A STUDY OF TESTING METHOD AND MECHANISMS OF STRESS CORROSION CRACKING PERFORMED ON STAINLESS STEEL

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ABSTRACT

It is desirable to know whether the selected materials are susceptible to stress corrosion cracking (SCC) in the expected environment or not. In this paper, modified C-ring specimens which are used for assessing the sensitivity of materials, especially for evaluating the parameter of critical SCC stress, *i.e.*, σ_{scc}, are proposed. Comparing with the results of the modified specimens and that of the normal C-ring specimens, both of them show no difference. But the former is much more convenient to be tested than the latter one. SUS 304 stainless steels with different heat treatment were used for confirming the modified SCC test and for analyzing the effect of heat treatment and microstructure on SCC behaviour in the magnesium chloride solutions. Test results show that the modified specimens are reliable and may be used for assessing the susceptibility to SCC of Ni-base alloys. The behaviour of transgranular fracture is due to carbon segregation or precipitated carbides at the intragranular slip bands.

KEYWORDS

Modified C-ring specimens, stress corrosion cracking, stress distribution, SCC testing method, stainless steel.

INTRODUCTION

In the design stages of many structures, it is desirable to know whether the construction materials being considered are susceptible to stress corrosion cracking (SCC) in the expected environment or not. For example, the assessment of SCC probabilities of piping materials in boiling water reactors (BWRs) and steam generator tube materials in pressurized water reactors (PWRs) in light water reactor systems (LWRs) is very significant to safety and economy in operation of the power plants. In reality, however, SCC failure takes a long time to occur and some times it seems to be impossible that such a kind of tests in the simulated environments could be done in laboratories. Ordinarily, accelerating test methods are taken into account in laboratories either harsh in environmental terms or in stressing conditions to the high resistant materials, such as Ni-base alloys . For instance, U-bend specimen (qualitative), C-ring specimen (quantitative), slow strain rate testing (SSRT) specimen (indirectly) *etc.* are usually applied to evaluate susceptibility of alloys to SCC properties. Recently, Lo *et al.* (1993) used a two legs touching (TLT) method as a modification of a C-ring specimen. International Standard Organization (ISO) (1993) is preparing to work out the Corrosion Testing of Materials for Nuclear Power Generation. Also, Zhao (1994) pointed out that there are still many things to do to evaluate the sensitivity of materials of environmentally assisted cracking quickly and conveniently.

It is well known that the parameter of critical SCC stress, *i.e.*, σ_{scc}, is a very important one in evaluating the SCC susceptibility of materials. The problem, however is that measurement of this kind of parameter takes many specimens and very long time by traditional testing methods. Although there are some accelerating testing methods, it also takes particularly long time to get such a kind of parameter. In addition, the relationship between the testing data obtained by accelerating method and LWRs environment condition, needs to be clarified.

This paper proposes an accelerated SCC testing method by using a modified C-ring specimen in which cracks are probably more easily and clearly initiated and distinguished. Then, calculation of stress fields on the specimens is done by the computer system in order to evaluate the critical SCC stress, σ_{scc} parameter. Furthermore, SUS 304 stainless steels with different heat treatment are used for confirming the modified SCC test and for analyzing the effect of heat treatment and microstructure on SCC behaviors in the boiling magnesium chloride solutions.

DEVELOPMENT OF MODIFIED SCC SPECIMENS

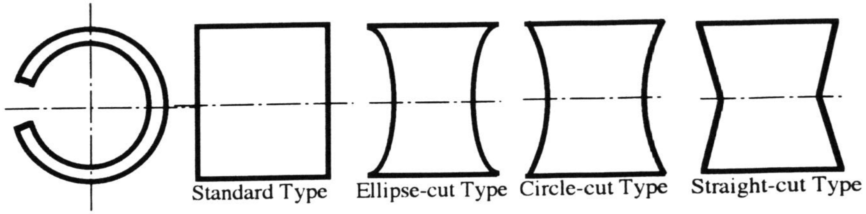
1. *Conception of Modified Specimens*

ASTM Designation G 38-73 (1990) describes making and using C-ring type of SCC test specimen in details. In this SCC test by using C-ring specimens, only the highest circumferential stress σ_{max} ($\Delta = \sigma_{max}\pi D^2/4EtZ$) is considered. Actually, such the characteristics of crack initiations and propagation in the C-ring specimens are that cracks do not always take place at the position of the highest stress. Usually, other places with lower stress are also easily cracked, *i.e.*, multi-cracks are frequently found on the different positions of C-ring specimens, so that, it is necessary to take the true stress as the cracking stress rather than only the σ_{max}.

