SCC INITIATION OF CW ALLOY TT690 AND ALLOY 600 IN PWR WATER

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ABSTRACT

The purpose of this work is to understand quantitative processes which underlie the initiation of stress corrosion after long term exposures in high temperature water. Long term stress corrosion cracking (SCC) initiation tests up to about 30,000 h were performed using blunt notch compact tension (CT) type specimens and CT specimens with a shallow depth of pre-crack (~0.1 mm) of cold worked (CW) thermally treated alloy 690 (UNS N06690, TT 690), which were exposed in the primary coolant environment in pressurized water reactors (PWR) under static load condition at 320 and 360˚C. Further, SCC initiation tests were also performed using blunt notch CT specimens of CW mill annealed Alloy 600 (UNS N06600, MA600) to consider the similarities and dissimilarities with Alloy TT690. Three important patterns were observed: first, intergranular (IG) cracking was observed in 20%CW TT690 from the shallow pre-crack at 360˚C in PWR primary water after 20,653 h under static load conditions. Second, clear evidence of cavities were identified in 20%CW TT690 ahead of SCC tip at 360˚C in PWR primary water after 20,653 h. The cavities seem to result from the condensation of vacancies and affected the bond strength of grain boundaries. Consequently, the bond strength is assumed to be weakened during the incubation period. Third, SCC initiated in 20%CW MA600 at 360˚C in PWR primary water after 1,522 h. The results suggested that the transition of SCC segments from initiation and steady growth seem to be occurred about 0.2 to 0.3 mm depth of intergranular cracking at high stress condition at 360˚C in PWR water.

As a model for the initiation of SCC after long term operations of CW TT690 in high temperature water, the combination of local corrosion and the formation of cavities from the collapse of vacancies seem to dominate the initiation of SCC after long times in high temperature water.

1. INTRODUCTION

The purpose of this work is to provide basis for quantitatively predicting the occurrence of SCC of cold worked Alloy TT690 in light water reactors (LWR); the ultimate aim of this work is to predict performance to at least 60 to 80 years. In particular, this work is focused on quantitative prediction of the initiation segment of SCC with Alloy TT690. Conditions considered here are: cold work, grain boundaries structures, temperatures in the range of 320˚C to 465˚C, and long testing times more than 20,000 h. Basically, the specimens used are of the “blunt notch” type. In addition, special designed compact tension type specimen with a shallow pre-crack of one grain size depth are used to examine the transition of SCC segments from initiation to growth. Crack initiation behaviors of Alloy MA600 was also studied in PWR water and in air to consider the similarities and dissimilarities with Alloy TT690.

The aim here is to focus on atomic processes which can be quantified in terms of coalescence of vacancies which produce cavities at grain boundaries.

The work here is in the category of proactive research where the aim is to predict processes before major damage occurs. As Staehle and Gorman1,2,3 have pointed out, such proactive research is crucial to
identifying problems before they occur in operating plants. Staehle describes such “proactivity” as “Serving to prepare for, intervene in, or control an expected occurrence or situation, especially, a negative one or a difficult one.

To assess degradation caused by SCC after long times of operation in light water reactors (LWRs), detailed knowledge of both initiation and growth over long times are necessary. Further, it is particularly difficult to predict long term initiation from conventional short terms accelerated experimental studies. Therefore, it is necessary to understand atomic identities and processes. Also. It is necessary to understand the underlined process by reasonably long term testing under simulated conditions in operating LWR’s considering the possible change in material during long term operation beyond 60 years.

The specific purpose of the present research is to identify and quantify the underlying atomic processes of initiation that provide bases for predicting the occurrence of SCC during very long term incubation periods in LWR environments. The main reasons for undertaking this study is related to the important applications of steam generator (SG) tubing and structural materials such as control rod drives as well as other components.

The excellent performance of Alloy TT690 is now widely recognized in primary systems in operating PWRs. However, Andresen et al., Paraventi et al., Toloczko et al., Alexandrea et al., and Arioka et al. have reported that intergranular SCC (IGSCC) growth occurs in Alloy TT690 in PWR primary water if the materials have been cold worked even though measured SCC growth rates were slower than mill annealed Alloy 600. However, there is little research on the SCC initiation precursors of these alloys in PWR environments that provides adequate information for predicting long term reliability.

