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Acta Materialia 60 (2012) 781–792

Acta MATERIALIA

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The study of stress application and corrosion cracking on Ni–16 Cr–9 Fe (Alloy 600) C-ring samples by polychromatic X-ray microdiffraction

Jing Chao [a], Marina L. Suominen Fuller [a], N. Stewart McIntyre [a,*], Anatolie G. Carcea [b], Roger C. Newman [b], Martin Kunz [c], Nobumichi Tamura [c]

[a] Department of Chemistry, University of Western Ontario, London, Ontario, Canada N6A 5B7

[b] Department of Chemical Engineering & Applied Chemistry, University of Toronto, Toronto, Ontario, Canada M5S 3E5

[c] Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, CA 94720-8229, USA

Received 7 October 2011; accepted 21 October 2011
Available online 14 December 2011

Abstract

Microscopic strains associated with stress corrosion cracks have been investigated in stressed C-rings of Ni–16 Cr–9 Fe (Alloy 600) boiler tubing. Polychromatic X-ray microdiffraction was used to measure deviatoric strain tensors and the distribution of dislocations near cracks that had been propagated in electrochemically accelerated corrosion tests. An associated investigation of the C-ring-induced strains prior to corrosion showed significant tensile strain in the stress axis direction by the torsional closure of the alloy tube section in the C-ring test. Significant grain lattice rotation and pronounced plastic strain at some grain boundaries were noted. Stress-corrosion-cracking-generated intergranular cracks were produced in two Alloy 600 specimens after 6 h and 18 h tests. The diffraction patterns and resultant strain tensors were mapped around the cracked area to a 1 μm spatial resolution. The strain tensor transverse to the crack growth direction showed tensile strain at the intergranular region just ahead of the crack tip for both specimens. Both cracks were found to follow grain boundary pathways that had the lowest angle of misorientation. Dislocation distributions within each grain were qualitatively obtained from the shapes of the diffraction spots and the effect of “hard” and “soft” grains on the crack pathway was explored for both 6 h and 18 h specimens. The Schmid factor of one of the grains adjacent to the crack at the 6 h and 18 h initiation sites was found to be the lowest, compared to Schmid factors calculated for surface grains away from the initiation site, and also along the crack path into the bulk.

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Keywords: Nickel alloy; Stress corrosion cracking; Polychromatic X-ray microdiffraction; Residual strain; Dislocations

1. Introduction

Ni–16 Cr–9 Fe (Alloy 600) boiler tubing, used in most of the older pressurized water reactor (PWR) power stations, is susceptible to stress corrosion cracking (SCC) under service conditions. In fact, SCC of Alloy 600 tubing is the single most important reason that nuclear steam generators are replaced. Local mechanical stresses from scratches and dents could result in strains at critical grain intersections, leading to enhanced initiation of chemical attack and crack propagation; however, there could be many other origins to the stresses leading to SCC. Thus, it is important to develop methods to measure such local stresses under cracking conditions.

Microscopic methods based on the use of electron backscatter diffraction (EBSD) have been used extensively to map stress magnitudes at or near the cracks produced in Alloy 600 tubing removed from service [1]. EBSD provides superb high-resolution imaging of the alloy microstructure, as well as detailed information on local grain misorientation. Information on plastic deformation and estimates of stress/strain magnitudes are possible with appropriate

* Corresponding author. Tel.: +1 519 661 2111x86703; fax: +1 519 661 3022.
E-mail address: smcintyr@uwo.ca (N.S. McIntyre).

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doi:10.1016/j.actamat.2011.10.040

calibration. An alternative approach uses polychromatic X-ray micro-diffraction (PXM). PXM uses a microscopic beam of polychromatic ("white") X-rays to produce a Laue diffraction pattern from which fundamental information can be derived from the measured local lattice distortions and thus deviatoric strain components [2,3]. All allowable planes in the volume, satisfying the Bragg condition, give reflections, and these are detected on an area X-ray detector above the sample. This Laue-based technique eliminates the need for sample rotation, thus simplifying spatial mapping compared to monochromatic single crystal diffraction.

