The Use of Electron Backscattered Diffraction for Material Characterization at Chalk River Laboratories

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Abstract

Electron Backscattered Diffraction (EBSD) is continuing to emerge as a valuable tool for characterizing polycrystalline engineering materials. This technique permits sub-micron measurements of materials to evaluate localized texture, grain size, grain boundary interactions, and residual plastic deformation. This paper presents the working principles and current potential for characterizing nuclear materials with EBSD.

1. Introduction

In the past few years EBSD has been emerging as a valuable tool in characterizing nuclear materials [1-9]. The capabilities of the technique include determining microstructural characteristics which contribute to failure.
This paper will discuss the capabilities at Chalk River Laboratories (CRL) for quantitatively analysing the residual plastic strain in an Alloy 600 material with a known through wall hardness gradient, the capabilities of mapping natural UO_2 for determining grain size statistics, and crack mapping of an environmentally assisted crack (EAC) in recrystallized Zircaloy-4 tubing.

2. Background

Figure 1 presents an EBSD schematic showing the incident electron beam and how the crystal diffracts as a Kikuchi pattern after electron excitation. Analysing Kikuchi patterns allows for crystal orientation with respect to the sample surface to be determined. The light bands on a Kikuchi pattern represent crystallographic planes, while the intersections of the bands correspond to a direction. For example, the (100) and (110) crystallographic planes (i.e. bands in Kikuchi pattern) both have the [001] crystallographic direction (i.e. band intersections in Kikuchi pattern) in common (refer to Figure 1). This Kikuchi pattern is automatically indexed with a pre-set Kikuchi pattern from the software (Oxford Channel 5). Once the orientation of that excited point on the specimen surface is indexed, the next adjacent point is excited, and a new Kikuchi pattern created. This is repeated until the entire scanning area is excited and characterized.

For each point on the map, a Kikuchi pattern image quality factor is recorded (Band Contrast), and using this quality factor, Band Contrast imaging can be utilized to show the grain structure of the entire scanning area mapped. Band Contrast is decreased at grain boundaries due to multiple Kikuchi patterns diffracting simultaneously. These areas appear as dark areas on Band Contrast images, and are what allows for the grain structure to become apparent.

The creation of pole figures (texture), grain size and grain boundary statistics are also a general technique available with Channel 5 software. For more advanced characterization with EBSD, such as characterization of intra-granular plastic strain, external data analysis is required using histogram output files from the Channel 5 software. Two quantitative methods are used to analyse data to represent intra-granular plastic strain:

1. Integrated Misorientation Density (IMD_{Φ}).
2. Average Intragrain Misorientation (AMIS).

The IMD_{Φ} has been used to show a strong linear relationship with strain for Alloy 600 [1]. Using grain boundary distribution histogram information obtained with EBSD, the IMD_{Φ} was calculated using Equation 1.

$$IMD_{\Phi} = \frac{\sum_{\Phi_0}^{\Phi max} \Phi \cdot MD(\Phi)}{N}$$

Equation 1

Where

IMD_{Φ}	=	Integrated Misorientation Density (rads/pixels).
Φ_0	=	Minimum resolvable angular deviation (1° for this study).
Φ_{max}	=	Maximum angular deviation ($\Phi \leq 15°$ for this study).
N	=	Number of pixel pairs (EBSD patterns) in the scan area (map).
$MD(\Phi)$	=	Number Distribution of misorientations of angular deviation Φ.
Φ	=	Magnitude of the angular deviation, in radians.

