Materials Science Forum *Online: 2011-12-06*
ISSN: 1662-9752, Vols. 702-703, pp 685-688
doi:10.4028/www.scientific.net/MSF.702-703.685

IGSCC of non-sensitised austenitic stainless steels in BWR environment – Effect of deformation mode on grain boundary susceptibility

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Keywords: Stainless steel, Non-sensitised and strain hardened, IGSCC, Crack growth rate, BWR environment

Abstract. Intergranular Stress Corrosion Cracking (IGSCC) of austenitic Stainless Steels (SS) in Boiling Water Reactor (BWR) environment is generic in nature in both the sensitised and the non-sensitized conditions. IGSCC in non-sensitized austenitic SS in the strain hardened condition has been reported without any grain boundary chromium depletion or impurity segregation. The present study ascertains the reason for IGSCC in BWR environment in non-sensitized condition and investigates the effect of nitrogen content in SS on the susceptibility to IGSCC. Two heats of type 304LN stainless steel with 0.08 and 0.16 wt. % nitrogen were used. Strain hardening was done by cross rolling at 200 °C to 20 % thickness reduction (warm rolling) to simulate the weld induced strain in constrained welds. Subsequently, Transmission Electron Microscopic (TEM) examination was carried out on the rolled SS. The deformation mode observed due to warm rolling was predominantly elongated twin and shear band (SB) formation in both the SS, terminating at the grain boundary regions. This resulted in higher stresses and strains making grain boundary susceptible to IGSCC. Presence of more dislocations at grain boundaries is a key feature for such enhancement in the susceptibility of non-sensitized SS to IGSCC. Formation of twins and SB occurred to a greater extent in the SS with higher nitrogen content indicating greater susceptibility to IGSCC in BWR environment. Crack growth studies done in simulated BWR environment at different Dissolved Oxygen (DO) levels showed higher crack growth rates in the SS with higher nitrogen content in the non-sensitised and strain hardened condition, confirming the higher susceptibility of SS with a higher level of nitrogen.

Introduction

Intergranular Stress Corrosion Cracking (IGSCC) of austenitic Stainless Steel (SS) and nickel based alloy components in Light Water Reactors (LWRs) has been an issue affecting the cost and reliability of nuclear power plants and such type of cracking is generic in nature [1]. Initial cracking incidences, which were attributed to weld induced sensitization and Low Temperature Sensitization (LTS), were mitigated by the use of molybdenum and nitrogen containing nuclear grade SS (type 316 NG). Non-sensitised grades of these sensitization resistant SS have also shown IGSCC in Boiling Water Reactor (BWR) environment which was attributed to strain hardening due to weld shrinkage of constrained welds and this is now the major cause of IGSCC incidences [2].

The low carbon austenitic SS grades used in BWRs have nitrogen addition to improve its resistance to sensitization and it also makes up for the loss in strength due to lower carbon levels. It is an important alloying addition in the austenitic SS used in LWRs and has a potential to affect the IGSCC susceptibility. Nitrogen addition in austenitic SS affects the Stacking Fault Energy (SFE) but the exact nature of the dependence of SFE on the nitrogen content is a contentious issue [3-5]. It

also increases the strain hardening rate and improves the work hardening potential [6, 7], improves resistance to pitting and crevice corrosion [8, 9], retards sensitization kinetics due to chromium carbide precipitation when present in the range of 0.04 – 0.16 wt. % [10]. Thus nitrogen affects the deformation and hardening behavior of austenitic SS which may significantly affect IGSCC susceptibility of the austenitic SS in the non-sensitized and the strain-hardened condition.

In the present investigation, two heats of type 304 LN stainless steel with two different levels of nitrogen were used. The CGR was measured in the non-sensitized and strain-hardened condition, in simulated BWR environment and has been reported earlier [11]. The present paper presents findings to explain the observed effect of nitrogen addition on CGR. In this paper the Transmission Electron Microscopic (TEM) investigation results of the as rolled plates has been reported and the observed deformation mechanisms are correlated to the earlier reported CGR results.

