**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.

Keywords: A. Alloy; B. SEM; C. stress corrosion; C. effects of strain

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 Cr-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 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 Figure 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 seconds 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 20000× for SACR sample and 10000× for TTCR sample.

2.2. Apparatus and Methodology

Tensile bars were strained to failure in 360 °C high purity water containing 18 cm³ (STP) H₂/kg H₂O 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 purify the water continuously. Samples were strained with a 5K 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 $1000\times$. The images were magnified to $4500\times$ 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

Figure 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 (Figure 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 (Figure 2c). On grain boundaries inclining to the rolling direction which were elongated, some voids, induced during cold rolling, appeared between the grain boundary carbides (Figure 2d). The coverage percentage of carbides on grain boundaries in TTCR is around 72% and the average carbide length is 0.32 μm .

Figure 3 shows the stress-strain curves of cold rolled samples strained to failure at different strain rates in 360 °C water containing 18 cm³ (STP) H₂/kg H₂O. The dip on the curves at a strain of 0.03 (for the higher strain rate) was due to a short power outage during the test that did not affect the outcome of the test. The curves acquired at two strain rates are consistent with each other for the same condition except that SACR-1 failed prematurely because of an error at 770 MPa in reloading. Nevertheless, the failure occurred after necking and shortly before failure was expected, so the cracking result on the uniformly strained gauge section was not affected.

Serrations due to dynamic strain aging (DSA) were observed on all the samples tested. The yield strengths and average stress amplitudes of serrations were extracted from the stress-strain curves and are listed in table 1. The yield strengths acquired at two different strain rates for both samples were quite consistent. The stress amplitude of serrations for the SACR samples changed only slightly with increasing strain rate while that for TTCR samples increased more notably as strain rate decreased.

The cracks on the samples are sparse and generally small, especially on the TTCR samples. Figure 4 shows some of the more readily observable cracks on sample

SACR-2. For TTCR samples, no cracks were found at 5×10^{-8} /s and a moderate number of cracks were visible on the sample strained at 1×10^{-8} /s, and they were very small. The cracking results are also listed in Table 1 and plotted in Figure 5. The unit of crack density (number of cracks per mm^2) is mm^{-2} and that of crack length per unit area (the total length of cracks in μm , per mm^2 of surface area) is $\mu\text{m}/\text{mm}^2$. For both strain rates, the thermally treated samples had lower degrees of cracking as measured by all of the parameters used to characterize cracks (Figure 5a and b). Further, for both conditions, the degree of cracking increased with decreasing strain rate (Figure 5c and d).

Figure 6 shows the fracture surfaces of each of the four cases. SACR sample fracture surfaces (Figure 6a and b) were dominated by dimples typical of ductile failure. The TTCR samples (Figure 6c and d) have a lower density of small dimples on the fracture surfaces. It should be noted that there are numerous IG cracks along the elongated direction on TTCR samples.

4. Discussion

Effect of grain boundary carbides

The yield strengths and serration amplitudes of SACR samples are slightly higher than those of TTCR, which may be due to the higher dissolved carbon content in solution annealed samples vs. thermally treated samples that underwent substantial carbide precipitation. From the cracking results at both strain rates (Figure 5a and b), the thermally treated samples had significantly lower crack density than the solution annealed samples. Considering that the thermally treated sample has much higher grain

boundary carbide coverage (72% vs. 12% for solution annealed samples) and larger carbide size (0.32 μm vs. 0.078 μm for solution annealed samples), the improvement of its SCC resistance over that of solution annealed samples is believed to be due to the mitigating effect of grain boundary carbides. TTCR-2 had a lower crack density and a smaller average crack length than SACR-2 suggesting that grain boundary carbides suppressed the SCC initiation and propagation of even small cracks.

