Journal of Nuclear Materials 492 (2017) 32–40

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Characterizing the effect of creep on stress corrosion cracking of cold worked Alloy 690 in supercritical water environment

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ARTICLE INFO

Article history:
Received 25 March 2017
Received in revised form
15 May 2017
Accepted 15 May 2017
Available online 17 May 2017

Keywords:
Alloy 690
Supercritical water
Stress corrosion cracking
Creep
Crack growth rates

ABSTRACT

The effect of creep on stress corrosion cracking (SCC) was studied by measuring crack growth rates (*CGRs*) of 30% cold worked (CW) Alloy 690 in supercritical water (SCW) and inert gas environments at temperatures ranging from 450 °C to 550 °C. The SCC crack growth rate under SCW environments can be regarded as the cracking induced by the combined effect of corrosion and creep, while the *CGR* in inert gas environment can be taken as the portion of creep induced cracking. Results showed that the CW Alloy 690 sustained high susceptibility to intergranular (IG) cracking, and creep played a dominant role in the SCC crack growth behavior, contributing more than 80% of the total crack growth rate at each testing temperature. The temperature dependence of creep induced *CGRs* follows an Arrhenius dependency, with an apparent activation energy (Q_E) of about 225 kJ/mol.

1. Introduction

Due to its system simplification, reduced plant layout and high thermal efficiency, the supercritical water cooled reactor (SCWR) is recognized as an advanced reactor concept by the Generation IV international forum since 2002 [1]. However, it is still difficult to build a demonstration SCWR, mainly because of the material problems caused by the high temperature, irradiation and the corrosive environment [2].

The operating temperature of primary component materials of a typical reactor vessel type SCWR has been increased to over 510 °C, and the water pressure to 25 MPa [3]. High temperature strength and corrosion resistance in the SCW environment are the basic requirements of a candidate material for SCWR primary components. Owing to its excellent SCC performances, Alloy 690 has been selected as one of the major nuclear grade materials for pressurized water reactors (PWR), such as pressure boundary components, reactor vessel head penetrations and steam generator heat exchange tubes, although a steam generator is no longer required in some SCWR designs.

So far, the existing SCC data for candidate materials in SCW environment are mostly obtained from constant extension rate tensile (CERT) tests or slow strain rate tests (SSRT) [4]. SCC susceptibility is usually measured by the reduction in ductility in the test environment [5], the percentage of intergranular (IG) cracking on the fracture surfaces [6,7], or by the crack depth and density on the gage surface [4,8]. These methods are mostly for quick qualitative comparison of the SCC susceptibility between materials or testing conditions, and often result in under-estimating or over-estimating the SCC behavior under plant operating conditions, and thus is not considered a reliable indication of SCC susceptibility except perhaps in extremely aggressive environments. SCC behavior can be quantitatively characterized by CGR experiments at a constant stress intensity factor (K), and Peng et al. obtained some SCC CGR data for 20% CW 316L stainless steel in SCW [9]. SCC *CGRs* data are of great importance both for the understanding of the SCC behavior and for engineering design of a SCWR, and are far more relevant and trustworthy than SSRT data. The SCC growth of Alloy 690 in SCWR environments remains unclear.

Arioka [10] studied the effect of creep on cracking of cold worked Alloy 690 in high temperature environments in the range of 360 °C–460 °C, and found that the intergranular creep cracking

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http://dx.doi.org/10.1016/j.jnucmat.2017.05.018
0022-3115/

(IG creep cracking) in inert and air environments has a similar morphology and temperature dependence as IGSCC in high temperature subcritical water. This suggests that creep is important in the growth of IGSCC for CW Alloy 690, consistent with the prevailing view that dynamic strain is fundamental to SCC in SCW.

SCWRs operate at temperatures higher than the temperatures studied by Arioka et al., so creep will play an even larger role in the cracking of Alloy 690. Cold work is inevitable during the construction of a large system such as a SCWR. For example, welding will introduce residual shrinkage strains, peaking near the fusion line and approximately equivalent to cold work levels up to 20% plastic deformation [11]. In this work, the SCC and creep induced crack growth rate of 30% cold worked Alloy 690 were measured in SCW environment by the reversing direct current potential drop (DCPD) method. The objective of this study was to evaluate the temperature dependence, as well as the creep effect on the cracking of cold worked Alloy 690 in SCW.

2. Experiments

2.1. Testing material

The test material is Alloy 690 bar with final mill anneal at 996 °C for 20 min followed by air cooling. The chemical composition is listed in Table 1.

