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Microstructure Engineering for Improved Intergranular Stress Corrosion Cracking Resistance of Stainless Steels

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ABSTRACT: Intergranular stress corrosion cracking (SCC) of austenitic stainless steel components is a life-limiting factor in nuclear power plant, in which failure of structural components presents a substantial hazard to both safety and economic performance.

This paper reports an on-going research programme into the mechanisms of intergranular stress corrosion cracking in austenitic stainless steels in simulated light water environments. These make use of new analytical and modelling techniques to develop an improved mechanistic understanding of the influence of materials, environment and stress on SCC. The work aims to establish new approaches for the engineering of microstructures and surfaces to develop stainless steels with improved SCC resistance.

KEYWORDS: Stress Corrosion Cracking, Austenitic Stainless Steels, Tomography, Electron Microscopy, Grain Boundary Engineering, Modeling.

I. INTRODUCTION

Stress corrosion cracking is a significant potential cause of failures in the nuclear power industry [1]. Cracking frequently nucleates from corrosion pits [2] and, depending on the material and environment, may be transgranular or intergranular in nature. The incubation period depends on factors including the rate of pit/localized corrosion formation and growth, the transition from pit/localized corrosion to crack initiation, and the propagation of short cracks with a size comparable to the microstructure scale. Although models for these processes have been developed [e.g. 3], the prediction of incubation periods remains uncertain due to the complex interaction between microstructure, environment and the mechanisms of localized corrosion and environmentally assisted cracking.

This paper reports an on-going research programme into the mechanisms of intergranular stress corrosion cracking in austenitic stainless steels in simulated light water environments. These make use of new analytical and modelling techniques to develop an improved mechanistic understanding of the influence of materials, environment and stress on SCC. The work aims to establish new approaches for the engineering of microstructures and surfaces to develop stainless steels with improved SCC resistance.

Recent results, which are described in this paper include

- Characterisation of the effects of surface preparation on the surface and near-surface distributions of residual stress and plastic strain.
- Analysis of the distributions of grain boundary character and grain boundary triple junctions have been combined with the new mechanistic understanding of IGSCC to develop trend models for the effects of grain boundary engineering on crack propagation resistance.
- In-situ, high resolution X-ray tomographic observations of intergranular stress corrosion crack nucleation and growth in sensitised austenitic stainless steel.
- Two-dimensional and three-dimensional intergranular finite element crack propagation models for short stress corrosion crack development and interactions.
- High resolution TEM characterisation of SCC crack-tip oxides.
II. STRESS CORROSION CRACKING RESEARCH

1. Surface Characterisation

In order to study the effect of the surface characteristics on test specimen behaviour, specimens with controlled values of the roughness, hardness and residual stress are required. Central composite design [4] was used to select combinations of parameters to minimise the number of experiments required [5]. The data was used to derive a response surface model [6] for the effects of the lathe turning parameters for a given tool on surface characteristics by statistical analysis (ANOVA) (Figure 1). The response surface model agreed well with data for axial residual stress and surface roughness (Figure 2). Interpolation, using the model, was used to prepare specimens with desired combinations of surface characteristics, which were then fully characterised (e.g. Figure 3). Design of specimens by this method enables systematic variation of the inputs into models for surface characterisation effects on stress corrosion and fatigue for the purposes of validation. Models are being developed for the effects residual stress on stress corrosion crack propagation. These are described in section 4.

Techniques for the quantification of near-surface plastic strain are being developed, such as the use of Electron backscatter diffraction analysis, with the aim of obtaining measurements which are in sensitive to the electron microscope and operating parameters. Two principle methods have been examined; the crystal deformation parameter (Cd) [7] and stored energy [8]. Typical data for a machined surface is given in Figure 4, which shows that plastic strain is localised within 80 µm of the surface. Discrepancies from the two methods are observed, which attributable to sampling size effects. Work is in progress to develop a fully quantitative technique with which to study the effects of machining induced plastic strain on corrosion and stress corrosion initiation.

