pictures are taken with a camera. Specimens are inspected under the Leica DM 2700 M microscope with a 5 Mpix camera for smaller details.

For specimens' general chemical surface composition, Energy Dispersive X-ray Fluorescence analysis (XRF) is done. The specimen is measured twice – once from each side. SEM (Scanning Electron Microscope) analysis requires specific preparation of samples. The first step of preparing specimens for SEM analysis is cutting each specimen into two pieces. The next step after cutting is casting the sample into a bakelite matrix. The copper tape is used in order to hold a specimen to enable analysis in a cross-section direction. For the casting itself, the phenolic hot mounting resin with carbon filler was used with a preset method (Cihlar 2020). The next steps in preparation are grinding, polishing, and etching (final polishing) of the sample. Grinding and polishing steps differ for different materials, and together with an even more detailed description of the process can be found in the Master's thesis (Cihlar 2020). After the polishing process is finalized, the specimens are washed with water, cleaned in an ethanol bath with ultrasonic waves, dried, and stored in a box with a low vacuum. The specimens are analyzed using a SEM.

Using the SEM, topographical pictures, maps of chemical composition, and line scans are taken. Topographical pictures and chemical maps give an idea about the overall surface and structural changes, whereas line scan analysis provides information about the changes at the surface layer and the depth of the corrosion layer. Afterward, all the analysis is used to calculate the depth of the affected surface layer. The results are compared to non-tested samples.

4. Methods for Data on Corrosion Tests Reliability Acquisition

The corrosion tests' reliability is performed using the risk engineering technique "safety audit" consisting of the process model of test experiments and checklist for risk judgment (Procházková 2018). We use a process model type, which describes the life cycle of the experiment at the type of level (a graphical representation of workflows) (Krogstie, Sindre and Jørgensen 2017). In our case, the goals of a process model are prescriptive, i.e., it defines the required processes and how they should/could/might be performed; and it establishes rules, guidelines, and behavior patterns which, if followed, would lead to the desired process performance. They can range from strict enforcement to flexible guidance. For check list criteria judgement is used scale 0 to 5 with principle "the lower the better" (Keene and Raiffa 1993).

5. Tool for Judgment of Corrosion Tests Uncertainties

The aim of each series of experiments is that the result is correct and has a specific informative value to the object of investigation. In the real world, the course of measurement processes is influenced by internal and external factors and, of course, by the human factor (Procházková and Procházka 2015), which causes the dispersion of measurement results. Of course, the fact in question affects the measurement result and can even lead to false conclusions. Therefore, reliability of measurements was assessed by safety audit.

In our experiments, we used the safety audit method to assess the reliability of measurements. Static corrosion tests of steel materials caused by molten salts are carried out according to a process model that has three basic parts:

- metal sample preparation for the test - the process model contains a selection of sample material; determination of sample size; metal sample abrading; experimental device preparation; preparation of container for sample; preparation of molten salt; and preparation of test unit,

- preparation of the experiment - the process model contains: selection of experiment conditions; checking the actual conditions; experiment start; monitoring the conditions at experiment life cycle; experiment termination; extraction of the container with a sample from the experimental device; and cleaning the metal sample,
- course and evaluation of the experiment – the process model contains the transfer of sample to measuring equipment; determining the sample weight; analyzing the weight change; photography of sample; analyzing the sample surface and its visual judgment; investigation of the sample surface by microscope; analyzing the changes on sample surface; the SEM preparation; execution of SEM; analyzing and evaluating the SEM results; and processing the professional record on test and its results.

The example of questions in checklist used is shown in Table 1. In total, 87 questions are used.

Table 1. Checklist for measuring the reliability of static molten salt corrosion experiments. Assessment is in form Yes or No; in Note, we give asterisk if an assessment is lower credibility.

No	Criterion	Assessment	Note
1	Is the choice of material determined by the results of previous experiments?		*
5	Is the same sample size always used for the experiment?		
20	Is the experimental equipment cleaned?		
25	Did the molten salt supplied for the experiment meet the purity requirements?		
34	Was enough molten salt poured into the metal sample container so that bubbles did not form?		
50	Was the extracted metal sample cleaned without the use of water?		
57	Did the analysis of the mass changes of the extracted metal sample comply with the regulations?		
58	Did the extracted metal sample be photographed under defined lighting conditions?		
64	Was the metal sample analyzed with a microscope according to the established regulations?		
65	Were apparatus for SEM calibrated before use?		
68	During the SEM preparation, was the grinding of the sealed metal sample into bakelite carried out according to the specified regulation?		
87	Has been report on the experiment prepared on an ongoing basis?		

6. Results

Most of the results of static corrosion experiments were very similar to each other (Cihlar 2020). However, some results diverged; examples are shown in the next subchapter. For practical purposes, we try to identify the cause of those differences. Applying the physics knowledge (Siegel 1981), the cause of differences is either material structure, lack of standardization, or uncertainty in experiment procedure. Therefore, the results of the experimental procedure test obtained using checklist follows.

6.1. Examples of unexpected experimental results

During the course of static corrosion tests of construction materials in molten salts through the past years, some unexpected results occurred, although all the procedures were done with systematic work and obeying all the standards for the experimental laboratory. Some of those unexpected results are summarized in the following paragraphs.

It is expected that the corrosion rate should be higher for higher temperatures and longer exposures. However, one group of stainless steel AISI 316L samples (Table 2) tested in NaF-$NaBF_4$ molten salt has the worst results for the least demanding scenario. Similar discrepancies occurred with MONICR T18/111 sheet samples (Table 3) tested in NaF-$NaBF_4$ molten salt. The samples tested with increased temperature have a lower corrosion rate. Moreover, two samples tested under the same conditions gave results that differed more than two times.

