



# Stress corrosion cracking susceptibility of oxide dispersion strengthened ferritic steel in supercritical pressurized water dissolved with different hydrogen and oxygen contents



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## ABSTRACT

Stress corrosion cracking (SCC) susceptibility was investigated by means of steady strain rate tests for a 15Cr–4Al–2W oxide dispersion strengthened (ODS) ferritic steel in supercritical pressurized water (SCPW) dissolved with different hydrogen (DH) and dissolved oxygen (DO) contents. All the specimens exhibit ductile fracture mode, regardless of the strain rate. The effect of DH and DO on the fracture behavior is negligible. Small cracks were observed at necking region but most of the cracks were identified as “corrosion layer cracking” by cross-sectional observation. The ODS ferritic steel shows no susceptibility to SCC in SCPW at this experimental conditions.

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## 1. Introduction

Supercritical pressurized water cooled reactor system has been considered as one of the candidates of Generation IV fission advanced nuclear systems as well as fusion systems because of its high thermal efficiency and simple reactor design [1]. In the supercritical water reactor (SCWR), the operation temperature is expected to be in the range 500–550 °C, and fuel claddings and the other core components are subjected to a potentially corrosive environment [2]. It is well known that the SCPW above critical point (374 °C, 22.1 MPa) is very severe environment for metallic materials being highly corrosive and it may cause stress corrosion cracking. For the application to the advanced nuclear systems, high performance is required for structural material in terms of its radiation resistance, weldability, creep strength, corrosion and stress corrosion cracking susceptibility, etc. [3]. To meet these requirements, oxide dispersion strengthened ferritic steels have been developed as a high performance structural material for nuclear applications. Recent study showed that the corrosion resistance of the ODS ferritic steels in SCPW significantly increased with increasing Cr concentration up to 16 wt.%. And the addition of Al to about 4 wt.% made the ODS steels show excellent corrosion resistance to Pb–Bi eutectic coolant and SCPW [4].

There are several works on the SCC susceptibility of ODS steels, ferritic–martensitic (F/M) and austenitic steels at room

and higher temperatures [5–9]. The existing data of austenitic and ferritic–martensitic steels at a temperature range from 380 °C to 550 °C in SCPW clearly showed a general decrease in the ultimate tensile stress with temperature. Was et al. [10] reported that the crack growth rate and maximum crack depth of austenitic stainless steels increased with increasing temperatures. In contrast, the observed dependence of strain to failure on temperature does not show any clear relationships with SCC susceptibility [1,6,10–14].

The selection of the strain rate is very important because the susceptibility to cracking may not be evident from the result of tests at too low or too high strain rate. In the previous works on SCC in a number of material–environment systems, strain rates in the range  $10^{-5}$ – $10^{-6}$  s<sup>−1</sup> are generally used and many of slow strain rate test (SSRT) in SCPW were conducted at strain rates faster than  $10^{-7}$  s<sup>−1</sup>. It was reported that low alloy steels become sensitive to the strain-induced corrosion cracking (SICC) at a strain rate of  $1 \times 10^{-6}$  s<sup>−1</sup> in light water reactor system [15]. Nishimura and Maeda proposed that SCC-dominated strain rate region is from  $10^{-4}$  s<sup>−1</sup> to  $10^{-6}$  s<sup>−1</sup> for types 304 and 316 austenitic stainless steels in acidic chloride solutions at 80 °C [16]. Serebrinsky and Galvele [17] investigated the effect of strain rate on the crack propagation rate (CPR), which was calculated by dividing the length of the brittle zone measured under the scanning electron microscope (SEM) by the fracture time, for FCC alloys in LiCl solution at 130 °C, and showed that an increase in the strain rate enhanced the CPR. Therefore, more various range of strain rate test in SCPW is required for careful consideration of SCC susceptibility.

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The dependence of SCC on DO content for ferritic–martensitic steel in SCPW [18] as well as 304 austenitic steel and alloy 690 in high temperature water [19,20] were investigated, indicating that the thickness of the oxide layer grown on the specimen surface depended on DO content of the water.

Similarly, several experiments have been conducted on ODS alloys in sub-critical water [21]. Previous works demonstrated that ODS steels showed no susceptibility to SCC in a simulated boiling water reactor condition dissolved with oxygen, but did show in a SCPW. Corrosion behavior and SCC susceptibility in SCPW have been evaluated for austenitic and ferritic–martensitic steels [1,6,7,10–13,18,21], but few research groups studied on corrosion behavior of ODS steels in a SCPW [4,7,9,21–25]. In general, nickel-base alloys and austenitic stainless steels exhibited higher susceptibility to intergranular stress corrosion cracking (IGSCC) than ferritic–martensitic alloys. Meanwhile, ferritic–martensitic alloys suffer from severe oxidation. On the other hand, outstanding corrosion resistance of ODS steels to SCPW has been presented by several works on corrosion behavior of ODS steels in a SCPW. Therefore, one of the solutions to prevent SCC is the use of ODS ferritic steels, which has been considered to be resistant to SCC. However, the experimental data on the SCC susceptibility of ODS steels in SCPW are so limited.