![Figure 1: Response surface diagram obtained for lathe turning of cylindrical specimens. Feed rate (mm/revolution) and cutting depth (mm). Selected conditions 1 to 6 are identified.](image1)

![Figure 2: Comparison between the predicted and the measured value for lathe turning of cylindrical specimen for a) axial residual stress, and b) surface roughness for conditions 1 to 6, identified in Figure 1](image2)

![Figure 3: Depth profiles of axial residual stress in lathe turned cylindrical specimens. The machining conditions (4, 5 and 6) are identified in Figure 1. Condition 0 was prepared using the tool manufacturers recommended parameters.](image3)
A microstructure engineering study has been performed to investigate the effects of thermo-mechanical processing on the parameters likely to improve stress corrosion cracking resistance in sensitised austenitic stainless steel [10, 11]. The study was based on the proposition that the resistance to stress corrosion cracking develops from grain boundary triple junctions with a resistant grain boundary (i.e. a non-sensitised boundary). These are boundaries with a high degree of coincidence between the crystal lattice orientations of the grains on either side of the boundary, defined using the coincidence site lattice (CSL) model. Twin boundaries (Σ3) and some low CSL boundaries with Σ≤29 are commonly observed as resistant to intergranular corrosion and stress corrosion cracking, although this may vary with environment [10]. Crack propagation depends on the frequency with resistant triple junctions are encountered by the crack tip, thus both grain size and the grain boundary character distribution are significant parameters.

The effect of grain boundary engineering on the population of cracks developed in stressed specimens has been investigated [Error! Reference source not found.]. The crack lengths have a log-normal population, and hence extreme value statistics, in the form of Gumbel distributions, were applied to evaluate the crack population. This method allows the crack population to be described by measuring the longest cracks in the sample, expressed as the reduced parameter, y. Typical results are given in Figure 6, which demonstrates the significant influence of microstructure on crack propagation. The 30%/900/30 microstructure has a reduced grain size and lower fraction of twin and low CSL boundaries than the as-received microstructure. It was cold worked by 30% and annealed at 900°C for 30 minutes. Mechanistic models for the effects of microstructure modification have been developed, based on experimental observations of crack behaviour. This is discussed in sections 3 and 4.
Figure 6: Crack population data (a) under an applied stress of 200 MPa for 144 hours in pH 2.5 K$_2$S$_4$O$_6$ solution, (b) under an applied stress of 244 MPa in 0.01M Na$_2$SO$_4$ electrolyte at 523 K (250°C) and a pressure of 40 bar for 300 hours. Solid lines are least square best fits to the data. Dashed lines are the 95% confidence limits.

3. Three Dimensional Characterisation

The interaction between a propagating intergranular stress corrosion crack and the microstructure has been investigated by in-situ three dimensional observations of intergranular stress corrosion cracking, using the ID19 X-ray microtomography beam line at the European Synchrotron Radiation Facility (ESRF), France [Error! Reference source not found.]. An annealed and fully sensitised 302 stainless steel wire (400 μm diameter) was tested in 0.15 M potassium tetrathionate (K$_2$S$_4$O$_6$) (pH 2).

The progressive failure of the bridging ligaments was observed at the different stages of the experiment (Figure 7). These tomographic observations enable three-dimensional visualization of the stress corrosion crack (e.g. Figure 8). Correlation of the location of the crack bridging ligaments with fractographic observations at the same locations, confirms that the ligaments are associated with ductile features, which have a morphology consistent with low CSL grain boundaries (Figure 9).

These observations imply that non-sensitised low CSL boundaries are resistant to stress corrosion cracking, and the crack propagates around the obstacle formed by these ligaments. The ductile failure of the ligaments is expected to shield the crack tip from the applied stress. Consequently, grain boundary engineering to increase the number and size of such ligaments may be expected to improve stress corrosion cracking resistance. This is considered in more detail in section 4.

