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Molten Chloride Salts for Next Generation CSP Plants: Selection of Promising Chloride Salts & Study on Corrosion of Alloys in Molten Chloride Salts

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Abstract. Molten chloride salts are promising high-temperature thermal energy storage (TES) and heat transfer fluid (HTF) materials in concentrated solar power (CSP) plants due to their high stability limits (>800°C) but low prices, compared to the commercial nitrate salt mixtures. However, severe corrosion of structural materials in contact with chloride salts is one of the most critical challenges to limit their applications at elevated temperatures. First, this work presents the properties (melting point, vapor pressure, heat capacity, hygroscopy) and large-scale prices of single chlorides and chloride mixtures for selecting the suitable molten chloride salt mixtures in next generation CSP. Then, based on our previous and current work, it discusses hot corrosion behaviors and mechanisms of metallic alloys in molten chloride salts, as well as corresponding corrosion mitigation strategies to develop the technology towards commercial applications of molten chlorides in next generation CSP.

INTRODUCTION

Recently, increasing attention is paid on applications of molten chloride salts in concentrated solar power (CSP) plants as thermal energy storage (TES) and heat transfer fluid (HTF) materials due to their high thermal stability limits (>800°C) and low prices, compared to the commercial nitrate salt mixtures (decomposition at ~550°C).

TABLE 1 compares the thermophysical properties and large-scale prices of commonly considered molten salts as TES and HTF materials in CSP. From the comparison, it can be concluded that [1]:

- All the molten salts have the similar density of ~2 g cm^{-3}.
- Commercial nitrate/nitrite salts have low melting points, high heat capacities and low prices, but low thermal stability limits (decomposed at ~550°C).
- Carbonate salts have moderate melting points (>390°C), high thermal stability limits (>650°C) and high heat capacities, but high prices due to utilization of expensive Li_2CO_3 for low melting points.
- Fluoride salts have high thermal stability limits (stable > 700°C) and high heat capacities, but are toxic and relatively expensive, and have high melting points (>450°C).
- Chloride salts have moderate melting points (<400°C), high thermal stability limits (>800°C), moderate heat capacities, and low prices.

Therefore, due to the satisfied thermophysical properties and low prices of chlorides, the U.S. Department of Energy (DOE) has supported the molten chloride salt development for next generation CSP plants over the course of the SunShot Initiative [2]. In next generation CSP, a higher efficiency of energy conversion is expected by increasing the TES/HTF operating temperature (T > 720°C) and using the sCO$_2$ Brayton cycle [2]. The further discussion in this paper is on chloride salts.

TABLE 1: Properties and large-scale prices of commonly used molten salts as TES/HTF in CSP [1].

Molten salts composition (wt. %)	Melting point (°C)	Stability limit (°C)	Density (g cm^{-3})	Heat capacity (kJ kg^{-1} K^{-1})	Material Cost (US $/kg)
Solar Salt KNO$_3$/NaNO$_3$ (40/60)	240	530-565	~1.8 (400°C)	~1.5 (400°C)	0.5-0.8
Hitec KNO$_3$/NaNO$_3$/NaNO$_2$ (53/7/40)	142	450-540	~1.8 (400°C)	1.5 (400°C)	0.9
LiNaK carbonates K$_2$CO$_3$/Li$_2$CO$_3$/Na$_2$CO$_3$ (32/35/33)	397	>650	2.0 (700°C)	1.9 (700°C)	1.3-2.5
LiNaK fluorides KF/LiF/NaF (59/29/12)	454	>700	2.0 (700°C)	1.9 (700°C)	>2
ZnNaK chlorides KCl/NaCl/ZnCl$_2$ (23.9/7.5/68.6)	204	850	~2.0 (600°C)	0.8 (300-600°C)	<1
MgNaK chlorides KCl/MgCl$_2$/NaCl (17.8/68.2/14.0)	380	>800	~1.7 (600°C)	~1.0 (500-800°C)	<0.35

