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著者	KAWASHIMA Asahi, TAKANO Michinori
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Stress Corrosion Cracking of Inconel 600 and Incoloy 800 in 50%NaOH at 140°C under Slow Strain Rate Condition*

Asahi KAWASHIMA and Michinori TAKANO

The Research Institute for Iron, Steel and Other Metals

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Synopsis

Stress Corrosion Cracking (SCC) behaviour of Inconel 600 and Incoloy 800 in aerated 50% NaOH at 140°C was investigated using a slow strain rate method under electrochemically controlled potentials. The susceptibility to cracking and the mode of cracking were changed depending upon the applied potential and heat-treatment. SCC occurred in a primary active-passive transition region and a secondary passive region at a strain rate of 4×10^{-3} min^{-1}. The former region: only intergranular cracking took place in both alloys for sensitized materials but not in solution annealed specimens, except for Inconel 600 which showed slight intergranular cracking. The latter region: SCC was found in both alloys and there was not so much difference in the susceptibility between annealed and sensitized materials. Transgranular cracking occurred around 0V (SCE) and intergranular cracking became predominant at a higher or lower potential than 0V. The mechanism of the SCC and the relationship between these results and earlier work were discussed.

I. Introduction

Inconel 600 (In 600) and Incoloy 800 (Iy 800), which are very resistant to chloride attack, have been widely used for PWR steam generator tubing materials. However, recently, failures have been reported in which In 600 was damaged by intergranular SCC in service(1,2). It appeared that the cause on these failures was the local concentration of free alkali which can be generated by condenser leaks and uncontrolled phosphate treatment(3,4).

SCC of high nickel alloys in caustic solution at high temparature and high pressure has been extensively investigated in laboratory experiments(5-13). The

* The **1697th** report of the Research Institute for Iron, Steel and Other Metals.

(1) S.H. Bush and R.L. Dillon, *Stress Corrosion Cracking and Hydrogen Embrittlement of Iron Base Alloys*, ed. by R.W. Staehle, J. Hochmann, R.D. McCright and J.E. Slater, NACE (1977), 61.
(2) J.R. Weeks, *Corrosion Problems in Energy Conversion and Generation*, ed by C.S. Tedman, *Jr.*, The Electrochemical Society, Princeton, New Jersey (1974), 322.
(3) J. Weber and P. Surg, Mater. Perform., **15** (1976), 34.
(4) J.R. Weeks, Nucl. Technol., **28** (1976), 348.
(5) I.L. Wilson and R.G. Aspden, Reference 1), 1189.
(6) A.J. Sedriks, S. Floreen and A.R. Mcllree, Corrosion, **32** (1976), 157.
(7) A.R. Mcllree and H.T. Michels, Corrosion, **33** (1977), 60.
(8) A.R. Mcllree and H.T. Michels, NACE Annual Corrosion Research Meeting (1974).
(9) H.R. Copson, D. Van Rooyen and A.R. Mcllree, *Proc. 5th Int. Cong. Metallic Corrosion*, Tokyo (1974), 376.
(10) R.C. Scarberry and S.C. Pearman, Corrosion, **32** (1976), 401.
(11) G.J. Theus, Nucl. Technol., **28** (1976), 388.
(12) J.R. Cels, J. Electrochem. Soc., **123** (1976), 1152.
(13) G.J. Theus, Corrosion, **33** (1977), 20.

results from various studies are not always consistant with one another. Previous studies suggest that SCC behaviour of high nickel alloys can be influenced by oxygen content in caustic solution, sodium hydroxide concentration, heat treatment and potential. In deaerated caustic solutions, increasing nickel content had a beneficial effect on stress corrosion resistance(5–7) and in oxygenated solutions, both high nickel and high chromium contents were necessary to increase stress corrosion resistance(7–9). Sensitization of In 600 was beneficial in promoting resistance to SCC in caustic solutions(7,10–13). In 600 and Iy 800 could be made to crack at small anodic potential in deaerated solution. At open circuit potential no crack was observed with In 600 but cracks formed in the Iy 800. The mode of cracking for In 600 and Iy 800 was intergranular and intergranular or transgranular, respectively(5,7–9,11).

The purpose of this study is to examine the effects of applied potential, strain rate and heat treatment on the SCC of In 600 and Iy 800 in 50% NaOH at 140°C using a slow straining method.

II. Experimental

Specimens for SCC tests were fabricated from In 600 and Iy 800 rods of 5 mm diameter. The chemical composition of these alloys are listed in Table 1. As shown in Figure 1 these were cut to give a 1.05 rad V-notch with an effective length of about 1 mm and a diameter of 3 mm at the neck. The heat treatment conditions are given in Table 2. The solution used was 50% NaOH, in which Type 304

Table 1. Chemical compositions of In 600 and Iy 800.