Table 2. Mass losses caused by corrosion for AISI 316L samples tested in molten NaF-$NaBF_4$ salt.

AISI 316L	30 days / 550 °C	30 days / 700 °C	90 days / 550 °C
Corrosion Mass Loss	0.8614 mg/cm^2	0.6597 mg/cm^2	0.6335 mg/cm^2

In some samples, cracks occur (Fig. 2). Even in samples that were not tested in a molten salt environment. Such cracks can have severe effects on the sample performance. Furthermore, it is not feasible to examine each and every sample for these cracks. Other samples evince extraordinary damage that is only apparent after the exposure (Fig. 3).

Table 3. Mass losses caused by corrosion for MONICR T18/111 sheet samples tested in molten NaF-$NaBF_4$ salt.

MONICR T18/111 sheet		**90 days / 550 °C**	**90 days / 700 °C**	
Corrosion Mass Loss		1.5321 mg/cm^2	0.5684 mg/cm^2	1.2298 mg/cm^2

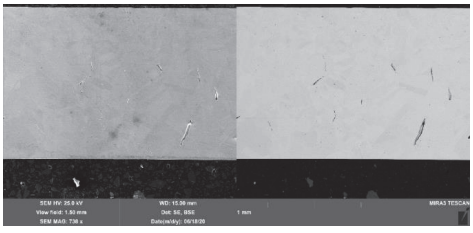

Fig.2. Topographical SEM picture of untested MONICR T18/111 sheet with cracks present.

Fig.3. The picture of AISI 316L sample after the 30 days / 550 °C exposure to molten NaF-$NaBF_4$ salt.

One of the most challenging parts of the corrosion rate evaluation is determining the corrosion layer depth. The corrosion depth layer is determined using a combination of different analysis and their results, including SEM chemical maps (Fig.4) and SEM topographical pictures (Fig. 5). The corrosion layer could be uneven, as seen in Fig.4, or an intercrystalline attack might occur (Fig. 5).

6.2. Results of experimental procedure test

From the safety audit by the help of the checklist in Table 1, it follows that the causes of significant differences can be caused by:

- Insufficient sample check before experiment
- Varying the sample preparation process: sample size; sample cutting; and manual gridding
- Inconsistent amount of molten salt poured into each ampule
- Complicated metal extraction and cleaning: metal extraction; and use of water for cleaning
- No standardized photography and visual inspection of samples, e.g. missing precise regulations and guidelines
- No guidelines for randomizing places for detailed SEM analysis
- No processing the running report on experiment history

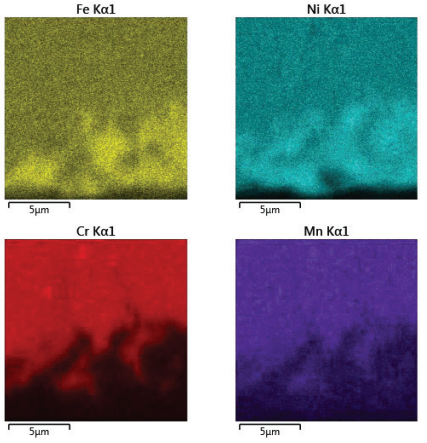

Fig. 4. Chemical maps of Inconel 600 after 30 days / 700 °C exposure to NaF-$NaBF_4$ taken with SEM.

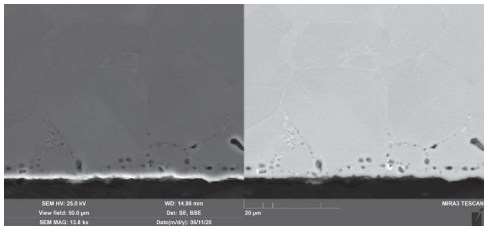

Fig. 5. Topographical SEM picture of stainless steel 316L sample after 30 days / 550 °C exposure to NaF-$NaBF_4$.

From this listing, it follows that in experiment process, it must be specified: consistent sample size; consistent cutting and grinding procedure; precise weighting of molten salt in ampule; careful extraction and cleaning; well defined visual analysis procedure; well defined site choice procedure for SEM analysis; and duty to process the report on experiments course.

From the knowledge of physics, the crystal defects surely play role in metal sample behavior. Crystal defects are interruptions of regular patterns in crystal solids. Defects or imperfections in crystals can be divided into four groups namely line defects, point defects, volume defects, and surface defects. These defects are responsible for large variation of electric and optical

properties and affects hardness of solid materials. They are starting points of microfractures from which during the time fractures are developed (Zhang et al. 2019). Therefore, it is necessary to include their study in searching the suitable material for practical use in reactors with molten salts.

Furthermore, from the chemistry point of view, there need to be a cleaning step including dissolving or molten salt remains on the samples. In this step there is no suitable replacement for water. Therefore, water cannot be completely eliminated from the cleaning process. At least, the water cleaning procedure has to have precisely defined procedure.

7. Conclusion

The risk engineering technique "safety audit" consisting of the process model of test experiments and checklist for risk judgment were used for the reliability analysis of molten salt static corrosion tests. The used process model type describes the life cycle of the experiment with a graphical representation of workflows. Sample questions of the checklist are at the Table 1. The need for this analysis is supported with examples of unexpected experimental result.

Based on the analysis critical points such as insufficient sample check before experiment, varying sample preparation process, complicated metal extraction and cleaning, missing standardization, regulations, and guidelines were identified, and some corrective measures were proposed. The proposed corrective measures include even more precise sample preparation standardization than the current one, definition of specific visual inspection photography analysis procedures, redefined procedure for molten salt weighting and ampule filling, and other regulations or guidelines.

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