The use of PXM has increased in the last decade due to the availability of bright, third-generation synchrotron sources, and progress in X-ray focusing optics. Automated indexing software has been developed to handle the copious amounts of data generated at these beamlines. The software packages, XMAS (X-ray Microdiffraction Analysis Software) and ThreeDimX_RayMicroscopy, used to process the data presented in this paper, were obtained from the group at beamline 12.3.2 at the Advanced Light Source (ALS) [3], and the Advanced Photon Source (APS)-Oak Ridge National Laboratory (ORNL) group at the APS beamline 34-ID-E [2], respectively. The CCD/sample geometry is determined using a silicon Laue pattern as a reference. The inter-ray angles for the Laue spots are determined and compared to known calculated values until a match is found. The lattice orientation is determined to within 0.01°. These software packages are capable of processing overlapping Laue patterns generated from multiple grains, and require only a few reflections (>4 per grain) for indexation.

In contrast to EBSD, PXM provides separate assessments of both residual elastic and plastic deformation in the crystal. Elastic deformation information is derived from deviations in crystallographic axes from those of an unstressed crystal; as such, directions of the strains as well as strain magnitude can be measured [2,3]. Knowledge of strain direction is particularly important to any attempt to model the forces leading to crack initiation and propagation. Plastic deformation information comes from assessment of the distortions in the Laue spots; from this, the direction(s) of any dislocation systems can be measured, as well as the magnitude of the dislocation displacement and the dislocation density in a particular volume [4–6].

In an earlier PXM study of microscopic strain distributions in Alloy 600, the effects of uniaxial external stresses were measured. The strain magnitudes and directions were found to agree approximately with those determined for the same material by neutron diffraction [7,8]. Since then, a method has been refined that has caused Alloy 600 C-rings to undergo SCC in an aqueous caustic solution. Some of the specimens from these tests have been studied using the PXM capabilities at the 12.3.2 beamline of the Advanced Light Source (ALS).

In this paper, we present an assessment of the strain tensors and dislocation distributions around small cracks in Alloy 600 tubing that had been induced during electrochemically accelerated caustic corrosion in a high-temperature autoclave. In contrast to the relatively simple stress patterns in the previous study, local mechanical stresses in this present study arise from many sources. Some of these are introduced by the action of the external force of the SCC on surface irregularities and on the chemical energy released during reaction between solution and metal; others come from rolling and swaging processes used in tube preparation. This paper first shows the effects of the application of C-ring stress on strain distributions in a section of Alloy 600 tubing. Because the action of this stress is rather less complex than that of the caustic cracking process, the changes to strain distribution are more apparent and interpretable. Another objective of the present study has been to identify internal factors that could contribute to crack initiation and propagation during SCC. The PXM studies here have shown that crack propagation may be supported at boundaries between grains that block the migration of dislocations. Such boundaries are fairly easy to identify by PXM and ultimately this could be used as a measure of cracking propensity for alloys already stressed by SCC and mechanical conditions.

2. Experimental

2.1. Sample preparation

Alloy 600 tubing material was obtained from Rolled Alloys in the mill-annealed condition. The average grain size was ~20 μm. Four samples – unstressed C-ring, stressed C-ring ("as-received"), 6 h SCC-cracked C-ring and 18 h SCC-cracked C-ring – are discussed in this paper. All C-ring samples were prepared from mill-annealed Alloy 600 tubing according to ASTM standard G-38. The latter three samples were stressed to a 2% plastic strain using bolt tightening. An optical image of one of the stressed C-rings is presented in Fig. 1a. In fact, the C-ring studied prior to SCC cracking was the same C-ring: a torsional stress corresponding to a nominal strain of 2% was applied immediately, after the same C-ring had been studied using PXM. In order to study a cross-section of each sample, one of the flat end faces was mechanically polished to 0.05 μm finish. Then, in an effort to reduce the contribution of polishing-induced strain, a focused beam of gallium ions (FIB) was used to remove such mechanical damage from the near surface region, by ion bombarding at a glancing angle, over an area close to the outer edge of the apex of the arch at the flat end face. Subsequently, this ion bombardment was found to have no measureable effect on the strain measurements of the surface. Also, using the ion beam, cuts were made in the surface as markers of features in the microstructures; these could be identified by scanning electron microscopy (SEM) and optical microscopy. As well, localized platinum deposition was used to mark regions on the sample where cracking was noticeable. These regions could be identified by X-ray microfluorescence spectroscopy

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