	C	Si	Mn	P	S	Cu	Ni	Cr	Ti	Al	Fe
In 600	0.030	0.33	0.33	0.005	0.004	0.02	75.55	15.70	0.21	0.12	7.63
Iy 800	0.032	0.61	0.74	0.003	0.004	0.01	32.78	21.44	0.32	0.19	—

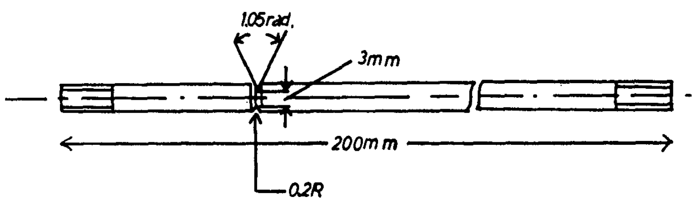


Fig. 1. Specimen used for SCC test.

Table 2. Heat treatment conditions of test materials.

	A	S_1	S_2	S_3	S_4
In 600	1100°C 30 min WQ	A+ 610°C 2hr	A+ 710°C 2hr	A+ 610°C 21hr	A+ 710°C 21hr
Iy 800	1050°C 30 min WQ		A+ 710°C 2hr		

and 316 stainless steels showed the highest susceptibility to SCC at high temperature and atmospheric pressure[14]. The solution was in naturally aerated condition and it was maintained at a temperature of 140°C to avoid boiling. The corrosion cell was made of teflon. The applied potential was controlled at a selected constant value with a potentiostat. A saturated calomel electrode was used as a reference electrode. The Luggin probe was made from a teflon U-tube with a teflon wick running inside it. The SCC tests were carried out using a slow straining machine which can provide a constant cross-head speed. For most experiments, the load was applied by an Instron-type tensile machine (Tensilon Model UTM-1) at a cross-head speed of 4×10^{-3} mm/min. Some experiments were run at a reduced strain rate to examine the effect of strain rate on the SCC behaviour by using another slow straining machine[15] which was built in our laboratory and which has continuously controlled corss-head speeds over the range of 9×10^{-2} to 5×10^{-6} mm/min.

Polarization curves were measured by a potentiodynamic method. The potential was scanned at 21.7 mV/min either in the noble direction or in the active direction. Fracture surfaces were examined under the light microscope and SEM.

III. Results

Polarization curves

The potentiodynamic polarization curves of In 600 and Iy 800 in 50% NaOH at 140°C are shown in Figure 2. Included for comparison in the figure are those of A and S_2 treated specimen for both alloys. Corrosion potentials (E_c) of both alloys lie −1.10∼−1.15V (SCE). E_c of In 600 has a more noble value than that of Iy 800. It should be noted that the polarization curves of the specimens in A and S_2 conditions of heat treatment for both alloys show similar behaviour. The anodic polarization curves of both alloys show three distinct anodic current peaks and two passive regions below the oxygen evolution potential. The current density at the primary active peak for In 600 is higher than that for Iy 800 and the current density at the secondary active peak for Iy 800 is higher than that for In 600. The current density in the secondary passive region is generally higher than that in the primary passive region for both alloys and in the case of Iy-800, the current density in the secondary passive region is one order of magnitude higher than that in the primary passive region.

SCC tests

Figure 3 shows the dependence of susceptibility to SCC of A and S_2 treated specimens for both alloys in applied potential in 50% NaOH at 140°C with the

(14) H.L. Logan, *The Stress Corrosion of Metals*, John Wiley and Sons, New York (1966), 100.

(15) M. Takano, K. Teramoto, T. Nakayama and H. Yamaguchi, J. Iron, Steel Inst. Jpn., **65** (1979), 212.

