Microstructural Changes on Tensile Property of Austenitic Alloys Exposed to High Temperature Supercritical-CO₂ Environment

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1. Introduction

Several studies have been conducted on corrosion and mechanical properties of ferritic martensitic steels (FMSs) in liquid sodium coolant environments [1]. However, compatibility study of austenitic alloys in high temperature supercritical-CO₂ (S-CO₂) Brayton cycle environment (550 °C, 20 MPa) is limited [2]. As candidate materials for S-CO₂ intermediate heat exchanger (IHX), corrosion study on tensile property for long-term integrity of austenitic alloys is in great demand.

Therefore, in this study, corrosion behavior on tensile property of austenitic alloys after exposure to high temperature S-CO₂ is presented. Microstructural changes are related to the changes in tensile property.

2. Experiment and Results

2.1 Test Materials

Test materials used in this study are commercialgrade alloys and can be divided into three major types based on the base metal: 1) Fe-base austenitic alloys (SS 310S, SS 316H, SS 316LN, SS 347H, and Alloy 800HT), 2) Ni-base austenitic alloys (Alloy 600, Alloy 625, and Alloy 690), and 3) FMS (G91) as a reference of ferritic metal to the other two group of austenitic alloys. The selection of the subject materials are carefully made based on the following aspects: 1) Anticipated structural materials for SFR (SS 316H, SS316LN, and G91), 2) HX materials in the PWR reactors (Alloy 600 and Alloy 690), and 3) structural materials in commercial high temperature fossil power plants (SS 310S, SS 347H, Alloy 800HT and Alloy 625).

2.2 S-CO₂ corrosion experiment and analysis

Both corrosion coupons and mini-sized tensile specimens of for each material are prepared and aged in $S-CO_2$ (200 bar) at 550, 600 and 650 °C for 1000 h. After the exposure, the weight change of both coupon specimens for each material is measured using a microbalance with a resolution of 0.001 mg. Then one of the samples is selected for the subsequent analyses using scanning electron microscope (SEM). Tensile property of mini-sized (16 mm length and 0.5 mm thickness) specimens after exposure is conducted at room temperature with the cross-head speed (strain rate)

of 3.33 x 10^{-4} /s following the procedures of ASTM E8/E8M-13a.

2.3 Corrosion Weight Gain

The weight change of materials is presented in Fig. 1. A general tendency of increase in weight change is observed as proportional to both time and temperature variables. As a reference grade, FMS G91 shows the worst corrosion resistance compared to the austenitic alloys. Among the austenitic alloys, Fe-base alloys (SS 310S, SS 316H, SS 316LN, SS 347H, and Alloy 800HT) show the poor corrosion resistance as compared to Ni-base alloys (Alloy 600, 625, and 690). Among Fe-base alloys, temperature dependency can be noticed especially for SS 316H and 316LN. A great increase in weight gain at 650 °C for 316H and after 600 °C for 316LN is observed.



Fig. 1. Results of weight gain after exposure in S-CO₂ at 550, 600 and 650 $^{\circ}$ C (200 bar) for 1000 h.

2.4 Changes in Tensile Property



Fig. 2. Changes in tensile property at room temperature after exposure in S-CO₂ at 550, 600 and 650 $^{\circ}$ C (200 bar) for 1000 h.

Changes in tensile properties, ultimate tensile strength (UTS) and elongation, are shown in Fig. 2. Unlike the weight change result in which a similar behavior is shown within the same group of materials, there is no general trend in tensile property depending on the base metal or phase of the materials. Instead, SS316 H, Alloy 625 and 800HT show a sudden reduction in ductility

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Fig. 3. Microstructure of (A) SS 316H and (B) Alloy 600 in S-CO2 at 550, 600 and 650 °C (200 bar) for 1000 h.

at 650 °C. Among the three materials, UTS for SS 316H is maintained while UTS for the other two seems to be gradually increased proportional to temperature.

2.5 Microstructure

A representative SEM image of microstructural changes for Fe-base (SS 316H) and Ni-base (alloy 800HT) is presented in Fig. 3. As the exposure temperature is increased from 550 to 650 $^{\circ}$ C, more precipitates can be observed for both SS 316H and alloy 800HT.

In case of SS 316H, a large string-type residual ferrite is found along with a few precipitates within/along the grain boundaries. This microstructural characteristic of SS 316H was consistently found in other study after exposure to S-CO₂ at 650 °C for 250 h [3] in which it caused the sudden decrease in elongation of SS 316H. In consideration of volume fraction of this residual ferrite to precipitates formed after exposure, it is highly suspected that the residual ferrite within SS 316H plays a dominant role in the reduction of ductility over newly formed precipitates after exposure to high temperature S-CO₂ environment.

Alloy 800HT, on the other hand, forms a large quantity of precipitates at the higher temperatures compared to 550 °C. At 600 °C, a formation of grain boundary precipitates seems to be dominant while large platelet-like precipitates are also found within the grain along with grain boundary precipitates at 650 °C. This formation of large quantity of precipitates at higher temperatures is also observed in alloy 625 in which it is suspected to cause the reduction of elongation for alloy 625 and 800HT.

3. Conclusions

The following conclusions can be drawn from this study of corrosion behavior on tensile property of austenitic alloys after exposure to high temperature S- CO_2 :

- 1. Both Fe-base and Ni-base austenitic alloys showed a good corrosion resistance at 550 °C, whereas at higher temperatures (over 600°C) the corrosion characteristics of the Fe-base alloys were severely worsened compared to the Ni-base.
- Changes in tensile property seemed to have no effects of base elements. Rather, SS 316H, Alloy 625 and 800HT —showed a reduced ductility at over 600 °C regardless of their base elements.
- 3. SS 316H showed grain boundary precipitates while a large quantity of precipitates were found within/along the grain boundary for Alloy 625 and 800HT after ageing at higher temperatures.

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