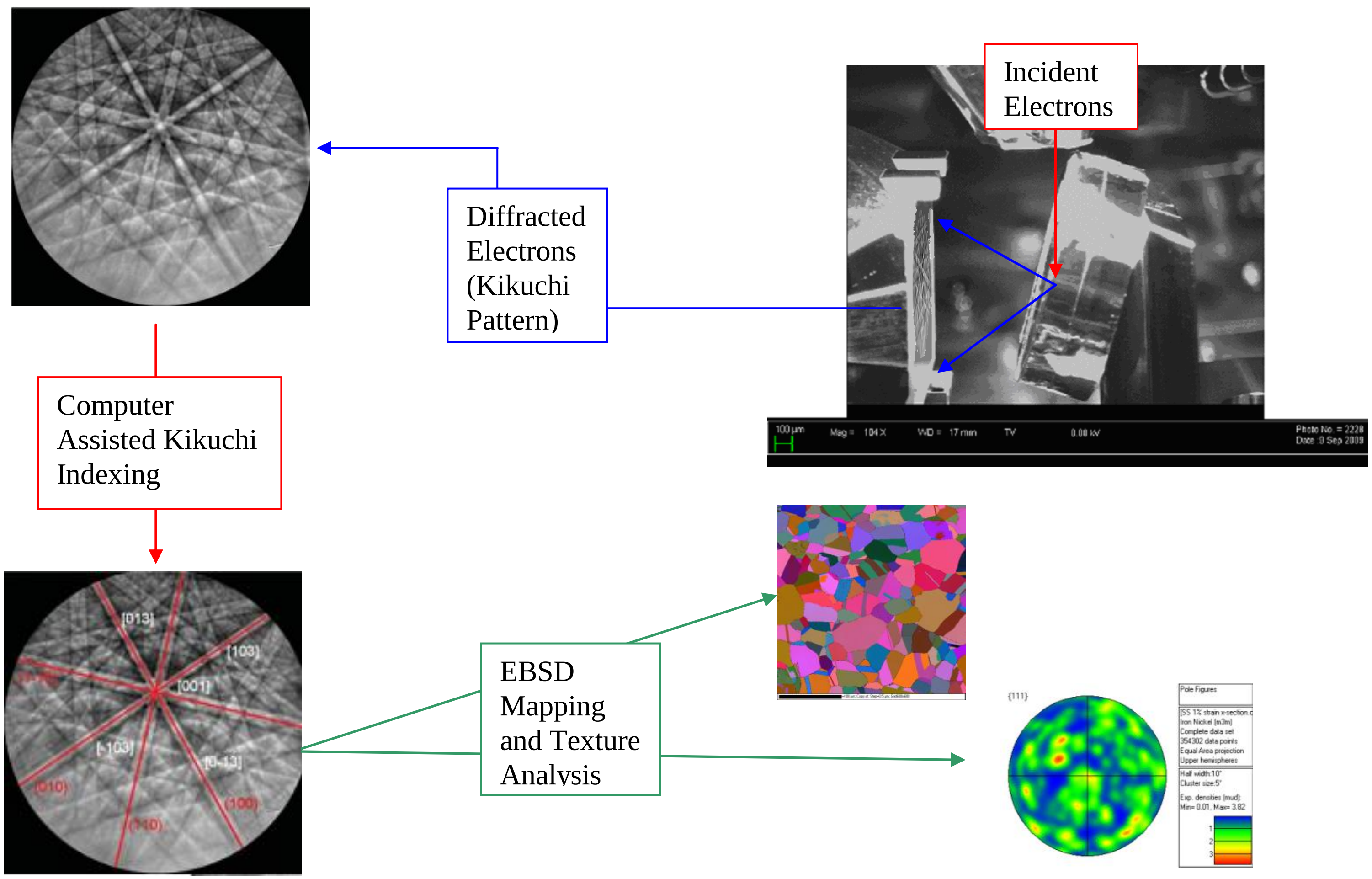


Figure 1 EBSD schematic showing the incident electron beam which diffracts from the specimen creating the Kikuchi pattern which is then automatically indexed using the computer. The incident electron beam then moves across the specimen, allowing for the area of interest to become mapped using EBSD.

Equation 1 gives a weighted distribution of misorientation between Φ_0 and Φ_{max}. This misorientation density is averaged over the entire scan area (map) to give one representative number for every measured EBSD map. If small numbers of dislocations are present in the material, the IMD_Φ will be a low number, but as the dislocations begin to accumulate, and potentially cause medium-angle grain boundaries, the IMD_Φ increases, and therefore has a strong relationship to strain. Lehockey has shown a linear relationship between IMD_Φ and strain for Alloy 600 up to nominal strains of 30% [1].