Grain boundary carbide precipitation has long been found to enhance the SCC resistance of Ni base alloys. The SSRT results in pure deaerated water from Bulischeck and Van Rooyen [7] show that heat treatment at 700 °C for 20 h increased the SCC resistance of Alloy 600 significantly. The reverse U-bend tests in 365 °C hydrogenated pure water from Norring et al. [8] consistently indicated that the initiation time of Alloy 600 increased with increasing grain boundary carbide density. Rios et al. [30] did U-bend tests in 360 °C primary water and found that 1h sensitization at 700 °C increased the initiation time of Alloy 600. Was and Lian [9] reported that the crack length per unit area and maximum crack depth of Alloy 600 after SSRT tests in 360 °C primary water decreased with increasing grain boundary carbide coverage and the mitigating effect of grain boundary carbide on SCC was also revealed in Ni-16Cr-9Fe-xC alloys under similar environments [10, 11]. Very limited results about the effects of grain boundary carbide density on the SCC initiation of Alloy 690 have been reported due to the difficulty in cracking this material. Sarver et al. [13] performed U-bend tests on Alloy 690 in 350 °C deaerated 10% sodium hydroxide and found that thermal treatments

between 704 and 927 °C increased the resistance to SCC initiation. Sui et al. [31] also reported that cracked Alloy 600 and 690 contained very little grain boundary precipitation while those heavily decorated grain boundaries did not crack when tested in 380 °C hydrogen/steam. The results shown here further confirmed that the beneficial effect of carbide in suppressing SCC initiation still exists in cold rolled Alloy 690.

It is interesting to note that both SACR samples had a larger density of dimples on the fracture surface, typical features of ductile failure (Figure 6a and b), than the TTCR samples. However, TTCR-1 and TTCR-2 had numerous secondary IG cracks between elongated grains (Figure 6c and d). It should be noted that those IG cracks are not SCC cracks because they were not exposed to the external environment before the sample failed. When viewed from the rolling direction, the grains would be squeezed in the short direction (indicated by “S” in Figure 6) and elongated in the transverse direction. Considering that these secondary cracks are mainly along the elongated grain boundaries, it is inferred that they are roughly perpendicular to the short direction of the cold rolled sheet. These observations suggest that grain boundary carbides decrease the cohesion strength between those elongated boundary surfaces after cold rolling and the complex stress state fractured the weakened grain boundaries during necking. So the carbides could enhance the deformation near grain boundaries during cold rolling and the enhancement is dependent on the orientation of the grain boundary surface with respect to the cold rolling process. Here, the grain boundary surfaces are perpendicular to the short direction and seem to have higher local deformation. Such enhanced deformation

near grain boundaries may decrease the SCC resistance of cold rolled material and the heterogeneous distribution of deformation would result in orientation dependent SCC susceptibility. Some previous SCC growth rate results confirm such a deduction. Toloczko et al. [19] found that the crack growth rates of thermally treated and cold rolled samples were much faster than those of solution annealed and cold rolled samples in both S-L and T-L orientations. Arioka et al. [21] reported that the SCC growth rate of cold rolled Alloy 690 tested in T-L orientation increased with carbide coverage. Recent work from Andresen et al. [32] also showed that solution annealing prior to cold work reduces crack growth rate by more than an order of magnitude for S-L oriented samples. Bruemmer et al. [33] proposed that grain boundary carbides enhance localized grain boundary deformation and induce high interfacial strain during cold rolling, which increases the susceptibility to crack growth. Their work also [20] indicated that high grain boundary strains raised by carbides promoted SCC propagation rates, not the preexisting voids and cracked carbides as IG cracks seemed to blunt at the cracked carbides although Peng et al. [34] suggested that fractured carbide and void at the grain boundary might promote IG cracking. So grain boundary carbides seem to have detrimental effect in SCC growth due to the enhanced local strains near grain boundaries. Interestingly, the crack growth rates of T-S orientated cold rolled stainless steel in 320 °C simulated primary water from Arioka et al.[35] still reveal the mitigating effect of carbide precipitation. This seemingly conflicting result attests to the orientation dependence of SCC susceptibility of cold rolled material with carbide precipitation.