However, the higher operating temperature of TES/HTF materials (in this work, i.e., molten chlorides) causes additional challenges, particularly increased corrosiveness of metallic alloys used as containers and structural materials. This significantly limits the lifetime of the structural materials and increases the levelized cost of electricity (LCOE). Fe, Cr and Ni are the common elements for commercial alloys as structural materials. Since the free energy of formation of CrCl$_2$, FeCl$_2$ and NiCl$_2$ is higher than that of the chloride salts (e.g., ZnCl$_2$, MgCl$_2$, KCl and NaCl) in the chloride mixtures, which are used as TES/HTF materials, the corrosion of alloys cannot be caused by these chloride salts in the chloride mixtures [3]. Other mechanism driving the corrosion process, e.g. impurity-driven corrosion mechanism, should be considered [3]. Addition of MgCl$_2$ or ZnCl$_2$ in chloride salt mixtures leads to low melting points. However, MgCl$_2$ and ZnCl$_2$ are strongly hygroscopic chlorides. A small amount of water adsorbed in them leads to a severe corrosion of alloys in the molten chlorides [1, 3]. Moreover, water or O$_2$ in the atmosphere above the molten salts accelerates corrosion of alloys [1, 3]. Thus, studies on corrosion behavior and mitigation strategies of alloys in molten chloride salts with corrosive impurities at high operating temperatures (500-800°C) are essential to realize the applications of molten chlorides in CSP and high-temperature industrial processes [2].

This work presents the properties and large-scale prices of single chlorides and chloride mixtures for selecting molten chloride salt mixtures, which are promising for next generation CSP. After that, it discusses hot corrosion behaviors and mechanisms of metallic alloys in molten chlorides, as well as corresponding corrosion mitigation strategies to realize the commercial applications of molten chlorides in CSP, based on our previous and current work in these fields [2-7].

SELECTING CHLORIDE SALT MIXTURES

In order to select the promising molten chloride salt mixtures for next generation CSP, the properties and large-scale prices of single chlorides and chloride mixtures are compared in this work. **TABLE 2 s**hows thermophysical properties, hydrates phases and large-scale prices of selected single chlorides, while **FIGURE 1** presents the vapor pressures of some representative single chlorides.

From **TABLE 2** and **FIGURE 1**, it can be concluded that:

- AlCl$_3$, FeCl$_3$ and ZnCl$_2$ have low melting points compared to other chloride salts (see **TABLE 2**). Low melting points are favored to have larger operation temperature ranges of TES/HTF, as well as to reduce the salt freezing risk during the CSP operation [2].
- However, at operating temperatures of the sCO$_2$ Brayton cycle for high efficiencies (T > 720°C), AlCl$_3$, FeCl$_3$ and ZnCl$_2$ have high vapor pressures (> 1 bar), while other chlorides like MgCl$_2$ have low vapor pressures (< 0.01 bar) (see **FIGURE 1**). Low vapor pressure is an advantage for application of molten chlorides as TES/HTF, as evaporation and condensation of salt in the storage system is less serve and a pressurized storage tank is not required.
- Chlorides like AlCl$_3$, CaCl$_2$, MgCl$_2$, SrCl$_2$, FeCl$_3$ and ZnCl$_2$ are strongly hygroscopic (see their hydrates phases in **TABLE 2**). They should be purified, i.e., the hydrated water should be removed, as the thermal

hydrolysis reaction of hydrated water during heating can produce corrosive HCl and hydroxyl ions harmful to metallic storage tanks and structural materials during the operation.

- $AlCl_3$, $CaCl_2$, $MgCl_2$, KCl, LiCl and NaCl have high heat capacities (> 0.9 kJ kg^{-1} K^{-1}) compared to other chlorides (see **TABLE 2**).

Alkali metal chloride salts like KCl and NaCl have high heat capacities, low vapor pressures at high temperatures, weak hygroscopy and low prices, but high melting temperatures (> 750°C). As shown in **TABLE 3**, melting temperatures of single alkali metal chloride salts can be significantly reduced by mixing with alkaline earth metal chlorides (e.g., $MgCl_2$, $CaCl_2$, $SrCl_2$) or other metal chlorides like $ZnCl_2$ and $AlCl_3$, in order to have larger operation temperature ranges. Among the binary chloride mixtures in **TABLE 3**, $MgCl_2$/KCl mixture has relatively low melting temperature (426°C), low vapor pressures at high temperatures (**FIGURE 1**) and low price (~0.35 US $/kg [2]). It can be further improved by addition of inexpensive NaCl (**TABLE 2**) to have a lower melting temperature, reduced cost and increased heat capacity (

TABLE 1). The eutectic ternary mixture $MgCl_2$/KCl/NaCl has a melting temperature of < 380°C (

TABLE 1).

