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Corrosion in Molten Salts for Solar Thermal Power

by Kodi Summers and Dev Chidambaram

Global energy needs continue to grow steadily with increasing population and standard of living. While fossil fuels have been a major source of our energy, they are no longer favorable due to their greenhouse gas emissions and atmospheric pollution; the development of carbon-free sources of energy is crucial. Solar energy promises unlimited amounts of emissions-free energy but suffers from intermittency issues that require expensive secondary energy storage systems to act as baseload power. A thermal energy storage (TES) system can store heat and provide a continuous source of power. Concentrated solar power (CSP) is carbon-free and can ideally be combined with a TES system to provide continuous power. These combined systems operate by focusing a large area of sunlight onto a receiver that contains a TES material to convert the sunlight into thermal energy. Thermal energy can be stored as either sensible heat, latent heat, or thermochemical heat. A heat transfer fluid (HTF) is utilized, sometimes in conjunction with a heat exchanger system, to drive a Rankine cycle heat engine (a turbine) to generate electricity, as shown in Fig. 1. In commonly installed configurations, the molten salt acts as the primary TES/HTF and is stored in two separate tanks for the cold leg and the hot leg.

The efficiency of a heat engine is limited by Carnot's theorem:

$$\eta_{\max} = 1 - (T_c/T_h)$$

where η_{\max} is the maximum theoretical efficiency, and T_c and T_h are absolute temperatures of the cold and hot reservoirs. Thus, increasing the operating temperature (T_h) will improve maximum efficiency and is a major focus of current studies. Additionally, an increased outlet temperature will reduce the amount of HTF required. An ideal HTF would have a very high boiling point, low vapor pressure, high heat capacity, high thermal conductivity, thermochemical stability during repeated temperature cycling, and not be corrosive to the systems carrying the fluid. Various materials can be used as HTF, including water, oil, and molten salts.

Molten salts systems have been considered as HTFs for over 60 years¹ and studied extensively for CSP systems since the 1980s² as they inherently meet many of the desirable HTF characteristics mentioned. Due to these properties, molten salts can operate at high temperatures while remaining at or near atmospheric pressure, lowering the cost of pressure vessels and other handling components. Additionally, molten salts also have excellent physical properties for heat transfer, storage, and fluid flow in these environments. While initial studies and most existing power plants use molten nitrate salts, the push to higher operating temperatures has led to consideration of chlorides and carbonates based salts. This article will discuss the general understanding of the corrosion behavior in these media.

Corrosion in Molten Nitrate Salts

In 1982, Sandia National Laboratory (SNL) conducted the Molten Salt Electric Experiment (MSEE) that used molten nitrate salt as TES/HTF to produce electricity. While pure alkali nitrate salts have higher melting points, combining nitrate salts with different alkali cations leads to a reduction in melting point. Because these were initial studies, the focus was more on proof of principle and design, which is easier to accomplish with a low melting salt. Hence, lower temperature salts and low-cost materials were originally preferred. A common molten nitrate salt mixture was a composition of 53% KNO_3 , 7% NaNO_3 , and 40% NaNO_2 (by weight), known as HITEC, which has an extremely low melting point of 143°C.³ However, the

cost of producing the HITEC salt was not economical due to the high proportions of NaNO_2 . Therefore, a composition of 60% NaNO_3 and 40% KNO_3 ultimately became the popular choice.^{3,4} The elimination of NaNO_2 along with the reduction in the proportion of KNO_3 meant significant savings that outweighed the downsides of working with a higher melting HTF.⁴

The MSEE program sought to qualify the 40/60 wt% $\text{KNO}_3/\text{NaNO}_3$ alkali molten nitrate salt mixture as an HTF and for TES for use in CSP plants. The long-term feasibility of an HTF and, by extension, its use in a TES system, is based on its thermochemical stability under operating conditions. Molten nitrate salts may undergo chemical changes due to the cyclic heating nature of an HTF and the composition of the cover gas in the system. The most prevalent reaction that nitrate salts undergo is the decomposition of the nitrate anion into nitrite (NO_2^-) and oxygen, which was determined to be mitigated by a cover gas of air at standard conditions.⁴⁻⁶ The oxygen partial pressure of the cover gas along with the temperature dependence of the decomposition reaction influenced the rate at which nitrite was formed. As expected, an increase in temperature and/or a decrease in oxygen partial pressure in the cover gas resulted in an increased rate of decomposition.⁷ Despite the formation of oxide ions as a result of nitrate decomposition; a study determined that the concentration of dissolved oxide ions was not significant.⁷

