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STRESS CORROSION CRACKING OF AUSTENITIC STAINLESS STEELS IN CHLORIDE ENVIRONMENT

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ABSTRACT
This study was carried out to assess the effect of applied potentials on stress corrosion cracking (SCC) of Austenitic Stainless Steels (ASS), Type 304L in a potentiostatically controlled chloride environment at ambient temperature. The stress corrosion cracking tests were carried out on annealed ASS using a Slow Strain Rate Test (SSRT) technique in sodium chloride solution acidified with hydrochloric acid at ambient temperature. Analyses of failed specimens were carried out using optical microscope. The study showed that plastic elongation, ultimate tensile strength and time to failure decrease as the applied potential increases during the slow strain rate test. The study showed that immunity of ASS to chloride SCC was improved when the electrochemical potential was maintained in the primary passive potential range. Analysis of failed samples showed evidences of SCC.

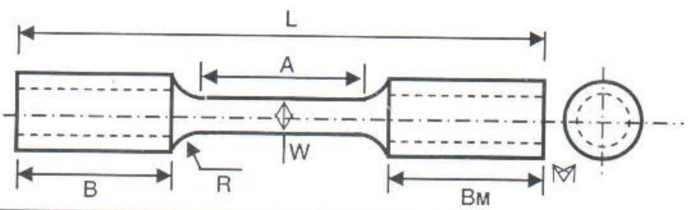
INTRODUCTION
The performance of Austenitic Stainless Steels (ASS) is considered satisfactory due to their good combination of high strength, ductility and general corrosion resistance even at elevated temperature(Couvant *et al.*; 2005, Garda, 2000).They are widely used in process plants, cryogenic plants, food industry and nuclear industry(Arioka *et al.*, 2007). However, austenitic stainless steels are susceptible to localised corrosion failure including pitting corrosion, crevice corrosion and stress corrosion cracking(Lai *et al.*, 2009, Garcia *et al.*, 2002).

Stress Corrosion Cracking (SCC) occurs when a susceptible material under tensile stress state is exposed to corrosive environment (Sedriks, 1996). SCC is such a dangerous failure that may occur within design stress and in an environment that may not be considered aggressive. The SCC occurs at a stress intensity factor which is well below the critical stress intensity factor determined by fracture mechanics(Craig and Lane, 2005). The damage caused by SCC occurs in stages namely; the crack nucleation stage which may occur at the root of the corrosion pit or area of stress concentration, the transition stage and the crack propagation stage. The crack propagation mode may be intergranular or transgranular, if it propagates along the grain boundaries or through the grain matrix, respectively (Sedriks, 1996). The SCC susceptibility of material can be influenced by factors which directly affect the three prerequisites for SCC namely: susceptible material, applied tensile stress and the presence of corrosive environment.

The long held tradition was that SCC does not occur in non-sensitised ASS at temperature below about 60°C in near neutral chloride solutions(Sedriks, 1996). It is now known, that chloride SCC can indeed occur at ambient temperature in a severe test conditions such as in slow strain rate tests and U-bend tests. Ghosh and Kain (2010) reported the observation of transgranular cracking in U-bend tests carried out on annealed, machined and 10% cold worked, 304L samples tested in 1M hydrochloric acid at room temperature. The objective of this study was to assess the SCC of ASS in a potentiostatically controlled chloride environment at ambient temperature and to investigate the effects of Applied Potentials (AP) on SCC susceptibility of the material.

Experimental Procedure SSRT in Potentiostatically Controlled Chloride Environment
The material used was austenitic stainless steels, Type 304L with chemical composition in Table 1.

The ASS plate (300mm × 100mm × 30mm) as received was initially solution annealed at 1050°C for 30 minutes as described elsewhere (Garcia *et al.*, 2002, Kain *et al.*, 2004, Ahmed *et al.*, 2013). The slow strain rate test was carried out on 100kN Instron 5500 Reequipped with Bluehill software. The schematic diagram of the tensile specimen is shown in Figure 1. Typical sample name used include ANL, interpreted as annealed sample made from longitudinal direction of as-received material. The sample preparation included wet grinding and polishing with carbide paper in order of 400, 600, 800 and 1200 grit sizes. The sample was then degreased with acetone and later rinsed with deionised water. The sample surface area to be exposed to electrolyte was measured for calculation of corrosion current density, and other area insulated from environment with wax.