Experimental

Two heats of type 304 LN stainless steels were used in this study with different levels of nitrogen, 0.08 and 0.16 wt. % (referred to as SS 304 LN1 and SS 304 LN2, respectively). The material was in the form of plates of 25 mm thickness in the solution annealed condition. The chemical compositions of both the grades are given in Table I. Strain hardening of the solution annealed plates was done by cross-rolling to 20 % thickness reduction at 200 °C (warm rolling). Details about the rolling process and the subsequent CGR results have been described earlier [11].

Table I. Chemical composition (in wt %) of the SS used in this study.

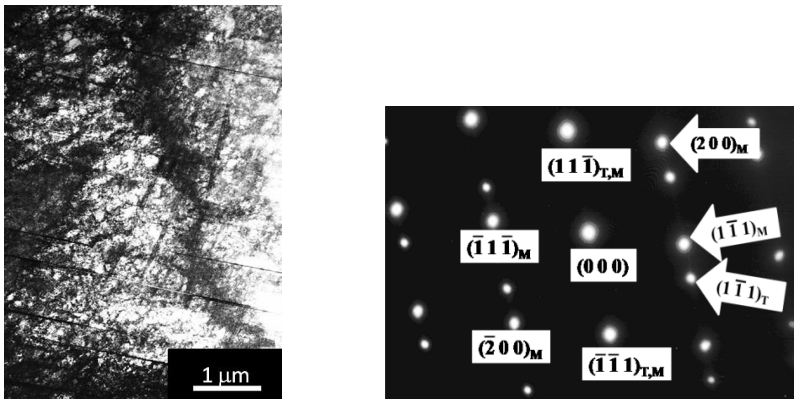
	C	S	P	Mn	Si	Cr	Ni	Mo	N
SS 304 LN1	0.028	0.004	0.033	1.63	0.49	18.82	9.20	0.11	0.08
SS 304 LN2	0.022	0.005	0.024	1.63	0.42	18.77	9.47	0.30	0.16

Samples for TEM examination were prepared from the mid-thickness of the as-rolled plates. Strips of approximate thickness 0.5 mm were cut from the rolled plates using slow speed abrasive precision cutting machine. The thickness was reduced further to 0.10 mm by polishing with 600 grit emery paper. Subsequently small discs of 3 mm diameter were punched from the thinned strip. Dual jet thinning was done at - 40 °C using a solution of 90 % Methanol and 10 % Perchloric acid at 20 V. The plane of the TEM specimens was parallel to the rolling plane. Subsequently, these specimens were used for TEM examination using JEOL 2000 FX microscope. A number of TEM micrographs and selected area diffraction (SAD) patterns were recorded and analyzed.

Results

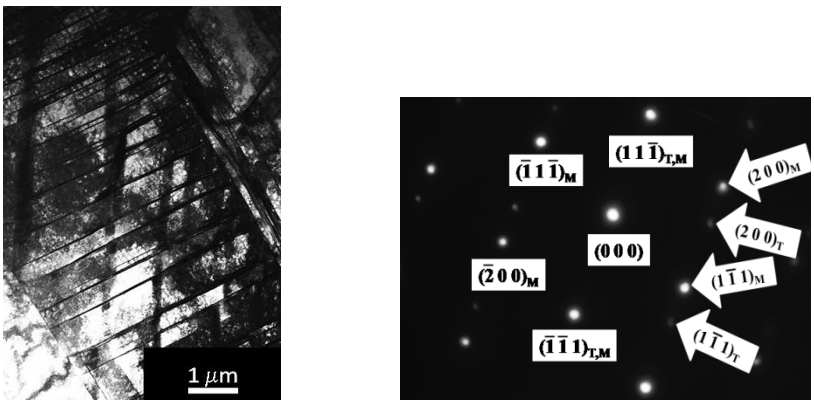
The TEM examination of the as rolled material showed the formation of narrow twins which were confirmed by the SAD pattern analysis. Dislocation entanglement and cell formation to a limited extent was also observed in both SS 304 LN1 and SS 304 LN2. Fig. 1 (a) shows the bright field image of the as rolled SS 304 LN1 along with the indexed SAD pattern confirming formation of twins. Twins, dislocation entanglement and cell formation can be seen as marked by arrows in fig. 1. The indexed SAD pattern confirming the formation of twins is shown in fig. 1 (b). Fig. 2 shows the bright TEM image for SS 304 LN2 after warm rolling at 200 °C. In fig. 2 (a) extensive twinning was observed and the twins were observed to be crossing each other. Twinning was confirmed based on the analysis of the SAD pattern as shown in fig. 2 (b).

