# Chloride-Induced Stress Corrosion Cracking of Oxide-Dispersion-Strengthened Austenitic Steels

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## ABSTRACT

The stress corrosion cracking (SCC) behavior of oxidedispersion-strengthened (ODS) 304 austenitic steels has been investigated in a chloride-rich aqueous environment at 143°C. ODS 304 alloys are found to be more resistant to SCC than the commercial AISI 304 steels. Under a constant tensile load of 177 MPa, the crack growth rate in ODS 304 steels is about one fourth of AISI 304 steels, and the time-to-failure of ODS 304 steels is 7.5 times of AISI 304 steels. Intergranular SCC dominates the fracture surface of AISI 304 steel, while in ODS 304 steel both intergranular and transgranular SCC occur. Electrochemical reactivity tests show ODS 304 steel is less sensitized than AISI 304, likely a result of a low carbon concentration and small grain size.

KEY WORDS: corrosion, fracture, ODS austenitic steels, stress corrosion cracking

#### INTRODUCTION

Oxide-dispersion-strengthened (ODS) austenitic steels, such as ODS 304 and 316, are promising candidate materials for advanced fossil power plants and Generation-IV nuclear energy systems (such as veryhigh-temperature reactor, VHTR). <sup>1-2</sup> They have a homogeneous dispersion of nanoscale oxide particles with a particle size of several to tens of nanometers, in a matrix of austenitic steel.<sup>3-4</sup> The oxide systems include: Y-Ti-O (Y<sub>2</sub>TiO<sub>5</sub> and Y<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub>), <sup>5</sup> Y-Al-O (YAlO<sub>3</sub>, Y<sub>2</sub>Al<sub>2</sub>O<sub>9</sub>, and Y<sub>5</sub>Al<sub>3</sub>O<sub>12</sub>), <sup>2,4</sup> Y-Hf-O (Y<sub>2</sub>Hf<sub>2</sub>O<sub>7</sub>), <sup>3</sup> Y-Ti-Hf-O (Y<sub>2</sub>(Ti,Hf)<sub>2-x</sub>O<sub>7-x</sub>), <sup>1</sup> and also Y-Zr-O (Y<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub>), <sup>6</sup> depending on the minor alloying element design. The dispersion of these nanoparticles can impede the motion of dislocations and reduce grain coarsening, improving the thermomechanical properties at elevated temperatures. <sup>4,7-8</sup> Thus, the mechanical strength and creep resistance of ODS austenitic steels are much higher than the conventional austenitic steels.

However, a concern is raised for the potential applications of ODS austenitic steels in its stress corrosion cracking (SCC) behavior in high-temperature water coolant environments in pipelines or heat exchangers, because its austenitic steel matrix is susceptible to SCC in these environments. SCC occurs as a brittle failure of a ductile material at relatively low applied or residual stress. Cracks initiate at a stress level that is much lower than those required to cause normal tensile failure, and propagate following a general macroscopic path that is approximately normal to the tensile component of stress. In practice, SCC of austenitic steels can occur in a water environment with a few ppm chloride at above 60°C.<sup>9-10</sup> Although the roles of temperature, chloride concentration, pH, alloy composition, and microstructures on the SCC susceptibility of austenitic steels have been extensively studied,<sup>11-13</sup> the SCC mechanisms are not well understood.<sup>14-15</sup> It is widely accepted that chloride-induced

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The Chemical Composition of ODS 304 and AISI 304 Steels Composition (wt%) Cr Ni Mn Ti Si Ρ С Y<sub>2</sub>O<sub>3</sub> Fe 0.06 Bal. ODS 304 steel 18.2 8.2 1.20 0.7 0.35 AISI 304 steel 8.5 0.06 0.03 0.04 18.3 1.38 Bal.

