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Effect of surface machining and cold working on the ambient temperature chloride stress corrosion cracking susceptibility of AISI 304L stainless steel

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1. Introduction

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ABSTRACT

Effect of plastic deformation induced by cold rolling or surface machining on the susceptibility to chloride-induced stress corrosion cracking at ambient temperature of 304L austenitic stainless steel was investigated in this study. The test material was subjected to three treatments: (a) solution annealed, (b) cold rolled and (c) surface machined to induce different levels of strain/stresses in the material. Subsequently constant strained samples were produced as per ASTM G30 for each condition and these were exposed to 1 M HCl at ambient temperature until cracking occurred. Subsequently the cracked samples were characterized using stereo microscopy, optical microscopy and atomic force microscopy to understand the effect of microstructural changes produced by straining on the susceptibility to stress corrosion cracking at ambient temperature. Strained surface produced by machining accelerated the process of crack initiation resulting in densely distributed shallow surface cracks in a very short period of time as compared to solution annealed and cold worked sample. Crack propagation in cold worked sample was along the slip lines and cracking occurred much earlier than in the solution annealed sample.

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Instances of chloride-induced stress corrosion cracking (SCC) in components of 300 series austenitic stainless steels at ambient temperature in the past two decades [1–3] have led to considerable research in this field. The effect of environmental parameters like pH, chloride content, temperature and relative humidity on the SCC susceptibility, crack propagation rate, and time to failure have been critically examined. These studies clearly delineated two necessary conditions for ambient (or below 60 °C [4]) temperature SCC; (a) pH in the range of -0.5 to 0.5 and (b) chloride ion concentration from 0.5 to 5 M [5-10]. Since stainless steels under these conditions are in active state, this mode of SCC is accompanied by considerable general corrosion with the corrosion rate ranging typically in mm per year [11]. Extensive low temperature stress corrosion cracking in a large number of End Shields have been observed [12] during extended periods of storage in open atmospheric conditions. These were partially fabricated and stored at ambient temperature in coastal regions for 6-8 years. The cracks in this case were shallow with a depth not more than 2-3 mm. These End Shields are meant for use in pressurized heavy water reactors (PHWR) and are made from 304L austenitic stainless steels. These components during fabrication, undergo a number of stages like heavy machining, welding (in many cases in constraint geometry), etc. which result in heavy residual stresses and retained strain in the material. The cold plastic deformation induced as a result of these processes leads to important microstructural modifications in the material. The effect of cold rolling on sensitization and IGSCC of AISI 304L aged at 500 °C has been reported [13]. It is now well established that in high-temperature (oxygenated) water environments of nuclear power reactors at ~288 °C, work hardening induced by manufacturing processes leads to intergranular stress corrosion cracking of even non-sensitized austenitic stainless steels [14]. However, the effect of work hardening and its resulting residual stress and strain on the ambient temperature stress corrosion cracking susceptibility of austenitic stainless steel is yet to be clearly demonstrated and understood. The aim of this paper is to correlate the effect of microstructural changes brought about by machining and cold working to the chloride-induced stress corrosion cracking susceptibility and nature of cracking of 304L austenitic stainless steel at ambient temperature (at room temperature, ~300 K).

2. Experimental techniques

2.1. Materials and methods

To study the effect of different levels of residual stress/strain induced in the material during different stages of fabrication on its SCC susceptibility, the as-received stainless steel 304L (0.023C, 17.14Cr, 9.13Ni, 0.29Si, 0.99Mn, 0.035P, 0.004S, in wt%) has been

