

# The corrosion and stress corrosion cracking behavior of a novel alumina-forming austenitic stainless steel in supercritical water



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## HIGHLIGHTS

- The general corrosion and SCC in SCW of the AFA steel have been limited reported.
- Fe-rich inner and Al-Cr-rich outer layers are formed in 650 °C/25 MPa/10 ppb SCW.
- The SCC behavior exhibits a combination of high strength and good ductility.
- Strength and elongation are lowered by increase of temperature and oxygen content.
- The AFA steel shows low SCC susceptibility and a superior corrosion resistance.

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## ABSTRACT

The general corrosion and stress corrosion behavior of Fe-27Ni-15Cr-5Al-2Mo-0.4Nb alumina-forming austenitic (AFA) steel were investigated in supercritical water under different conditions. A double layer oxide structure was formed: a Fe-rich outer layer (Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub>) and an Al-Cr-rich inner layer. And the inner layer has a low growth rate with exposing time, which is good for improvement of corrosion resistance. Additionally, some internal nodular Al-Cr-rich oxides were also observed, which resulted in a local absence of inner layer. Stress corrosion specimens exhibited a combination of high strength, good ductility and low susceptibility. The stress strength and elongation was reduced by increasing temperature and amount of dissolved oxygen. In addition, the corresponding susceptibility was increased with decreased temperatures and increased oxygen contents.

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## 1. Introduction

Compared with the other five concept reactors of the future Generation IV nuclear reactor systems, supercritical water (SCW) reactor has exceptional thermal efficiency, smaller volume and simplified design [1,2]. Accordingly, it is one of the most promising reactor in China. However, SCW environments are extremely corrosive to materials that will be applied on the core's internals and fuel cladding [3]. Ferritic/martensitic steels, austenitic steels, Ni

based alloys, and oxide dispersion strengthened (ODS) steels are proposed as candidates materials for future SCW reactor systems [4,5]. The primordial characteristic for any candidate material should be a high corrosion resistance in order to guarantee a safe operation of the reactor. Usually, a good corrosion resistance is achieved by forming a continuous Cr<sub>2</sub>O<sub>3</sub> protective scale on traditional alloys. However, such Cr<sub>2</sub>O<sub>3</sub> scale will be substantially compromised as exposed in environments containing water vapor, which causes losing their protectiveness and shortening material life [6,7]. Therefore, a next generation candidate material with higher strength and better corrosion resistance in the presence of water vapor is needed.

A new family of alumina-forming austenitic (AFA) stainless steels have been initially developed at Oak Ridge National

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**Table 1**  
Actual chemical compositions of the tested steels (wt.%).

Alloy	Alloying elements									
	Ni	Cr	Si	Mo	C	Nb	B	Al	Mn	Fe
AFA	25.38	14.55	0.35	2.15	0.007	0.41	0.01	4.25	0.15	Bal.
Modified 310	16.56	22.18	0.65	2.06	0.12	0.19Ti	0.21Zr	0.15V	0.13	Bal.

Laboratory (ORNL) [8–13], which have superior overall mechanical properties strengthened by stable nano-scale NbC, submicron B2-NiAl phase and Laves-Fe<sub>2</sub>Nb precipitates and improved corrosion resistances by the formation of a dense and continued Al<sub>2</sub>O<sub>3</sub> scale at high temperatures (600–900 °C).

In the past few years, AFA steels have become of great importance for the development of anticorrosive materials. Considerable attention has been focused on the oxidation behavior of the AFA steels in the presence of water vapor. Intensive research has shown that a protective Al<sub>2</sub>O<sub>3</sub> scale exhibits a good adhesion with matrix phase. An Al<sub>2</sub>O<sub>3</sub> scale has not only a low growth rate but also shows better thermo-dynamical stability than the Cr<sub>2</sub>O<sub>3</sub> scale. The corrosion behavior of AFA steels and the formation of Al<sub>2</sub>O<sub>3</sub> scale under different environmental conditions have been of particular interest. Studies related to chemical processing and energy production applications [6,14,15] have shown that AFA steels have good corrosion resistance in various simulated environments (aggressive carbon-/sulfur-species sulfidation-oxidation, metal dusting, steam, and air with 10% water vapor). For example, Nie et al. [16] reported that Fe-20Ni-14Cr-3Al-0.6Nb-0.1Ti (wt.%) AFA steel shows superior oxidation resistance in supercritical water (500 °C/25 MPa/25 wppb) than other austenitic alloys, such as 800H, D9 and 316 stainless steel. It is noted that a protective Al-Cr-Fe-rich oxide layer was formed on AFA steel under similar conditions. Recently, He et al. [17] studied the corrosion behavior of AFA steel in supercritical carbon dioxide under temperatures ranging from 450 to 650 °C and a pressure of 20 MPa. It is reported that protective and continuous Al<sub>2</sub>O<sub>3</sub> and (Cr, Mn)-rich oxide layers are formed at low temperatures and short times, but a breakaway oxidation occurs at high enough temperatures and long enough exposure times.