Accordingly, Arioka et al. examined the crack initiation and growth in cold-worked carbon steels in a hydrogenated pure water and in air to assess the reliability of piping during its life in LWRs. The clear evidence of cavity formation resulting from vacancy clusters was recognized at grain boundaries before crack initiation and growth both in the water and air. The cavities were assumed to weaken the grain boundary bonding strength.

Arioka et al. also reported that cavities, which resulted from vacancy coalescence, were formed more rapidly when material was exposed to water rather than to air environments at the same temperature. The main cause of this more rapid cavity formation is probably due to the effect of super abundant vacancies, that result from absorbed hydrogen generated from the exposure to high temperature water. Regarding the mechanism of IGSCC of cold-worked materials in high temperature water, Arioka et al. reported, on the basis of creep studies and crack tip analyses, that diffusion of vacancies, which were driven by stress gradients near grain boundaries, produced cavities.

Based on these results, cavities, especially in cold worked materials, might form prior to crack initiation especially in the case of a long term operation at LWR operating temperatures. Further, Arioka et al. reported that it is necessary to take into account the possible change in bonding strength at grain boundaries caused by cavity formation during long term operations. Further, Bruemmer recently reported that SCC initiation occurred in CW TT690 at 360˚C in PWR primary water under dynamic loading conditions, and they also recognized cavities ahead of SCC-tip. However, there are no data on the initiation segment of Alloy TT690 in PWR water under static constant load condition. Furthermore, there are no mechanistic studies on the initiation segment of Alloy TT 690 in PWR environments; therefore, initiation of SCC, caused by the formation of cavities in cold worked Alloy TT 690 is a focus of this study.
2. EXPERIMENTAL PROCEDURE

2.1 Materials

The following materials were prepared for experimental studies to determine the rate of SCC initiation and cavity formation: commercial purity thermally-treated Alloy 690 and mill-annealed Alloy 600 (UNS N06600, MA600). Tests of TT690 were performed mainly using material, which is used in operating PWR plants. In addition, some tests of TT690 were performed using material, which was prepared in laboratory: the plate of TT690 was solution-treated at 1,075˚C for 0.5h in air and water-cooled; it was then heat-treated at 700˚C for 15 hours, and air-cooled. A plate of MA600 was mill-annealed in air at 930˚C for 1h and air-cooled. Almost all carbide precipitated at grain boundaries in TT690. On the other hand, both intergranular and intragranular carbides precipitated in MA600.

Grain sizes of TT690 and MA600 were approximately 100 μm. Cold work in the specimens was produced by rolling at room temperature in one direction to reduce the thickness by 20%. The chemical compositions and mechanical properties of the materials are described in Tables 1 and 2.

2.2 Crack Initiation Testing in PWR Primary Water and Air

There were two objectives of the experiments: (a) predict times of crack initiation at PWR operating temperatures; (b) determine the phenomena that precede SCC at initiation sites during long-term incubation periods. This information would determine major variables that dominate prediction of initiation. For this objective, blunt notch 0.5TCT specimens with 12.5mm thickness were prepared using 20% cold worked TT690 and MA600 to simulate the region where stress is concentrated in piping and tubing.

Specimens were prepared in the T-L orientation, i.e., the direction of crack initiation was parallel to the rolling direction. The radius of the round notches was 0.5 mm. The surfaces of the notch areas were polished by buffing with diamond paste. Elastic-plastic analyses were performed on this type of specimen using a commercially available finite element program. The calculated results of the local stress distributions are shown in a previous published paper. Further, additional testing was performed to examine the transition from SCC initiation to SCC growth using 0.5T CT with very shallow fatigue pre-crack. A shallow fatigue pre-crack of about one grain size (~0.1 mm) was produced using a load ratio (R=K_{min}/K_{max}=0.1) with 8 Hz and a K_{max} below the stress intensity for testing. All tests were performed under constant load.