TABLE 1

SCC in austenitic stainless steels is initiated by localized corrosion such as pitting, but the mechanism of crack propagation is less understood.<sup>14-15</sup> The chloride-induced SCC initiates in active corrosion sites and occurs when the rate of crack growth is higher than the rate of localized corrosion.<sup>14</sup> The crack propagation in austenitic stainless steels involves a combination of electrochemistry and fracture mechanics, which may involve the mechanisms such as slip dissolution,<sup>16</sup> film-induced cleavage,<sup>14</sup> and hydrogen embrittlement (HE).<sup>17</sup>

The current research on SCC of ODS steels has been limited to ODS ferritic steels such as 19Cr-ODS steel and 19Cr-4.5Al-ODS steel, which show low SCC susceptibility.<sup>18</sup> This is not surprising as ferritic steels are more resistant to SCC than austenitic steels.<sup>9,19</sup> In addition, ODS ferritic steels are corrosion resistant in pressurized water environment because of the formation of a thin Cr/Al-rich oxide layer.<sup>20-22</sup> However, SCC is a concern for ODS austenitic steels because austenitic steels exhibit greater susceptibility for SCC than ferritic steels.<sup>23</sup> The published research data on SCC of ODS austenitic steels are rare. Li, et al., investigated the corrosion and SCC behaviors of ODS 304 austenitic steels in a supercritical water (SCW) environment at 600°C by slow strain rate tensile (SSRT) tests.<sup>24</sup> The corrosion kinetics of ODS 304 steels followed a parabolic law, as a result of the formation of a multilayer protective oxide scale. The fracture mode of ODS 304 steels was unchanged from the conventional 304 steels, with a mixed fracture mode of brittle cleavage and ductile dimple in the SCW environment.

In this study, the SCC behavior of ODS 304 austenitic stainless steels was examined using a constant load test in a 42% MgCl<sub>2</sub> aqueous solution at 143°C and compared with AISI 304 steels (UNS S30400<sup>(1)</sup>). The crack growth rate, fracture morphology, and sensitization of ODS 304 steel samples were investigated to reveal the potential SCC mechanisms in this chloride-rich aqueous environment.

#### EXPERIMENTAL PROCEDURES

ODS 304 austenitic steels were prepared from Fe, Cr, Ni, W, Ti, and Y<sub>2</sub>O<sub>3</sub> powders, with a nominal composition of Fe-18Cr-8Ni-2W-1Ti-0.35Y<sub>2</sub>O<sub>3</sub> in weight

percent. The mechanical alloying of the powder mixture was conducted by high-energy mechanical milling in argon atmosphere for 60 h. The as-milled powders were consolidated by hot isostatic pressing (HIP) for 3 h at 1,150°C under a pressure of 200 MPa. The asconsolidated ODS 304 steels were then annealed at 900°C for 2 h. More details of sample processing can be found in Xu, et al.<sup>5</sup> For comparison, AISI 304 austenitic steels were provided by Alfa Aesar. The chemical compositions of ODS 304 steels and AISI 304 steels are listed in Table 1.

Microstructures of ODS 304 and AISI 304 steels were characterized by electron backscattering diffraction (EBSD) and transmission electron microscopy (TEM). The samples for EBSD characterization were electrochemically polished using a solution of 5% HClO<sub>4</sub> and 95% C<sub>2</sub>H<sub>5</sub>OH. EBSD analysis was performed on a Helios 660<sup>†</sup> focused ion beam/scanning electron microscope (FIB/SEM) dual beam workstation that was equipped with an EBSD detector (Hikari XP  $2^{\dagger}$ , AMETEK) with a step size of 0.01 µm. The accelerating voltage used for EBSD was 30 kV. During the TEM sample preparation, 3 mm-diameter disks were thinned to a thickness of less than 100 mm by mechanical polishing. Final thinning to electron transparency was accomplished by electrochemical polishing in a twin jet polisher (TenuPol- $5^{\dagger}$ , Struers) using an electrolyte of 5% HClO<sub>4</sub> and 95% CH<sub>3</sub>OH at -20°C. TEM analysis was performed in a JEOL 2010  $\text{LaB}_6^\dagger$  operated at 200 kV using the bright-field (BF) imaging mode. Energy dispersive spectroscopy (EDS) was performed using a Super-X<sup>†</sup> windowless EDX detector in a FEI Tecnai Osiris<sup>†</sup> S/TEM.

