Effect of Pre-strain on Stress Corrosion Cracking of Type 304 Stainless Steel in High Temperature Water*

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Synopsis

The effect of pre-strain on intergranular stress corrosion cracking (IGSCC) susceptibility of Type 304 stainless steel has been studied using a constant load tensile specimen in 288 °C water containing 26 ppm dissolved oxygen. The results obtained are summarized as follows:

- (1) The time to failure of the specimens pre-strained at room temperature or 288 °C are longer than those of non pre-strained specimens.
- (2) The stress-strain behaviour of materials changes by pre-straining. The effect of pre-strain on IGSCC initiation could be explained in terms of a relation between the rupture of surface oxide film and the stress-strain behaviour of materials.

I. Introduction

The occurrence of Intergranular Stress Corrosion Cracking (IGSCC) of Type 304 stainless steel in oxygenated high temperature water has been known for several years. The key factors required for the occurrence of IGSCC of Type 304 stainless steel include the coincidence of three critical factors:

- 1) sensitization of material,
- 2) high tensile applied stress, and
- 3) corrosion environment.¹⁾

The critical value of each factor depends on the degree of other factors. When high stress is working in heavy sensitized material, IGSCC may be initiated and propagate even in comparatively less aggressive environment such as the reactor water of BWR. The critical value of stress/strain for IGSCC initiation also varied from the degree of sensitization of material.

A number of studies including the effect of sensitization,^{2,3)} cold work,⁴⁻⁶⁾ surface grinding¹⁾ and applied stress⁷⁾ on IGSCC of Type 304 stainless steel have been carried out over the past several years. The effect of deformation martensite on IGSCC has been also studied^{6,8)} related to the effect of cold work. As the results of the above studies, it has been demonstrated that Type 304 stainless steel becomes more susceptible to IGSCC by acceleration of degree of sen-

sitization and high applied stresses over yield strength of material.

However, many studies have been carried out on the evaluation of metallurgical aspect under fixed applied stress condition so that the effect of stress/strain behaviour on IGSCC have not been clarified fully at this time.

Furthermore, the oxide film rupture theory has been proposed⁹⁾ for mechanism of IGSCC initiation, but no initiation studies related to the oxide film rupture and stress-strain behaviour of materials have been carried out.

This study examines IGSCC susceptibility of Type 304 varying the stress-strain characteristic by pre-straining to make clear the role of strain from the viewpoint of the oxide film rupture by plastic strain.

II. Experimental Procedures

Type 304 stainless steel was used in this experiment. The chemical compositions and mechanical properties of the material used are given in Table 1. The steel was received in the form of 9 mm plates. These plates were solution annealed at 1 050 °C for 50 min.

Figure 1 shows the procedure of specimen preparation. After cutting the 9 mm plate, sensitization heat treatments at 621 °C for 4 hr or at 700 °C for 30 min plus at 500 °C for 24 hr were given, and then the specimens for pre-straining were machined with emery paper #04 finishing as shown in Fig. 2.

The uniaxial constant load specimens for the evaluation of IGSCC susceptibility shown as dotted line in Fig. 2 were cut from the pre-strained material with an intact surface. The amount of pre-strain is calculated from the following equation.

$$\varepsilon = \frac{l-l_0}{l_0} \times 100(\%)$$

where, l_0 : initial gauge length (50 mm)
 l : gauge length after pre-straining (mm).

Table 1. Chemical composition (wt%) and mechanical properties of alloy.

Alloy	C	Si	Mn	P	S	Ni	Cr	Co	B
304	0.06	0.52	1.21	0.032	0.006	8.62	18.17	0.21	0.0001
	Tensile strength (kg/mm ²)		0.2 % offset yield strength (kg/mm ²)		Elongation (%)		Reduction of area (%)		ASTM grain size
Room temp.	74.4		23.8		79.0		81.5		5.5
288 °C	46.2		17.7		45.4		71.3		—

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The specimens were pre-strained at room temperature or 288 °C. In the case of the specimens pre-strained at room temperature, the martensite will be induced by plastic deformation, and IGSCC initiation could be affected by the deformation martensite. Therefore, the pre-straining was also given at 288 °C which is far above Md point, the maximum temperature for martensite formation by deformation. The specimens pre-strained at room temperature were heat treated at 621 °C for 4 hr and the specimens pre-strained at 288 °C were heat treated at 700 °C for 30 min plus at 500 °C for 24 hr.

The stress-strain curve at 288 °C, the amount of deformation martensite, and the surface roughness of the specimens pre-strained at either room temperature or 288 °C were measured.