Thus, $MgCl_2$/KCl/NaCl mixtures are promising chloride mixtures as high-temperature TES/HTF materials in next generation CSP. DLR is developing such chloride mixtures towards the CSP application by studying corrosion behavior and mitigation strategies of alloys.

CORROSION BEHAVIOR AND MECHANISM

The research work [1, 3-4, 17-18] indicates that corrosion of metallic alloys in molten chlorides at high temperatures is a complex process comprising interaction between atmospheric gases with molten chlorides, interaction of species in the molten chlorides and interaction between molten chlorides with metallic alloys. The corrosion is mainly driven by the corrosive impurities in the molten chlorides, which originate from the atmosphere or hydrated water and adsorbed O_2 in the chloride salts [4, 18]. Corrosion of alloys in molten chlorides under air and inert atmosphere has shown different corrosion rates and mechanisms due to the interaction between atmosphere (impurities like O_2 and H_2O) and molten chlorides [18]. Corrosive impurities (e.g., oxygen dissolved, oxygen or hydrogen containing ions like OH$^-$, H$^+$) dissolved in the molten salts can also affect corrosion rates significantly [1, 3, 18]. These corrosion behaviors and mechanisms should be considered in the development of corrosion mitigation methods.

TABLE 2: comparison of thermal properties, hydrates phases and large-scale prices of single chlorides.

Chloride salts	T_m [8] (°C)	T_b [8] (°C)	Hydrates phases	Heat capacity (liquid) (kJ kg^{-1} K^{-1})	Material Cost (US $/kg)
$AlCl_3$	193	180*	6 [8]	0.94 [10]	~0.9§
$BaCl_2$	961	1560	2 [8]	0.52 [10]	~0.5§
$CaCl_2$	775	1935	1, 2, 4, 6 [8]	0.92 [10]	~0.3§
$CrCl_2$ **($CrCl_3$)** **($CrCl_4$)**	824 (1152) (gas)	1120 (1300 dec) (>600 dec)	N/A (6) [8] (N/A)	N/A N/A N/A	N/A N/A N/A
$FeCl_3$ **($FeCl_2$)**	308 (677)	316 (1023)	6 [8] (2, 4) [8]	0.83 [10] (0.81) [10]	~0.5§
KCl	771	N/A	no [8]	0.99 [10]	0.43 [2]
LiCl	610	1383	1 [8]	1.5 [10]	>3§
$MgCl_2$	714	1412	1, 2, 4, 6 [8]	0.97 [10]	0.2 [2]
NaCl	801	1465	no [8]	1.1 [10]	0.06 [2]
$SrCl_2$	874	1250	6 [8]	0.66 [10]	N/A
$ZnCl_2$	290	732	1, 1.5, 2.5, 3, 4 [9]	0.74 [11]	1 [2]

*: sublimation point, dec: decomposition, §: The prices are estimated with the approximate large-scale prices for salts.

TABLE 3: Melting temperatures of eutectic binary chloride mixtures.