The tensile specimens for SCC tests were machined using electron beam machining to the dimensions shown in Figure 1, according to ASTM standard G49-85. A notch of 2 mm in length and 0.5 mm in width was machined. The surface was processed by mechanical polishing using SiC paper down to 1200 grit, then electrochemical polishing in an electrolyte of 5% HClO<sub>4</sub> and 95% C<sub>2</sub>H<sub>5</sub>OH, and degreasing by ultrasonic cleaning in ethanol. A constant tensile stress of 177 MPa was applied to the specimens, which were immersed in a 42% MgCl<sub>2</sub> aqueous solution holding at a constant boiling temperature of 143±1°C. The concentrated MgCl<sub>2</sub> solution was used to accelerate the SCC of austenitic steels.<sup>25</sup> The solution was prepared by adding reagent grade MgCl<sub>2</sub> to distilled water into the container. When the solution began boiling, it was carefully adjusted to keep the boiling

<sup>&</sup>lt;sup>(1)</sup> UNS numbers are listed in Metals and Alloys in the Unified Numbering System, published by the Society of Automotive Engineers (SAE International) and cosponsored by ASTM International. <sup>†</sup> Trade name.



temperature at 143±1°C by adding distilled water. The ODS 304 steel and AISI 304 steel specimens were tested under the same conditions.

The SCC tests were interrupted about every 5 min after 30 min for the AISI 304 steel specimen and about every 30 min after 6 h for the ODS 304 steel specimen. During the interruptions, the specimens were taken out for examination in an optical microscope (BX51<sup>†</sup>, Olympus). The crack length (a) was measured a function of the test time (t), which was used to determine the crack growth rate ( $\frac{da}{dt}$ ). The crack growth rate was expressed as a function of the stress intensity factor (K) of crack tip. The Model I stress intensity factor, K<sub>I</sub>, of the tensile specimens can be calculated in the "edge crack in a plate under uniaxial stress" model by the following equation:<sup>26</sup>

$$K_{I} = \sigma \sqrt{\pi a} \left[ \frac{1 + 3\frac{a}{b}}{2\sqrt{\pi \frac{a}{b}(1 - \frac{a}{b})^{3/2}}} \right]$$
(1)

where  $\sigma$  is the applied stress (177 MPa) and b is the width of the gauge (6 mm). After the specimen failed, the fracture surface was examined using the secondary electron mode in the FIB/SEM.

The electrochemical potentiodynamic reactivation (EPR) tests were performed following the ASTM standard G108-94 on an electrochemical workstation (CHI6062E<sup> $\dagger$ </sup>, CH Instruments). The samples of ODS 304 steel or AISI 304 steel was set as the working electrode, a platinum foil as the auxiliary electrode, and the saturated calomel electrode (SCE) as the reference electrode. The electrodes of ODS 304 steels and AISI 304 steels were machined to a crosssection area of 1 cm<sup>2</sup>. The surface was mechanically polished using SiC paper down to 1200 grit, and then degreased by ultrasonic cleaning in ethanol. The electrolyte was 0.5 mol/L sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and 0.01 mol/L potassium thiocyanate (KSCN) at  $30\pm1^{\circ}$ C. The three electrodes were immersed into the electrolyte for 5 min at the open-circuit potential (OCP) which is approximately  $-450 \text{ mV}_{SCE}$ . Passivation was accomplished by applying a potential of 200 mV<sub>SCE</sub> and held for 2 min. The reactivation scan started at 200 mV in the active direction at a rate of 1.67 mV/s(100 mV/min) until 50 mV above the OCP. The current was recorded as a function of the potential versus SCE.

### RESULTS AND DISCUSSION

Microstructures of AISI 304 and ODS 304 steels were characterized by EBSD and TEM. Figures 2(a) and (b) show the inverse pole figures of AISI 304 and ODS 304 steels, respectively, suggesting the austenitic grains in both alloys have no preferred crystallographic orientation. The average grain size of ODS 304 steel (7 µm) is much smaller than that of AISI 304 steel (30  $\mu$ m), according to the statistic measurements in EBSD images. Another difference is that ODS 304 steels have Y-Ti-O dispersion particles inside the grains of austenitic matrix (Figure 2[c]). The size of dispersion particles is 10 nm to 80 nm according to the statistic measurements of the TEM images. EDS chemical analysis indicates the particles are Y- and Ti-rich, suggesting they are Y-Ti-O (presumably Y<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub> and Y<sub>2</sub>TiO<sub>5</sub>).<sup>2</sup> The Y-Ti-O oxide particles were evolved from  $Y_2O_3$  particles during the mechanical alloying procedure.<sup>27-29</sup> Recent experimental investigations reveal that during the ball milling procedure, Y<sub>2</sub>O<sub>3</sub> dissolve and form amorphous oxygen-rich regions as a result of the diffusion of dissolved  $Y_2O_3$  and Ti.<sup>14</sup> These amorphous oxygen-rich regions were crystallized to form Y-T-O particles during the heating process in the HIP procedure.<sup>10,30</sup>