Using this type of specimen, SCC initiation tests of 20%CW TT690 and 20%CW MA600 were performed at 360˚C, 340˚C, 320˚C, and 290˚C in PWR water with 500 ppm B, 2 ppm Li, and 30 cc H_{2}/kgH_{2}O under constant load conditions. In addition, crack initiation tests of 20%CW MA600 were performed also in air under constant load conditions to compare the rate of cavity formation with 20%CW TT690 and to determine effects of environment on initiation time in the range of temperature between 435˚C to 465˚C. Static loads were 6.91kN (30MPa.m^{1/2}) in water and 9.16kN (40 MPa.m^{1/2}) in air in testing using blunt notch specimens. On the other hands, initial stress intensity factor value (K) in test with a shallow fatigue pre-crack CT specimen was 24 MPa.m^{1/2}. The change in displacement between the loading pins was monitored continuously. Testing was terminated based on the change in displacement; specimens were then examined destructively to determine whether cracking had initiated.

2.3 Measurement of Population of Cavities

The objective of the measurements was to quantify the role of cavity formation on crack initiation in air of 20%CW TT690 and 20%CW MA 600, and then to compare the rate of cavity formation of these alloys. The formation of cavities was determined by using scanning electron microscopy (SEM) of the cross sectional view at the round notched area after testing. Then, the area of cavities (S_i) was measured by
image processing using SEM images to quantify the population of cavities in various locations. The area fraction of cavities was obtained using Equation (1) to quantify the population of cavities in each position.

\[
\text{Area fraction of cavity} = \frac{S_c}{S_0}
\]  

where, \( S_c \) = Area of cavities measured by image processing; \( S_0 \) = Area of measured positions.

\( S_0 \) is 160 \( \mu \)m x 228 \( \mu \)m in the case of CW MA600. The reason why \( S_0 \) is large is its large grain size of more than 100 \( \mu \)m. The area fractions of cavities were measured as a function of the distance from the surface of the notch to determine the stress dependence on the processes of cavity formation prior to crack initiation.

### 2.4 Scanning Electron Microscopy Observation and Auger Electron Spectroscopy Analysis

The principal objective of these observations was to clarify whether intergranular penetrations occur as IGSCC initiation site resulted from cavity formation and local oxidation. Careful observation by Scanning Electron Microscopy (SEM) was performed on all the fracture surface to examine the role of cavity formation and local corrosion in processes of long term SCC initiation. Then, some specimens were examined in cross sectional view to examine the role of cavity formation and intergranular corrosion on SCC initiation. Auger Electron Spectroscopy Analysis (AES) were performed to examine the cause of intergranular penetration by cavity formation. Further, SEM observation was performed to examine the role of intergranular corrosion on SCC initiation. The samples were prepared by gallium ion sputtering in a focused ion beam apparatus (FIB).

### 3. RESULTS AND DISCUSSION

#### 3.1 Crack Initiation of 20% CW MA600 and TT690 in Air

Crack initiation time was measured in air using 20%CW MA600 and compared with the already obtained results\(^\text{15}\) of 20%CW TT690. Significant depths of crack initiation were observed in 20%CW MA600 near the surface of the notch in the range of temperature between 435 to 465\(^\circ\)C in air. Crack initiated mainly just below the surface of the notch in the range between about 0.1 to 0.8 mm although shallow cracking also initiated at the surface as shown in Figure 3.1(a). Further, clear evidence of cavities was also observed as shown in Figure 3.1(b). Those results were completely similar with behaviors\(^\text{15}\) of CW TT690 in gas environments. The reason why cracks initiated just below the surface is assumed to be related with the mechanism of cavity formation as described in our previous publication\(^\text{21}\): cold worked induced vacancies diffuse to high stress area driven by stress gradient controlled by equation (2).

\[
\left( \frac{\partial c}{\partial t} \right) = D \left( \frac{\partial^2 c}{\partial x^2} \right) - \left( \frac{D}{kT} \right) \left( \frac{\partial}{\partial x} \left( c \frac{\partial \sigma}{\partial x} \right) \right)
\]  

where, \( c \) : concentration, \( t \) : time, \( x \) : distance, \( D \) : diffusion coefficient, \( k \) : constant, \( \sigma \) : stress