AMIS is an approach that gives the mean misorientation per grain within the scan area. This is done by calculating the average intra-granular misorientation for each grain with respect to a reference Kikuchi pattern for each grain. A distribution of misorientation angles are made in Channel 5 analysis software and a weighted average is made for all grains in the scanned area. Similarl to the IMD_Φ approach, as the strain is increased, the dislocations will create larger intra-granular misorientations. AMIS is a representative angular misorientation for the scanned area. Using this method, a strong linear correlation between AMIS and plastic strain has been found for 304 and 316 stainless steel [10].

3. Results

3.1 Residual Plastic Strain

Alloy 600 is a nickel-based material used in the nuclear industry for steam generator tubing. This is an austenitic material with a face centred cubic (FCC) microstructure. A piece of Alloy 600 material which had a known through-wall hardness gradient was measured using EBSD. The main focus was to characterize the level of intra-granular misorientation, and correlate this with hardness measurements.

As discussed earlier, the level of misorientation within grains is directly proportional to the dislocation density. Figure 2 shows a local misorientation map from the OD towards the ID of an Alloy 600 pipe with a known hardness gradient. The EBSD map was split into 10 equal vertical subsets for analysis. Each subset was analysed for IMD_Φ and AMIS, as tabulated in Table 1. After the EBSD mapping was performed, Vickers hardness measurements were done on the same through-wall specimen in order to make a direct comparison between hardness, and the IMD_Φ and AMIS findings. Figure 3 is a plot of hardness with an IMD_Φ overlay on the secondary vertical axis. There is a strong correlation between the misorientation density and the hardness, as expected. Figure 4 is a plot of the hardness with an AMIS overlay on the secondary vertical axis. As with the IMD_Φ, there is a strong correlation with the hardness.

The IMD_Φ approach takes into account the total level of small angle misorientations, but its shortfall is that it fails to indicate the amount of built up dislocations creating higher angle intra-granular misorientations. The AMIS approach gives an idea as to the level of built up dislocations creating higher angle intra-granular misorientations; however, as higher levels of plastic strain are achieved, the misorientation angles can become greater than the cut-off limit for our measurement (i.e. >15°). For this reason, both techniques are used in order to gain as much knowledge from the EBSD data with respect to the level of plastic deformation in the material.