Dimensions (mm)	
A	26
B	20
L	82
R	8
W	8

Figure 1: Schematic Diagram of the Slow Strain Rate Test Specimen and Dimension

Table 1: Chemical Composition (wt%) of as-received Austenitic Stainless Steel used

C	Cr	Mn	N	Ni	P	S	Si
0.030	18.387	1.804	0.086	8.133	0.034	0.005	0.411

The SCC susceptibility test in a chloride environment was carried out using SSRT method in a solution containing 1M sodium chloride acidified to the pH of 1.5 with hydrochloric acid. The volume of custom made SCC cell used was 250ml. The chloride environment was potentiostatically controlled with the aid of an ACM instruments Research Potentiostat. The Working Electrode was connected through a stainless steel wire spot welded to the test sample. The Auxiliary Electrode (AE) was connected to cathode made from 1m Platinum wire wound round a plastic cylinder placed within the SCC cell. The Saturated Calomel Electrode was used as the Reference Electrode (RE). Tests were carried out at three different potentials namely: -125mV, -100mV and -50mV with respect to the SCE at ambient temperature. Two of the applied potentials (-100mV and -50mV) fall within the metastable pitting potential region, while -125mV corresponds to the passive potential that allows the material to maintain a protective corrosion resistance passive film. The test was carried out at the cross-head speed 6.3 x 10^{-6}mm/s in line with the ASTM standard (2000).

Post-Mortem Optical Microscopy

The post-mortem optical microscopy was carried out on failed samples using an Olympus light microscope installed with AxioVision software. The gauge length surface area of failed samples was observed to determine the extent of cracks after the SCC test, and to be used as guide for sectioning across cracks for the subsequent SEM analysis.

RESULTS AND DISCUSSION

Effect of Applied Potentials on SCC Susceptibility

The results of SCC tests are shown in Figure 2. The plots of applied stress against engineering strain and the plot of current density against Time To Failure (TTF) are shown in Figure 2A & B respectively. The effect of AP was quite obvious from the Plastic elongation (ductility), the Ultimate Tensile Strength (UTS) and from TTF.

The ductility and UTS of materials decreased as the AP was increased. Similarly, TTF also decreased as AP increased. The decrease in the TTF and mechanical properties of the material may be attributed to significant increase in the corrosion rate caused by increase in the AP. The plots of corrosion current density against TTF in Figure 2B showed that corrosion current density increased as AP was increased. The increased corrosion current density with TTF was attributed to crevice corrosion between the elongated sample and the SCC cell. The plot of corrosion current density against TTF also showed that first current peak coincided with onset of plastic elongation. The curve showed a repeated pattern of sudden increase in corrosion current density followed by gradual decay with time, which is characteristic of SCC failure.

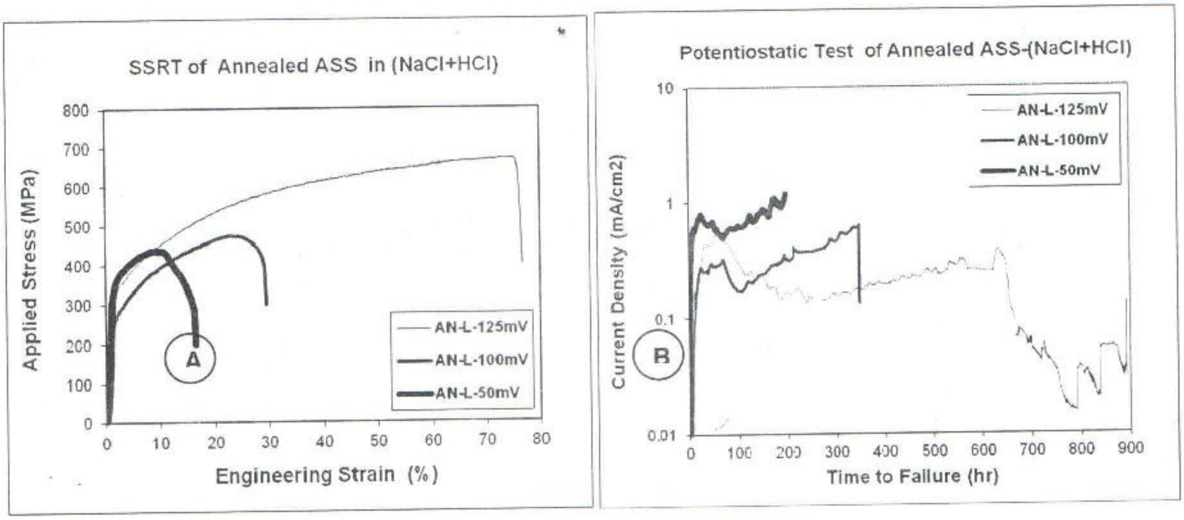


Figure 2: SSRT Curves for Samples Tested at Strain Rate of 2.4×10-7s-1, pH of 1.5, and Applied Potentials of -125mV, -100mV and -50mV at 25°C: Applied Stress Vs Strain (A) and Current Density Vs Time to Failure (B)