One example of the relationship between the stress gradient and the population of cavities is shown in Figure 3.2 for 20%CW TT690. Peak in cavity density was located near the peak of the stress gradient in each test. The location of crack initiation is almost the same as the peak of population density of cavities, as shown in Figures 3.1 and 3.2. This suggested that cracking first initiated from just below the surface of the notch as a result of the coalescence of cavities at grain boundaries. Results also suggest that cavities seem to nucleate in the high stress area by the diffusion of vacancies driven by the stress gradient, as shown in equation (2). The rate limiting process for cavity formation seem to be lattice diffusion of cold work induced vacancies driven by the stress gradient. The vacancy concentration, \( C_v \), induced in metals by plastic deformation can be estimated\(^\text{23-24}\) by equation (3).
\[ C_v = \eta \varepsilon^n \quad (3) \]

where, \( \varepsilon \) : strain given by the plastic deformation, \( \eta \) and \( n \): constants

In the early stage of deformation, the constants are reported\(^{23-24} \) to be \( \eta = 10^{-4} \) and \( n = 5/4 \), while in the later stage of deformation, the constants are \( \eta = 10^{-2} \) and \( n = 2 \). Therefore, the vacancy concentration in atomic fraction induced by 20% cold worked specimen is estimated to be \( 4 \times 10^{-4} \). The concentration of thermal equilibrium vacancies in Ni at 400˚C is estimated about \( 10^{-8} \) from the literature\(^{25} \) data. This suggests that cold work strongly enhances the rate of cavity formation resulting from the increase in the concentration of vacancies in materials. Further, about \( 10^4 \) times faster lattice diffusivity was observed\(^{26} \) in 20% cold work carbon steel at 320˚C. Those results suggest that crack initiation from cavity formation is enhanced by cold work.

The effects of temperature (as 1/T) for crack initiation in air of 20%CW MA600 are shown in Figure 3.3 together with results\(^{15} \) of 20%CW TT690. Crack initiation time of 20%CW MA600 in gas environments was almost same as 20%CW TT690, as shown in Figure 3.3. The extrapolated crack initiation time at PWR operating temperature seems to have insufficient margin for extended life, noting that additional factors should be considered for prediction, such as the grain size effect, the environmental effect in PWR water, and others factors. The rate of cavity formation is assumed to be enhanced in fine grain materials as reported\(^{15} \) in our previous literature, although direct evidence to show the effect of grain size is necessary to assess the crack initiation of fine grained steam generator tubing. Furthermore, effects of environment in reactor coolant should be evaluated such as corrosion at the crack tip/crack flank, absorbed hydrogen, etc.

Therefore, careful attention to the effect of cavity formation on the process of crack initiation should be given to SCC initiation especially after long term operation even at operating temperatures of water cooled reactors if material is heavily cold worked and the stress is high. Detailed studies in reactor coolant environment are crucial to assess the precise SCC initiation time.

3.2 SCC Initiation in PWR Primary Water

3.2.1 SCC initiation of 20%CW MA600 in PWR Primary Water

The crack initiation time was measured using 20%CW MA600 in the range of temperature between 290˚C to 360˚C in PWR primary water. Significant depths of crack initiation were observed at 360˚C after 1,522 h exposure in PWR primary water. Intergranular SCC initiated from the surface of about the center of notch as shown in Figure 3.4. This is a completely different pattern than crack initiation in air caused by cavity formation, as described in the previous section in which the first cracks initiated from inside the surface. Small number of shallow cracks are observed near the deep main SCC from the surface, as shown in Figure 3.5. The depth of the shallow cracks was about 0.2 to 0.3 mm, as shown in Figure 3.4. The results suggest that the transition of SCC segments from initiation to steady SCC growth occurs after about 0.2 mm depth of intergranular cracking. At the present time, no clear evidence of SCC initiation is observed in 20%CW MA600 at 340˚C, 320˚C, and 290˚C up to 2,000 h. The SCC initiation times in PWR primary water are summarized as a function of temperature together with the crack initiation times in air at 360˚C, as shown in Figure 3.6. More than 60 times faster SCC initiation occurred in PWR primary water than the extrapolated initiation time in air at 360˚C, as shown in Figure 3.6. The results suggest that the rate limited process of SCC initiation in PWR primary water of 20%CW MA600 is not cavity formation, but some reactions in water such as intergranular corrosion.