The material was compressed by one step hydraulic forging up to 30% thickness reduction at room temperature. High-resolution scanning electron microscopy (SEM) backscatter electron (BSE) imaging under low kV conditions proved to be the most effective method for identifying fine intergranular cavities and grain boundary carbides [12,13]. Fig. 1 shows the micrographs of grain boundaries. It can be observed that primary carbides precipitate at grain boundaries semi-continuously along with small isolated TiN particles. These primary carbides are formed during original melting/solidification, usually MC, as well as TiN, and will precipitate along the grain boundaries, even during normal cooling from the annealing temperature, but more so during the 705 C/12 h typical thermal treatment. No micro-cracks or voids can be observed at the interfaces between matrix and carbides although the material was cold worked to 30% reduction in thickness. Fig. 2 shows the electron backscatter diffraction (EBSD) images of as-received and 30% CW Alloy 690. The EBSD inverse pole figure maps reveal that grains are highly compressed in the CW material as compared with the as-received. Moreover, high misorientation is observed near grain boundaries of CW material, indicating high residual strain along the grain boundaries, which may accelerate IGSCC growth rate.

Compact tension (CT) specimens with 12.7 mm thickness and 5% thickness side grooves were prepared in the S-L orientation to obtain the highest SCC CGR under testing corrosive environments [11]. The dimensions of the CT specimen are shown in Fig. 3. Vickers Hardness (HV) of as-received and CW specimens in three different faces were measured. The 1000 g-force was applied on the sample surface and held for 10 s before removal. A HV can then be calculated in units of kilogram-force per square millimeter (kg/mm^2). Each value was averaged by at least three individual measurements on the same surface, and the results are given in Table 2. Hardness increases in all three orientations after cold deformation, especially in the front and back faces (as shown in Fig. 3). Yield strength (YS) increased remarkably after the 30% cold working, which could significantly affect its SCC behavior in SCW environment.

Table 1
Chemical composition (*wt.* %) of as-received Alloy 690.

Elements	Cr	Ni	Mo	P	Ti	C	Fe	Mn	Si
wt %	29.30	60.42	0.01	0.006	0.37	0.034	9.21	0.22	0.06

2.2. Experimental procedure

The crack growth rate measurements were performed with one specimen using “on the fly” changes to study the effects of environment to obtain consistent data. The SCC tests were conducted at constant stress intensity factor (K) in SCW environment at temperatures between 400 and 550 °C and at a pressure of 25 MPa. Creep crack growth rate tests were carried out in high purity argon at the same temperature and at the same K as SCC tests. The crack length was measured by the reversing DCPD system [14]. The feed water to the autoclave was pumped by a double diaphragm high pressure metering pump, and the autoclave pressure was maintained by a precision back pressure regulator, which maintains pressure at ±0.1 MPa fluctuation at 25 MPa. The water chemistry was controlled by the recirculating a water loop connected to the autoclave with a flow rate of about 1 L/h, corresponding to an autoclave refresh rate of 1 vol/h. The feed water to the autoclave was purified by nuclear grade polishing mixed bed resin (Rohm and Hass DS160). High purity argon was continuously bubbled into the water tank to maintain a deaerated water environment. The dissolved oxygen (DO) in the feed water to the autoclave was monitored by using an inline DO sensor, with the value well below 10 ppb. Water purity was continuously monitored by two conductivity meters installed at the inlet and outlet of autoclave. The inlet conductivity was controlled below 0.06 μS/cm and the outlet water conductivity was about 0.2 μS/cm. A pressure balanced seal eliminated the force on the pull rod that would have been induced by the pressure differential between inside and outside of the autoclave.

Before SCC testing in SCW, the CT specimen was fatigue precracked by 1 mm in room temperature air at $K_{max} = 25$ MPa$\sqrt{m}$, load ratio $R = 0.3$ and frequency $f = 1$ Hz. Then the pre-cracked specimen was loaded in autoclave for the subsequent transitioning stages in SCW to change the cyclic plastic zone and TG morphology at fatigue crack tip to the monotonic plastic zone and IG morphology of an SCC crack tip. Transitioning was performed in SCW at 400 °C and 25 MPa, with R increased from 0.3 to 0.7, and f decreased from 1 Hz to 0.001 Hz, until cracking linear response (which usually was close to the SCC crack growth rate). Then a change was made to constant K conditions to evaluate the SCC crack growth response. The transitioning stages are among the most important elements of a successful SCC crack growth rate test. A hold time of 3000–9000 s at the maximum fatigue load (K_{max}) is ideally introduced if the transitioning behavior looks abnormal. The corrosion crack was propagated a minimum length of one grain size (~50 μm) before finishing the transitioning step.

To evaluate the contribution of creep to SCC growth rate quantitatively, the creep test was conducted on the same specimen after the SCC crack growth rate was measured in SCW. Before the creep induced crack growth rate measurement, the load on the specimen was released, the high pressure pump was stopped, and the autoclave was depressurization then evacuated using high purity argon for a minimum of 10 h to establish a non-corrosive inert environment in the autoclave for the following creep test. Then the specimen was reloaded up to $K = 25$ MPa$\sqrt{m}$ to measure the creep induce crack growth rate at each given temperature.

3. SCC and creep *CGRs*

After testing, each specimen was sliced into two pieces along the center plane of the thickness. One piece was fatigued apart at room