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Using transmission Kikuchi diffraction to study intergranular stress corrosion cracking in type 316 stainless steels

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ABSTRACT

Transmission Kikuchi diffraction (TKD), also known as transmission-electron backscatter diffraction (t-EBSD) is a novel method for orientation mapping of electron transparent transmission electron microscopy specimen in the scanning electron microscope and has been utilized for stress corrosion cracking characterization of type 316 stainless steels. The main advantage of TKD is a significantly higher spatial resolution compared to the conventional EBSD due to the smaller interaction volume of the incident beam with the specimen.

Two 316 stainless steel specimen, tested for stress corrosion cracking in hydrogenated and oxygenated pressurized water reactor chemistry, were characterized via TKD. The results include inverse pole figure (IPFZ) maps, image quality maps and misorientation maps, all acquired in very short time (<60 min) and with remarkable spatial resolution (up to 5 nm step size possible). They have been used in order to determine the location of the open crack with respect to the grain boundary, deformation bands, twinning and slip. Furthermore, TKD has been used to measure the grain boundary misorientation and establish a gauge for quantifying plastic deformation at the crack tip and other regions in the surrounding matrix. Both grain boundary migration and slip transfer have been detected as well.

1. Introduction

Stainless steel alloys such as SUS316 are widely used for applications in nuclear power plants because of their excellent performance in high-temperature and corrosive environments (Arioka et al., 2006a). For decades, they have been commonly used as the main constituents of structural components in the primary circuit of Pressurized Water Reactors (PWRs) and Boiling Water Reactors (BWRs) (Karlsen et al., 2010). However, despite their corrosion resistance, these materials have shown susceptibility to intergranular stress corrosion cracking (IGSCC) after long service periods (Terachi et al., 2008; Arioka et al., 2007).

Hence, for at least two decades stress corrosion cracking (SCC) in stainless steels has been the focus of increasing research efforts (Andresen, 2013; Arioka, 2013; Dietzel and Bala Srinivasan, 2011; Kain, 2011; Lynch, 2011; Shoji et al., 2011; Staehle, 2010; Das et al., 2009; Lozano-Perez et al., 2009a; Couvant et al., 2007; Terachi et al., 2007). However, there is still only limited understanding of the mechanisms underlying SCC and there is no general model for the initiation and propagation of SCC yet (Bruemmer and Thomas, 2010; Guerre et al., 2007; Vaillant et al., 2004; Scott and Le Calvar, 1993). Most studies are complicated by the fact that there are several parameters potentially simultaneously influencing the occurring crack initiation and propagation processes, including water chemistry, temperature and matrix composition (Terachi et al., 2007; Lozano-Perez et al., 2009b; Arioka et al., 2006b; Terachi and Arioka, 2006; Langevoort et al., 1984).

These previous SCC propagation studies have focused on the oxide structure and chemistry, the crack morphology and the matrix surrounding the crack tip, such as changes in the local matrix composition and deformations. These features have been studied using a variety of electron microscopy (EM) methods, including scanning electron microscopy (SEM), auger electron spectroscopy (AES), transmission electron microscopy (TEM) (Kruska et al., 2011;

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Lozano-Perez and Gontard, 2008; Lozano-Perez and Titchmarsh, 2003; Lozano-Perez et al., 2004; Olszta et al., 2011) as well as NanoSIMS (Lozano-Perez et al., 2008a,b). To complement these mechanical and chemical studies of SCC, higher resolution characterization from a crystallographic point of view (such as orientation mapping) is also necessary.

There is limited information on the relationship between SCC crack propagation and crystallographic features in the sample surrounding the crack tip. This includes the misorientation of the grains on either side of the crack, the plastic deformation before, around and ahead of the crack tip as well as the existence of deformation structures such as twinning, slip and deformation bands. Comparing these features in different types of SCC specimen (with regard to water chemistry, temperature or matrix composition) may provide significant new insights with respect to the propagation of SCC.

This work represents a novel study of crystallographic features in two different types of samples, tested in hydrogenated and oxygenated PWR primary water chemistry, utilizing a new method of high-resolution orientation mapping: transmission Kikuchi diffraction (TKD) or sometimes also known as transmission-electron backscatter diffraction (t-EBSD). The samples were chosen because they manifest two different types of crack propagation. It is shown that, with this novel technique, high-resolution crystallographic data can be easily acquired which may contribute to solving some remaining questions about the SCC crack propagation process (such as the impact of plastic deformation around the crack tip).

2. Material

The material used for this study was type 316 (reactor grade) stainless steel. Two different types of specimen were characterized: type 316 tested under oxygenated PWR water conditions, provided by AREVA; and type 316 tested under hydrogenated PWR water conditions, provided by INSS. Both steels exhibited SCC after autoclave testing. The compositions of each alloy are listed in Table 1.