3.2.2 SCC initiation of 20%CW TT690 in PWR Primary Water

\[ \text{SCC initiation in blunt notch CT specimen} \]

No SCC initiation is detectable on 20%CW TT690 in PWR water at the current exposure times of 27,481 h at 360˚C and 29,927 h at 320˚C. However, evidence of intergranular corrosion was observed by SEM observation from the surface at the surface of the notch, as shown in Figure 3.7. The current results of the
SCC initiation time in PWR primary water are summarized as a function of temperature together with the crack initiation time in air in Figure 3.8. A testing period over 30,000 h is long for a laboratory test. However, it seems to be shorter than the extrapolated value from high temperature tests in air, as shown in Figure 3.8. Accordingly, we decided to continue those tests in PWR primary water. Some tests will be terminated in near future for destructive measurements to examine the effect of the PWR water environment.

❖ SCC initiation from one grain size shallow pre-crack

Tests have been performed for 20,653 h to examine the transition behavior of SCC from initiation to steady growth in 360˚C PWR primary water using a special designed CT specimen with shallow fatigue pre-crack depth about one grain size (~0.1 mm depth). The initial K value of the testing is 24 MPa m$^{1/2}$. The local stress at the tip of these specimens is assumed to be lower than in conventional CT specimens with about 2 mm depth of pre-crack for SCC growth measurement considering the shallow depth of transgranular pre-crack and the low initial K value. Under those circumstances, one tests was terminated for destructive examinations to obtain information on the effect of environment on SCC initiation of CW TT690 after long term exposure. Figure 3.9 show the cross sectional view of 20%CW TT690 after test in PWR water for 20,653 h. A high density of cavities was observed at grain boundaries ahead of shallow pre-crack as shown in Figure 3.9. Furthermore, intergranular cracking covered by oxide were observed just ahead of pre-crack together with intergranular cracking not covered by oxide, as shown in Figure 3.10. The most important implication of this finding is that intergranular SCC initiated from the shallow pre-crack in 360˚C PWR primary water after 20,653 h at 24 MPa m$^{1/2}$ although 20,653 h is significantly shorter than the extrapolated initiation time at 360˚C from the data of crack initiation in air. This result suggests that some enhancement mechanism might be active in PWR water such as corrosion in water and hydrogen enhanced localized plasticity (HELP), although more detailed studies and examination are crucial. Another important implication of this finding is that crack embryos are nucleated before SCC advance just ahead of SCC, probably caused by cavity formation, as shown in Figure 3.9 and Figure 3.10.

Then, detailed examinations were performed by high resolution SEM observation and AES measurement to study the origin of the intergranular crack embryo formation before crack advance. About 200 nm size of tiny pores and same size of particles were observed on the intergranular surface without oxide, as shown in Figure 3.11. Comparing the results of cross sectional view and fracture surface shown in Figure 3.12, the pores are assumed to be cavities formed near carbides and particles are assumed to be carbide precipitated on grain boundaries. Results of AES analysis also support this view as shown in Figure 3.13. Results$^{21}$ of our observation in creep cracking show that cavities were formed at carbide interfaces, in other words, carbides play a role as the nucleation site for cavities. Also, the measured coverage of carbide on grain boundaries in TT690 is about 50% as shown$^{12}$ in our previous literature. Furthermore, similar size of cavities with carbides seem to be nucleated near the carbides as shown in Figure 3.12. The results suggested that bonding strength of grain boundaries of CW TT 690 weakens with time by cavity formation prior to crack advance. Detailed and much longer time testing should be performed using blunt notch CT specimens in PWR primary water to assess SCC initiation in PWR primary water. However, the results described in this section suggest that a faster rate of cavity formation might occur in PWR water than in air. In other words, more rapid crack initiation caused by cavity formation might occur in PWR water if the degree of cold work and applied stress are high enough. Furthermore, we should pay careful attention to the effect of grain size to assess SCC initiation of steam generator tubing with fine grain. Rapid cavity formation is assumed to occur as described$^{21}$ in our previous literature. Therefore, careful attention after long term operation should be given to grain size.

