



Corrosion behavior of stainless and low-chromium steels and IN625 in molten nitrate salts at 600 °C



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ABSTRACT

The corrosion resistance of two ferritic steels P91 and X20CrMoV11-1, two austenitic steels SS316 and SS347H and a Ni-alloy IN625 is studied during long term isothermal immersion in molten nitrate salts consisting of 40% KNO₃ and 60% NaNO₃. The corrosion test was conducted at 600 °C for a maximum immersion time of 5000 h. The corrosion behavior was determined by recording the weight changes of samples at different time intervals and metallographic methods. The corrosion products were investigated in detail via electron probe micro-analysis (EPMA), and scanning electron microscopy (SEM). Results showed that P91 and X20CrMoV11-1 do not offer a sufficient long term corrosion resistance in molten nitrate salts. Stainless steels SS316 and SS347H instead formed protective oxide scales in both salts with similar kinetics. However, dissolution of the formed oxide scale in the form of alkali ferrite was observed on stainless steels. A linear weight loss with a very slow kinetics was evident for IN625. However, IN625 showed the best protective behavior among alloys. The qualitative and quantitative EPMA results revealed that a multiphase oxide layer composed of iron–chromium spinel, iron oxides, and sodium ferrite formed on the stainless steel SS347H. A dense NiO layer was primarily formed on the IN625 surface.

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1. Introduction

The increasing interest for perusing clean, renewable, reliable, and efficient energy sources is recently emerged in concentrated solar power (CSP). Although the solar power industries have existed since 1970s, most of its commercialization steps were conducted during the last decades [1].

The common CSP technologies are (a) parabolic dish systems (PDS), (b) parabolic trough collector (PTC), (c) solar power tower (SPT) and (d) linear Fresnel reflector (LFR). To date, the parabolic trough collector is used in more than 95% of the CSP plants [1]. Concentrated solar power technology is based on basic principles. The solar radiation is concentrated by program focused mirrors (heliostats) to a receiver. The solar heat is stored by the heat transport fluids (HTF) in the receiver and is further transported to the hot storage tank [1]. The thermal energy is used to generate steam for use in an ordinary turbine generator. After exchanging the thermal energy, the cold salt is transported to the receiver to repeat the cycle [1].

Molten nitrate salts (KNO₃–NaNO₃) are used as HTF and for energy storage in solar power technology. Their characteristic

properties such as high specific heat, high thermal conductivity, high boiling point, low vapor pressure, and high density made them desired candidates for heat transfer applications [2]. In solar power plants, the eutectic composition of 40 wt% KNO₃–60 wt% NaNO₃ (so called solar salt) is the preferred molten salt mixture due to its low melting point of 223 °C. Such solar salts are thermally stable up to 600 °C and they circulate between the hot storage and the cold storage tank in a temperature range of 290–565 °C [3]. Increasing the thermal efficiency in this cycle requires lower melting points of the salt mixtures (in cold tank) and higher maximum operation temperature limits (in hot tank) [4,5]. These characteristics, however, rely on the salt characteristics. The cold tank temperature cannot go below the melting point of the salt as a continuous salt flow is essential in CSP applications. On the other hand, the hot tank temperature is limited by the stability of the salt and the stability of salt containing materials. It is known that the binary nitrate salts are not stable beyond 600 °C in oxidizing atmospheres (e.g. air) [6]. Moreover, the combination of high operating temperature and salt composition presents a severe corrosive environment [7–10]. Molten nitrate salts contain oxidizing species such as NO₂⁻ and NO₃⁻ and O₂ (from air). In addition, commercial salts contain levels of impurities such as chlorides, nitrites, sulfates etc. which can induce more corrosion. Therefore, before increasing the operating temperature the corrosion behavior of the storage tanks and transport tube materials must be

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examined. At high temperatures, molten solar salts act as ionic electrolytes leading to the transportation of oxidizing agents to the metal and metal ions into the salts.

Alloys such as low alloy steels P91 and X20CrMoV11-1, stainless steels 316, 304 and nickel alloys Incoloy 800 and IN625 are candidates for this application [9,11–13]. Recently, several studies have been conducted to understand the corrosion behavior of such materials in molten salts at higher temperatures [7,9,10,12,13].

In the following, the corrosion behavior of five materials including two ferritic steels, two austenitic stainless steels and nickel base IN625 alloy during isothermal exposure for up to 5000 h at 600 °C in molten nitrite salt will be addressed. The corrosion kinetics and the resulting corrosion products are evaluated and discussed.

