

Corrosion Behavior of UNS S31600 in a Purified Molten Salt

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ABSTRACT

Chloride salt mixtures have been proposed as candidates to improve the efficiency of CSP plants, due to their high thermal stability, thermal conductivity, heat capacity and low cost. However, molten chlorides are highly corrosive, and impurities in salts like magnesium chloride can increase the corrosivity of this salt mixture. Therefore, an in situ method of purifying the mixture was applied to mitigate the severity of their corrosive effects on the containment materials. The molten salt corrosion behavior of UNS S31600 at 700°C was evaluated in (a) the purified KCl-20 mol% NaCl- 20 mol% MgCl₂ salt mixture and (b) in mixtures using as-received salts. Electrochemical tests were performed according to the ASTM G59-97 standard to determine the corrosion behavior of metallic alloy pre- and post- salt purification.

Key words: Concentrated Solar Power, Molten Salt Corrosion, Stainless Steel, Electrochemistry

INTRODUCTION

Concentrated solar power (CSP) is one of the renewable energy industry's leading candidates for power generation due to its ability to harness solar energy along with its capacity to contain solar energy in the form of thermal energy storage (TES).^{1,2} CSP plants commonly use molten salts as the working fluid for the steam generation process to store thermal energy due to their favorable thermal properties.³

The salt selected as the working fluid for CSP plants needs to have desirable properties such as high heat capacity, thermal conductivity, low melting point, high boiling point, and low vapor pressure. The cost of the salts is another crucial factor that impacts the economic viability of a commercial CSP plant. Chloride-based salts are great candidates relative to nitrate and fluoride-based salts due to their favorable thermal properties and low material cost; however, chloride salts are corrosive.^{4,5,6,7} Moreover, the chloride salts of interest are hygroscopic and can contain impurities. Thus, the molten chloride salt may need to be purified, i.e., removal of contaminants, to mitigate the effect of corrosion from contaminants that could increase the corrosion rate.^{8,9} The selection of appropriate materials for components like piping and pipe fittings is crucial. Nickel-base alloys are potential candidates; however, they are quite expensive. A grade of austenitic stainless steel, UNS S31600, was chosen for testing due to its promising performance in nitrate molten salts and its cost effectiveness for the cost-conscious CSP industry.^{10,11} As examples of the cost-benefit value of UNS S31600: (a) the relative cost of UNS S31600 is only 1.29 times that of UNS S30403, thereby offering enhanced corrosion resistance for a small price, and (b) the cost

savings are very significant in comparison with UNS N0625, the latter being at least 6.01 times more expensive on a 30,000 lbs (13, 608 kg) basis for a plate geometry and assuming that the plates were hot rolled, annealed and pickled.^{11,12} Moreover, the choice of UNS S31600 also stems from the current use of UNS S31600 as an infrastructure material in current molten salt CSP plants.¹³

In this project, a KCl-20 mol% NaCl- 20 mol% MgCl₂ ternary eutectic salt mixture was purified using an electrolysis process, resulting in the removal of impurities like magnesium hydroxide. Electrochemical corrosion tests were conducted using both as-received salts and purified salts to evaluate the behavior of UNS S31600 at 700°C in each of the two environments.

EXPERIMENTAL PROCEDURE

Coupons of UNS S31600 were cut from rods nominally 1.03 – 1.25 cm in diameter, and nominally 0.2 – 0.28 cm thick, were ground to 600 grit with SiC grit paper. The surface area of the test coupon was in the range of 2.4 – 3.6 cm². These coupons were used for electrochemical test. The complete electrochemical setup consisted of an alumina crucible, flange, thermocouple sheath, inlet and outlet gas tubes, and tungsten counter and reference electrodes. The coupon was extended with tungsten wire and served as the working electrode in the electrochemical setup. An alumina crucible was suspended through the flange as well as the required electrodes and gas tubes. The electrodes were sheathed to avoid physical contact between the electrodes. The setup procedures were based on our previous work done in a high temperature corrosion research project.^{14,15} The molten salts were purified with electrolysis and verified with cyclic voltammetry techniques. A small amount of a eutectic chloride salt mixture (7 g) was placed evenly inside the crucible after setting up for each test. The salts were preheated in a furnace at approximately 110°C to avoid the presence of moisture since MgCl₂ is hygroscopic and it has a great tendency to absorb moisture. The entire system was tightly sealed inside a vertical tube furnace with a gasket using vacuum grease. The experimental preparations were the same for tests using unpurified and purified salt mixtures. A commercial potentiostat was used to conduct the electrochemical testing in accordance with ASTM Standard G59-97.¹⁶ The Open Circuit Potential (OCP) scan was run for one hour to determine the corrosion potential of the alloy and check for salt stability. A Linear Polarization Resistance (LPR) scan was conducted from -20 to +20 mV, followed by Cyclic Potentiodynamic Polarization (CPP) scan from -400 mV to +400 mV at a rate of 0.167 mV/s where both the LPR and CPP scans were performed with respect to OCP scan. The test was completed at 700°C in a stagnant argon environment, with the entire test lasting for about 3 hours. The post-corroded coupons were then examined and characterized with scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS).

RESULTS

Electrochemical Tests

Figure 1 shows two example Linear Polarization Resistance (LPR) plots for UNS S31600 tested in unpurified and purified salt mixtures at 700°C with stagnant argon. Examples of Cyclic Potentiodynamic Polarization (CPP) plots for UNS S31600 that were tested in both purified and unpurified salt mixtures at 700°C in stagnant argon are portrayed in Figure 2. Electrochemical calculations were calculated according to ASTM G59-97 standard.¹⁰ Polarization resistances (R_p) are obtained from the slope of Linear Polarization Resistance (LPR) plots.

Coupons tested in the purified salt mixture had a lower average R_p value, 41.6 $\Omega \cdot \text{cm}^2$, compared to the average value for the unpurified salt mixture, which has a higher R_p value, 421.2 $\Omega \cdot \text{cm}^2$. This was contrary to what was expected since it signifies that the alloy has greater corrosion resistance in the unpurified salt mixture than in the purified salt mixture. Measurements of the open circuit potentials showed that the unpurified coupons, on average, had higher nobilities (-62 mV) relative to the purified coupons (-142 mV). The corrosion potentials measured from the CPP plots showed that the potentials in the unpurified salts (-95 mV) were nobler than those in the purified salts (-185 mV). Thus, both in regards to the resistance