On the other hand, the gradient of stress field in the specimen plays very important role in the SCC behaviour. Therefore, if the gradient of stress field could be changed to the expected one freely by means of the modified specimens, it is convenient to assess the SCC properties of the testing materials. For example, if the σ_{max} of materials is to be evaluated, the best way is to enlarge the measurable stress ranges on the specimens. Another example is that if the SCC test of corrosion-resisting materials, such as, Inconel 600, 690, or Incoloy 800 *etc.*, is to be done, the expanding gradient of stress field of specimen is also beneficial to the assessment of SCC failures.

2. *Development of New SCC Specimens*

The modified samples are shown in figure 1 in which the both ends of C-ring specimens are respectively cut to the shape of ellipse, circle and straight types by using electric spark machine in order to minimise residual stress from the specimens. Here, they are respectively called to *standard type* (Fig. 1(a)), *ellipse-cutting type* (Fig. 1 (b)), *circle-cutting type* (Fig. 1(c)) and *straight-cutting type* C-ring specimens (Fig. 1 (d)). The purpose of these modified specimens is that the stress distribution on the specimen could be rearranged freely in the specimen for different SCC tests.



(a) (b) (c) (d)

Figure 1. Schematic drawing of the modified specimens

3. *Distribution of Stress in the Specimen*

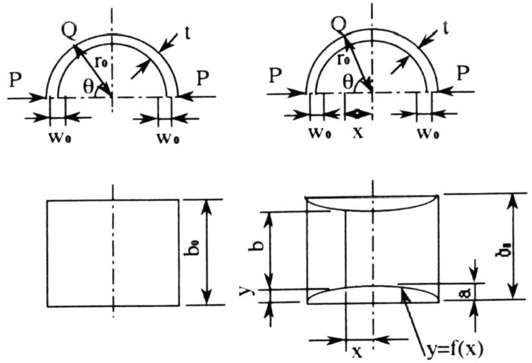


Figure 2. Calculation of stress distribution on specimens

The calculation of stress distribution can be done by means of the differential equations of bending condition. An expression for the stress equation will now be derived in terms of the bending moment at the section. First, consider the simple type, *i.e.*, the standard type C-ring specimen of figure 2a, which has the symmetrical cross section of half-tube. When force *P* is applied to the both sides as shown in figure 2, the bending moment at point Q of specimen is shown as

$$M = Pr_0\sin\theta \qquad \cdots\cdots\cdots\cdots (1)$$

If, taking W as displacement caused by force *P* (Fig. 2), then

$$\frac{d^2w}{d\theta^2} + w = -\frac{Mr_0^2}{EJ} = -\frac{Pr_0^3\sin\theta}{EJ} \qquad \cdots\cdots\cdots\cdots (2)$$

where *E* is the modulus of elasticity, *J* is the moment of inertia of areas, and *M* is the bending moment at any section of C-ring.

For the modified specimens, on the other hand, if, as shown in figure 2b, y = f(x) is the types of cut curves, and b as the width of specimen, then

$$b = b_0 - 2y = b_0 - 2f(x) = b_0 - 2f(r_0 \cot\theta)$$

$$\frac{dw}{d\theta} + w = -\frac{P r_0^3 \sin\theta}{\frac{t^3}{12} E\left[b_0 - 2f(x)\right]} = -\frac{P r_0^3 \sin\theta}{\frac{t^3}{12} E b_0\left[1 - \frac{2f(x)}{b_0}\right]} = -\frac{P r_0^3 \sin\theta}{E J_0\left[1 - \frac{2f(r_0\cos\theta)}{b_0}\right]} \quad \cdots\cdots (3)$$

When the above differential equations are solved, one obtains the following results.