2. Experimental

Three classes of alloys including two ferritic steels (P91 and X20CrMoV11-1), two austenitic stainless steel (SS316 and SS347H), and a Ni-alloy (IN625) were studied as candidate materials for solar salt applications. The compositions of the alloys were obtained using optical emission spectroscopy (OES) and are listed in Table 1. Rectangular coupons were ground to 320 grit SiC abrasive paper, degreased in alcohol and dried in air. The initial dimensions and weight of the coupons were recorded.

A nitrate salt mixture of 60 wt% NaNO₃–40 wt% KNO₃ composition was provided by Ferrostaal GmbH. The salt mixture contained different levels of impurities, primarily consisting of NaCl, MgNO₃, Na₂CO₃ and Na₂SO₄. The level of impurities in the salt mixture according to the salt provider is given in Table 2.

The corrosion test was conducted following the ISO/NP 17245 standard protocol (*Corrosion of Metals and Alloys-Test method for high-temperature corrosion testing of metallic materials by immersing in molten salt or other inorganic liquids*). The test was

performed in a horizontal tube furnace with samples placed in alumina boats and covered with the salt mixture. The boats were placed in the hot zone of the furnace which was calibrated at 600 °C (Fig. 1).

Flowing laboratory air was passed through the furnace with a linear flow rate of 6 cm h⁻¹. Five specimens were placed in each alumina boat and a sample was removed after each accumulative immersion intervals of 100, 500, 1000, 2500, and 5000 h (Fig. 2). Only one sample of each alloy was removed at each time interval.

After cooling, the exposed coupons were rinsed with warm distilled water to remove the remaining salt and were then weighed and stored in a desiccator. The as-removed specimens were investigated on a macroscopic and microscopic scale. A macrograph was taken from each specimen before metallographic investigations. Metallographic preparation of the immersed samples was commenced by wrapping the sample in a thin nickel foil. This was performed to protect the oxidation products during the subsequent metallographic preparation. Each coupon was then embedded and ground by various grades of silicon carbide paper using petroleum oil as a lubricant to avoid removal of chlorides. For final polishing different grades of diamond paste (down to 1 μm) were used and the polished sample was rinsed in molecular sieve dried acetone and then dried. Metallographic cross sections were microscopically studied using optical microscopy. A JEOL JXA-8100 Electron Probe Micro Analyzer (EPMA) was used to characterize the corrosion products of the alloys with better corrosion performance.

3. Results and discussion

3.1. Oxidation kinetics

Fig. 3 shows the plot of weight change data versus immersion time. These plots allow the determination of the reaction kinetics

Table 1
Chemical composition of the studied alloys (wt%).

Alloy	P91	X20CrMoV 11-1	SS347 H	SS316	IN625
EN number	1.4903	1.4922	1.455	1.4401	2.4856
Alloy type	Ferritic steel	Ferritic steel	Austenitic stainless steel	Austenitic stainless steel	Ni base alloy
Price Euro/ton ^a	450	500	1880	1925	7700
Composition (wt%)					
C	0.102	0.208	0.05	0.253	0.396
Si	0.356	0.274	0.35	0.485	0.176
Mn	0.412	0.58	1.24	1.79	0.089
P	0.014	0.022	0.034	0.031	< 0.001
S	0.01	0.005	0.004	0.004	0.003
Cr	8.75	10.37	17.06	17.05	22.85
Ni	0.17	0.693	9.84	12.03	59.7
Mo	0.944	0.852	0.341	2.37	8.6
Fe	88.7	86.5	70.1	65.8	3.41
Al	0.001	0.014	0.003	0.032	0.231
Ti	0.002	0.001	0.009	0.008	0.288
V	0.203	0.247	0.041	0.083	0.055
Co	< 0.002	0.025	0.052	0.036	0.221
W	0.008	0.045	0.022	0.022	0.08
Cu	0.119	0.056	0.288	0.097	0.055
Nb	0.08	< 0.004	0.523	0.015	4.07

^a According to www.dew-stahl.com, 04.2015.

Table 2
The reported level of impurities in the nitrate salt mixture.

Impurity	Nitrite (NO ₂ ⁻)	Chloride (Cl ⁻)	Magnesium (Mg ²⁺)	Carbonate (CO ₃ ²⁻)	Sulfate (SO ₄ ²⁻)	Insolubles	Moisture
Amount (wt%)	0.01	< 0.02	< 0.0007	< 0.1	< 0.01	< 0.01	< 0.2

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