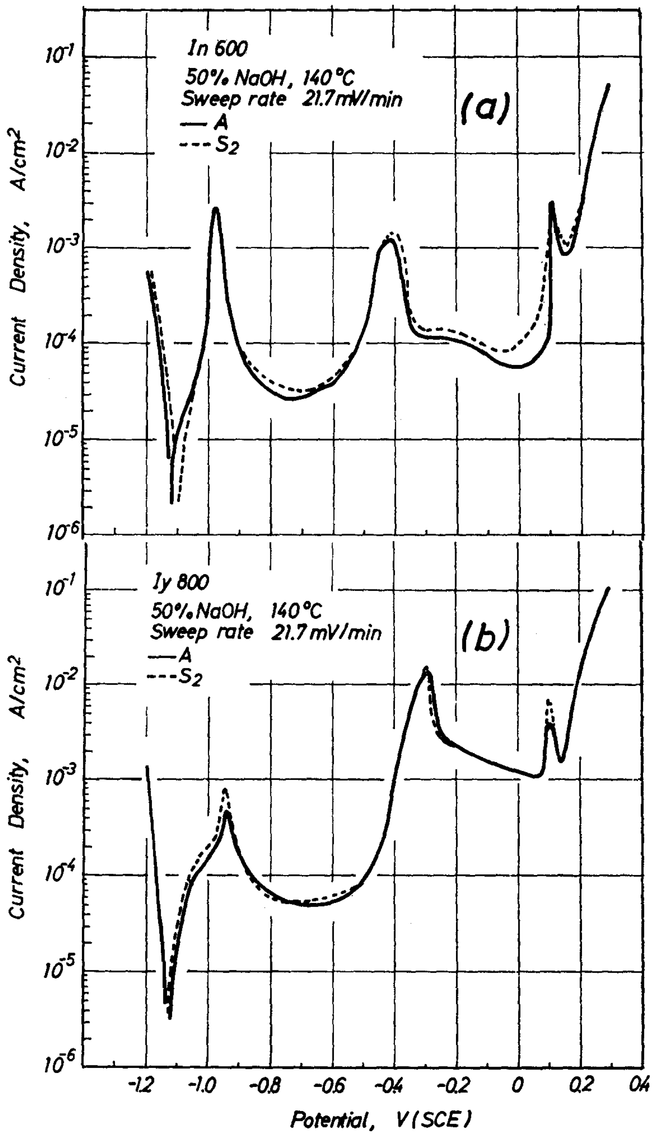


Fig. 2. Polarization curves in 50% NaOH at 140°C, (a) In 600, (b) Iy 800.

strain rate of $4\times10^{-3}\,\mathrm{min}^{-1}$. Included for comparison in the figure are those of various degrees of sensitization (S_1, S_3, S_4).

Solution annealed specimens: cracking occurred in the second passive region (−0.3V∼+0.05V) for both alloys. For Iy 800, cracking is more extensive in this region. Shallow cracks were observed for In 600 at −0.90 V which corresponds to the primary active-passive transition region in the polarization curve but no cracking was observed for Iy 800 in this region.

The cracking mode varied with applied potential in the secondary passive region; it was transgranular at 0V for both alloys. For In 600, intergranular cracking was predominant at higher or lower potentials than 0V. For Iy 800, intergranular cracking was also predominant at lower potential than 0V while transgranular cracking was still predominant above 0V. In the primary active-passive region, the cracking mode of In 600 was always intergranular. The potential for

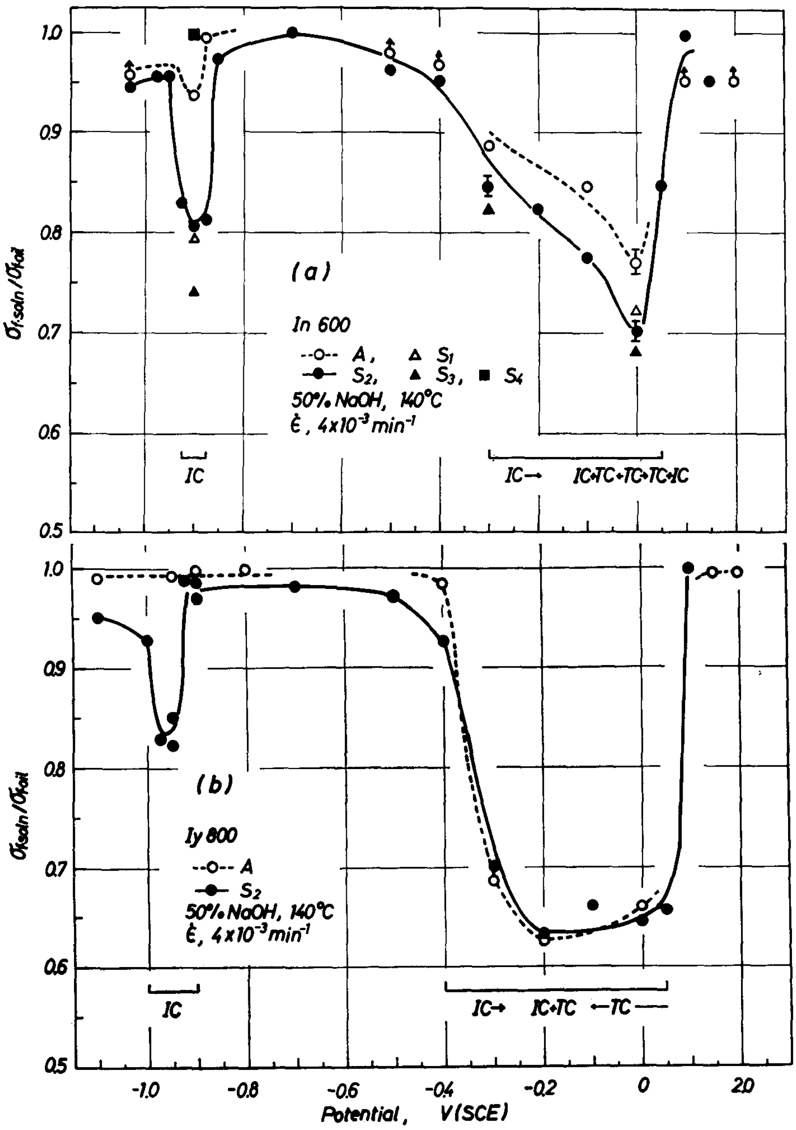


Fig. 3. Dependence of susceptibility to SCC of annealed and sensitized specimens on applied potential in 50% NaOH at 140°C and at $\dot{\varepsilon}=4\times10^{-3}$ min^{-1}, (a) In 600, (b) Iy 800.

maximum susceptibility to cracking was in the neighborhood of 0V.

