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Dark-field imaging based on post-processed electron backscatter diffraction patterns of bulk crystalline materials in a scanning electron microscope

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ABSTRACT

Dark-field (DF) images were acquired in the scanning electron microscope with an offline procedure based on electron backscatter diffraction (EBSD) patterns (EBSPs). These EBSD-DF images were generated by selecting a particular reflection on the electron backscatter diffraction pattern and by reporting the intensity of one or several pixels around this point at each pixel of the EBSD-DF image. Unlike previous studies, the diffraction information of the sample is the basis of the final image contrast with a pixel scale resolution at the EBSP providing DF imaging in the scanning electron microscope. The offline facility of this technique permits the selection of any diffraction condition available in the diffraction pattern and displaying the corresponding image. The high number of diffraction-based images available allows a better monitoring of deformation structures compared to electron channeling contrast imaging (ECCI) which is generally limited to a few images of the same area. This technique was applied to steel and iron specimens and showed its high capability in describing more rigorously the deformation structures around micro-hardness indents. Due to the offline relation between the reference EBSP and the EBSD-DF images, this new technique will undoubtedly greatly improve our knowledge of deformation mechanism and help to improve our understanding of the ECCI contrast mechanisms.

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1. Introduction

In transmission electron microscopy, a dark-field (DF) image is obtained when a specific diffraction reflection is excited. This can be accomplished either in the conventional transmission electron microscope (CTEM) or the scanning transmission electron microscope (STEM) by collecting the signal from the diffracted beam corresponding to the selected reflection. Practically, this is performed by placing an aperture (CTEM) or selecting a particular collection angle (STEM) to collect electrons scattered through the Bragg angle corresponding to the specific lattice planes selected.

In the scanning electron microscope (SEM), DF imaging can be achieved in STEM mode as in a dedicated STEM when thin specimens are used [1]. However, no DF imaging has been reported on bulk specimens. Only electron channeling contrast imaging (ECCI) provides a DF type contrast based on the electron channeling pattern (ECP). This Kikuchi-like pattern is an angular distribution of the backscattered electron (BSE) yield obtained when the primary electron beam is scanned over a large specimen area or rocked around the optic axis of the microscope at a specific point of the specimen surface [2]. In fact, when the angle between the beam and the lattice planes is close to the Bragg angle, the BSE yield is proportional to the probability of the electron to be backscattered inside the matter in addition to the Z^2 dependence of Rutherford scattering. This probability is based on the Bloch wave theory [3] and is the square of the coefficient of a Bloch wave contribution divided by the sum of all the square of each Bloch wave contribution. To simplify the calculations, only two Bloch waves are generally used to describe this probability. Bloch wave of type I has its maximum at the atom sites while type II has its maxima between the atom rows. At the exact Bragg angle the contribution of the two Bloch waves is equal. When the scan angle is small, typically at magnification higher than 100 times [4], the intensity at each pixel of the ECCI image is equal to that at the centre of the corresponding ECP at the same pixel position. This acts like a virtual aperture at the centre of the ECP. However, this virtual aperture, used to select the channeling pattern (ECP) area for the signal

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collection, is only limited to the centre of the ECP [4]. Then, the specimen needs to be tilted and rotated to change the region of the ECP that will be located at the centre point.

Payton and Nolze reported the use of diodes on top of the EBSD camera combined to EBSD scan to improve phase identification [5] following the work initiated by Prior et al. on using semi-conductor diodes attached to the EBSD camera [6]. Previous studies were also reported where large areas of the EBSPs were selected to reconstruct the image from an EBSD scan [7,8] and a similar technique was recently commercialized by EDAX researchers [9] during the course of our study, named PRIAS for Pattern Region of Interest Analysis System. However, in these techniques, because of the large regions of the EBSPs used to reconstruct an image, the intensity of one region is the average of several different diffraction conditions (area in the EBSP) and the original diffraction information present in the EBSPs is thus lost. In this work, we report on an innovating technique that provides controlled DF imaging in the SEM. At each pixel, the reported intensity is related to a specific diffraction condition with a better angular accuracy contrary to the previous works cited above. This technique is based on the post-processing of electron backscatter diffraction patterns (EBSPs) and may open interesting applications in the SEM.

2. Materials and instrumentation

The Si specimen used in this work was a 1 × 1 cm² [001] (001) silicon wafer prepared by the cleavage technique. The iron sample was cut from a polycrystalline pure iron rod with a diameter of 7.6 mm (Alfa Aesar, Ward Hill, MA, USA). The small disc was annealed in vacuum at 800 °C for 24 h followed by slow cooling in the furnace. The thickness of the sample was reduced by 4.7% using a compression machine. The compressed sample was then ground with 800 and 1200 grit papers, followed by polishing using 3 and 1 μm diamond suspensions with etching steps (5% and 2% nitric acid in water) in between. The sample was then electro-polished in 20% perchloric acid and 80% ethanol for 5 min (30 s interval) at room temperature. A final polishing step was manually performed with a 50 nm colloidal silica suspension. Indentations were carried out using a Clark Microhardness Tester CM-1000AT (Sun-Tec Corporation, Novi, MI, USA), with a peak load of 50 g. The steel sample was a stress relief annealed non-oriented Si–Fe electrical steel (NOES) cut in a 1.5 × 1.5 cm^2, ground with 800 and 1200 grit papers and polished with 3 and 1 μm diamond suspensions. Final polishing was performed with a 50 nm colloidal silica suspension for 1 h followed by Ar^+ ion beam milling using a Hitachi IM3000 flat milling system (Hitachi High-Technologies, Rexdale, Canada). The accelerating voltage was 3 kV and the angle of incidence in regard to the surface normal was 80°.