|	AlCl₃ 193°C	BaCl₂ 961°C	CaCl₂ 775°C	KCl 771°C	FeCl₃ 308°C	LiCl 610°C	MgCl₂ 714°C	NaCl 801°C	SrCl₂ 874°C
BaCl₂ 961°C	192°C [13]								
CaCl₂ 775°C	N/A	608°C [13, 14]							
FeCl₃ 308°C	N/A	N/A	N/A	235°C [15]					
KCl 771°C	128°C [13]	649°C [13, 14]	600°C [14]						
LiCl 610°C	145°C [13, 15]	514°C [14]	475°C [14]	355°C [14, 16]	N/A				
MgCl₂ 714°C	N/A	559°C [14]	617°C [14]	**426°C [14, 16]**	N/A	571°C [14]			
NaCl 801°C	105°C [13]	651°C [14]	504°C [14]	657°C [14, 15]	162°C [15]	554°C [14]	445°C [16]		
SrCl₂ 874°C	N/A	849°C [14]	662°C [14]	584°C [14]	N/A	492°C [14]	535°C [14]	565°C [14]	
ZnCl₂ 290°C	N/A	N/A	N/A	230°C [13]	N/A	N/A	N/A	262°C [13]	N/A
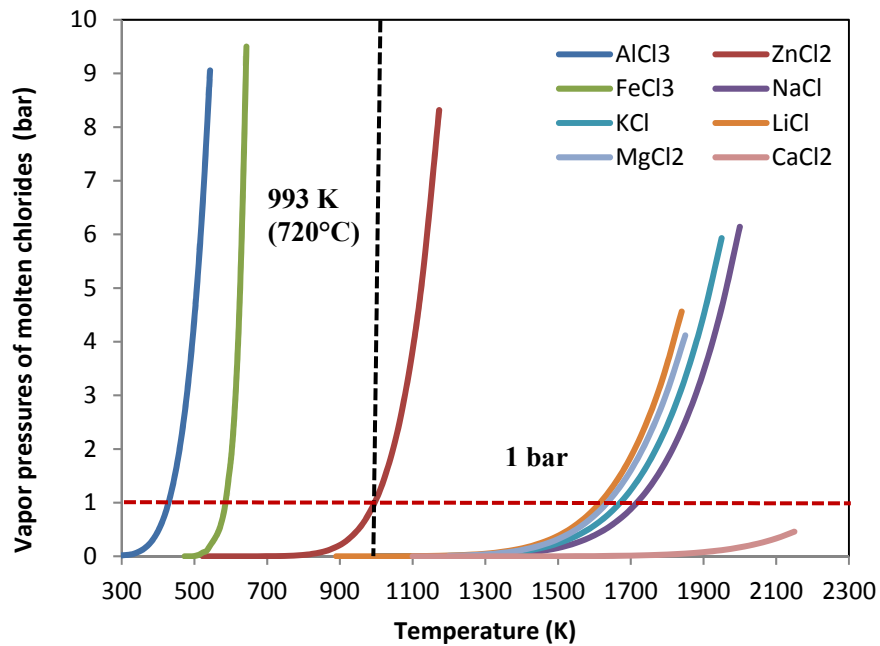
FIGURE 1: Vapor pressures of single chloride salts [11, 12].

An electrochemical method based on cyclic voltammetry (CV) has been developed at DLR to in-situ measure the concentration of the hydroxide impurities in molten MgCl₂/KCl/NaCl (60/20/20 mol%) salts at 500-700°C [5-6]. This CV method helps to verify quickly advanced purification approaches, which aim for an effective control of the

concentration of the impurities in molten chloride salts, and thus reduce corrosion of structural metallic components in contact with the molten salts.

The potentiodynamic polarization (PDP) is a useful tool for corrosion study, which has widely been used in-situ monitoring the corrosion rates of alloys in molten salts [17-20]. The corrosion current obtained from the polarization curve can be used to calculate the estimated corrosion rate according to Faraday law [17-20]:

$$CR = k\left[(I_{corr} \cdot EW)/\rho \cdot A\right], \tag{1}$$

where the corrosion rate (CR) has the unit of μm per year, $k = 3.27$ in μm g μA^{-1} cm^{-1} $year^{-1}$[20], I_{corr} is the corrosion current in μA, which could be determined from the PDP curve via Tafel-slopes (as shown in **FIGURE 2**), A is the contact area of the working electrode (studied alloys) with molten salts, EW and ρ are the equivalent weight (dimensionless) and density (g cm^{-3}) of the alloy sample, respectively [17].