The uniaxial constant load specimens as shown in Fig. 2 were used for IGSCC test in high temperature oxygenated water. The facility is capable of testing 150 uniaxial constant load specimens in the test vessel at a constant load. The specimens were loaded through a piston with a test pressure of 85 kg/cm² as shown in Fig. 3.¹¹⁾ The applied stress levels on the specimens were established by varying the cross section of the specimens at a constant load of 424 kg. In this experiment, the applied stress level was fixed at 30 kg/mm².

Fig. 1. Specimen preparation procedure.

$$\epsilon = \frac{l - l_0}{l_0} \times 100 (\%) \quad \text{----- (1)}$$

$l_0 = 50 \text{ mm}$
 $l = \text{gauge length after Pre-Straining}$

Fig. 2. Specimen configuration.

Fig. 3. Detail of uniaxial tensile specimen loading device.

Upon failure of a specimen, a numbered stainless steel ball indicating the identity of the failed specimen was ejected from the specimen holder and dropped down the manifold and out of the test vessel. The time to failure was indicated by a small fluctuation on the recorder caused by a limit switch when the indicator ball went through it.

The environmental conditions are as follows;

- Testing temperature: 288 °C
Dissolved oxygen : about 26 ppm
Conductivity : <1 μmho/cm
Flow rate : 100 l/h

III. Results

1. IGSCC Test Results

Figure 4 shows IGSCC test results. It is shown from Fig. 4 that the time to failure of the specimens pre-strained at room temperature or 288 °C is longer than those of the non pre-strained specimens. The specimens pre-strained by 16 and 20 % are still under testing over 7 000 hr. As shown above, the initiation of IGSCC of Type 304 is retarded by pre-straining.

2. Stress-Strain Characteristic of Pre-strained Specimens

Stress-strain curve of the pre-strained specimens at 288 °C are given in Fig. 5. In this experiment, the applied stress was fixed at 30 kg/mm² so that the amount of strain corresponding to the applied stress of 30 kg/mm² can be presented as shown in Fig. 6 based on the results shown in Fig. 5. The amount of strain corresponding to the applied stress of 30 kg/mm² at 288 °C decreases with increasing the amount of pre-strain, and it becomes less than about 0.2 % above 8 % pre-strain.

Figure 7 shows the relation between the amount of pre-strain and the mechanical properties of the material at 288 °C. 0.2 % off-set yield strength, σ_y and tensile strength, σ_u increase and reduction of area decreases with increasing the amount of pre-strain.

Fig. 4. Effect of pre-strain on time to failure of Type 304 stainless steel specimens in high temperature water.

The yield strength, σ_y and reduction of area of the specimens pre-strained at room temperature or 288 °C are almost the same, but the tensile strength, σ_u of the specimens pre-strained at room temperature becomes greater than those pre-strained at 288 °C above 10 % pre-strain.

3. Deformation Martensite and Surface Roughness of the Pre-strained Specimens

Figure 8 shows the relation between the amount of

Fig. 5. Stress-strain curve of pre-strained material.

Fig. 6. Relation between pre-strain and strain corresponding to applied stress, $\sigma = 30 \text{ kg/mm}^2$ (294.2 MPa) at 288 °C.

Fig. 7. Effect of pre-strain on mechanical properties at 288 °C. (Type 304)

deformation martensite and of pre-strain. In the case of the specimens pre-strained at room temperature, the amount of deformation martensite increases with increasing the pre-strain as shown in Fig. 8. On the other hand, the amount of martensite of the specimens pre-strained at 288 °C is about 25 % regardless the amount of pre-strain. It is estimated that the Md point of the material used in this experiment based on Angel's equation¹²⁾ is about 40 °C. It is considered, therefore, that the phase transformation will not occur at 288 °C even if the specimens are pre-strained. Then, the amount of deformation martensite of the specimen was re-examined after electropolishing which removed the surface layer introduced by emery paper finishing. After electropolishing, no martensite was observed in the specimen pre-strained at 288 °C. It is indicated that the observed deformation martensite of about 25 % was caused by emery paper #04 finishing at room temperature.

Figure 9 shows the relation between the maximum surface roughness and the amount of pre-strain. The maximum surface roughness of the pre-strained specimens became greater than that of non pre-strained specimens, but there is no significant changes of the maximum surface roughness above 5 % pre-strain.

Fig. 8. Relation between pre-strain and the amount of deformation martensite.

Fig. 9. Effect of pre-strain on the maximum surface roughness.

The maximum surface roughness of the specimen prestrained by 20 % is less than 10 μm at most.