Corrosion of structural components in molten nitrate salts is an important aspect to consider when qualifying an HTF-alloy combination for operation. The electrochemical reactions that occur at the interface of the molten nitrate salt HTF and the structural alloy modify the composition and properties of the alloy surface. Because electrochemical reactions are coupled reduction-oxidation reactions, the molten nitrate salt HTF will also undergo a compositional change. However, the volume of the HTF is much larger than the surface area of the structural components, and its influence on the alloy is more significant than the alloy's influence on the molten nitrate salt. Therefore, the selection of the alloy in contact with the molten nitrate salt is important to ensure that a protective, adherent oxide layer is formed that will slow the corrosion rate of the alloy. As if the high-temperature, oxidizing environment was not enough to contend with, the corrosion resistance of alloys is significantly influenced by

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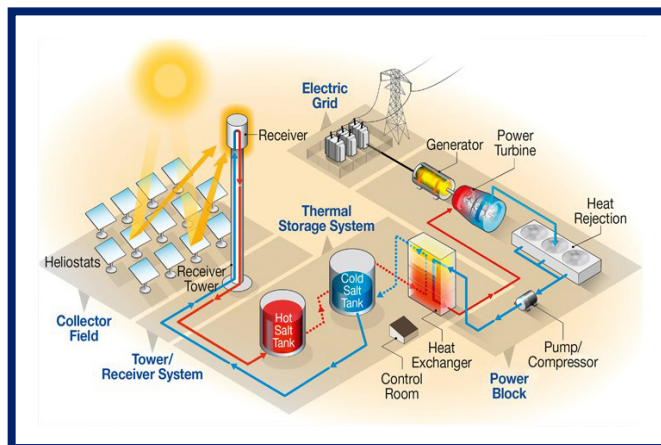


FIG. 1: Schematic of a solar thermal power system. (Courtesy of the U.S. Department of Energy.)

the level of impurities in the salt mixture. Due to the hygroscopic nature of molten nitrate salts, the most common impurity is water, and its presence significantly impacts the corrosion performance of most alloys. The oxidation rate of iron immersed in wet NaNO_3 and KNO_3 increased by 45% and 11% compared to their dry counterparts, respectively.⁸ Similar to aqueous corrosion, the presence of sodium chloride can lead to pitting in mild steels at elevated concentrations.⁹

Various alloys have been subjected to electrochemical studies in molten nitrate salt mixtures to understand the mechanisms of corrosion. The passivation of iron in molten nitrate and nitrite salts has been studied extensively in order to understand the mechanism of iron oxidation. Studies have shown that the formation of oxide ions occurs in the presence of iron, leading to the proposed mechanism shown in Fig. 2.^{5,9,10} Electrochemistry has also been used to understand the variable composition of the molten salt due to the thermal decomposition that occurs at high temperatures.¹¹⁻¹⁴ In addition to electrochemical studies, there have been numerous long-term exposure studies on the corrosion properties of Incoloy 800, SS304, and SS316 in the operating ranges of 300 to 600°C.^{4,15,16} Some key aspects of these long-term exposure studies revealed that some alloys such as Incoloy 800 and aluminized steels saw very little corrosion, while stainless steels corroded at a rate of 6-15 $\mu\text{m}/\text{year}$.^{4,17,18} Chemical analysis of molten nitrate salts following long-term exposure studies with alloys containing chromium showed that chromium dissolves significantly in these salts up to the solubility limit.⁴ The solubility of Cr corroborates findings that corrosion of Fe-Cr-Ni steel alloys in thermal convection loops exposed to thermal cycling exhibits a three-layer corrosion film consisting of a Cr-depleted outer layer, a Fe and Cr-rich oxide mid-layer, and a Ni-rich in contact with the base metal.^{16,18-21} Several studies of the oxide layer formed on Fe-Cr-Ni alloy systems generally show the formation of Fe-Cr spinels in duplex and/or triplex films with the formation of Ni-Fe-Cr oxide phases at the surface.^{17-19,22-25} High Ni-content alloys, such as Incoloy 800H, form a thicker Ni-Fe spinel compared to stainless and carbon steels as the Ni in the spinel composition are correlated to the Ni content of the alloy.^{19,20}