In the presented work, corrosion behavior and mechanism of a commercial Cr-Fe-Ni alloy (Incoloy 800 H) in molten $MgCl_2$/KCl/NaCl (60/20/20 mol%) chlorides under inert Ar atmosphere is investigated by monitoring the concentration of impurities via CV and the corrosion rate via PDP. **FIGURE 2** and **FIGURE 3** show the PDP diagrams and calculated corrosion rates of Incoloy 800 H exposed to molten $MgCl_2$/KCl/NaCl (60/20/20 mole %) at 700°C at different exposure times, respectively, while **FIGURE 4** presents the cyclic voltammograms of the studied molten chloride salt at 700°C at different exposure times of Incoloy 800 H. In the cyclic voltammograms, Peak B represents the electrochemical reduction reaction of the main corrosive impurity $MgOH^+$ to H_2 and MgO [5-6]. The peak current density of Peak B i_p(B) is proportional to the bulk concentration of $MgOH^+$ in the melt [5-6].
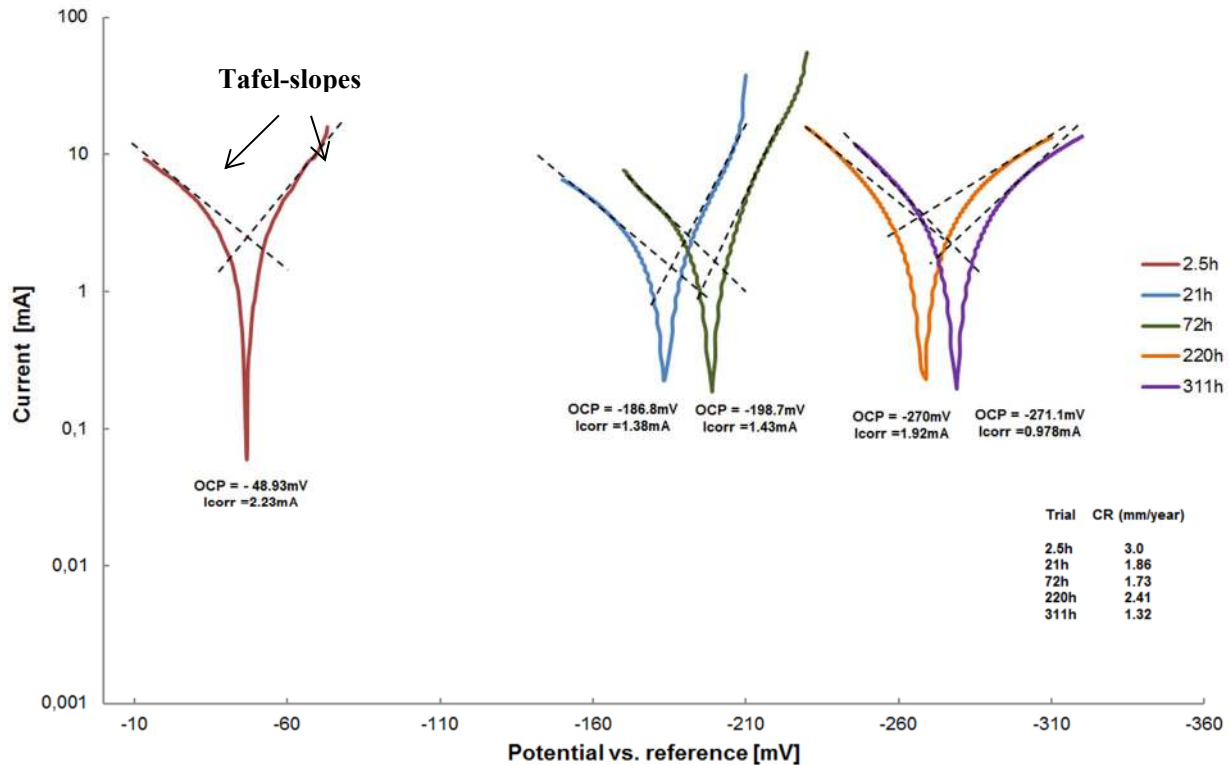