Sensitized specimens: SCC occurred in both alloys under S_2 condition in the secondary passive region and in the primary active-passive region. In the former region, there is not so much difference in the potential region where cracking occurs and in the susceptibility to SCC between S_2 and A treated specimens for each alloy. The dependence of sensitization treatment for In 600 upon SCC behaviour is presented in Table 3 and in Figure 3(a). The order of sensitization treatment in decreasing tendency to promote SCC for In 600 was; $S_3>S_1\approx S_2>A>S_4$.

Cracking mode of sensitized specimen was always intergranular in the primary active-passive region and it was the same tendency as solution annealed

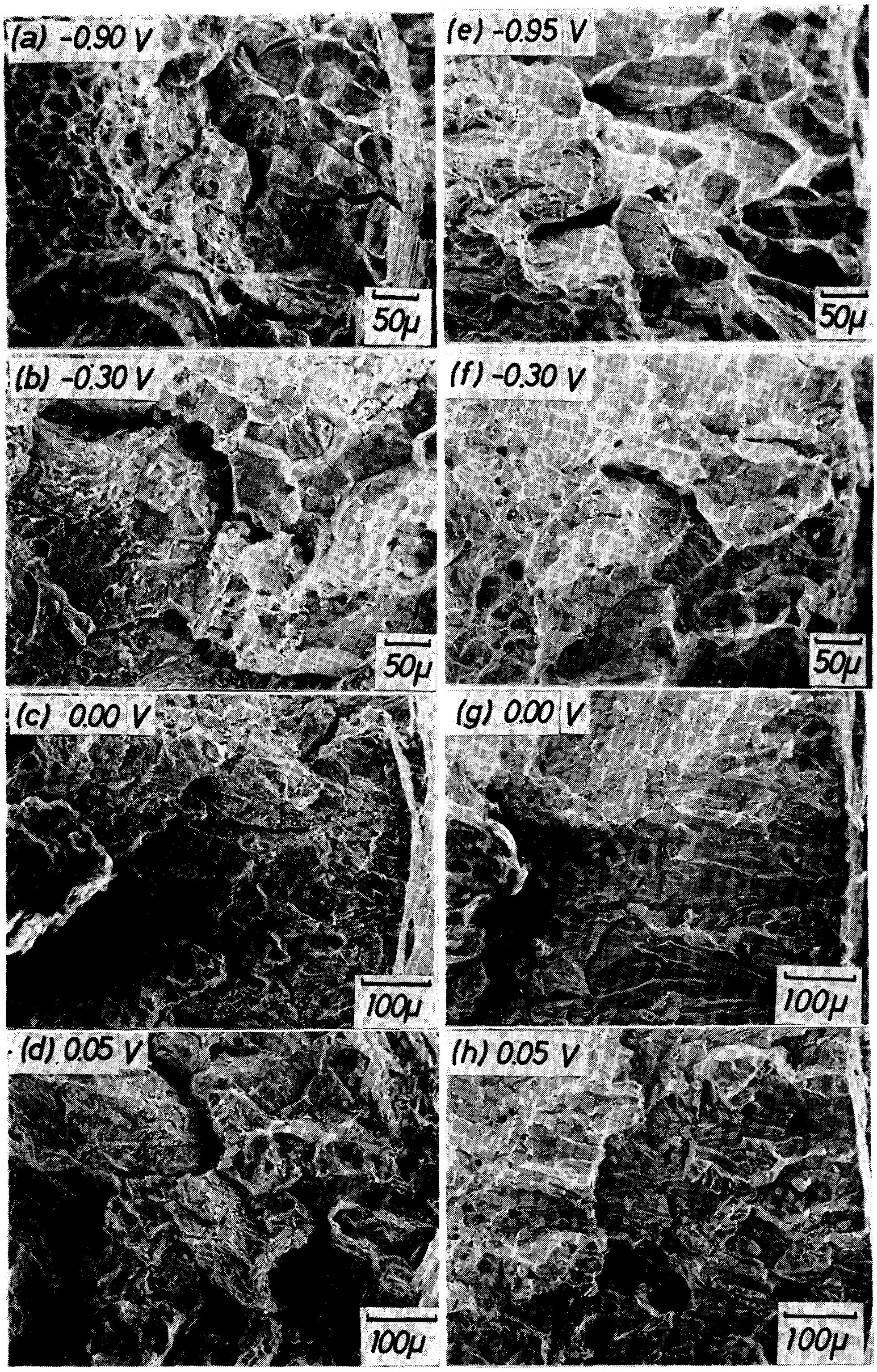


Fig. 4. Comparison of crack modes of sensitized specimens obtained in 140°C, 50% NaOH at different potentials, $\dot{\varepsilon}=4\times10^{-3}$ min. In 600; (a)~(d), Iy 800; (e)~(h).

specimen in the secondary passive region. Figure 4 shows fractographs of S_2 treated specimens for both alloys as a function of applied potential.

