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The effects of grain boundary carbide density and strain rate on the stress corrosion cracking behavior of cold rolled Alloy 690

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ABSTRACT

The effects of grain boundary carbide density and strain rate on stress corrosion cracking (SCC) initiation susceptibility of cold rolled 690 were evaluated in 360 °C hydrogenated water using slow strain rate tensile (SSRT) tests. The improvement in SCC resistance of a thermally treated microstructure over that of a solution annealed microstructure indicates that grain boundary carbides have a mitigating effect in cold rolled Alloy 690. The effect of grain boundary carbides is dependent on the cold rolling orientation of sample. Slower strain rate aggravates SCC initiation by enhancing the environmental component of SCC.

1. Introduction

Alloy 690 is widely used in nuclear power plants as a replacement for Alloy 600 due to its superior resistance to stress corrosion cracking (SCC) [1–3]. However, it is not totally immune to SCC as there are specific conditions that increase the susceptibility to cracking. For example, it has been reported that cold rolling boosted the crack growth rate of Alloy 690 significantly [4,5].

Much research has been conducted to investigate the possible effects of thermal mechanical factors on the SCC susceptibility of Alloy 690. It is recognized that semi-continuous grain boundary carbides induced by thermal treatment can enhance the SCC resistance of Alloy 600 in caustic environment [6] and reducing environment [7–11] although the intergranular carbides reportedly increased the susceptibility to SCC in oxidizing environment [12]. For Alloy 690, the beneficial effect of carbides in caustic has also been found in U-bend tests [13] although it was not revealed in C-ring tests due to insufficient stress applied [14]. Alloy 690 is typically processed in the thermally treated condition. The mechanism behind this improvement is still not clear. Bruemmer et al. [15] proposed that grain boundary carbides serve as effective dislocation sources which promote crack blunting and increase resistance to cracking. Hertzberg and Was [10] suggested that the improvement is due to the formation of a protective C-rich film at the crack tip resulting from chromium carbide dissolution at the grain boundary. Consistently, Persaud et al. [16] suggested that oxidation of Cr carbides at the grain boundary might impede inwards oxygen diffusion which would contribute to the SCC resistance. Concern has also been raised about the detrimental effects of cold work on the SCC resistance, induced during shrinkage in weld heat affected zones or during manufacturing. Relevant work on Alloy 600 revealed that the strengthening due to residual strain in heat affected zone could accelerate the growth rate of SCC in simulated pressurized water reactor (PWR) primary water [17,18]. Some studies [4,5,19] also indicate that cold working significantly increased the SCC susceptibility of Alloy 690. Whether the beneficial effect of grain boundary carbides in suppressing SCC could be retained in cold worked nickel base alloys has yet to be established. The results from Toloczko et al. [19,20] suggest that the grain boundary carbides may induce high grain boundary strain during cold rolling, which results in higher SCC propagation rate, while solution annealing prior to cold rolling improves the SCC resistance. Consistently, Arioka et al. [21] found that the SCC growth rate of cold rolled Alloy 690 tested in T–L orientation increases with increasing carbide coverage. So according to these studies, it appears that grain boundary carbides may be detrimental to SCC crack growth in cold rolled Alloy 690. There is no data on the effect of grain boundary carbides on the initiation of SCC in cold rolled Alloy 690.

The slow strain rate tensile (SSRT) test is an attractive option for assessing the SCC initiation susceptibility of a resistant material like Alloy 690 because the test time is relatively short compared to a constant load test or crack-propagation test [22], and it involves dynamic loading, which appears to be important in initiating intergranular SCC cracks. Some work [23,24] has been done to unify the SCC damage during SSRT and constant load tests and to further predict the long term behavior of materials based on

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results from short term SSRT tests. To initiate SCC on resistant material like Alloy 690, the strain rate needs to be carefully chosen to balance the environmental and mechanical components. The results from Kim and Van Rooyen [25] indicated that slower strain rate induced higher percentage of SCC area on the fracture surface of Alloy 600 strained to failure in 365 °C primary water environment. The model developed by Santarini [26] suggested that the number of cracks increased significantly on Alloy 600 strained to rupture in simulated PWR primary environment as strain rate was decreased. Tyler and Was [27] strained both Alloy 600 and 690 in the mill-annealed condition to the same strain using two different strain rates in 320 °C pure hydrogenated water and found that the crack density increased as the strain rate was decreased while the average crack length changed slightly. These data indicate that a lower strain rate is a more aggressive condition for initiating SCC on mill annealed or thermally treated alloys 600 and 690. The effect of strain rate on cold-rolled 690 has not yet been established.

In this work, SSRT tests at two different strain rates were performed to study the effects of strain rate on the SCC initiation susceptibility of cold rolled Alloy 690 in 360 °C hydrogenated water. The effect of carbides was also assessed by correlation of SCC with grain boundary carbide density.