Such enhanced local damage near grain boundaries may also increase the SCC initiation susceptibility of Alloy 690 as cold work was reported to reduce the crack initiation time as well [36-38]. So for cold rolled samples, the beneficial effect of grain boundary carbides in suppressing SCC may be countered by the localized grain boundary damage raised by carbides, and the overall effect of grain boundary carbides would be dependent on the severity of cold work. With increasing cold work, the effect of grain boundary carbide coverage would change from beneficial to detrimental as the localized grain boundary damage would be exacerbated accordingly. From the work of Toloczko et al. [19], 17% cold rolling boosted the crack growth rate of Alloy 690TT over Alloy 690SA by a factor of 5. However, the beneficial effect of grain boundary carbides in suppressing SCC initiation is still observed in 20% cold rolled samples in this work. It seems that the orientation of the sample with respect to the cold rolling process is important. In this work, the tensile bars were machined and loaded in the rolling direction such that cracking occurred in the short and transverse directions, while in the crack growth work just described, the samples were loaded perpendicular to the rolling direction and the crack growth direction was in the rolling direction. As mentioned above, the local damage raised by grain boundary carbides during cold rolling is not homogeneous and the SCC susceptibility of cold rolled material would depend on the orientation of the grain boundary relative to the cold rolling process. The dependence of SCC susceptibility on the roll processing orientation has also been reported in numerous studies [35, 39, 40]. It would be of interest to test samples in other directions to see how

the orientation affects the SCC initiation susceptibility.

Effect of strain rate

For both conditions, the crack length per unit area increased significantly when the strain rate was decreased from $5 \times 10^{-8}/s$ to $1 \times 10^{-8}/s$ (Figure 5c and d). Consistent results have been reported on Alloy 600 [25] showing that a slower strain rate induced a larger percentage of SCC area on the fracture surface. In SSRT test, the severity of environmental degradation is reflected by the test time and that of mechanical loading is mainly controlled by the magnitude of the uniform strain. For a given strain, a lower strain rate allows a longer time for the environment to act. For a given time, higher strain rate produces larger strain, which increases the severity of mechanical loading. An enhancement of either factor would promote SCC. So the crack growth in unit time increases with strain rate due to increasing strain applied while that in unit strain decreases with strain rate due to decreasing test time. The work of Was and Lian [9] on Alloy 600 in 360 °C primary water confirms this deduction and the results from Boursier et al. [23, 24] on both Alloy 600 and 690 also show that crack growth rate in $\mu m/h$ increased with strain rate.

For both material conditions, the increase in cracking severity at lower strain rate is due to enhanced corrosion, which stimulates SCC initiation. Calculations by Santarini also suggest that both average and maximum crack depths increased with decreasing strain rate for Alloy 600 in primary water strained up to rupture [26]. For SACR samples, the crack density increased significantly while the average crack length changed

very little as the strain rate was decreased (table 1). The crack density increased by a factor of 3.64, which is close to the ratio of time spent in uniform strain (4.15) for the two cases. Similar results have been found on Alloy 600 samples that were strained to the same level at different rates [27] and showed that the increase in crack density is very close to the increase in time spent in uniform strain. So the crack density is in rough proportion to the active straining time while the crack size barely changes for a given applied strain. These data suggest that SCC initiation is mainly controlled by the time-dependent corrosion process under fixed severity of mechanical loading, while crack growth is mostly dependent on the severity of mechanical loading. If a sample is incrementally strained at a constant rate without necking, both crack density and length would increase.

5. Conclusion

SCC initiation susceptibility of cold rolled 690 samples with different grain boundary carbide densities in 360 °C water containing 18 cm³ (STP) H₂/kg H₂O H₂ were evaluated using the SSRT technique at two different strain rates. The improvement of thermally treated samples over solution annealed samples in SCC resistance is due to the beneficial effect of grain boundary carbides in mitigating SCC of cold rolled Alloy 690.