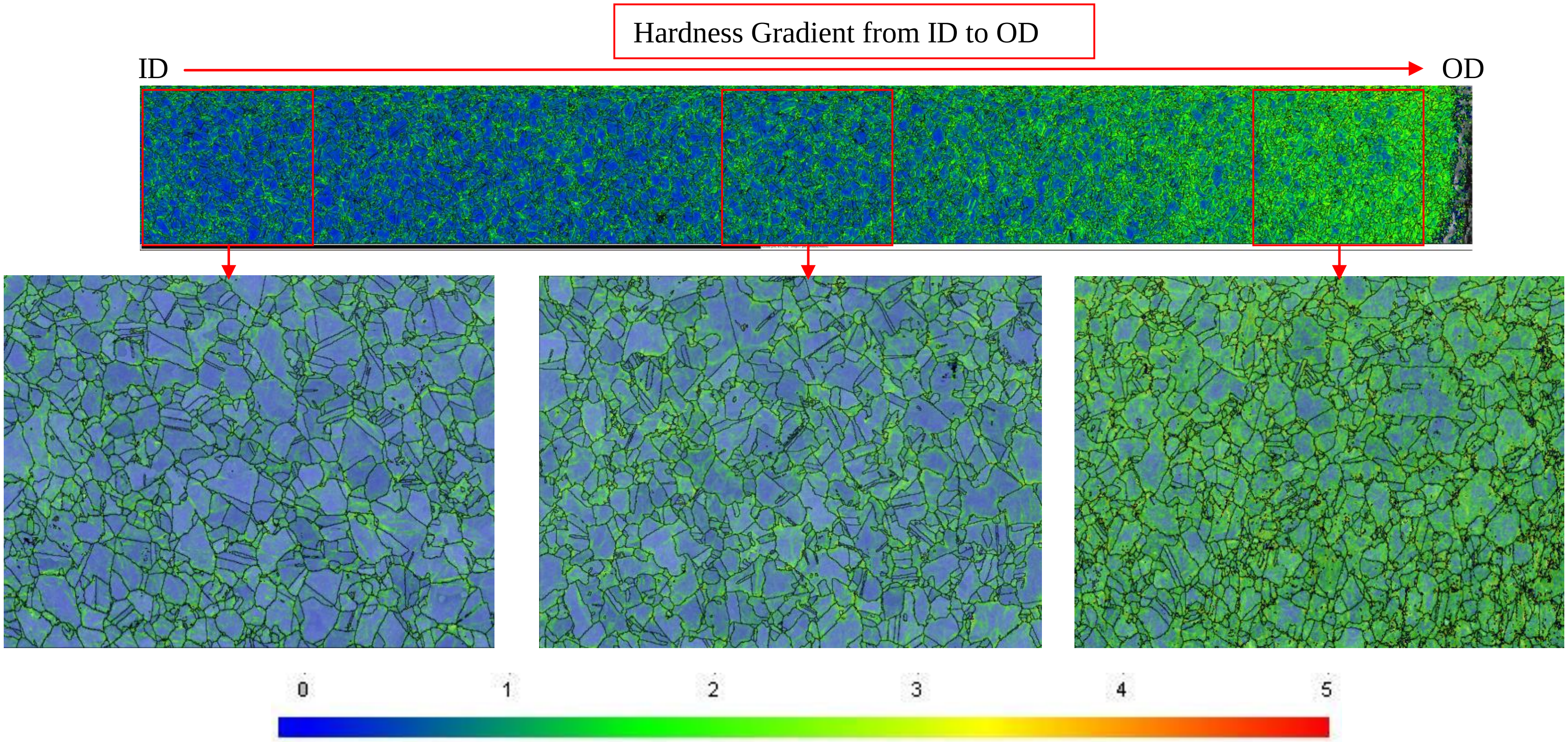


Figure 2 Through-wall misorientation plot of Alloy 600 pipe.

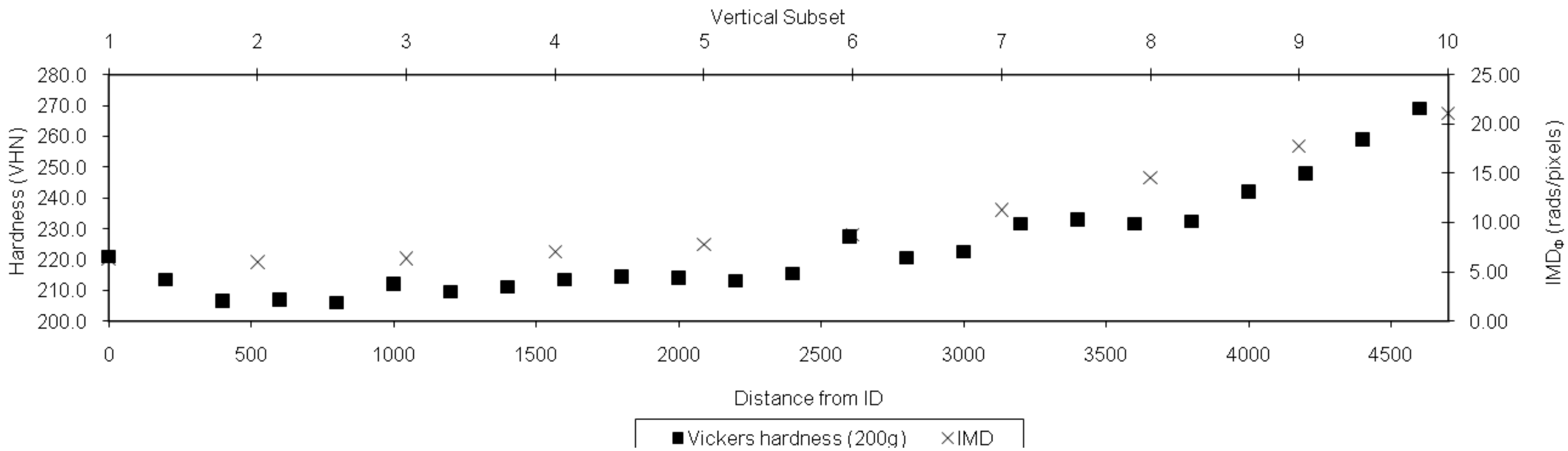


Figure 3 Alloy 600 through-wall hardness profile with IMD overlay.