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given three different treatments; (a) solution annealed (held at 1298 K for 15 min in vacuum of 10^{-6} Torr and water quenched), (b) cold worked: reduction in thickness of 10% by rolling subsequent to solution annealing and (c) surface machined (as-received sample machine ground to remove a depth of 0.5 mm from the surface). Subsequently the hardness of the surface of each of the samples was measured by microhardness tester using a load of 200 gf and dwell time 10s. The wt% of martensite produced in the material during cold working and surface machining was measured by ferrite meter. Constant strained samples (U bend: width 5 mm, thickness 1.5 mm, radius of curvature 10 mm) prepared as per ASTM G30 [15] were made out of solution annealed, machined and cold worked 304L stainless steel respectively for stress corrosion cracking studies. The standard ASTM G30 procedure for preparing U bend sample gives the average strain in the sample to be T/2R (where T is the thickness and R is the radius of curvature of the U bend), and assumes it to be constant throughout the sample. The average strain in these samples was 10%. The stress distribution in the U bend sample is as follows: the upper membrane of the U bend sample is under tensile stress, the lower and the inner most membrane is under compressive stress and the mid-region in between the upper and lower membrane lie the neutral zone where the tensile and compressive stresses are nullified.

2.2. Stress corrosion cracking test

Ambient temperature stress corrosion cracking susceptibility of the solution annealed, surface machined and 10% cold worked 304L stainless steel sample was studied by exposing the constant strained samples to 1 M HCl solution at room temperature (\sim 300 K). Selection of the concentration of this environment was based on the previous work [16,17], which gives the range of concentration of hydrochloric acid that induces stress corrosion cracking in 304L austenitic stainless steel at ambient temperature. It has been reported that at concentrations lower than 0.6 M HCl, only pitting (and no SCC) occurs while at concentrations higher than 1.2 M HCl, uneven general corrosion occurs instead of cracking [16]. The strained samples were taken out from the environment periodically (after every 24h), washed with water thoroughly, dried with acetone and the upper surface of the U bend (which is under tensile stress) was examined under stereo microscope at magnifications $10-175 \times$ for detecting cracks if present. The experiment was stopped once cracks were detected. The assessment of the susceptibility of austenitic stainless steel to ambient temperature stress corrosion cracking in solution annealed, cold worked and surface

machined conditions were based on the time for initiation of cracks on the surfaces of the U bend samples.

2.3. Characterization

Detailed characterization of the stress corrosion cracks generated on the surface in terms of mode of cracking and the microstructural features favoring preferential crack propagation in the samples under different levels of residual stress/strain were determined by the cross-sectional examination of the exposed U bend samples using characterization tools like optical microscopy and scanning probe microscopy. After cracks were detected on the outer surface of the U bend samples exposed to corrosive environment at room temperature, the cross-section of the samples were mounted, polished up to 1 μ m surface finish, electrochemically etched with oxalic acid for 15 s at 1 A/cm² and were examined under a optical microscope for cracks. Topographic imaging of the cross-section of the exposed samples was performed using atomic force microscopy (AFM) in semi-contact mode using silicon tip.

3. Results and discussion

Extensive cracking was observed on the upper surface (outer membranes under tensile stress) of all the U bend samples under different stress levels exposed to 1 M HCl but the time to cracking was minimum in case of surface machined sample followed by cold worked and finally solution annealed sample. The hardness of solution annealed, cold worked and machined 304L stainless steel was found to be 180HV, 250HV and 320HV, respectively.

The time of detection of cracking (as detected by stereo microscopic examination) was recorded in each case and it was approximately 170 h for solution annealed samples, 96 h for the cold worked samples and 48 h for the machined samples. A typical stereo micrograph of the cracked surface of U bend region of the solution annealed sample is shown in Fig. 1(a). As is evident from the figure, cracks initiated simultaneously at different sites, grew and finally coalesced to produce a large crack, which is the characteristic of stress corrosion cracking. The cracks on the surface of the U bend run parallel to each other. In addition to cracking, general corrosion of the surface also occurred which is in line with previous observations in literature [12,16,17] and is attributed to the highly aggressive nature of the environment. Cross-sectional examination showed that cracking is transgranular in nature for solution annealed, machined and cold worked sample and that it initiated from the surface under tensile stress and the cracks moved down



Fig. 1. (a) Stress corrosion cracks on the upper surface of the constant strained sample of SS 304L on exposure to 1 M HCl at room temperature for 144 h. (b) Cross-sectional microstructure showing transgranular stress corrosion cracking in thickness direction.

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