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journal homepage: www.elsevier.com/locate/solmatCorrosion of steel alloys in molten NaCl + Na₂SO₄ at 700 °C for thermal energy storage

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ABSTRACT

Stainless steel 316, duplex steel 2205 and carbon steel 1008 were examined for compatibility with the eutectic mixture of NaCl + Na₂SO₄ at 700 °C in air for thermal energy storage. Electrochemical measurements combined with advanced microscopy techniques were employed. Results confirmed oxidation to be the primary attack to all metals. Ferritic alloy 1008 was found the most vulnerable alloy with severe oxidative attack. Alloys 316 and 2205 showed close performance while grain boundary oxidative and sulfur attack threatened these materials. Alloy 2205 showed chromium depletion followed by selective oxidation of ferrite in the presence of a semi-protective film on the surface.

1. Introduction

Recent interest in raising the working temperature of Concentrating Solar Thermal Power (CSP) plants requires considerable improvement in material compatibility used for Thermal Energy Storage (TES) [1–6]. TES is a technology comprised of a storage medium retained in a containment material (tank) [7–11]. Whilst insulating the system from its surroundings, containment systems hold storage medium and energy transfer equipment. TES systems commonly take advantage of Phase Change Materials (PCM) with high thermal capacity which store energy as both sensible heat and phase change [7,11]. Corrosion behavior of the containment systems is a key issue affecting material selection, fabrication, maintenance costs and overall life cycle of CSP plants [12]. Steel alloys are considered as economic candidates for containment materials in TES systems while eutectic composition of molten chlorides, nitrates, carbonates and fluorides are favorite candidates as PCM [11,13–18].

Based on the working temperature low-alloyed carbon steels, Cr-Mo steels and Cr-Ni stainless steels might be employed in combination with PCMs [11]. The compatibility of structural metals with molten salts depends on the metals potential for oxidation to the corresponding salt, passivating nature and the solubility of corrosion products in the salt [19]. Corrosion resistance in many high-temperature environments is achieved by the formation of a protective oxide scale on the material surface [20]. Cr, Al, and Si are widely used in alloys to promote the development of self-healing protective oxide films on the material surface acting as diffusion barriers against further oxidation [14]. The stability of oxide layers and the impact of salt impurities and the

atmosphere should be taken into consideration in molten salt exposure [11]. The corrosion behavior of Fe-based alloys at 450 °C in ZnCl₂-KCl deposits in flowing pure oxygen showed that the material suffers from intensive corrosion characterized by the separation and spallation of oxide scales [21]. Studies on molten nitrate salts at 570 °C and 316 °C have also revealed the formation of complex and multiphase oxides, like Fe-Cr spinel or iron oxide, on the steel alloy surface [22].

Since the next generation of CSP plants are expected to operate at temperatures higher than those currently in use, PCMs with higher melting points are required to be employed in the storage system [23]. The compatibility of containment materials with PCMs and its stability during the cyclic operation of CSP plants is a concerning issue [9]. The selection of appropriate and optimal structural materials as thermal energy storage vessels is essential in developing economic and functionally efficient TES systems. This study will examine the corrosion behavior of three commercial steel alloys in a eutectic mixture of molten sodium chloride + sodium sulfate salt for the next generation of CSP applications.

2. Experimental procedure

Sodium chloride AR (CAS No. 7647-14-5) and sodium sulfate AR anhydrous powder (CAS No. 7757-82-6) were placed for 24 h in a 180 °C furnace to dry and then were measured and mixed according to the salt composition (in wt%) 26.5 NaCl + 73.5 Na₂SO₄. This eutectic melts at 626 °C, which seems to be close enough to the temperatures required for the next generation of CSP plants [24].

Commercial structural alloys of austenitic stainless steel 316

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Table 1
Nominal elemental composition and crystal structure of the steel alloys (wt%).

Alloy	Structure	Fe	Ni	Cr	C	Mo	Mn	S	P
SS316	fcc	Bal	12	17.5	0.05	3
DS2205	fcc/bcc	Bal	6.5	23	0.03	3.5
CS1008	bcc	Bal	0.14	...	0.5	0.04	0.04

(SS316), ferritic carbon steel 1008 (CS1008) and ferritic/austenitic duplex steel 2205 (DS2205) were then examined in the salt mixture at 700 °C in air. This temperature is close to the salt melting point assuming that the salt will be used as PCM in a CSP plant. Although it is unlikely for alloy CS1008 to be used at such a high temperature, we decided to study this alloy for a better understanding of the behavior of ferrite compared to austenite in steel alloys in molten salt environments. Structures and nominal compositions of the metals used in the current study are summarized in Table 1.