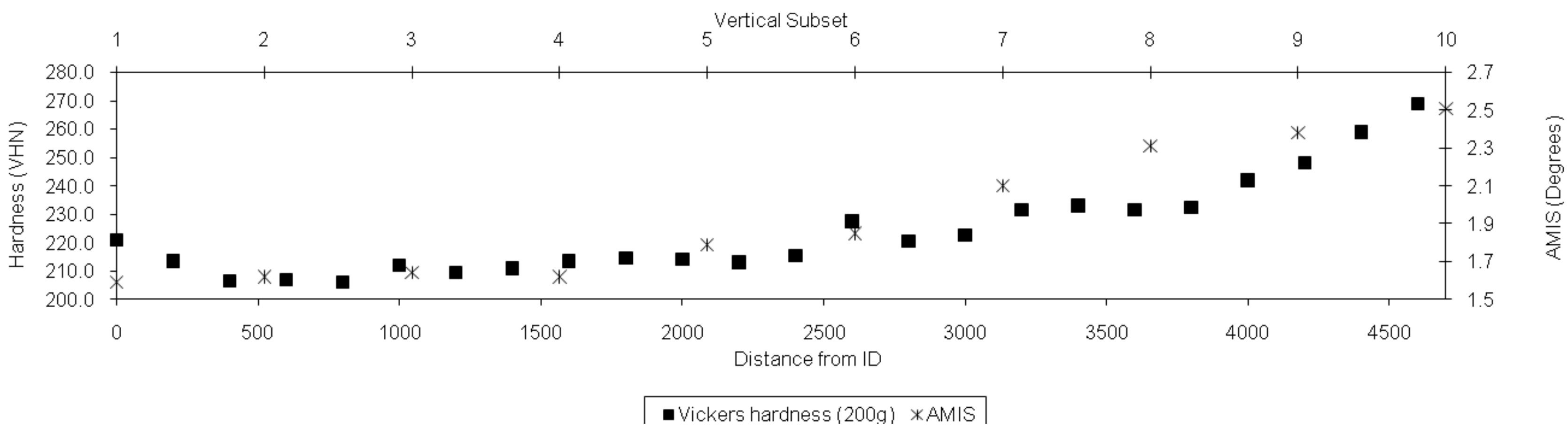


Figure 4 Alloy 600 through-wall hardness profile with AMIS overlay.

This technique can be useful in determining the fitness for service of materials prior to implementation in a nuclear facility, and can assist in component failure analysis for nuclear materials. This characterization technique is useful in combination with hardness measurements. Hardness measurements may be affected by more than just the level of deformation, and can therefore be checked with EBSD. Hardness measurements are limited with respect to characterizing residual surface damage from material manufacturing because measurements cannot be taken close to a sample surface, according to ASTM E 92-82 [11]. Because the level of surface deformation in materials is of great importance with respect to the materials performance in a reactor, EBSD can be used as a valuable tool for assessing fitness for service of materials prior to implementation in a nuclear facility, and can assist in component failure analysis.

Table 1 IMD_{Φ} and AMIS results from ten equal subsets of the through-wall Alloy 600 specimen indicated in Figure 2.