Further comprehensive basic modeling, including effects of electrochemical reactions occurring at the surface, are crucial for precisely predicting long term behavior for more than 60 years in LWR’s and PHWR’s.
4. CONCLUSIONS

1. Intergranular SCC advance occurred in 20%CW TT 690 from a shallow fatigue pre-crack of about one grain size depth after 20,653 h in 360˚C PWR primary water. Also, intergranular cracking without oxide was observed ahead of the SCC-tip. The fracture surface was almost completely covered by cavities. This suggests that the observed intergranular cracking is formed as a crack embryo before SCC advance in 360˚C PWR primary water during 20,653 h.

2. A high density of cavities are observed at intergranular locations in 20%CW TT690 ahead of SCC tip after 20,653 h in 360˚C PWR primary water. Cavities appear to be nucleated near carbides by the condensation of vacancies. This result suggests that diffusion of vacancies, which are driven by stress gradients, occurs during the incubation period even in 360˚C PWR water. Eventually, the bonding strength of grain boundaries seem to weaken before cracks initiate.

3. Clear evidence of cavities formation of 20%CW TT690 after 20,653 h in 360˚C PWR primary water suggests that the rates of cavity formation might be enhanced in 360˚C PWR primary water although further comprehensive studies are crucial.

4. IGSCC of 20%CW MA600 seems to initiate mainly by localized intergranular corrosion from the surface in PWR primary water. The transition of SCC segments from initiation to growth seems to occur above about 0.2 to 0.3 mm depth of intergranular cracking at high stresses.

5. The effect of cavity formation should be taken into account to assess SCC initiation of CW TT690 fine grained steam generator tubing after long term operation such as beyond 60 years considering possible enhancement by the fine grain size on the rate of cavity formation.

5. ACKNOWLEDGEMENTS

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6. REFERENCES


Table 1 Chemical composition of test materials. (Weight percent)

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<th>C</th>
<th>Cr</th>
<th>Fe</th>
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Table 2 Mechanical properties of test materials at room temperature and 320° C.

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<th>Yield strength (MPa)</th>
<th>Tensile strength (MPa)</th>
<th>Elongation (%)</th>
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Figure 3.1 Cross sectional view after test of 20%CW MA600 in air at 435°C for 2,928 h

Figure 3.2 Distribution of population of cavities and stress gradient under test condition
Figure 3.3 Crack initiation time vs. $1/\text{Temp.}$ of 20% CW MA600 and TT690 in air.

Figure 3.4 Cross sectional view of 20% CW MA600 after test

$[360^\circ\text{C in PWR primary water, 1,522 h}]$
Figure 3.5 Appearance of SCC initiated site of 20% CW MA600 after test (360°C in PWR primary water, 1,522 h)

Figure 3.6 Crack initiation time vs. 1/Temp. of 20% CW MA600 in air and in PWR water
Figure 3.7 Inter-granular corrosion in 20% CW TT690 after test in PWR water for 29,927 h

Figure 3.8 Crack initiation time vs. 1/Temp. of 20% CW TT690 in air and in PWR water
Figure 3.9 Cross sectional view of 20% CW TT690 after test in PWR primary water

(360°C for 20,653 h, Pre-crack depth: about 0.1 mm, K: 24 MPa m¹/²)

Figure 3.10 Fracture surface of 20% CW TT690 after test in PWR primary water

(360°C for 20,653 h, Pre-crack depth: about 0.1 mm, K: 24 MPa m¹/²)
Figure 3.11 Fracture surface of 20%CW TT690 after test in PWR primary water
(360°C for 20,653 h, Pre-crack depth : about 0.1 mm, K : 24 MPa m^{1/2})

Figure 3.12 Comparison between fracture surface and cross sectional view after test in PWR primary water
(20%CW TT690, 360°C for 20,653 h, Pre-crack depth : about 0.1 mm, K : 24 MPa m^{1/2})
Figure 3.13  AES mapping of fracture surface of 20% CW TT690 after test in PWR primary water
(360°C for 20,653 h, Pre-crack depth: about 0.1 mm, K: 24 MPa m¹/²)