For the *standard type* (C-ring)

$$\sigma = \sigma_{max} \sin\theta \quad \cdots\cdots (4)$$

$$\Delta = \frac{\sigma_{max}\Big|_{\frac{\pi}{2}} \cdot \pi D^2}{4Etz} \quad \cdots\cdots (5)$$

For the *ellipse-cutting type*

$$\sigma = \sigma_{max}(b_0 - 2a)\frac{\sin\theta}{b_0 - 2a\sin\theta} \quad \cdots\cdots (6)$$

$$\Delta = -\frac{\sigma_{max} D^2}{Et}(b_0 - 2a)\left\{\frac{4a + \pi b_0}{8a^2} + \frac{b_0^2}{2a^2\sqrt{b_0^2 - 4a^2}}\left[\arctan\sqrt{\frac{b_0 - 2a}{b_0 + 2a}} - \arctan\left(\frac{-2a}{\sqrt{b_0^2 - 4a^2}}\right)\right]\right\} \quad \cdots\cdots (7)$$

For the *circle-cutting type*

$$\sigma = \sigma_{max}(b_0 - 2a)\frac{\sin\theta}{\left(b_0 + \frac{r_0^2 - a^2}{a} - \frac{r_0^2 + a^2}{a}\sqrt{1 - k^2\cos^2\theta}\right)} \quad \cdots\cdots (8)$$

$$\Delta = -\frac{\sigma_{max} D^2}{ET}(b_0 - 2a)\int_0^{\frac{\pi}{2}} \frac{\sin^2\theta}{b_0 + \frac{r_0^2 + a^2}{a}\sqrt{1 - k^2\cos^2\theta} + \frac{r_0^2 - a^2}{a}} d\theta \quad \cdots\cdots (9)$$

And for the *straight-cutting type*

$$\sigma = \sigma_{max}(b_0 - 2a)\frac{\sin\theta}{b_0 - 2a + 2a\cos\theta} \quad \cdots\cdots (10)$$

$$\Delta = -\frac{\sigma_{max} D^2}{Et}(b_0 - 2a)\int_0^{\frac{\pi}{2}} \frac{\sin^2\theta}{(b_0 - 2a) + 2a\cos\theta} d\theta \quad \cdots\cdots (11)$$

4. *Calculation and Measurement of Stress*

Based on the above equations, stress distributions in specimens obtained are shown in figure 3. It is easily seen that not only the measurable stress ranges on the modified specimens become wider than that of the standard one, but also the gradients of stress distribution of the modified specimens are enlarged under condition of same cut width (Fig. 2b). Here, the mostly expanded measurable ranges of stress, in sequence, are found in the straight-cutting type, circle-cutting type, and the ellipse-cutting type. It must be pointed out that with the change of cut with of *a*, stress distributions can be changed to different conditions freely. Therefore, one can select the different conditions for different purposes.

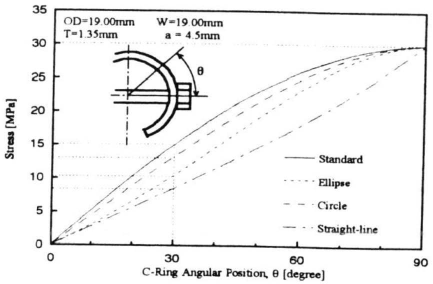


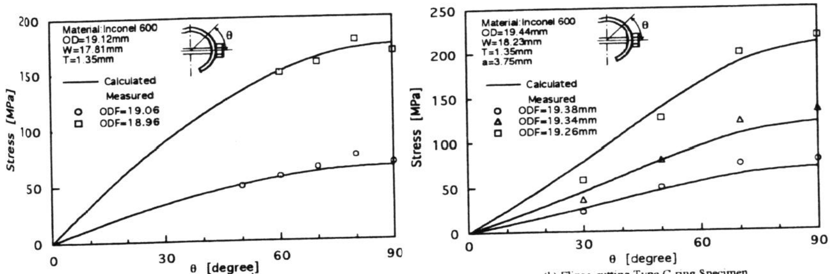
Figure 3. Calculated Stress Distributions of the Modified and Standard Specimens

In order to confirm the calculated results of the stress distribution of the specimens, true stress is need to be measured by strain gauges with 200 μm size in length (Yonetani, 1975 and Sugano, 1986). Strain gauges were mounted at the different locations on the specimen surface. A switch box is used for measuring multi-points stresses in the same time. The results are shown in figure 4. It can be seen that although there are some differences between the experiment and the theory results, the shapes of stress distribution are very similar to each other, and the difference is also small. The explanations of this difference is probably because the strain gauges were sticked on the camber of the surface of the specimen. Therefore, generally, the calculated results are believable.