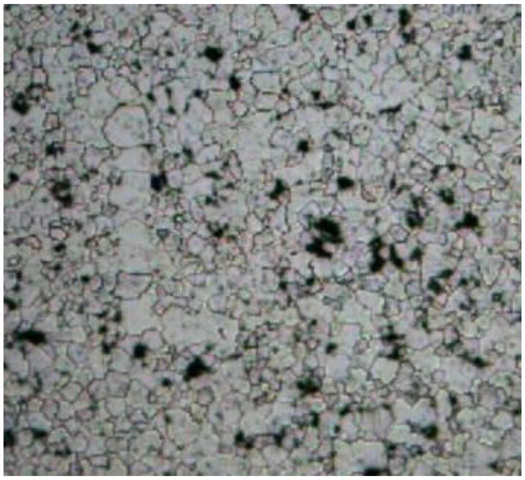
Subset	IMD_{Φ}	AMIS
Vertical 1	6.33	1.59
Vertical 2	5.98	1.62
Vertical 3	6.36	1.64
Vertical 4	7.03	1.62
Vertical 5	7.80	1.79
Vertical 6	8.78	1.85
Vertical 7	11.30	2.10
Vertical 8	14.59	2.31
Vertical 9	17.77	2.38
Vertical 10	21.06	2.51

3.2 Fuel Characterization

EBSD is useful in characterization of metals such as Zircaloy-4, and it can also be useful in characterization of ceramic materials such as UO_2. The capability of characterizing fuel materials with EBSD is currently being developed at AECL. Examples of how EBSD can be used for fuel characterization of UO_2, and Zircaloy-4 are discussed in the sections below.

3.2.1 UO_2

Figure 5 shows the microstructure and a representative Kikuchi pattern (indexed) from an un-irradiated UO_2 specimen. This indicates the capabilities at CRL in characterizing uranium oxide fuel materials. Utilizing EBSD for fuel characterizations will allow an in-depth examination into the crystallographic relationship between grains and relationships with material properties. A previous EBSD study on erbium additions to uranium oxide showed the effect on grain growth and sintering behaviour with small additions of Er to UO_2 [12].



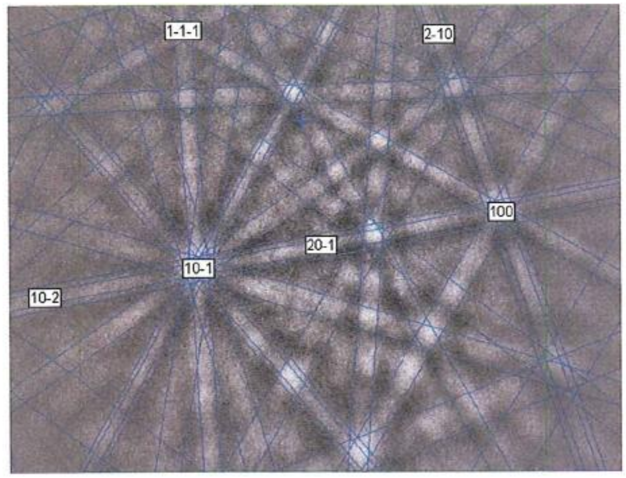


Figure 5 Microstructure of natural UO_2 with the representative indexed Kikuchi pattern.

3.2.2 Zircaloy-4

Recently, EBSD has been utilized in characterizing zirconium alloys [3, 7-9]. These studies have been primarily on twinning structure in zirconium alloys and grain boundary determination. Currently at CRL, the grain boundary relationship with EAC is being investigated in Zircaloy-4 fuel sheathing. A recrystallized Zircaloy-4 fuel sheathing specimen was strained to failure (prior to the 0.2% YS being reached) in an iodine environment. The specimen was sectioned and the outside surface examined, see Figure 6. The fine step size used for EBSD investigation allows for detailed examinations of micro-cracking surrounding the main outside surface crack. EBSD characterization of EAC cracking will allow for an in-depth study of the microstructural characteristics which govern the fracture properties of the sheathing. As shown in Figure 6, cracking propagates along certain grain boundaries, and through certain grains, while not others. With the use of EBSD, the crystallographic relationship with crack propagation, and grain boundary relations can be examined in further detail.