The crack lengths in ODS 304 and AISI 304 specimens under a constant tensile stress of 177 MPa in 42% MgCl<sub>2</sub> aqueous solution at 143±1°C were measured as a function of time by optical microscopy (Figure 3[a]). The crack growth in ODS 304 steel is much slower than in AISI 304 steel. The time-to-failure of ODS 304 steel is 7.5 times of that of AISI 304 steel (Table 2). Figure 3(b) shows the effect of stress intensity factor (K<sub>I</sub>) at the crack tip on the crack growth rate  $\left(\frac{da}{dt}\right)$  of SCC, which shows three typical regions. At the same  $K_I$ , the crack growth rate in ODS 304 steel is much lower than in AISI 304 steel. At low levels of K<sub>I</sub> (within Region I), the crack growth rate is highly sensitive to K<sub>I</sub>. It is well established that the corrosive environment can cause a subcritical crack growth above a threshold stress intensity factor  $(K_{ISCC})$ .<sup>28,31</sup> In Region II, the crack growth rate is independent of K<sub>I</sub>. In this region, the crack growth rate of ODS 304 steel is only about one fourth of the AISI 304 steel (Table 2). At high levels of K<sub>I</sub> (within Region III), cracks grow at critical velocity above the fracture toughness. The Region III of the ODS 304 steel was not measured from the data points, presumably because it was too short. All of these



FIGURE 2. Microstructures of AISI 304 and ODS 304 steels. (a) EBSD inverse pole figure of AISI 304 steels. (b) EBSD inverse pole figure of ODS 304 steels. Inset: color key for crystallographic orientation. (c) BF-TEM image of the dispersion particles in ODS 304 steels.



**FIGURE 3.** The comparison of crack growth behavior during SCC of ODS 304 and AISI 304 steels: (a) crack length (a) versus time (t), and (b) crack growth rate (da/dt) versus stress intensity factor ( $K_1$ ).

| TABLE 2                                       |  |  |
|---|--|--|
| Comparison of SCC Test Results of ODS 304 and |  |  |
| AISI 304 Steels                               |  |  |

| Material                        | Time to<br>Failure (h) | Crack Growth Rate in<br>Region II (μm/h) |
|---------------------------------|------------------------|--|
| ODS 304 steel<br>AISI 304 steel | 10<br>1.33             | 501<br>2,138                             |

experimental results demonstrate that the ODS 304 steel is more resistant to SCC than AISI 304 steel, by lengthening the time-to-failure and decreasing the crack growth rate.

The SEM images of the fracture surfaces of AISI 304 steel and ODS 304 steel after the completion of SCC tests are shown in Figure 4. In the AISI 304 steel, the

fracture surface is dominated by intergranular cracks (Figure 4[a]). In addition to the primary crack that causes the fracture, secondary intergranular cracks can be observed (indicated by white arrows). In the ODS 304 steel, the fracture surface shows characteristics of both intergranular and transgranular cracks (Figure 4[b]). The statistic characterization shows that about 70% of the cracks are intergranular and 30% are transgranular. The transgranular cracks can be indicated by the river marks on the fracture surface (Figure 4[c]).<sup>32</sup> The oxide particles are difficult to detect on the fracture surface. However, the mechanical properties of the dispersion particles (e.g., for  $Y_2Ti_2O_7$ , hardness  $H_v = 12.1$  GPa, Young's modulus E =262 GPa, shear modulus G = 101 GPa<sup>19</sup> exceed those of the 304 austenitic steel matrix ( $H_v = 1.7$  GPa to 2.1 GPa, E = 190 GPa to 203 GPa, G = 74 GPa to 81 GPa),



FIGURE 4. (a) Fracture surface of AISI 304 steel that shows a primary intergranular crack with secondary cracks along grain boundaries. (b) Fracture surface of ODS 304 steel which shows a mixed fracture mode with both intergranular and transgranular cracks. (c) River marks on the fracture surface of ODS 304 steel which indicate the characteristic of transgranular crack.

so the dispersion particles are less likely to fracture than the steel matrix.