4. Modelling of Stress Corrosion Cracking

Two-dimensional [13] and three-dimensional [Error! Reference source not found.] finite element intergranular crack propagation models, which quantify the effect of crack bridging ligaments on short crack development and interactions have been developed. These models have been used to assess the effects of crack bridging crack branching for a range of fractions of susceptible boundaries. The models, which assume a critical crack tip strain for crack propagation, show that both the fraction of susceptible boundaries and the ductile ligament failure strain control the shielding effect of bridges. Typical data, obtained by Monte-Carlo methods for randomized distributions of resistant boundaries using the 2D model, is shown in Figure 10. The effects of surface residual stresses, such as those described in section 2 are being investigated and typical results are given in Figure 11. The model predicts crack retardation and arrest, which is also observed in experiments. The three-dimensional model predicts similar behaviour and is being applied to study the interaction and coalescence of stress corrosion cracks. Typical data is given in Figure 12, and is consistent with the behaviour of stress corrosion cracks.

Figure 7: Tomographic data obtained as successive periods (a and b) during stress corrosion cracking. Progressive failure of crack bridging ligaments is observed.

Figure 8: Three dimensional reconstruction of part of a stress corrosion crack. Features CA1 ands CA2 are identified in Figure 7.
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Figure 9: Ductile failure of a crack bridging ligament. The morphology is consistent with low CSL grain boundaries.

The modeling methodology developed therefore appears to satisfactorily reproduce the observed effects of microstructure and residual stress on stress corrosion crack behaviour. Further work is in progress to validate the model against a range of thermo-mechanically processed microstructures, with varying surface residual stress state. The model does not currently address the kinetic aspects of stress corrosion crack propagation, for which a detailed understanding of crack tip processes is required. Work in this area is described in section 5.

Figure 10: Two dimensional model for the development of the crack tip stress intensity factor (K) with applied stress ($\sigma_{\infty}$) for a microstructure with grain size (D) as the crack length (a) increases (W=50D). The theoretical stress intensity factor for an unbridged crack is compared against the effects of different fractions (f) of susceptible grain boundaries. The failure strain of susceptible boundaries is 1% of the failure strain of resistant boundaries.

5. High Resolution Characterisation

High resolution TEM characterisation of layered SCC crack-tip and crack-flank oxides from samples prepared by focused ion beam milling, is providing new insights into SCC mechanisms in stainless steels.

For example energy dispersive X-ray spectra (EDX) line profiles, performed using a scanning transmission electron microscopy (STEM) with a spot size of 1 nm, provide evidence for a layered structure in the oxides at the tip of a stress corrosion crack in 30-4 austenitic stainless steel, which propagated in a simulated light water environment. Figure 13 shows a typical elemental line profile. Within the oxide (~200–320 nm on the horizontal axis), the material is Fe-O rich. Adjacent to the Fe-O rich region, there are alternate Cr-O and Ni rich layers.

Figure 11: Typical results for the development of cracks using the 2D model with crack bridge development for a) applied tensile stress, b) applied tensile stress with sub-surface compressive residual stress.

Figure 12: Three-dimensional model for the growth and coalescence of intergranular stress corrosion cracks. Crack bridging (open spaces) is indicated by an arrow.

Detailed analysis of this nature is required to understand, and ultimately predict, the effects of crack tip environment on the kinetics of crack propagation, and the relationship between the bulk environment and the electrochemical conditions that
develop at the crack tip.

III. CONCLUSIONS

The propagation of short intergranular stress corrosion cracks is influenced by microstructure and residual stress. Two and three-dimensional models for crack behaviour have been developed, guided by high resolution observations of the mechanism of crack propagation. These models are being validated using test specimens with well characterized microstructures and surface residual stress distributions. Further developments to include the effects of crack growth kinetics require high resolution studies of processes such as oxide development.

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Figure 13: High resolution characterisation of stress corrosion crack tip oxides. The composition obtained by EDX along line 1 (a) is shown in (b).

V. REFERENCES