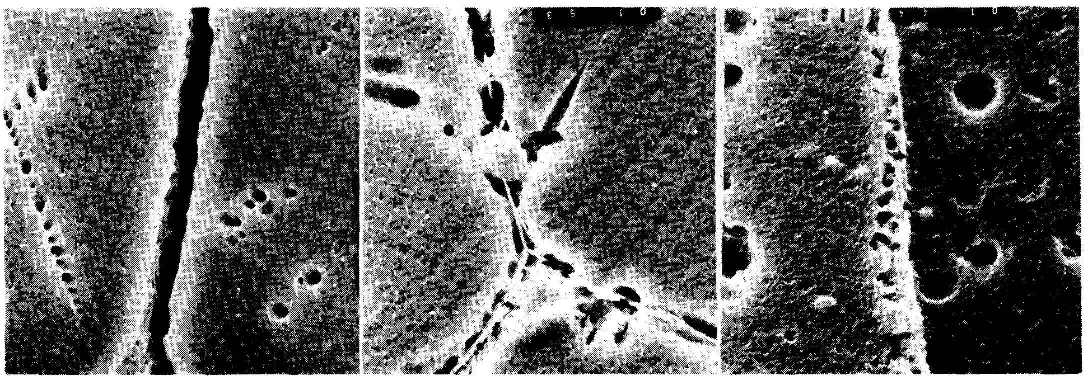
Table 3. Results of caustic SCC test at –0.9V and of modified Streicher test for In 600 alloy.

Heat treatment	Caustic SCC $\sigma_{f.soln}/\sigma_{f.oil}$	Modified Streicher test (ipm)	Crack
A (1100°C×30 min, W.Q)	0.95	0.002	NO
S_1 (A+610°C×2 hr)	0.80	0.020	YES, IC
S_2 (A+710°C×2 hr)	0.81	>0.720	YES, IC
S_3 (A+610°C×21 hr)	0.74	0.200	YES, IC
S_4 (A+710°C×21 hr)	0.98	0.015	NO

Unstressed specimens

In order to investigate the relationship between SCC behaviour and composition of surface film, an attempt was made to identify the surface film by means of a reflection electron diffraction method for both alloys in solution annealed materials which were exposed to 50% NaOH at 140°C for 24 hr in the four potential regions; the primary active and passive potentials and the secondary active and passive potentials. The results show that $Ni(OH)_2$ can exist at all potentials examined for both alloys and that metallic nickel was identified at the primary active potential and NiO at higher potentials in the secondary passive region. These results suggest no direct relationship between SCC behaviour and surface film composition. SEM examination of the surface of the above specimens revealed that no intergranular attack was observed.

In order to estimate the extent of sensitization, 10% oxalic acid etched surfaces of In 600 were examined in the SEM and a modified Streicher test was carried out. Photographs of different sensitized In 600 surfaces etched in 10% oxalic acid are shown in Figure 5. The continuous film of thin chromium carbides at the grain boundaries are seen in the S_3 treated specimen which is the most sensitive to cracking at –0.90V, while it appeared that the carbides at the grain



S1 1.6μ S3 2μ S4 1.6μ

Fig. 5. Photographs of different sensitized In 600 (unload) obtained in 10% oxalic acid etch.

boundaries dissolves away for S_1 treated specimen. Corrosion attacks occurred not only in grain boundaries but also within the grains in S_4 treated specimen in which no SCC was found at -0.90V. This might be due to fast diffusion of chromium to chromium depleted zones. Grain boundary attack of S_4 treated specimen appeared to be less severe than that of other specimens.

The results of the modified Streicher test for In 600 are presented in Table 3. All sensitized specimen showed intergranular attack. S_2 treated specimen was subject to gross intergranular attack and fell apart when compressed with the finger tip. Solution annealed specimen showed slight or no intergranular attack.

The results of SEM and LM examination of In 600 in the S_2 condition showed that no intergranular attack was observed at -0.90V and -0.30V where intergranular SCC occurred.

Effect of strain rate

The SCC behaviour of the alloys has been studied at various strain rates to investigate the effect of the mechanical factor in SCC. Experiments were carried out at a controlled potential of 0V where solution annealed specimens for both alloys were found to be the most susceptible to cracking. The results are shown in Figure 6. The susceptibility to cracking is strongly dependent upon strain rate and has a maximum value at $\dot{\varepsilon}=10^{-4}\text{min}^{-1}$. SEM examination of fracture surfaces for both alloys are presented in Figure 7. It should be noted that cracking mode was

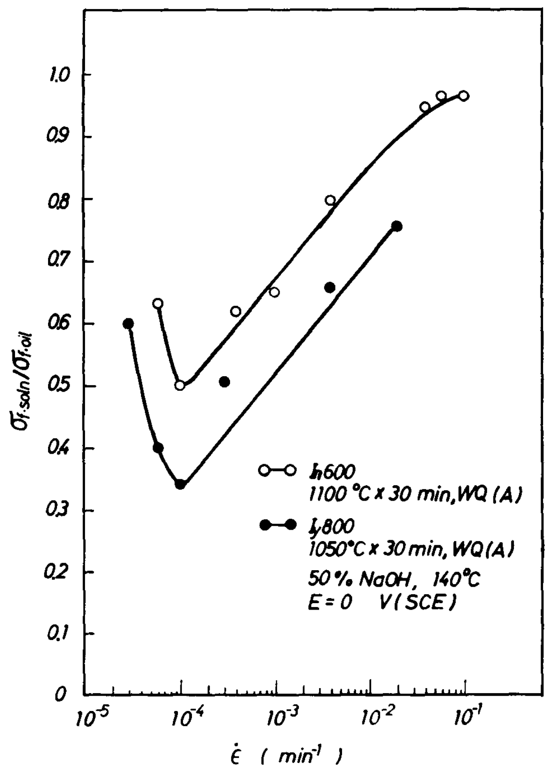


Fig. 6. Effect of strain rate on susceptibility to SCC in 50% NaOH at 140°C and at 0V (SCE) for annealed In 600 and Iy 800.