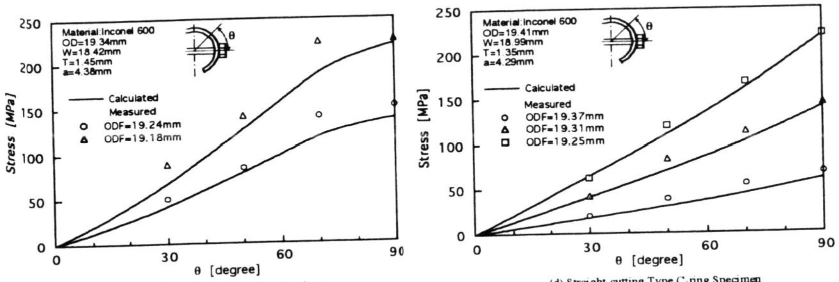
RESULTS OF SCC TESTS AND DISCUSSION

1. *SCC Tests*

In order to assess the practicality of the modified specimens, as a simulation test, specimens of SUS 304 stainless steels with different heat treatments were used in the SCC test. Specimens with an outside diameter of 19 mm and a 1.35 mm wall thickness were machined from the ϕ21 tube with 2 mm thickness. The corrosive environment was selected as 30 *wt% $MgCl_2$* at 100°C and 42 *wt% $MgCl_2$* at boiling conditions. The SCC tests were carried out in the testing facility in which there is a teflon chamber with mantel heater. Specimens were stressed in the elastic range, and continuously immersed in the corrosive solution. With the modified specimens, failure criterion was crack initiation, therefore, detection of a crack on the surface was regularly carried on at the optical microscope.

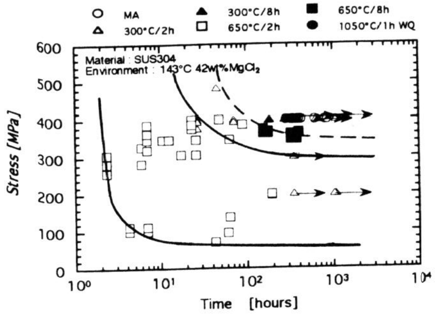


(a) Standard Type C-ring Specimen (b) Elipse-cutting Type C-ring Specimen



(c) Circle-cutting Type C-ring Specimen (d) Straight-cutting Type C-ring Specimen

Figure 4. Comparison of the Results between the Theory and Experiments



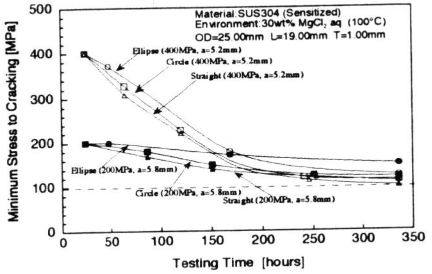


Figure 5. Influence of Heat Treatment on σ_{scc} **Figure 6.** σ_{scc} Results of the Modified Samples

2. *Results*

The results of SCC tests with the standard type specimens are shown in figure 5. It can be seen that the critical stress of σ_{scc} of the material (650°C/2hrs) is about 90 MPa (~ 9 kg/mm²) by using 30 specimens. On the other hand, for the modified specimen (figure 6) in the same SCC test, measured σ_{scc} also shows about 90 MPa (~ 9 kg/mm²) by only respectively using one modified specimen of each type.