For a given uniform strain applied, a slower strain rate results in greater SCC degradation by enhancing the environmental component of the process. SCC initiation is dominated by the time-dependent corrosion process under fixed severity of mechanical

loading, while the crack growth is mostly dependent on the severity of mechanical loading.

Acknowledgements

The authors gratefully acknowledge financial support through the DOE I-NERI program contract 2011-01-K. The authors would like to thank Young Suk Kim and Sung Soo Kim from Korea Atomic Energy Research Institute for providing the materials for this study, and Alex Flick from the University of Michigan for his assistance with preparation of the high temperature autoclave systems.

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Figure captions:

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

Figure 2. SEM images of (a), (b) SACR and (c), (d) TTCR samples.

Figure 3. Stress-strain curves of (a) solution annealed, cold rolled, and (b) thermally treated, cold rolled samples, strained to failure at different strain rates in 360 °C water containing 18 cm³ (STP) H₂/kg H₂O.

Figure 4. Cracks on SACR-2 strained to failure at $1 \times 10^{-8} \text{ s}^{-1}$ in 360 °C water containing 18 cm³ (STP) H₂/kg H₂O.

Figure 5. Cracking results of cold rolled samples strained to failure in 360 °C water containing 18 cm³ (STP) H₂/kg H₂O. (a) and (b) show the effect of grain boundary carbides; (c) and (d) show the effect of strain rate.

Figure 6. Fracture morphologies of (a) SACR-1, (b) SACR-2, (c) TTCR-1 and (d) TTCR-2 strained to failure in 360 °C water containing 18 cm³ (STP) H₂/kg H₂O.

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

Rolling direction

Figure 2. SEM images of (a), (b) SACR and (c), (d) TTCR samples.

Figure 3. Stress-strain curves of (a) solution annealed, cold rolled, and (b) thermally treated, cold rolled samples, strained to failure at different strain rates in 360 °C water containing 18 cm³ (STP) H₂/kg H₂O.

Loading direction

Figure 4. Cracks on SACR-2 strained to failure at $1 \times 10^{-8} \text{ s}^{-1}$ in 360 °C water containing 18 cm³ (STP) H₂/kg H₂O.

Figure 5. Cracking results of cold rolled samples strained to failure in 360 °C water containing 18 cm³ (STP) H₂/kg H₂O. (a) and (b) show the effect of grain boundary carbides; (c) and (d) show the effect of strain rate.

Figure 6. Fracture morphologies of (a) SACR-1, (b) SACR-2, (c) TTCR-1 and (d) TTCR-2 strained to failure in 360 °C water containing 18 cm³ (STP) H₂/kg H₂O.

Tables:

Table 1. Mechanical properties and cracking results of cold rolled samples strained to failure in 360 °C water containing 18 cm³ (STP) H₂/kg H₂O.

Sample Designation	Strain rate (s ⁻¹)	Yield strength (MPa)	Average stress amplitude (MPa)	Uniform strain at UTS ^a (%)	Time to reach UTS (h)	Number of cracks	Crack length per unit area (µm/mm ²)	Crack density (mm ⁻²)	Average crack length (µm)
SACR-1	5 × 10 ⁻⁸	852.3±11.47	35.4±3.94	2.36	131	13	156.8±84.8	25.2±13.6	6.2±0.272
SACR-2	1 × 10 ⁻⁸	839.7±13.86	35.4±5.72	1.90	528	53	679.2±177.7	91.8±24.0	7.4±0.135
TTCR-1	5 × 10 ⁻⁸	801.7±14.20	28.5±4.40	1.22	68	0	none	none	none
TTCR-2	1 × 10 ⁻⁸	798.6±11.86	33.5±4.49	1.48	411	32	230.9±79.1	62.0±21.0	3.7±0.173

^aUTS- ultimate tensile strength.