transgranular at high strain rate, while it changed to intergranular with decreasing strain rate. For example, In 600 shows transgranular cracking at $\dot{\varepsilon}=4\times10^{-2}$ min^{-1} and intergranular at $\dot{\varepsilon}=1\times10^{-4}$ min^{-1}.

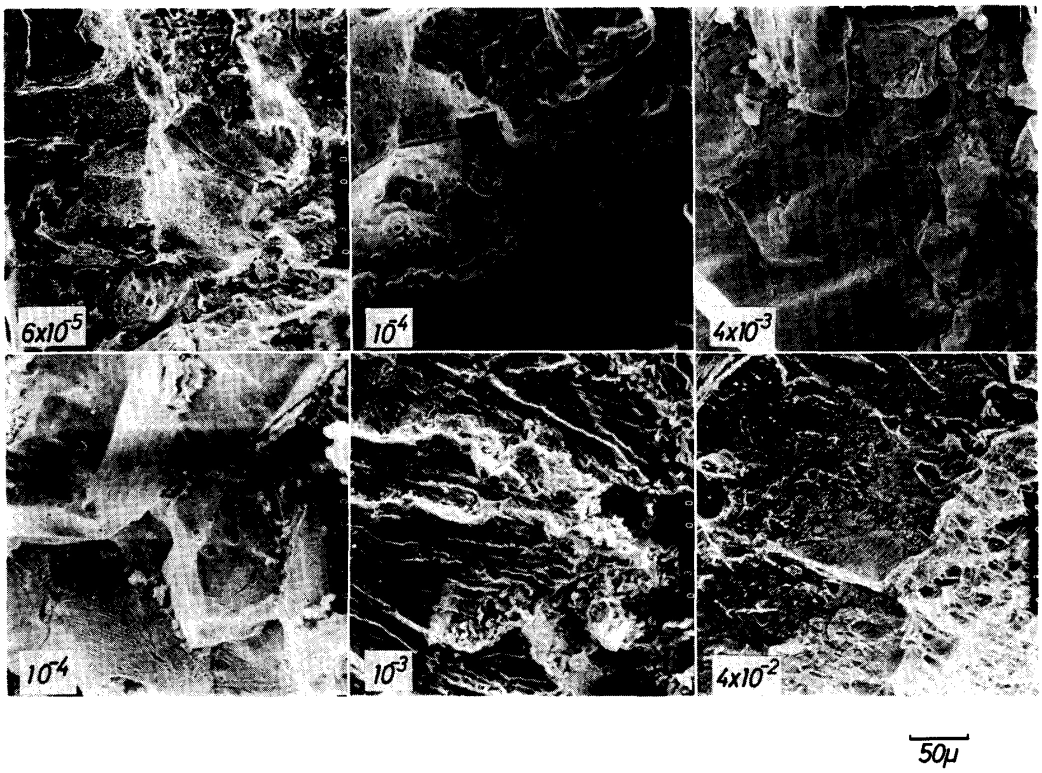


Fig. 7. Comparison of crack modes of annealed specimens in 140°C, 50% NaOH at 0V (SCE) and at different strain rates. Upper; In 800, lower; In 600.

IV. Discussion

SCC

In order to study SCC behaviour, it is necessary to take into account three factors; material, environment and stress or strain. In this study, the effect of applied potential (environment) on the susceptibility to SCC has been investigated at a controlled strain rate ($\dot{\varepsilon}=4\times10^{-3}$ min^{-1}).

Solution annealed specimens; From Figures 2 and 3, the characteristics in the potential regions where SCC occurs are as follows. In the primary active-passive transition region; cracking occurred only in In 600 which showed a higher current density in the polarization curve. In the secondary passive region; both alloys were susceptible to cracking and Iy 800 which showed higher current density was more sensitive to cracking. These facts agree with the previous report(16) that SCC is expected in the potential regions where the surface film shows kinetic instability in the polarization curve. However, it is considered that this concept means that

(16) R.W. Staehle, Reference 1), 180.

whether SCC occurs or not might be strongly dependent upon the kinetics of formation of slip caused by dynamic strain and of the subsequent repassivation process in the environment. In this study, it appears that the two potential regions of the primary active-passive transition and the secondary passive regions would satisfy the kinetic conditions at $\dot{\varepsilon}=4\times10^{-3}$ min^{-1} and therefore SCC occurred.