Specimen 316INSS underwent solution treatment followed by water quenching and uni-directional cold-rolling to a thickness reduction of 20%. The SCC test (CGR) was performed at the INSS (Japan) laboratories using a pre-cracked 1/2 CT specimen in the T–S direction in an autoclave under constant load (30 MPa $m^{1/2}$). The sample was exposed to a testing environment of simulated PWR water chemistry (hydrogenated water: 500 ppm B + 2 ppm Li, +30 cm^3-STP/kg-H_2O DH_2) for 700 h at 360 °C. More information about the standard methods for stress corrosion cracking testing can be found on the American Society of Testing and Materials website (www.astm.org).

Specimen 316AREVA was a reverse U-bend specimen with a shot-peened surface and was tested at the AREVA Laboratories in France. The specimen was exposed to a PWR primary water chemistry in shutdown conditions with increased oxygen content (oxygenated conditions: 10 ppm O + 1200 B + 2 ppm Li) for 1500 h at 345 °C.

The cross-sectioned surface of both samples was ground with SiC paper and polished with 1-μm diamond suspension. Mirror-finish was achieved by final treatment (15 min) with colloidal silica. Subsequently, the bulk specimens were screened with an optical microscope and the SEM to locate the crack tips. These crack tips have then been lifted out in situ in plan-view orientation in a focused ion beam (FIB) instrument. The lift-outs were then mounted on TEM Cu grids and thinned to electron transparency (<100 nm). Before the actual TKD acquisition, the TEM foils were plasma cleaned for 5 min (Fishione 1020 plasma cleaner). Initial TEM observations were made with a JEOL 2100 LaB_6 TEM and EELS spectra were acquired with a JEOL ARM200F (cold-FEG gun, 200 kV).

3. TKD methodology

For decades, electron backscatter diffraction (EBSD) has been a very important and commonly used tool for the gathering of crystallographic data for many materials scientists (Dingley, 2004; Schwartz et al., 2009). Like many other techniques, recent progress in nano-science has required orientation mapping to improve in terms of spatial resolution. However, reports suggest that EBSD appears to have reached its threshold at around 50–100 nm, depending on the analyzed material (Keller and Geiss, 2012; Trimby, 2012; Zaefferer, 2011).

In 2012, Keller et al. first reported of a new technique called transmission-electron backscatter diffraction (t-EBSD) or later also known as transmission Kikuchi diffraction (TKD) which uses the scanning electron microscope (SEM) with a conventional EBSD detector to perform orientation mapping on electron transparent TEM foils (Keller and Geiss, 2012). While the exact same hardware as for EBSD was used, the TKD setup changed slightly with regard to the specimen position. The EBSD software accounts for this by adapting the pattern recognition algorithm. The TKD geometry is derived from the basic EBSD setup with the main difference being the specimen position and its orientation with respect to the SEM column. Instead of the 70° tilt of the sample toward the EBSD camera, the TEM foil (mounted on a standard TEM Cu grid) is almost horizontal (max. 10° tilt) with respect to the incident electron beam. Due to this setup and the thin TEM foil, the interaction volume of the electron beam with the sample is minimized in TKD. This is because of the sample thickness and the fact that the cone of the incident electron beam in EBSD is much larger due to the tilt angle of 70° which increases the interaction volume [as shown via Monte Carlo simulations in Keller and Geiss (2012)]. Hence, the lateral resolution is therefore significantly improved (Trimby, 2012). In addition, the almost-horizontal mounting of the sample reduces the need for dynamical focus or tilt correction.

The ideal thickness of the TEM foil for TKD depends on the material composition; good quality results have been achieved at under 100 nm thickness (Rice et al., 2014). In the first years of application of this technique, it has become apparent that the quality of TKD patterns is very much dependent on the thickness of the specimen, which also has a strong effect on the achieved spatial resolution. Therefore, TEM sample preparation is key for the successful and reliable data acquisition via TKD (Suzuki, 2013).

The TKD patterns originate from the volume very close to the bottom surface of the sample (Trimby, 2012). No additions to the common EBSD system are necessary: the Kikuchi patterns are recorded with the EBSD camera and the (for TKD slightly adapted) EBSD software is responsible, as usual, for orientation mapping.

There is a general consensus from researchers applying TKD to the characterization of a variety of materials that this technique offers a significant improvement in spatial resolution (Keller and Geiss, 2012; Trimby, 2012; Rice et al., 2014; Brodusch et al., 2013; Trimby and Cairney, 2014; Trimby et al., 2014; Babinsky et al., 2014). The authors concur that in most cases that the advantages offered by this improved resolution outweighs the extra effort of having to prepare electron transparent TEM foils.

In this study, all TKD maps were collected using a Zeiss Merlin FEG-SEM and an eFlashHR Bruker EBSD detector system. An accelerating voltage of 20 kV and a probe current of 3 nA were used. Reports (Trimby, 2012) suggest that in thicker sample regions 30 kV works best, but the used specimens were very thin (60–80 nm) and 20 kV produced sufficiently good pattern quality. The TEM foil was mounted on a special Bruker TKD sample holder with an intrinsic