ECPs were recorded with a Hitachi SU-3500 thermo-ionic emission gun SEM equipped with a tungsten filament (Hitachi High-Technologies, Rexdale, Canada) and EBSPs were recorded with either a Hitachi SU-70 or a SU-8000 field-emission SEMs (Hitachi High-Technologies, Rexdale, Canada). Both were equipped with a HKL Nordlys electron backscatter diffraction (EBSD) system (Oxford Instruments, Concord, USA) controlled with the Channel 5 package. The EBSD camera screen resolution was 640 × 480 and 1344 × 1024 pixels for the SU-70 and SU-8000, respectively. Pattern acquisition, indexing and simulations were carried out with the Flamenco software which is part of the Channel 5 package. The EBSPs were recorded and stored following the same procedure as for standard EBSD acquisition, i.e., background subtraction and flat fielding was applied. The ECP was acquired with an accelerating voltage of 20 kV at normal incidence with a solid state semi-conductor backscattered electron detector placed on top of the specimen surface and normal to the beam (PD-BSE). The ECP image resolution was 1280 × 960 pixels. The EBSPs were recorded with accelerating voltages of 20 and 30 kV as specified in the text and a tilt angle of 70°, except in Fig. 1 where the tilt angle was 80°. The distance

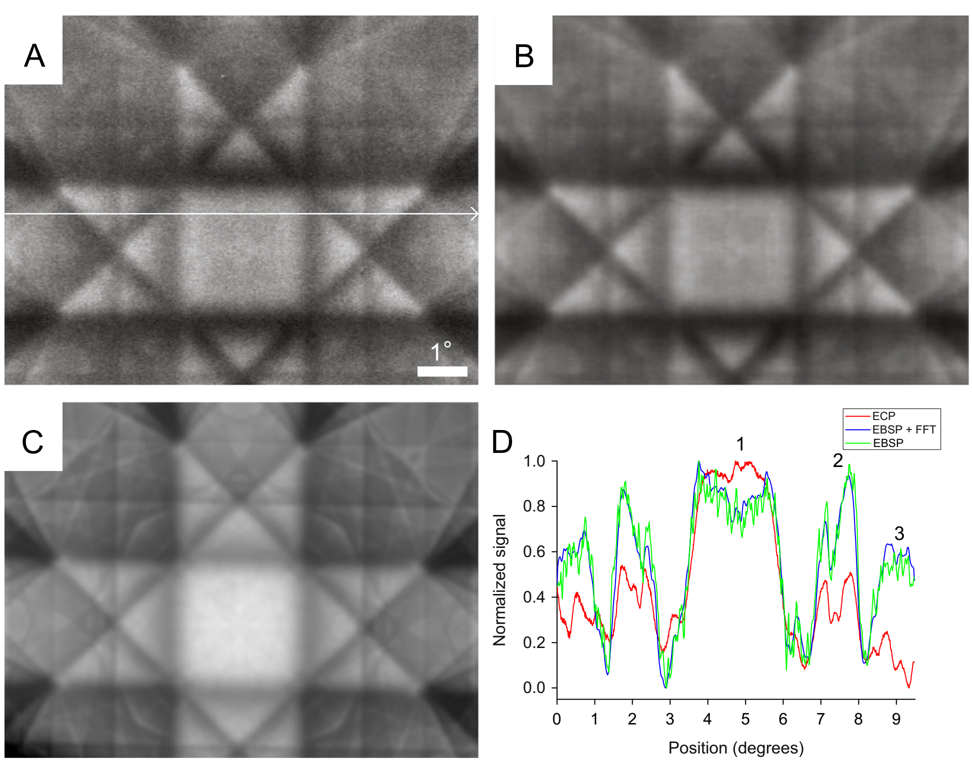

Fig. 1. Comparison between an electron backscatter diffraction pattern (EBSP) and an electron channeling pattern (ECP) of a [001] (001) silicon wafer. (A) Raw and (B) digitally filtered EBSP and (C) ECP. Both were recorded with an accelerating voltage of 20 kV. The working distance was 10 mm for the ECP and the detector distance was 80 mm for the EBSP. Tilt angles were 0° and 80° for the ECP and the EBSP, respectively. (D) Line profiles extracted from (A–C) show the higher resolution obtained with the ECP compared to the EBSP.

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