The test at AP of -125mV took 891 hours before failure occurred. This was obviously too long, while failure occurred too quickly at 197 hours when the AP was increased to -50mV (see Figure2) due to accelerated corrosion process. The AP of -125mV represents the passive potential where corrosion process was expected to be at minimal level. Hence, the SCC test was largely dominated by mechanical process. Comparing and contrasting the effects of AP have shown that SCC susceptibility can be mitigated and held under control if electrochemical potential is maintained in the passive potential region. On the other hand, when AP of -100mV was used, failure time was considerably reduced at 348 hours. Therefore, tests at -100mV appeared to strike a balance between competing processes dominated by corrosion and mechanical effects. The correlation of plastic elongation with AP is shown in the Figure 2. The significant decrease in plastic elongation (ductility) was probably caused by increase in the corrosion rate as the AP was increased from -125mV to -50mV.

Post-Mortem Optical Microscopy of the SCC Samples

The results of optical microscopy at different AP are shown in the Figure. The three samples failed at a similar location, probably facilitated by crevice corrosion between the cell and sample due to reduction in cross-sectional area as the sample was elongated. However, the cracks and corrosion pits shown in the Figure 3 were found on the gauge length area. All three samples showed evidence of cracks perpendicular to the loading (longitudinal) direction. The cracks on sample tested at applied potential -50mV are quite faint and thinner than other two samples (Figure 3 C). This probably occurred due to lower plastic elongation and accelerated corrosion process that dominated the test. In addition to the observed cracks on the samples, corrosion pits of about 0.8mm diameter were also observed on the sample (see Figure D).

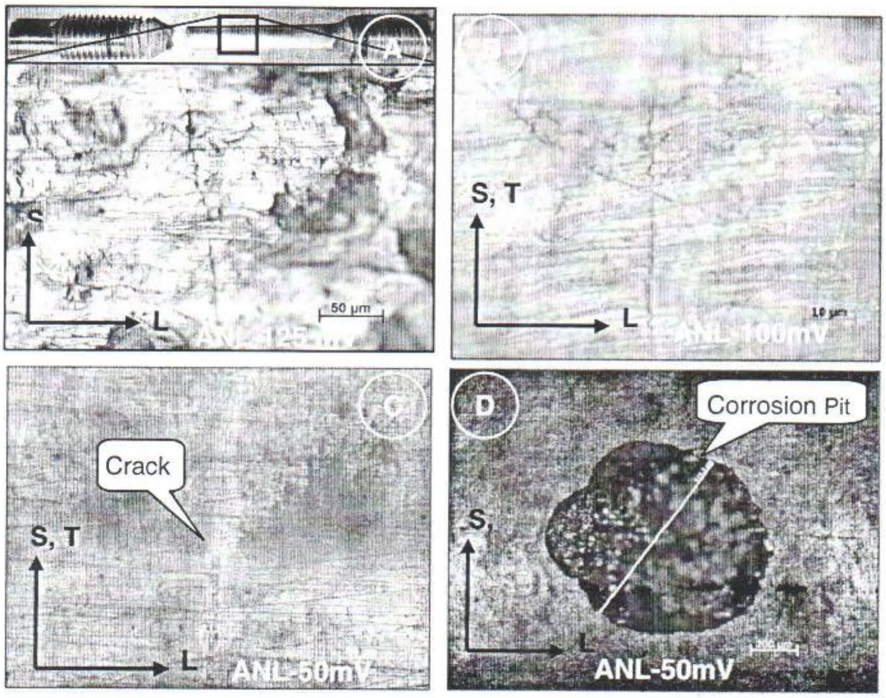


Figure 3: Optical Micrographs of Annealed Samples Tested at Strain Rate of 2.4×10-7s-1, pH of 1.5, and Applied Potentials of: -125mV (A), -100mV (B) and -50mV (C and D) at Ambient Temperature.

CONCLUSIONS

The following are conclusions made from the study of the effect of applied potential on the SCC susceptibility of austenitic stainless steels, Type 304L:

1. The SCC study showed that plastic elongation, UTS and TTF decreased as the applied potential was increased during the SSRT carried out on annealed samples.
2. The decrease in SCC with applied potential was attributed to significant increase in corrosion rate due to transpassive dissolution of primary passive film.
3. The immunity of ASS to chloride SCC was improved when the potential was maintained at primary passive potential range. The improvement was attributed to formation of stable chromium-rich passive film that continuously help to heal the substrate when crack opens.
4. The optical micrographs of the failed samples showed that cracks propagated perpendicular to loading axis and cracks observed were predominantly transgranular SCC.

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