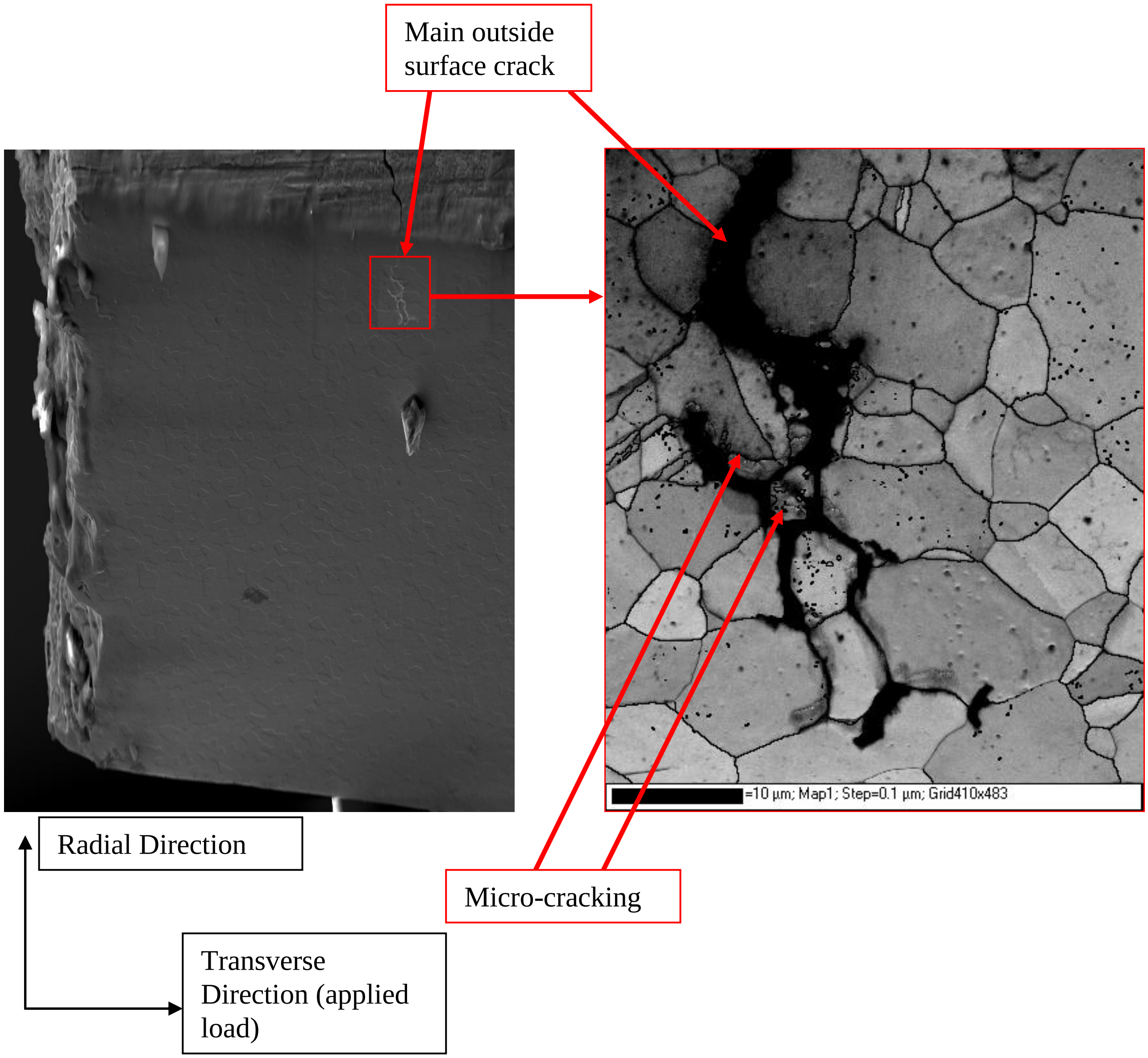


Figure 6 SEM image of the fractured surface and OD crack of interest, and band contrast image of an outside surface crack in Zircaloy-4 failed in an iodine environment.

4. Conclusion

The examples discussed in this paper show how EBSD is being developed at CRL for nuclear materials characterization. The ability to characterize the level of residual plastic deformation in materials was shown with an Alloy 600 steam generator material, where a known hardness gradient between the outside and inside surfaces were characterized. A strong correlation was found between the hardness measurements and the quantitative analysis from EBSD mapping. CRL has investigated the capabilities in examining natural UO_2 ceramic, and EAC cracking in Zircaloy-4 tubing materials. This characterization technique is capable of being used for fitness for service, failure examination, and optimizing material design of nuclear components.

5. References

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