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Crystal orientation mapping via ion channeling: An alternative to EBSD

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ABSTRACT

A new method, which we name ion CHanneling ORientation Determination (iCHORD), is proposed to obtain orientation maps on polycrystals via ion channeling. The iChord method exploits the dependence between grain orientation and ion beam induced secondary electron image contrast. At each position of the region of interest, intensity profiles are obtained from a series of images acquired with different orientations with respect to the ion beam. The profiles are then compared to a database of theoretical profiles of known orientation. The Euler triplet associated to the most similar theoretical profile gives the orientation at that position. The proof-of-concept is obtained on a titanium nitride sample. The potentialities of iCHORD as an alternative to EBSD are then discussed.

1. Introduction

Electron backscatter diffraction (EBSD) is routinely employed as a characterization tool coupled with a scanning electron microscope (SEM) to obtain individual grain orientations, local texture, point-to-point orientation correlations, and phase identification and distributions on the surfaces of bulk polycrystals [1]. As commercial EBSD systems are now readily and widely available, the technique is no longer reserved to specialized research laboratories. Consequently EBSD has become a tool of choice for process development and quality control. With the goal of full automation in mind, efforts are being made to increase the speed of the technique [2] to obtain orientation maps. Several avenues are currently being explored, such as increasing the sensitivity and speed of the digital camera [3] as proposed by the manufacturers, as well as improving the indexation algorithms for treating and indexing patterns [4]. In this context, any new technique that can ease the implementation of orientation mapping at an industrial scale would be welcome. The precision of the indexation is also the subject of intense research efforts. Indexing algorithms tend to be more and more precise, revisiting the classical Hough transform used to extract the diffraction lines from the Kikuchi patterns [5,6]. Other approaches are currently explored, using for instance the pattern matching technique, which consist in comparing directly the Kikuchi patterns to a database of simulated ones. This idea was proposed originally by Wright et al. in 1991 [7] and was recently implemented again by Chen et al. with the benefits of modern computational power [8]. The same technique has been used in Transmission Electron Microscopy to obtain orientation maps using precession diffraction [9,10].

We propose in this paper a new technique for orientation mapping based on the well-known channeling contrast phenomenon [11,12]. As the channeling contrast is emphasized in ionic images [13], the experiments were conducted in a FIB-SEM instrument to acquire ionic image series as well as comparative EBSD orientation maps. According to the channeling theory for ions and electrons, the intensity of induced secondary electrons (SE) at a given position on the surface of a polycrystalline sample will change if the orientation of the primary incident beam with respect to the sample surface is changed [14]. An intensity profile can then be drawn as a function of the sample orientation, from which information about the orientation of the crystalline lattice at this position can be retrieved. This idea has been suggested several times in the literature [14–16], but, to our knowledge, no comprehensive method currently exists that is capable of yielding full orientation maps on common polycrystalline samples, such as pure metals or metallic alloys. A relevant contribution was recently brought by Veligura et al. [16] but orientation maps were

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obtained only for particles deposited in epitaxy on a substrate, with only a single degree of freedom in rotation for the lattice orientation of the different grains.

This paper is divided into several sections. In Section 2, the approach we used to obtain ionic image series is outlined, with a comparison between a tilting image series and a rotation image series. Section 3 deals with the experimental details and Section 4 addresses the question of the different possible ways to recover the crystallographic orientation associated with an experimental intensity profile. The results and performances obtained with our method on a TiN sample are exposed in Section 5. The following discussion (Section 6) addresses several important points such as the spatial resolution, the time to obtain an orientation map and the applicability of this technique to non-cubic materials as well as multi-phased materials. We conclude this study in Section 7, with an outline of our key results.