FIGURE 2: Potentiodynamic polarization (PDP) curves of Incoloy 800 H exposed to molten $MgCl_2$/KCl/NaCl (60/20/20 mole %) at 700°C at different exposure times (2.5, 21, 72, 220 and 311 hours).
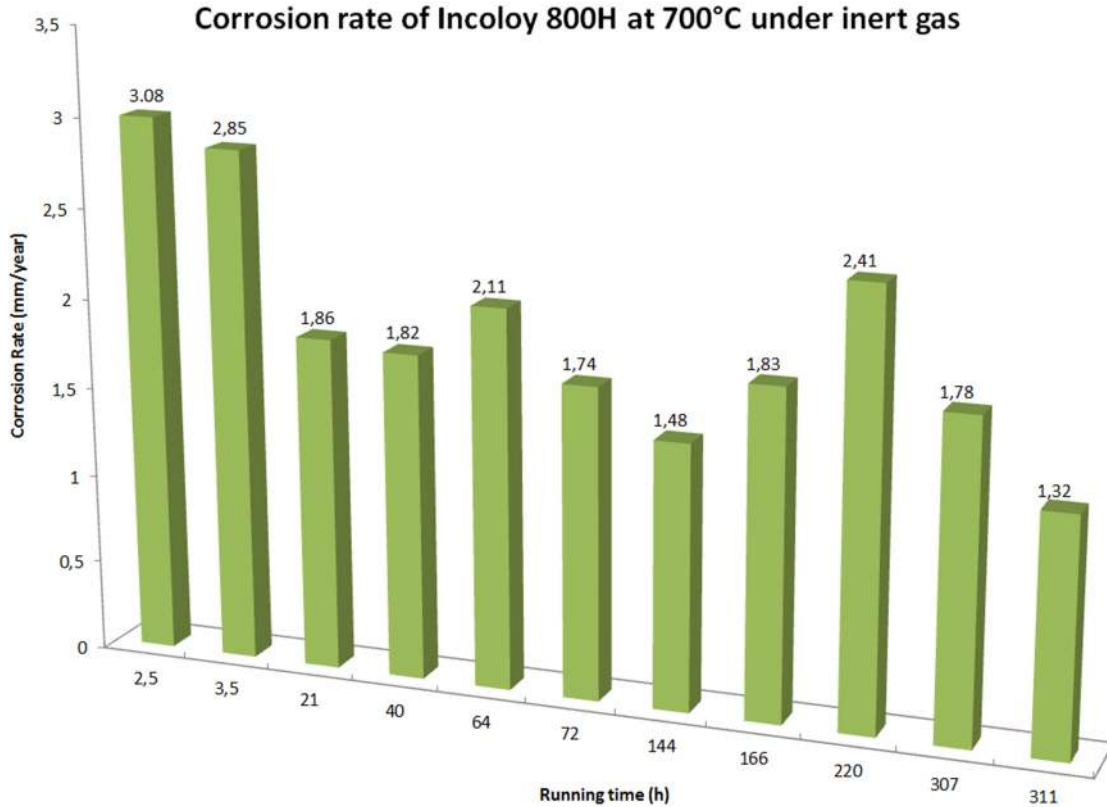OCP: Open Circuit Potential. Icorr: Corrosion current. Sweep rate of PDP: 1 mV/s.

FIGURE 3: Estimated corrosion rates of Incoloy 800 H exposed to molten $MgCl_2$/KCl/NaCl (60/20/20 mole %) at 700°C with the exposure time.

As shown in **FIGURE 3**, in the initial stage (exposure time 0-40 h), the corrosion rate decreases with the exposure time significantly from 3.08 to 1.82 mm/year. In the second stage (exposure time > 40 h), the corrosion rate decreases slowly. Moreover, the fluctuation of the corrosion rate is observed, e.g., at exposure times of 64 and 220 h. This may be explained with that the fresh surface of the studied alloy to expose in the melt is produced due to the corrosion. After 311 h exposure, the corrosion rate decreases to 1.32 mm/year. Overall, it is found that the corrosion rate trends to be lower with the increasing exposure time due to the decreasing amount of the corrosive impurities in the melt. This is indicated by the CV measurements (see **FIGURE 4**) that the concentration of the main corrosive impurity $MgOH^+$ (i.e., $i_p(B)$) is shown to decrease with the exposure time, due to its reaction with the elements like Cr in the alloy. Moreover, some additional peaks (Peak E and F) are observed in the CVs after ~100 h exposure, which indicate that some metal ions such as Cr^{x+} may exist in the melt due to the corrosion (x $MgOH^+$ + Cr → x MgO + x/2 H_2 + Cr^{x+}). The atomic absorption spectroscopy (AAS) on the salt after the exposure test also indicates that the salt contains 0.041% Cr and 0.018 % Fe.