Corrosion in Molten Chloride Salts

The advantage of molten salts for CSP plants is the ability to increase the Carnot efficiency of power generation. Additionally, integrating a Brayton power cycle that uses supercritical CO_2 will

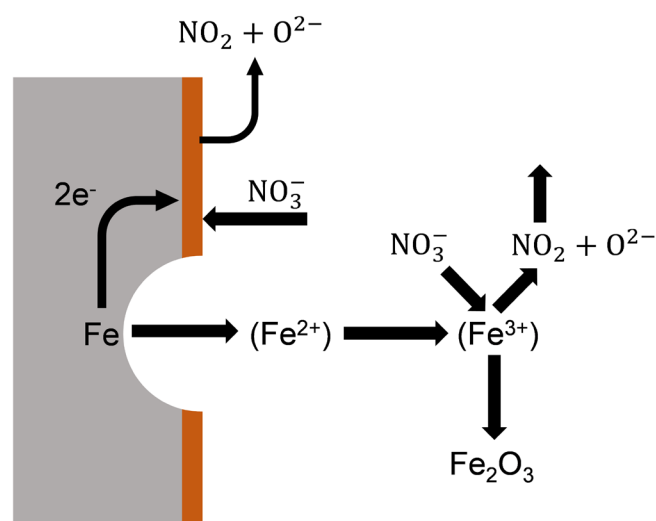


FIG. 2: Oxidation and pitting mechanism of an iron electrode exposed to molten nitrate salts.⁹

lead to further improvement in efficiencies but will also require an HTF that can operate above 560°C. Molten nitrate salts are limited to a maximum operating temperature of 565°C due to the decomposition of the nitrate ions.^{6,26} Due to this limitation, the desire to improve efficiency, and store thermal energy for longer periods, the implementation of chloride salts as HTFs has been considered due to their ability to operate at temperatures up to and beyond 800°C while maintaining thermal stability.²⁷ Due to attractive thermophysical properties and low cost, MgCl_2 and ZnCl_2 are also being considered, in addition to LiCl , NaCl , and KCl .²⁷

There are relatively fewer studies on the corrosion resistance of alloys in mixtures of molten chloride salts at various operating temperatures. The results from these studies do not follow a trend, likely due to differences in experimental conditions (such as impurities in salt), and some have shown concerning corrosion rates of ~8000 $\mu\text{m}/\text{year}$.²⁸⁻³⁰ For example, SS316 and aluminum exposed to mixtures of MgCl_2 - KCl - NaCl and MgCl_2 - KCl - LiCl were found to be severely corroded after just 1 and 3.5 hours at 500°C, respectively.³¹ Similarly, high corrosion rates have been seen in other studies for stainless steels,³² Ni-based alloys,^{32,33} and various alumina forming alloys.^{33,34} Conditions have been varied as well to study under different cover gases,^{35,36} in reducing conditions by adding Li ³⁷ or Mg .³⁸

The type and concentration of alloying elements and their effect on corrosion behavior have also been studied.^{39,40} In general, chromia forming alloys, as well as Fe/Ni-based alloys, do not perform well in molten chlorides. Cr/Ni/Fe oxides are generally soluble in molten chlorides with oxides of Cr having higher solubility than the oxides of Fe or Ni.⁴¹ Therefore, chromia-forming alloys generally perform poorly. Corrosion is exacerbated by the fact that when chromia dissolves, it liberates oxygen, thus forming a vicious cycle of chromia formation and dissolution. Increasing chromium content in alloys leads to an increase in corrosion.