2. Intensity profiles as a signature of the orientation

As mentioned in the introduction, the channeling effect has been theoretically modeled both for ions and electrons [17]. These models include a significant role of lattice plane orientation with respect to the charged particle beam and channeling contrast has frequently been applied to enhance microstructural details, especially in SEM [18,19,13,15,16,20]. The work of Yahiro et al. clearly illustrates the variation of the SE signal under a Ga^+ probe, with minima appearing when the beam arrives parallel to low index crystallographic planes [14]. However, if the aim of an experiment is to recover the crystallographic orientation of the sample, acquiring a tilt series of images is clearly not the best choice for symmetry reasons. Usually, the tilt axis lies in the sample surface, let say along the $\mathbf{e_x}$ direction as represented in the drawing of Fig. 1a and b which represent respectively the reference frame, common to the sample and the stage, as well as its position in a stereographic projection. Within a tilt series, the ion or electron beam originally perpendicular to the sample surface along $\mathbf{e_z}$ direction crosses the stereographic projection linked to the reference frame along a plane passing through the center of the projection, containing $\mathbf{e_y}$ and $\mathbf{e_z}$ directions. If we assume that the signal received only depends on the intersections between the beam and the crystallographic planes or directions, the same signal evolution will be obtained for two different crystals with orientations linked by mirror symmetry with respect to the geometric plane in which the beam travels. An example is given in Fig. 1c, showing two stereographic projections corresponding to such mirror orientations, with the beam path represented by a vertical dashed line. A beam tilted around the $\mathbf{e_x}$ direction will cross the same planes at exactly the same angular positions on the two stereographic projections, giving the same intensity variations. This kind of situation does not allow an unambiguous determination of the crystallographic orientation from the obtained intensity profile and additional experiments would then be required. On the contrary, if we give a fixed tilt to the sample, for arguments sake 40°, and then rotate the sample around its tilted normal ($\mathbf{e_z}$ direction), the beam will circulate along a cone figured by the dotted circle in the stereographic projections of a given grain (see Fig. 1c). A series of image acquired during such a rotation of the sample (around a rotation axis not parallel to the beam) then results in unique intensity profiles for a given orientation because there is no other stereographic projection that would result in intersections with the same planes at the same angular positions, with the same sense of rotation. In this work, we used rotation image series to obtain the orientation of the crystal for each point of the region of interest (ROI). Concerning the initial tilt, because of the necessity to cross enough planes to obtain a significant signature, we have chosen rather arbitrarily 40°. It is worth noting that a correction of beam deflection during the image scan has to be included if working at low magnifications [17]. In the present work, the magnification is 500 ×, with an horizontal field of view of 230 μm. In these conditions, the beam deflection is low enough to ignore the effect.

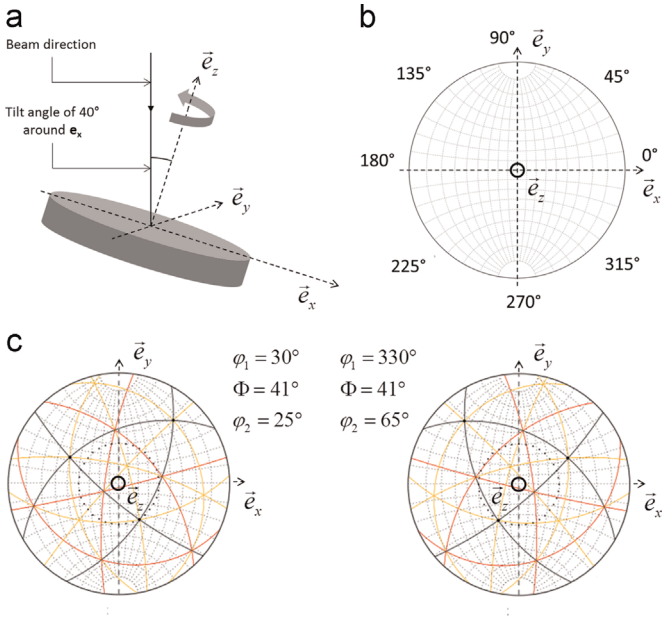

Fig. 1. (a) schematic of the sample together with its orthonormal reference frame ($\mathbf{e_x}$; $\mathbf{e_y}$; $\mathbf{e_z}$) attached to the surface. The beam direction is represented vertically and the tilt angle is defined as the angle between the beam direction and the direction $\mathbf{e_z}$ normal to the surface of the sample. (b) Stereographic projection attached to the sample with the representation of its reference frame. (c) stereographic projections attached to the sample, with the crystallographic plane families {100} {110} and {111} represented respectively in black, yellow and red (color online) for the two orientations Euler triplets indicated. The path followed by the beam during a tilt series around the $\mathbf{e_x}$ axis is represented as a dashed line. The two crystallographic orientations represented are linked by a mirror symmetry with respect to the geometric plane ($\mathbf{e_y}$; $\mathbf{e_z}$) in which the beam travels. *Note*: the dotted circles on the two stereographic projections correspond to the beam path during a rotation series around $\mathbf{e_z}$ assuming a fixed tilt of 40°.

3. Experimental setup

Experiments were carried out on a ZEISS NVision40 FIB-SEM machine in order to obtain both ion-induced electron images and reference orientation maps, acquired with an Oxford Instruments F$^+$™ EBSD system (fast camera NordlysII and Channel 5 software). Concerning the ionic source, the microscope is equipped with a Seiko Zeta column and a Ga liquid metal ion source. FIB images were acquired at 30 kV and a current of 3 nA. This quite high current was chosen to have a probe size of 170 nm that matched the pixel size in the images. Matching the probe size and the image pixel size allows milling the surface evenly, without localized craters during the acquisition of an image series [21]. Moreover, the dwell time per pixel was 0.1 μs, which limits milling, amorphisation and Ga implantation. With these conditions, the ion channeling does not fade out, even after completing the whole series of 360 images. A titanium nitride (TiN) sample was used to acquire ion image series and EBSD maps. Titanium nitride has a face-centered cubic structure with a TiN motif localized at the origin of the crystallographic cell, and in the middle of each face. The sample does not present any strain or any precipitate in its microstructure. The surface of the sample was prepared as for classical EBSD: mechanical polishing, and vibratory polishing