The structure observed after rolling shows a heavy banded structure, indicating heterogeneous deformation. Such banded structure is a typical feature of low SFE alloys and the formation of such bands and the extent of heterogeneity in deformation in SS 304 LN2 are significantly more than that observed in SS 304 LN1. Formation of such banded structure due to rolling in austenitic SS has been reported earlier and these bands have been classified into various categories [12]. Such bands can be commonly called deformation bands and it can be regions containing either twins or stacking



(a) (b)

Figure 1. a) Bright field TEM image of SS 304 LN1 after warm rolling showing the formation of narrow twins and b) indexed SAD pattern confirming the formation of twins.



(a) (b)

Figure 2. a) Bright field TEM image of SS 304 LN2 after warm rolling showing extensive twinning and shear band formation and b) indexed SAD pattern confirming the formation of twins.

faults. These bands are defined as regions where the strain state is different from the surrounding regions. Blicharski et al. [12] made a specific classification of such bands as shear bands which produces large shear but the crystallographic orientation on both sides of the bands are the same, that is, there is no change in the SAD pattern from the banded region and the remaining matrix. Similar formation of banded structure in low SFE austenitic alloys have been reported earlier [13-17].

The CGR results in simulated BWR environment at different dissolved oxygen (DO) levels of these two SS in the warm rolled condition have been reported earlier [11]. It was shown that the CGR in SS304LN2 was 3 times higher as compared to SS304LN1 at all the DO levels. The TEM examination clearly showed that SS in the warm rolled condition lead to the formation of twining and shear band formation to a markedly larger extent in SS 304 LN2 as compared to that in SS 304 LN1. Twins and shear bands are linear features which have been observed to terminate at the grain boundaries. Thus grain boundaries act as barriers which restrict the strain induced due to the formation of these features. Karlsen et al. [18] have shown that inhomogeneous deformation in the low SFE austenitic SS leads to an enhanced susceptibility to IGSCC in LWR environment. Shear bands terminating at the grain boundary leading to a step formation was observed in the warm rolled SS as shown in fig. 3. Since the bands terminate at the grain boundary preventing the transmission of strain to the adjacent grain, local stresses as well as strain are expected to increase at the grain boundary and the grain boundary also gets disrupted as shown in fig. 3. Such increased localised stresses and strains at the grain boundary and more dislocations at grain boundaries are expected to make these regions the preferred path for crack growth in simulated BWR conditions water in the non-sensitized condition. Increased localised stresses and strains at the grain boundary make these

regions the preferred path for crack growth in simulated BWR conditions for the non-sensitised warm rolled SS. The extent of the residual stresses and strains at the grain boundary regions is expected to be higher in SS 304 LN2 as compared to SS 304 LN1 as the banded structure is formation is more pronounced in SS304LN2.

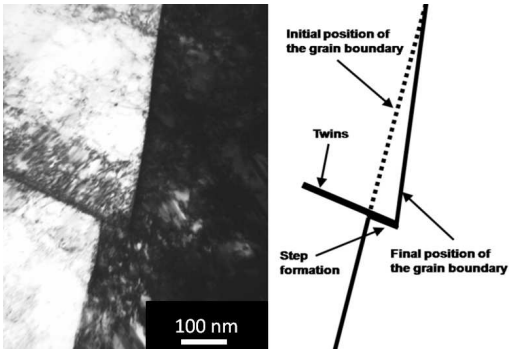


Figure 3. Step formation at the region where the bands intersect the grain boundary and a schematic explaining the step formation.

Conclusions

In this study the effect of deformation mode in austenitic SS with two different nitrogen levels was shown. The effect of deformation mode on the grain boundary susceptibility to IGSCC in simulated BWR environment was reported. Higher grain boundary strains and stresses in the SS containing higher level of nitrogen was the cause of higher susceptibility to IGSCC and consequently higher CGR in simulated BWR environment.

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