IV. Discussion

It has demonstrated from IGSCC test results shown in Fig. 4 that the initiation of IGSCC of Type 304 is retarded by pre-straining. The effect of pre-strain on IGSCC will be discussed here taking into consideration that the stress-strain behaviour, the change of surface roughness and the change of deformation martensite by pre-straining.

Figure 10 shows the relation between the strain corresponding to the applied stress, $\sigma = 30 \text{ kg/mm}^2$ at 288°C and the time to failure from the results shown in Fig. 6. It is shown that the initiation of IGSCC is retarded when the strain is less than about 0.2 % (the range of elastic deformation). Rupture of the surface oxide film of the pre-strained specimens could be retarded when the deformation of specimens is within elastic range.

Figure 11(a) shows the SEM observations of the specimens loaded at 30 kg/mm² and 14 kg/mm², respectively. In the case of the specimen loaded at 30 kg/mm² ($\epsilon=4.8\%$), intergranular cracking is observed. On the other hand, there are no cracks in the specimen loaded at 14 kg/mm² ($\epsilon=0.5\%$). Fig-

ure 11(b) shows the SEM observations of the specimen loaded after pre-straining of 23 %. The specimen pre-strained by 23 % extend elastically even if the stress is applied up to 30 kg/mm², and the amount

Fig. 10. Relation between time to failure and strain corresponding to applied stress, $\sigma=30$ kg/mm² at 288 °C.

Fig. 11(a).
SEM observations of specimen surface
after unloading. ($\times 7/10$)

of strain corresponding to 30 kg/mm² is about 0.45 %. In this case, IGSCC initiation was not observed as shown in Fig. 11(b). From the above SEM observations, it is supported that the rupture of surface oxide film will be suppressed when the amount of strain is within elastic deformation range.

Figure 12 shows the relation between the applied stresses and the time to failure of Type 304 sensitized heavily. As a result, 2 of 3 specimens loaded below nominal 0.2 % off-set yield strength, σ_y at 288 °C are still on test without failure even if they are sensitized heavily. But the specimen heat treated at 700 °C for 30 min plus at 500 °C for 24 hr failed after about 6 000 hr even if it was loaded below nominal 0.2 % off-set yield strength, σ_y . As above indicated, IGSCC of Type 304 stainless steel can occur under the applied stress level of below nominal 0.2 % off-set yield strength, σ_y when the material is sensitized severely by heat treatment such as at 700 °C for 30 min plus at 500 °C for 24 hr. It is indicated that the critical value of stress/strain factor may vary with the degree of sensitization of material as mentioned earlier in this paper.

Next, the effect of the deformation martensite and the surface roughness on IGSCC will be discussed. From Fig. 8, the amount of deformation martensite of the specimens pre-strained at room temperature are

larger than those of the specimens pre-strained at 288 °C. In the case of the specimens pre-strained by 6 to 14 %, the time to failure of the specimens pre-strained at room temperature seems to be longer than those of the specimens pre-strained at 288 °C as shown in Fig. 4. The reasons for the above phenomena do not clear at this time, however, it could be thought that the deformation of specimen such as micro-creep is controlled by the deformation martensite and then IGSCC is controlled. Much more work is needed, however, to develop the quantitative inter-

Fig. 12. Effect of applied stress on time to failure. (Type 304, C=0.06 %, 288 °C, 26 ppm O₂ water)

Fig. 11(b). SEM observations of pre-strained specimen surface after unloading. ($\times 2/3$)

play between the deformation martensite and IGSCC susceptibility.

The surface roughness of the specimens pre-strained over 5 % fall about 5 to 8 μm at most as shown in Fig. 9. The surface roughness of the non pre-strained specimens also becomes about 5 μm after loading in uniaxial constant load test. Therefore, in this experiment, the surface roughness of the non pre-strained specimens is almost the same order as that of the specimens pre-strained during uniaxial constant load test. It could be thought that there is no significant effect of surface roughness on IGSCC test results.

From the above discussion, it could be concluded that the strain caused in the materials is a primary factor for IGSCC initiation of Type 304.

V. Conclusions

Conclusions drawn from this study are as follows:

(1) The time to failure of the specimens pre-strained at room temperature or 288 °C become more longer than those of the non pre-strained specimens in IGSCC test. IGSCC initiation is retarded by pre-straining.

(2) The changes of stress-strain characteristic of the materials due to pre-straining is a primary factor for IGSCC initiation of Type 304. Since the rupture of the surface oxide film is retarded when the deformation of the materials is within an elastic range,

the initiation of IGSCC is retarded.

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