Similar to molten nitrate salts, impurities in molten chloride salts have a significant influence on the corrosion properties of alloys. Impurity-driven corrosion is a primary mechanism of corrosion in molten salt systems, with water being a major and unavoidable source, even in high-purity salts.⁴² SS316L and alloy-N exposed to varying purities of 68KCl:32MgCl₂ at 700°C for 100 hours showed increased corrosion for low purity salt compared to moderate and high purity salts.⁴³ A common impurity in molten chlorides is an oxide of one or more of the constituent salt cations that are the result of hydrolysis of the salt that occurs at high temperatures. For example, hydrated MgCl_2 will decompose into MgOHCl at temperatures above 300°C and MgO above 550°C.⁴³ These oxidizing impurities will react with alloying elements and deplete them from alloys by oxidizing the alloying element, followed by dissolution, precipitation, and/or vaporization of the oxidized alloying element. Al, Mn, and Cr are particularly affected and are reflected by the poor performance of stainless steels in various molten chloride mixtures with corrosion rates ranging from 2 to 54,000 $\mu\text{m}/\text{year}$.⁴²

Overall, certain conditions have been observed that result in lower corrosion rates: use of high purity salts with low moisture content, use of a reducing agent in the salt, or the presence of Al or other passivating layer forming elements such as Ta in the alloy. Some studies have shown results that are contrary to the generalized behavior characterized above. However, there are relatively fewer studies of corrosion properties in molten chloride systems than nitrate systems, and our understanding of the surface films is less comprehensive.

Corrosion in Molten Carbonate Salts

As mentioned above, higher operating temperatures can lead to higher efficiencies. Carbonates (Li/Na/K ternary carbonate mixture) have been shown to be thermally stable for up to at least 1000°C under a CO_2 cover gas.⁴⁴ These types of ternary carbonate mixtures have a higher specific heat of ~4.9J/g-K in comparison to ~1.54 for binary nitrates. The ~3x higher specific heat leads to a higher heat capacity and the need for smaller storage tanks for similar thermal loads. Hence, carbonates are attracting more attention for use in CSP applications. Most of the molten carbonate knowledge comes

from their applications in molten carbonate fuel cells, where they are intentionally set up as an electrochemical cell. However, the conditions are quite different for CSP.⁴⁵

Corrosion in these carbonate systems has been shown to be influenced by the amount and transport of O₂/CO₂ that is dissolved in the melt and the dissolution of oxide scale and its solubility in the melt. The ability to form a protective surface film is primarily determined by O₂ content. For example, exposure temperatures below 580°C have been shown to lead to the formation of a porous LiFe₃O₈ film on SS316L, while higher temperatures led to the formation of a protective LiFeO₂ layer.⁴⁶ Several Ni-based alloys were studied via long-term exposure in molten eutectic Na/K binary carbonate salt at 900°C, and corrosion rates of up to several thousand µm/year were observed.⁴⁷ Alumina forming alloys were studied in ternary (Li/Na/K) carbonate mixtures at a temperature of 650°C, and corrosion rates were determined to be as low as 5 µm/year.⁴⁸ SS316 and Ni alloys were also studied in this ternary mixture, and corrosion rates were seen to vary with alloy 718 performing the best.⁴⁹ SS316, in general, has shown less than desirable performance under these conditions.

Similar to other molten salts, impurities in carbonate salts have been shown to play a significant role in corrosion behavior. Presence of moisture leads to the significant dissolution of Cr, but further studies are needed. The effect of other impurities such as chloride also needs to be evaluated. Further, the protectiveness of chromia is not fully understood as its effectiveness is likely linked to the activity of oxygen. While the formation of alumina has been seen to be protective, the formation of oxides of manganese, zirconium, or titanium is not necessarily equally protective.⁴⁵ There are very few studies on the corrosion behavior of alloys in molten carbonates, and our understanding is evolving.

Conclusions

Concentrated solar power is attractive due to the high Carnot efficiencies that can be achieved for power plants taking advantage of the higher operating temperatures allowed by molten salts. The added benefit of using molten salts is their ability to be used as both the heat transfer fluid and the thermal energy storage. While most molten salt-based plants use nitrate-based salts, the ability to achieve even higher efficiencies with molten chlorides and carbonates is appealing. Corrosion mechanisms of alloys in molten nitrate salts have been reasonably well understood, and corrosion rates are generally low. Fewer studies have been conducted in chloride and carbonate molten salts, and the observed corrosion rates were generally high in those studies. However, there are promising materials such as alumina forming alloys and those with a protective film-forming capability that show high corrosion resistance in these salts. Impurity and moisture content in the molten salt has been shown to lead to higher corrosion rates. Further studies under controlled environments are needed to develop a comprehensive understanding of corrosion in chloride and carbonate molten salts. ■

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