2.1. Electrochemical corrosion investigation

Electrochemical experiments were conducted using a three electrode cell containing the molten salt in alumina crucibles open to air at 700 °C in a preheated cylindrical furnace. Test coupons of 25 mm long, 5 mm wide and 1.4 mm thick were mechanically wet ground and polished down to 0.04 μm by colloidal silica, washed with ethanol and dried in air. Measurements were implemented by means of a VMP3-based BioLogic instrument controlled by EC-Lab® software. The three electrode cell was implemented with the polished sample as the working electrode and two same sized platinum sheets (25 \times 5 \times 1 mm) as pseudo reference and counter electrodes [25–28]. Samples were immersed into the molten salt for 1 h to stabilize and then were subjected to electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PDP) measurements. EIS measurements were obtained using a frequency range of 200 kHz–100 mHz with the amplitude of ± 10 mV. PDP was conducted at the potential scan rate of 10 mV/min and potential range of -750 to 200 mV with respect to the Pt pseudo reference electrode. ZFit analysis of EC-Lab software was the used to fit impedance data.

2.2. Static corrosion

Fresh metal coupons were cut to 25 \times 7 \times 1.4 mm for static corrosion and were mechanically wet polished down to 1 μm in colloidal silica using standard grinding and polishing procedures. A schematic representation of the test condition and alignment of samples in the furnace have been already shown [29]. Cylindrical alumina crucibles were used as salt vessels and the furnace temperature was set to 700 \pm 10 °C.

The vessels containing the eutectic salt were placed into the furnace at room temperature and then gradually heated up to the test temperature. Once the salt melted and the chamber conditions stabilized, the metal samples were immersed so that the top half was exposed to the air and bottom half submerged into the molten salt. Such a configuration enabled us to make a comparison between the impact of the salt and that of the oxygen from the environment in the presence of decomposition gases of the salt, which are expected to increase the corrosive impact in this atmosphere. Metal coupons were removed after 120 h of exposure. All coupons were then mounted exposing the side indicated previously [29] into a conductive resin, ground and polished down to 0.04 μm from the side of the sample in colloidal silica using standard procedures, washed with ethanol and finally dried in air. The selected microscopy points [29] provide us with a view of the material close to the polished side. It is worthy to note that although 120 h does not seem long enough to study corrosion rate and related phenomena, the aggressive nature of molten salts makes studying short-term impacts

like attack morphology and corrosion mechanisms possible.

2.3. Metallography samples

Small coupons of each metal were selected and mechanically wet ground and polished down to 0.04 μm by colloidal silica, washed with ethanol and finally dried in air. These coupons were used to study the corroded metal morphology and short-term impact of the molten salt on the microstructure. The coupons were submerged into the molten salt for only 3 min and then were ultrasonically cleaned for 15 min in demineralized water for corrosion product residues to be removed from the surface. The samples were then studied under an optic microscope from the surface.

2.4. Macro and micro-structural investigations

2.4.1. Optical microscopy analysis

Optic microscopes model Leica DMi8A (magnification 1.25 \times –50 \times) and Leica M125 (magnification 0.8 \times –10 \times) both equipped with Leica Application Suite software were used to take macro and micro-images for microstructural and corrosion observations.

2.4.2. SEM, EDS and EBSD analysis

Complimentary techniques like scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS) and electron backscatter diffraction (EBSD) were employed for further microstructural investigations using a field emission SEM (model: JEOL 7001F, with automated feature detection equipped with secondary electron, EDS analysis system, OXFORD EBSD pattern analyzer and Channel 5 analysis software).

3. Results and discussion

3.1. Electrochemistry

PDP graphs of the alloy specimens in the molten salt at 700 °C are represented in Fig. 1. Plots show the least E_{corr} values for CS1008 and SS316 with nobler values for DS2205. This emphasizes that the duplex steel is the more noble alloy to the molten salt at the test temperature probably because of its high Cr content [30]. Data extracted from PDP curves is summarized in Table 2. Maximum I_{corr} value belongs to CS1008 while SS316 and DS2205 represent the least (and very close) current density values.

Cathodic and anodic branches of PDP plots provide more

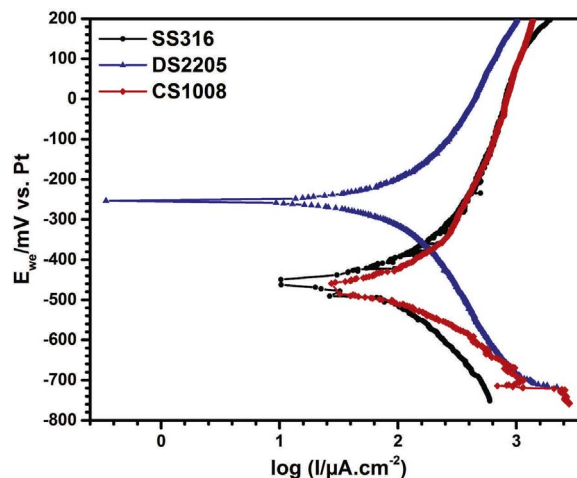


Fig. 1. Potentiodynamic polarization (PDP) curves of samples in molten eutectic NaCl + Na₂SO₄ at 700 °C in air. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

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