2. Experimental

2.1. Material

The chemical composition of Alloy 690 used in this work is 57.6 wt.% Ni, 32.7% Cr, 8.64% Fe, 0.25% Mn, 0.315% Al, 0.08% Si and 0.02% C. The material was a forged bar with a diameter of 185 mm. It was solution annealed (SA) at 1100 °C for 1 h and then water quenched. For some samples, the solution annealed alloy was thermally treated (TT) at 700 °C for 17 h and cooled in air. Both SA and SA+TT samples were cold rolled (CR) to 20% thickness reduction, resulting in a sheet of approximately 8 mm in thickness. SACR denotes solution annealed and cold rolled and TTCR denotes thermally treated and cold rolled. The cold rolled sheet was machined into round tensile bars with the sample axis in the cold rolled direction as shown in Fig. 1. The gage section of the tensile bar is 20 mm in length and 2 mm in diameter. Samples were mechanically abraded up to 4000 grit and then electropolished for 30 s at 30 V in a solution of 10% (volume fraction) perchloric acid in methanol solution. Some coupons were also prepared for carbide analysis using the same procedure.

Grain boundary carbides were characterized with FEI Helios Nanolab 650. The imaged surfaces were perpendicular to the transverse direction of cold rolled bulk sample. The extent of grain boundary carbide coverage in % coverage was measured from images taken at 20,000× for SACR sample and 10,000× for TTCR sample.

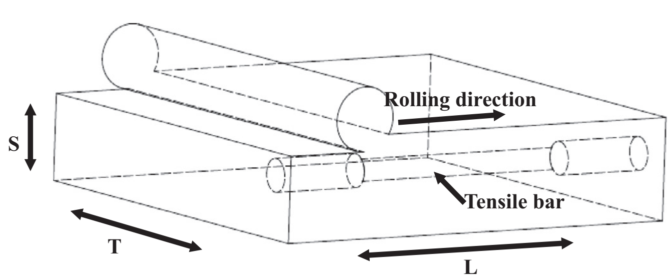

Fig. 1. The orientation of tensile bar in cold rolled bulk sample.

2.2. Apparatus and methodology

Tensile bars were strained to failure in 360 °C high purity water containing 18 cm^3 (STP) H_2/kg H_2O which is approximately the electrochemical potential at the Ni/NiO boundary. The inlet resistivity was above 18 MΩ cm and the outlet resistivity was above 5 MΩ cm. A recirculating water loop equipped with a 4 L stainless steel autoclave was used to maintain the required water environment. Both inlet and outlet dissolved oxygen concentrations and water conductivities were monitored during the test. Ion exchange columns were used to purity the water continuously. Samples were strained with a 5 K servo via a crosshead that was capable of loading a maximum of 4 tensile bars.

After the desired water environment stabilized, the samples were first loaded to just below the yield point at a rate of 1.24×10^{-5}/s and then strained to failure at the specified rates. Two nominal strain rates were used: 5×10^{-8}/s and 1×10^{-8}/s. The samples were designated by the thermal mechanical treatment and applied strain rate. For example, a solution annealed and cold rolled sample strained at 5×10^{-8}/s was designated as SACR-1 and the one strained at 1×10^{-8}/s was designated as SACR-2. Once necking occurred, strain was restricted to the necked region and the rest gage section was not actively strained. So the degree of cracking was mainly dependent on the uniform strain applied.

After the straining tests, the gage sections of tensile bars were examined with a JOEL JSM-6480 scanning electron microscope (SEM). More than 40 equally spaced areas on the uniformly strained gauge section were imaged at 1000×. The images were magnified to 4500× to characterize intergranular (IG) cracks. Crack lengths were measured and the crack numbers were counted. Characterization of cracking was always done at or near the point of uniform strain. Beyond this point, strain is confined to the necked region in which the strain rate increased rapidly. From those data, crack length per unit area, crack density (number of cracks per unit area) and averaged crack length were calculated. These parameters especially crack length per unit area have long been used to assess the SCC initiation susceptibility of materials [27,28]. The validity of these data may be questioned due to the fact that the surface SCC cracks are scarce and shallow. Page and Mcminn [29] also used SSRT to evaluate SCC susceptibilities of Alloys 600 and 690 in simulated boiling water reactor environments and found the fracture surfaces were ductile and only surface cracks could be found. However, the focus of SCC initiation study is to characterize the cracks after they initiate with minimal crack growth. Crack depth measurement could add the validity of initiation study. But for materials highly resistant to SCC like Alloy 690, the statistics of crack depth measurement on cross section is very poor and could not support the analysis of crack initiation propensity.

3. Results

Fig. 2 shows the SEM images of the microstructure of SACR and TTCR samples. The grains on both samples were elongated in the rolling direction and squeezed in the short direction. Small carbides were found on some grain boundaries in the SACR sample (Fig. 2a and b). The measured grain boundary coverage of carbide is about 12% and the average carbide length is around 0.078 μm. For the TTCR sample, most grain boundaries were heavily decorated by semi-continuous carbides while some boundaries were only slightly covered by small carbide particles. Some carbide clusters intruding into the grains were occasionally found (Fig. 2c). On grain boundaries inclining to the rolling direction which were elongated, some voids, induced during cold rolling, appeared

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