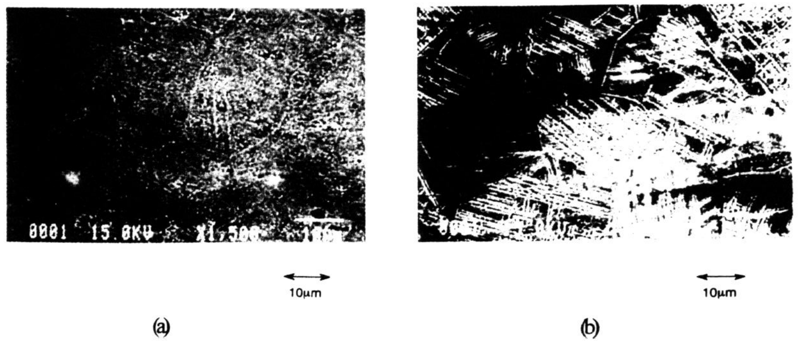
As a formal test, influence of heat treatment on σ_{scc} was investigated by using the standard type C-ring and the modified specimens.

The results are as follows:

Heat treatment	Critical stress
650°C/2hrs (sensitization)	σ_{scc} ≈ 70 MPa (~ 7 kg/mm²)
300°C/2hrs (removing residual stress)	σ_{scc} ≈ 300 MPa (~ 30 kg/mm²)
650°C/8hrs (removing sensitization)	σ_{scc} ≈ 320 MPa (~ 32 kg/mm²)

3. *Discussion*

The test results show that the modified specimens are applicable and reliable in assessing the SCC behaviour, especially for measuring the critical stress of SCC, *i.e.*, σ_{scc}. For the next stage, by using the modified specimens, Ni-base alloys which would take a long time for SCC failure to occur in reality, and are mainly used in atomic power plants, will be evaluated.



(a) (b)

Figure 7. SEM Photographs of Cross Sections (a). 650°C/2hrs, (b). 650°C/8hrs

Figure 7 shows some of SEM photographs of cross sections. These photographs revealed on effect of heating time during heat treatment. As observed by electron probe micro analyzer (EMPA), carbon was probably re-segregated or precipitated in carbides at not only grain boundaries, but also on the intragranular slip bands with increasing heating time.Fractographs of failed specimens also showed that intergranular cracking (IGC) was mainly found in specimens with sensitization treatment (650°C/2hrs), and transgranular cracking (TGC) was observed when the treatment of removing sensitization was done (650°C/8hrs). This means that carbon segregation or precipitation plays an important role to SCC behaviors. According to the test results, the following supposition could be proposed.

When the specimen was heated at 650°C, carbon was segregated and carbides were precipitated on grain boundaries from grains. This brought about the chromium depleted layers at the boundaries which became susceptible to SCC. With increasing heating time at 650°C, carbon began to diffuse in reverse and segregated at the intragranular slip bands. Therefore, cracks could easily propagate in transgranular way.

4. *The Problems*

It must be pointed out that during the SCC test with the modified specimens, it was realized that there were some problems to be studied further. Firstly, it was difficult to evaluate quantitatively the released stress when a crack was propagating. Generally, multi-cracks are initiated on the surface of specimen. At this stage, there is few effect of stress-releasing. Then, one of the cracks began to propagate. In this case, there is an obvious effect of stress-releasing. Therefore, in this test, only the data of crack initiation stage was selected. Secondly, crack is not usually found at the place at which there was a maximum stress ($\theta = 90°$). Although this phenomena is also realized by other researchers, there has not been a satisfactory explanation. The authors propose that provided $\sigma \geq \sigma_{scc}$, the probability of crack initiation is nearly same no matter what stress level is. Thirdly, it is necessary to set up the inspection system of crack initiation. Up till now, crack initiation is inspected by optical microscope after a fixed testing time. However, if the crack were initiated in a fixed testing time, there would be an error to SCC test. These problems need to be discussed further.

SUMMARY

1. The stress distribution of the modified specimens can be regulated freely by changing the cutting depth and the shape.
2. By using the modified specimens, the critical stress of SCC, *i.e.*, σ_{scc} is more easily measured.
3. The effect of heating time on SCC behaviour is mainly the change of carbon segregation or carbide precipitation at the different positions. When the heating time is longer than 8 hours, SUS 304 steel was significantly improved SCC resistance.
4. The behaviour of transgranular fracture of SUS 304 steel was due to carbon segregation or precipitated at the intragranular slip bands.

ACKNOWLEDGMENT

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