In this study, the effect of strain rate on the deformation and fracture behavior was investigated to evaluate the susceptibility of ODS steel to SCC in SCPW dissolved with different hydrogen and oxygen contents.

## 2. Experimental

### 2.1. Specimen preparation

The material used in this study is a high Cr (15Cr) ODS ferritic steel (SOC-16) of which the chemical compositions are shown in Table 1. SOC-16 was manufactured by mechanical alloying method using a high speed attritor in KOBELCO. The mixed powders were encapsulated, hot extruded at 1150 °C and subsequently annealed at 1150 °C for 60 min followed by air cooling. The details of the fabrication process of the ODS steel are given in the previous papers [26,27]. Tensile specimens were sampled from the extruded bar with the loading direction parallel to the extruded direction. Round bar tensile specimens which measure a gauge section of 10 mm with 2.1 mm of diameter were used for SSRT. The specimen geometry is shown in Fig. 1. The surface of the round bar specimens was mechanically polished by SiC abrasive paper to 4000 grit.

### 2.2. SSRT conditions

In order to evaluate the susceptibility to SCC in SCPW, SSRT was carried out at strain rates of  $1 \times 10^{-3} \text{ s}^{-1}$  and  $1 \times 10^{-6} \text{ s}^{-1}$  or  $5 \times 10^{-7} \text{ s}^{-1}$  in SCPW at 500 °C under a pressure of 25 MPa with different contents of dissolved oxygen and dissolved hydrogen. The DO and DH contents of the water were DH = 0.4 ppm, DO and DH < 0.01 ppm and DO = 8 ppm (mg/L). The water chemistry was controlled by injections of high purity nitrogen and hydrogen gas to a conditioning tank and the conductivity was lower than 0.10  $\mu\text{S}/\text{cm}$  during experiment. In addition, tensile tests in a vacuum of  $2.0 \times 10^{-3} \text{ Pa}$  were also performed at 500 °C to compare

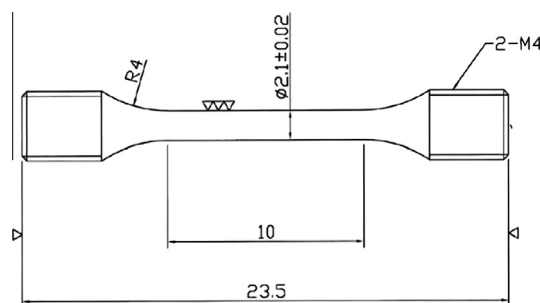


Fig. 1. The geometry of tensile specimens for SSRT in SCPW.

with the deformation and fracture behavior in SCPW. After the SSRT, fractured surface and side surface of the gauge section were observed by SEM to investigate the fracture mode in detail. Electron probe microanalyzer (EPMA) analysis was also conducted for the cross-sectional areas of the tensile specimens to understand the roll of corrosion products. The cracking pass way was examined by the cross-sectional observation to measure the crack depth and to count the number of cracks.

## 3. Results and discussion

### 3.1. Stress–strain behavior

The SSRT stress–strain curves of SOC-16 deformed in a vacuum and SCPW dissolved with different DO and DH are shown in Fig. 2(a) for the test at a higher strain rate ( $1 \times 10^{-3} \text{ s}^{-1}$ ) and Fig. 2(b) for the test at a lower strain rate ( $1 \times 10^{-6} \text{ s}^{-1}$  or  $5 \times 10^{-7} \text{ s}^{-1}$ ). Table 2 summarized the numerical values of the tensile properties obtained by the SSRT. Both the figures indicate that there is no significant effect of test environment on the stress–strain behavior of the ODS steel irrespective of the strain rate, although the yield stress and ultimate tensile stress are decreased with decreasing strain rate, while the total elongation tends to increase with decreasing strain rate. This behavior is a typical strain rate effect for metallic materials because a decreased strain rate gives more time for dislocations to move past obstacles as a thermally activation process. The obtained tensile test results suggest that there is no susceptibility to SCC of the ODS steel in the SCPW.

Generally, the susceptibility to SCC can be evaluated by measuring the reduction of total elongation and the change in the fracture mode from ductile to brittle fracture, IGSCC and transgranular stress corrosion cracking (TGSCC). In our previous work on the SCC of a martensitic steel in SCPW, a remarkable fracture mode change from ductile to brittle mode was accompanied by lowering strain rate, while the total elongation of the steel was not affected by the strain rate change [1]. Therefore, the susceptibility to SCC can be more clearly evaluated by the observation of fracture mode change and the measurement of reduction in area (RA). Fig. 3 shows the RA of the tensile specimens after SSRT at different strain rates, indicating that the RA is almost independent of the test environment and strain rate. This also suggests that the ODS steel is not susceptible to SCC in SCPW at this experimental condition.

Table 1  
Chemical compositions of ODS ferritic steel (SOC-16) wt.%.

Material	C	Cr	W	Al	Ti	Y	O	N	Ar	Hf	Y <sub>2</sub> O <sub>3</sub>
SOC-16	0.043	14.54	1.93	3.01	0.13	0.28	0.17	0.006	0.0064	0.62	0.36

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