The intergranular stress corrosion cracking (IGSCC) of austenitic stainless steels has been generally attributed to the chromium depletion because of sensitization, <sup>33-34</sup> or impurity element segregation (e.g., Si)<sup>35</sup> at the grain boundaries. The transgranular stress corrosion cracking (TGSCC) of austenitic stainless steels can be explained by the film-induced cleavage, <sup>36-37</sup> or HE mechanism. <sup>17,38</sup> In this test, the



sample was immersed in a saturated  $MgCl_2$  solution at 143°C, which provides a slightly acidic environment by the hydrolysis of  $MgCl_2$  following the electrochemical reaction:

$$Mg^{2+} + 2H_2O = Mg(OH)_2 + 2H^+$$
 (2)

The cathodic reduction of protons to hydrogen ions on the metal surface will then promote the uptake of hydrogen atoms into the crack tip.<sup>17</sup> It has been found that hydrogen could diffuse and enrich in the crack tips during the rupture of the surface film.<sup>33,37,39-40</sup> It is suggested that hydrogen can assist the crack propagation during SCC by either the anodic dissolution at the crack tip sustained by the cathodic reduction of hydrogen ions,<sup>41-42</sup> or the martensite phase transformation in unstable austenitic steels resulting from hydrogen absorption.<sup>43-44</sup>

The EPR test results of ODS 304 and AISI 304 steels are presented in Figure 5. The maximum anodic current densities (Ir) for AISI 304 steel and ODS 304 steel are 0.67 A and 0.14 A, respectively, indicating AISI 304 steel is highly sensitized, while the ODS 304 stainless steel is not. It is well established that in austenitic steels such as AISI 304. the sensitization involves (Fe,Cr)<sub>23</sub>C<sub>6</sub> precipitation and Cr-depleted regions in the grain boundaries, which can lead to intergranular corrosion and IGSCC. The low sensitization of ODS 304 steel compared with AISI 304 stainless steel can be attributed to two reasons. First, there is a lower carbon content in the ODS 304 steel because of the highly pure powders of metal elements used for mechanical alloying. Second, the smaller grain size of ODS 304 steel (7 µm) compared to AISI 304 steel (30 µm) can provide much larger grain boundary areas, thus reducing the sensitization effect.

Based on the EPR test results, the AISI 304 steel is highly sensitized, which is the probable cause of IGSCC of AISI 304 steel sample. The higher degree of sensitization means that the precipitation of Cr-rich carbides and the associated Cr depletion in the adjacent region of grain boundaries are more significant in AISI 304 steel, because the EPR results are dependent on the width and length of the Cr-depleted zone.<sup>45</sup> This will cause the preferential dissolution of the Cr-depleted zone in grain boundaries, serving as the crack nucleation sites for IGSCC.<sup>46-47</sup> In contrast, the ODS 304 steel is less sensitized and shows both IGSCC and TGSCC, and its TGSCC feature may be caused by the film-induced cleavage or HE mechanisms, as discussed before. The reduced sensitization decreases the susceptibility of ODS 304 steels to IGSCC. Solomon suggested that the fracture mode in SCC of austenitic steels depends on the degree of coverage of grain boundaries by Cr-depleted zones, and with the increased coverage the fracture mode transitions from TGSCC, to a mixed mode of TGSCC and IGSCC, and then IGSCC.<sup>48</sup> The observations of ODS 304 and AISI 304 are consistent with this trend.

## CONCLUSIONS

♦ The SCC behavior of ODS 304 austenitic steels has been investigated in 42% MgCl<sub>2</sub> aqueous environment at 143°C under a constant load of 177 MPa. ODS 304 alloys are found to be more resistant to SCC than the commercial AISI 304 steels. The measured crack growth rate in ODS 304 steels is about one fourth of AISI 304 steels, and the time-to-failure of ODS 304 steels is 7.5 times of AISI 304 steels, suggesting a much better SCC resistance of the ODS 304 steel. The fracture surfaces show that IGSCC dominates AISI 304 steel, while IGSCC and TGSCC coexist in ODS 304 steel. Electrochemical reactivation tests suggest ODS 304 steel is less sensitized than AISI 304 steel likely because of a lower carbon content and smaller grain size, which may explain their difference in the fracture surface.

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