

Effects of Solution Depletion and Segregation Oxidation on Morphology of Modified 310 Austenitic Stainless Steel

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Abstract: The oxidation morphologies of modified 310 steel exposed in 900 and 1100 °C air were investigated. A double layer morphology consisting of a (Cr, Mn)-rich outer layer and a fine Cr-rich inner layer was formed at 900 °C. It was related to the breakaway oxidation induced by the Cr-depletion and the Mn-segregation in inner layer. Some Cr-rich oxides with amorphous state were formed along grain boundaries. And some new finer oxide grains, voids and Cr-rich precipitates were observed in spallation areas at 1100 °C. Correspondingly, the oxidation kinetic curve dropped with the spallation of scale and increased with the formation of some new oxide grains. It was caused by segregation of Cr and the transformation of oxides from Cr₂O₃ to the volatile oxides at elevated temperature. XRD analysis showed that the precipitates were mainly composed of CrO₃. Segregation and depletion for solutions were also discussed by oxidation diffusion mechanisms.

Key words: austenitic stainless steel; SEM; morphology; oxidation; segregation; element

Austenitic stainless steels have diverse applications due to their combination of good high-temperature creep strength and oxidation resistance^[1-3]. The reactive elements of Cr, Al and Si contribute significantly to enhancing the oxidation resistance of austenitic stainless steels. Although these steels have excellent general oxidation resistance, they are susceptible to “breakaway” oxidation in some stringent conditions. As one of the most common service failures, “breakaway” oxidation causes unacceptable loss for metallic materials. This localized accelerated oxidation often takes place in the specific aggressive environments, such as high temperature and nuclear radiation. The “breakaway” process is sporadic, random and stochastic. So it is difficult to predicate the time and the location of events^[4]. Although the mechanism of breakaway oxidation is unclear, there is no doubt that it is related to the microstructure of steels. The growth of oxides is based on diffusion

process, which relates to the characteristics of substrate (chemical composition, grain size, grain boundaries character distribution and solute segregation) in addition to the oxidizing environments (medium and temperature)^[3,5]. Several breakaway oxidations that link to grain boundaries, chemical failure^[4] or mechanical failure^[6] have been observed, while the specific process is still unclear.

The diffusion of minor alloy additions affects the establishment and growth of the protective oxide scales, sometimes even plays a significant role in the oxidation process^[7]. Wang^[8] and Paúl et al.^[7,9] studied the diffusion mechanisms of the reactive elements and the results showed that diffusion paths of beneficial elements might be changed because of the chromium depletion in substrate and the segregation on grain boundaries. Reactive elements are progressively consumed during oxidation proceeding, which induces the solution depletion in Cr, Mo, Nb, Zr

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and Si-contained stainless steel. In many cases, the solute precipitation is demonstrated by the changes of composition, structure of the multi-layer scales and the evaporation of some low-melting oxides^[10]. For example, evaporable oxides of Cr lead to Cr-depletion in matrix, which induces a transformation of oxides layer from Cr-rich oxides to the less protective Cr, Fe or Mn spinel.

In the past several decades, many works have been carried out and considerable progress has been achieved in understanding the mechanical failure of oxide scales^[4]. However, chemical failure of the protective oxide scale, which related to solute-depletion and segregation, was reported in an incomprehensive way^[4,8,10,11]. The aim of this work is to study the oxidation behavior of a modified 310 austenitic stainless steel by investigating the surface morphologies of oxide scales. The mechanism of slow breakaway oxidation at 900 °C illustrated by means of solution diffusion. The influences of solute precipitation and segregation on the spallation and formation of network oxidation formed at 1100 °C were also discussed.

1 Experimental Procedures

Substrate material was a modified 310 austenitic steel developed in the laboratory. The chemical compositions (mass%) were designed as: Cr 25, Ni 20, Si 0.65, C 0.1, Mo 2.0, Ti 0.2, Zr 0.2, W 0.15 and V 0.15.

The alloy was fabricated to a steel plate with a thickness of 6 mm via metallurgy processes of vacuum-melting, mold-casting, forging and rolling. Small

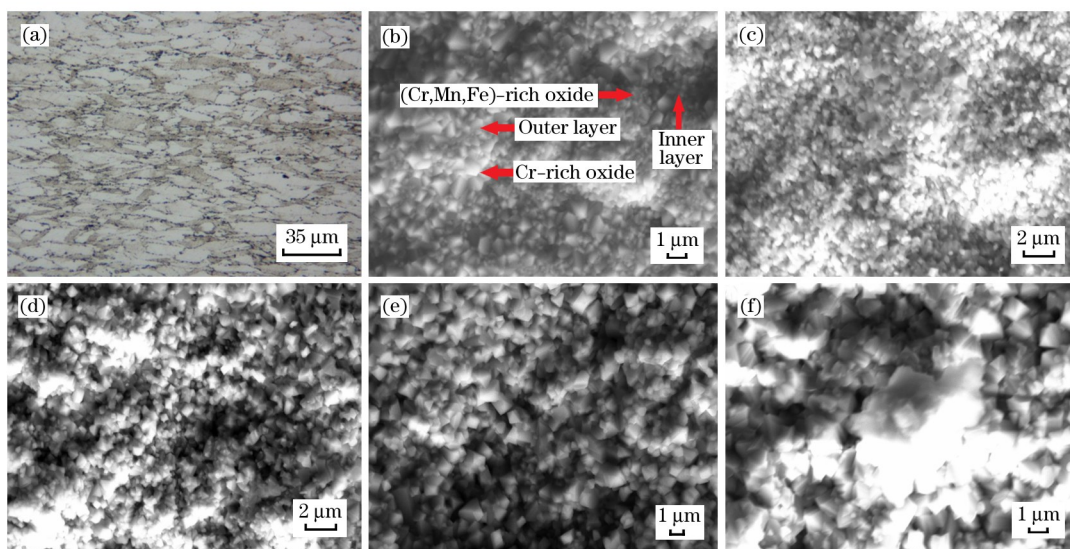
oxidized specimens were cut from the rolled plate with a size of 1.5 mm×15 mm×25 mm (thickness×width×length). Specimens were polished to 2000 grit. Then, they were thoroughly degreased with anhydrous ethanol by ultrasonic cleaning for 20 min. Subsequently, specimens were dried in a vacuum box at 175 °C for 4 h. After specimens were cooled to room temperature, their sizes and masses were measured. Oxidation test was carried out in a muffle furnace with free flowing air at 900 and 1100 °C for 5, 10, 25, 50 and 100 h, respectively. After finishing the oxidation process, samples were cooled to ambient temperature in a ventilated place. The mass changes of specimens were measured after the oxidation tests.

Substrate microstructures were investigated by optical microscope (OM, ZEISS Observer. A1m). Oxide morphologies were examined using a Leo 1530 VP scanning electron microscope (SEM). The coupled energy dispersive spectroscopy (EDS) detector was applied for chemical composition analysis. The oxide phases were analyzed by X-ray diffraction (XRD, D/MAX-RB).

2 Results and Discussion

2.1 Microstructural evolution at 900 °C

The typical microstructure of the substrate material is shown in Fig. 1(a). It consists of homogeneous equiaxed austenite grains and some fine grain boundary precipitates. The average grain size is about 10.4 μm which is measured by the method of interruption lines. In Fig. 1(b), the 5 h oxidized specimen exhibits two layers. The outer oxides are large,



(a) Substrate microstructure; (b) 5 h; (c) 10 h; (d) 25 h; (e) 50 h; (f) 100 h.

Fig. 1 Optical micrographs of the substrate microstructure and oxide morphology after different time at 900 °C

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