CORROSION MITIGATION

Corrosion rates of alloys in molten chlorides can be reduced via corrosion mitigation methods such as adding **corrosion inhibitors** or **forming a protective layer** on alloys [21-23]. Cathodic protection is a well-known method for preventing metallic corrosion in aqueous solutions. The protection method of using a sacrificial anode has been used to reduce the corrosiveness of the molten chloride salts by saturation of a salt with a liquid metal (i.e., LiCl with Li metal [21]), or adding active metals such as Mg [22] in the molten chlorides, to reduce the redox potential of the melts, i.e., the concentration of the corrosive impurities like metal-hydroxyl ions (e.g., $MgOH^+$) and dissolved O_2.
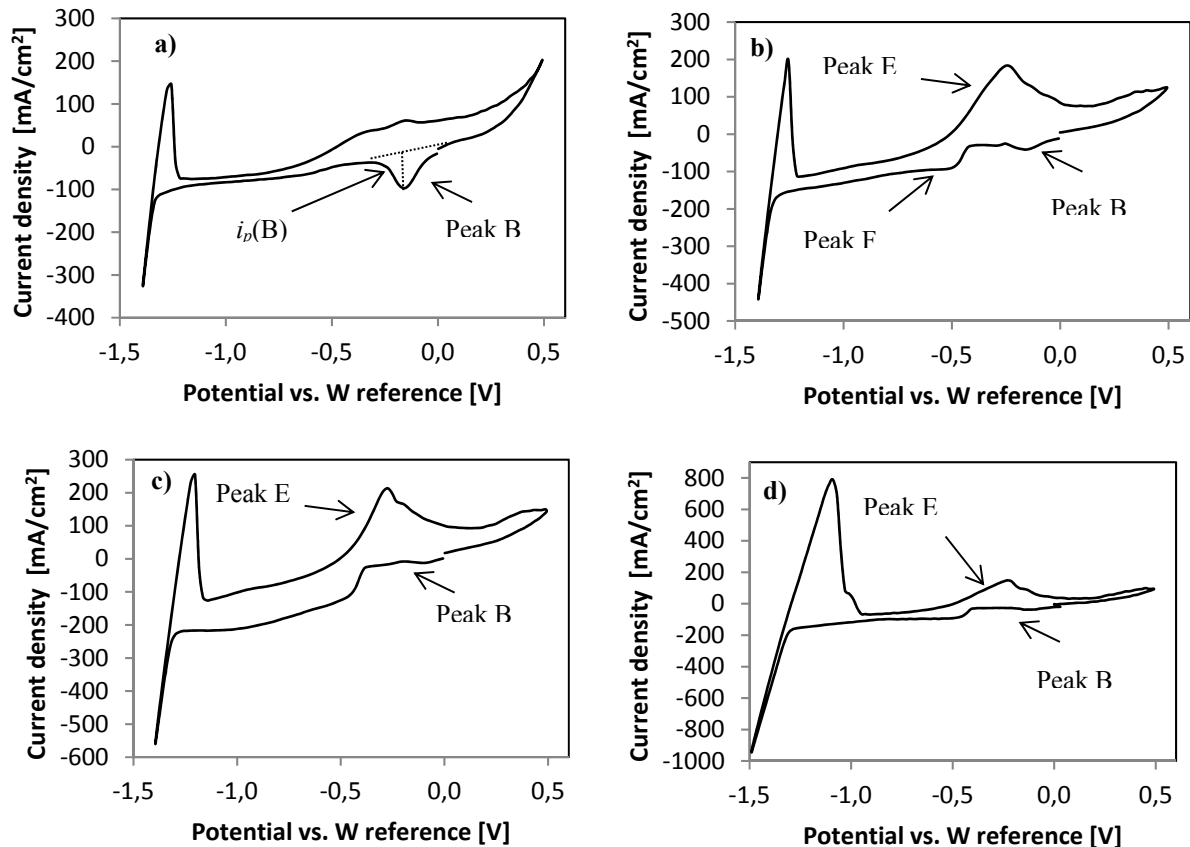
FIGURE 4: Cyclic voltammograms of the studied molten chloride salt at 700°C at the exposure time of Incoloy 800 H: a) 2.5 h, b) 144 h, c) 190 h and d) 311 h. Sweep rate: 200 mV/s. Sweep direction: 0 V → -1.5 V → +0.5 V → 0 V.