However, there are few investigations on the corrosion resistance and stress corrosion crack (SCC) susceptibility of AFA steels exposed in SCW. Thus, to ensure the safe application of structure material in aggressive environments similar to SCW, it is necessary to evaluate the corrosion and SCC resistance of AFA steels. Recently, a novel AFA steel Fe-27Ni-15Cr-5Al-2Mo-0.4Nb for advanced nuclear industries or power plants was developed and tested. This AFA steel was developed with low Nb and Si alloying elements and without artificial addition of C and Mn. This AFA steel composition has never been studied before. In this investigation, we examine the general corrosion resistance of the developed AFA steel in a 650 °C/25 MPa/10 ppb SCW environment. Furthermore, the SCC susceptibility of such AFA steel is investigated when exposed to SCW of 650 °C/25 MPa/10 ppb, 550 °C/25 MPa/10 ppb, and 550 °C/25 MPa/200 ppb environments.

## 2. Experimental methodology

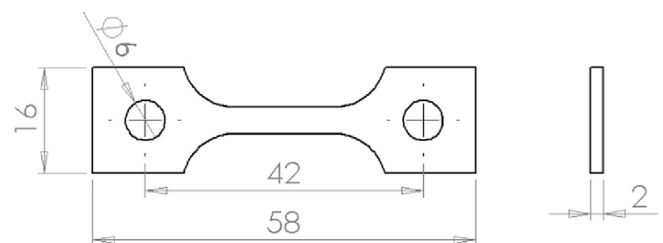
AFA and modified-310 steels were fabricated using commercially high purity metals as the starting materials. The chemical compositions of the AFA and modified-310 steels in wt.% are listed in Table 1. The modified-310 steel is popularly used because of its anticorrosive properties, and it is a good parameter to determine the capabilities of the novel AFA steel. Both steels were prepared by the next successive methodology and follow the same testing procedures: vacuum-induction melting, mold-casting, forging and

rolling at 1050–1180 °C to a thickness of 7 mm. Finally, the steel plates were homogenized at 1250 °C for 30 min, and then quenched into water. The received steels showed a single-phase of austenite with grain size of around 60 μm.

Both general corrosion and SCC specimens were machined from the as-solution plate. General corrosion specimens were square coupons with sizes of 20 mm × 10 mm × 2 mm. Several small plate tensile specimens were also machined for SCC tests. The schematic of the SCC sample is shown in Fig. 1, its specific size has been presented in Ref. [18]. All specimens were progressively ground to 2000 grit SiC paper, and then mechanically polished with 0.5 μm diamond pastes. Then specimens were ultrasonically cleaned in acetone and methanol before being exposed to SCW. Prior to corrosion testing, general corrosion specimens were measured by using a calipers (±0.01 mm) and weighed (±0.1 mg) to determine weight change per unit area after corrosion. Additionally, the modified-310 steel was also examined under the same conditions for comparison.

General corrosion tests were performed in SCW at 650 °C/25 MPa/10 ppb with a flow rate of 1.2–2.0 L/h. Exposure times were 100 h, 600 h, 1000 h, and 1500 h. Environmental parameters of SCW were as follows: the inlet conductivity was less than 0.06 μS/cm, and the dissolved oxygen (DO) concentration was about 10 ppb (weight fraction, controlled by passing over the argon). After the corrosion tests, the specimens were measured by a microbalance (Mettler Toledo, AL104) and its precision is 0.1 mg. Based on the metallographic preparing, the surface and the cross section morphology of the corrosive specimens were investigated using scanning electron microscope (SEM, LEO 1450). The composition of the oxide scales was analyzed by energy dispersion spectroscopy (EDS, Inca X-Max). Also phase structures were characterized by using an X-ray diffractometer (XRD, RIGAKU D/MAX-2500), carrying out at a scanning rate of 1°/min with a step size of 0.01°.

The slow strain rate tension (SSRT) was conducted in the SCW autoclave at a pressure of 25 ± 0.1 MPa and a strain rate of 9.26 × 10<sup>-7</sup> s<sup>-1</sup>. The electric conductivity was less than 0.06 μS/cm at the inlet of the autoclave. Tests were carried out at temperatures of 650 °C and 550 °C. Two tests were done at a temperature of 550 °C. The DO was controlled to be approximately 10 ppb in one of the tests and 200 ppb in the other test. Another test was performed at a temperature of 650 °C and a DO of 10 ppb. The DO content was controlled by bubbling pure argon into the autoclave. These temperature and conditions were selected because the AFA steel was designed for similar application environments. After the tests, strain-stress curves were generated, and the fracture surface



**Fig. 1.** The schematic of the SCC sample.

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