The main chemical composition of the surface film for both alloys was $Ni(OH)_2$ over the range of anodic potential and NiO in the seconary passive region. These facts indicate that there would be no particular relation between the surface film composition and susceptibility to cracking. Although there has been a report [17] that indicated a particular relation between surface film composition and SCC, it does not appear to be essential for the mechanism of SCC. SEM and LM observations revealed that no intergranular attacks were shown in unstressed specimens which were exposed for 24 hr at the potentials where SCC occurred. This fact suggests that SCC for solution annealed material is not "stress assisted cracking", in which stress promotes grain boundary attack.

The primary active-passive region and the secondary passive region where SCC took place correspond to that of the dissolution of Ni, Fe and Cr, respectively [18,19] and the tertiary active dissolution peak (it appears at about 0.1V) corresponds to the dissolution of Ni[18]. In primary active-passive region, In 600 is only susceptible to intergranular cracking. This might be due to the fact that In 600 has a much higher nickel content than Iy 800. It is considered that since the chromium content of both alloys is not so much different as the nickel content, both alloys in the secondary passive region are subject to SCC. That Iy 800 is somewhat more susceptible to cracking than In 600 might be due to the fact that the chromium content of Iy 800 is slightly higher than that of In 600.

Sensitized specimens: The large difference in SCC behaviour between S_2 and A conditions exists in primary active-passive region (Figure 3). Iy 800 in the S_2 condition failed in this region but no cracking occurred in the A condition. Similarly In 600 in the S_2 condition was more susceptible to cracking than in the A condition. Since chromium depleted zones are formed at the grain boundaries for S_2 treated specimen, the nickel content in this zone could be relatively higher than that in the matrix. As primary active-passive region corresponds to nickel dissolution potential and easier slip tendency in chromium depleted zone, it appears to be understood that intergranular SCC occurs for both alloys in this region.

The susceptibility to cracking for various heat treated specimens is not always coincident with the tendency of intergranular attack evaluated from the modified Streicher test as shown in Table 3. The S_2 treated specimen, which is very sensitive to intergranular attack, shows the same susceptibility to intergranular SCC as S_1

(17) J. Flis, Corros. Sci., **15** (1975), 553.
(18) M.F. Long, A.K. Agrawal and R.W. Staehle, *High Temp. High Press. Electrochem. in Aqueous Soln., NACE-4*, ed. by R.W. Staehle, D. de G. Jones and J.E. Slater, (1973), 524.
(19) K. Hashimoto and K. Asami, Corros. Sci., **19** (1979), 427.

treated specimen which shows very slight intergranular attack. It is considered that a severely sensitized specimen like S_2 for In 600 might produce a blunt crack in hot caustic solution resulting in less sensitivity to intergranular SCC than moderate sensitized specimen like S_3. The resistance to SCC for S_4 treated specimen may be due to the formation of large amounts of $Cr_{23}C_6$[(20)] not only at grain boundaries but also within grains. It is considered that these chromium carbides might not affect the corrosion behaviour in the primary active-passive region, but they could make a barrier against the moving dislocations under stress, further this specimen has no chromium depleted zone because of rediffusion of chromium. Therefore S_4 treated specimen shows resistance to SCC.

Taking into account that the secondary passive region corresponds to the region for dissolution of chromium, as mentioned before, it is thought that grain boundary under sensitized conditions dissolved more slowly than that under solution annealed condition. As seen in Figure 3, however, experimental results show that the susceptibility to SCC for both alloys in the sensitized condition is almost the same as that in the solution annealed condition. Although the fact that Iy 800 is more susceptible to SCC than In 600 for all specimens tested could be explained by chromium content, the reason that there is not so much difference in susceptibility to SCC and crack mode for each alloy is not clear. This might depend upon strain rate used.

Effect of strain rate

Figure 6 shows strain rate dependence for solution annealed specimens on susceptibility to cracking. It is seen that the maximum SCC susceptibility exists at $\dot{\varepsilon}=10^{-4}$ min^{-1} for both alloys, which suggests that SCC behaviour depends strongly on slip step formation rate and repassivation rate of the fresh surface. The susceptibility to cracking again decreases with further decrease of strain rate. This might be due to the assumption that repassivation rate could exceed slip formation rate under above condition. Stress-time curves during the SCC tests for solution annealed In 600 are shown in Fugre 8. Comparing the curve at $\dot{\varepsilon}=10^{-4}$ min^{-1} with the curve at $\dot{\varepsilon}=6\times10^{-5}$ min^{-1} in this figure, it can be seen that the resistance to SCC at $\dot{\varepsilon}=6\times10^{-5}$ min^{-1} is higher than that at $\dot{\varepsilon}=10^{-4}$ min^{-1}, in spite of the fact that at the strain rate of 6×10^{-5} min^{-1}, the specimen is exposed for a longer time at a higher stress level where failure occur at the strain rate of 10^{-4} min^{-1}. This result shows that the important factor in SCC is not stress (or load) itself but is the appropriate slip formation rate in the system.