In our current work [7], the cathodic protection method was used to mitigate corrosion of commercial Cr-Fe-Ni alloys in molten chloride salts (MgCl$_2$/NaCl/KCl 60/20/20 mol%) with 1 wt.% Mg addition under inert atmosphere . **FIGURE 5** shows the salts and Incoloy 800 H samples after exposure to molten chlorides without (left) and with Mg corrosion inhibitor (right) at 700°C for 500 h. It is cleanly found that the salt with Mg addition keeps white, while the salt without Mg addition is changed due to the new impurities e.g., Cr and Fe species from the corrosion process (AAS indicates that it contains 0.175% Cr and 0.045% Fe). Additionally, the corrosion mitigation mechanisms were further studied via microstructural analysis on exposed alloy samples and corrosion products with SEM, EDX and XRD [7]. The cross-sectional SEM and EDX images of the exposed alloy samples indicate that the corrosion rates of In 800 H in the molten chlorides with Mg corrosion inhibitor were reduced to ~10% compared to the molten chloride mixture without Mg [7].

Surface passivation by forming a protective layer on the alloys is another promising corrosion mitigation approach, as the alloy with a protective layer could be exposed to both the liquid and the vapor phases of the molten chlorides [23]. Some effort has been made on the development of a protective layer on alloys for molten chlorides, e.g., a ceramic layer like Al$_2$O$_3$ and Yttria-stabilized zirconia (YSZ) [23], and a metallic layer such as nickel based alloy coating [23]. In our current work [7], a **dense protective Al$_2$O$_3$ layer** was formed on the model Cr-Fe-Al alloys via pre-oxidation in air. The experimental results in molten chloride salts at 700°C for 500 h show that it can successfully inhibit the outer diffusion of Cr and inner penetration of corrosive impurities [7].
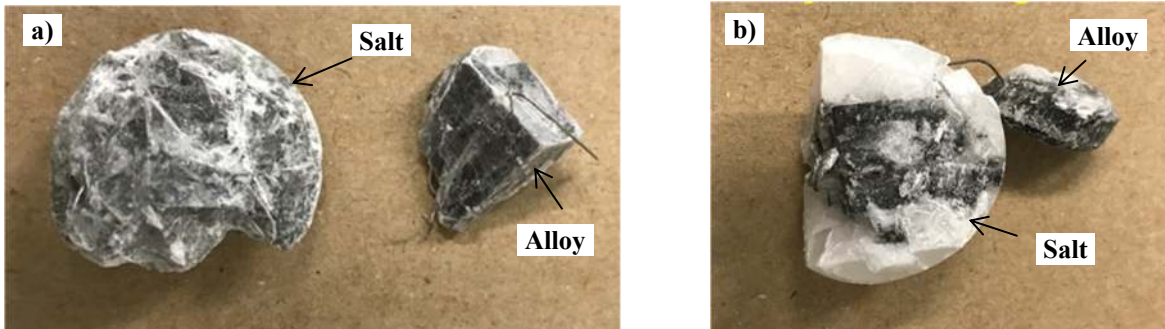
FIGURE 5: Salts and Incoloy 800 H samples after exposure to molten $MgCl_2/KCl/NaCl$ (60/20/20 mole %) at 700°C for 500 h: (a) without Mg corrosion inhibitor; (b) with Mg corrosion inhibitor.

SUMMARY

- Chloride salts are promising TES/HTF materials in next generation CSP due to moderate melting points (<400°C), high thermal stability limits (>800°C), moderate heat capacities, and low prices, compared to nitrate/nitrite, carbonate, and fluoride salts.
- $MgCl_2/KCl/NaCl$ salt mixtures are selected for further study due to low prices, high heat capacities, low melting temperatures and low vapor pressures, compared to other chloride salt mixtures.
- Electrochemical methods are established for monitoring corrosion: CV for impurities in molten chloride salts, and PDP for corrosion rates of alloys in molten chloride salts.
- Corrosion mitigation methods are investigated:
    - Mg is added as the corrosion inhibitor in the molten chloride salts. The corrosion rate of the alloy is significantly reduced.
    - Surface passivation by forming a protective Al_2O_3 layer on Cr-Fe-Al alloys shows the promising corrosion mitigation.
- In future work, we continue to develop these techniques for monitoring chloride salt quality and corrosion, and corrosion mitigation techniques to increase the Technology Readiness Level for CSP applications.

ACKNOWLEDGMENTS

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