Crack morphology

SCC morphologies for both alloys under A and S_2 conditions in the secondary passive region changed from transgranular cracking around 0V where the passive

(20) C.S. Tedman, *Jr.* and D.A. Vermilyea, Corrosion, **27** (1971), 376.

film appeared to be the most stable to intergranular cracking at the potentials where the passive film appeared to be unstable. Transgranular cracking was predominant at 0V and at $\dot{\varepsilon}=4\times10^{-3}$ min^{-1} but intergranular cracking became predominant with further decrease of strain rate at the same potential. In the latter case, repassivation ability would be sufficient to inhibit the corrosion of the subsequent slip steps within grains produced by straining, resulting in intergranular cracking. This situation agrees with previous result[21]. However, like in the former case, the phenomenon that intergranular cracking becomes predominant with decrease of passivation ability would not agree with the results shown in previous reports[22,23].

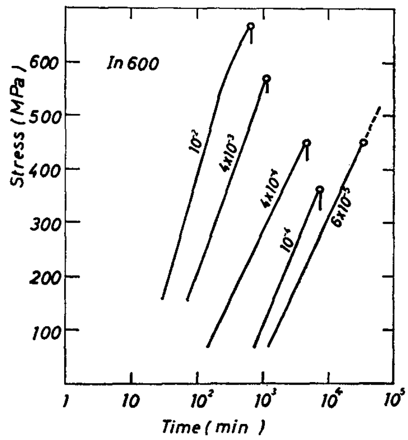


Fig. 8. Stress-time curves during extension tests for annealed In 600 in 50% NaOH at 140°C at different strain rates.

Correlation with previous work

It is important to compare the results obtained in the present study with previous work in SCC for high nickel alloys/high temperature caustic solutions in order to clarify the mechanism of SCC and establish the SCC test method.

In the case of solution annealed specimens, In 600 was only subject to intergranular cracking in the primary active-passive region in this experiment. This result agrees with those of Theus[11] and McIlree and Michels[8]. They found that in oxygenated caustic solutions, solution annealed In 600 failed from intergranular SCC but Iy 800 was resistant to SCC. However, the result that SCC occurred for both alloys under sensitized condition in the same potential region in this experiment is not in agreement with the previous papers[5,11,13] which shows the sensitized In 600 reduces the severity of SCC in caustic solutions.

Considering that the secondary passive region corresponds to a potential of chromium dissolution[18,19], one could expect that sensitized specimens (S_2) are more resistant to cracking than annealed specimen (A). In 600 and Iy 800 under

(21) M. Takano, Trans. Jpn. Inst. Met., **18** (1977), 787.
(22) M. Takano, Corrosion, **30** (1974), 441.
(23) M. Takano and R.W. Staehle, Trans. Jpn. Inst. Met., **19** (1978), 1.

S_2 condition, however, showed almost the same susceptibility to SCC as material A as seen in Figure 3.

Tsujikawa *et al.*[24] have conducted corrosion fatigue tests of In 600 in 1 N H_2SO_4 at room temperature. They reported that intergranular cracking occurred at a high potential of 1.02 V SCE, and that sensitized material was more resistant to cracking than annealed material. These results represent the characteristic of SCC in service environment and in high temperature high pressure caustic environments in laboratories[5,8,11,13]. From above resuls, they concluded that SCC of In 600 and service environment would occur in high potential region (around IV SCE) of chromium dissolution. Long *et al.*[18] have shown that the potential for chromium dissolution in highly concentrated caustic solutions shifted in the less noble direction with increasing temperature, especially above 240°C the potential became less noble than that of nickel dissolution. These results suggest that SCC of In 600 in service environment and in concentrated caustic solution at high temperature high pressure would occur at the potential of chromium dissolution, which is less noble potential than that of nickel dissolution.

In the present study, which was conducted at 140°C under atmospheric pressure, no chromium dissolution occurred at the less noble potential, so the same condition as that in service environment could not be obtained. If only chromium dissolution would be considered, then SCC in service environment would also be able to reappear under a suitable crosshead speed test in a potential region from secondary active-passive transition to secondary passive, where chromium dissolution occurred.

V. Conclusions

SCC of Inconel 600 and Incoloy 800 in aerated 50% NaOH at 140°C was investigated using a slow straining method under electrochemically controlled potentials. The following conclusions may be drawn from the results presented.

1. SCC took place in the primary active-passive transition region and in the secondary passive region in polarization curves at $\dot{\varepsilon}=4\times10^{-3}$ min^{-1}. The primary active-passive transition region; intergranular SCC was found in solution annealed In 600 and in both alloys under sensitized condition. The secondary passive region; SCC was found in both alloys and there was not so much difference in susceptibility to SCC and in crack mode for annealed and/or sensitized specimens of each alloy. Transgranular cracking occurred around 0V corresponding to the potential for passive film stability and intergranular cracking occurred at a higher or lower potential than 0V, corresponding to the potential for passive instability. 2. In the strain rate range from 6×10^{-5} to 10^{-2} min^{-1} for annealed materials at 0V maximum susceptibility to SCC was obtained at $\dot{\varepsilon}=10^{-4}$ min^{-1} for

(24) S. Tsujikawa, Y. Hisamatsu, T. Endo and K. Furuse, JSCE. Corrosion Research Meeting (1977), 70.

both alloys and intergranular cracking became predominant as the susceptibility to cracking increased. **3.** The important factor in SCC is not stress itself but is the appropriate formation of dynamic slip.

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