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Sub-micron resolution selected area electron channeling patterns

J. Guyon [a,b], H. Mansour [a], N. Gey [a,b], M.A. Crimp [c], S. Chalal [d], N. Maloufi [a,b,*]

[a] Laboratoire d'Étude des Microstructures et de Mécanique des Matériaux (LEM3), UMR CNRS 7239, Université de Lorraine, 57045 Metz, France
[b] Laboratory of Excellence on Design of Alloy Metals for low-mAss Structures (DAMAS), Université de Lorraine, France
[c] Department of Chemical Engineering & Materials Science, Michigan State University, East Lansing, MI 48824, USA
[d] Carl Zeiss S.A.S, Division Microscopie et Systèmes, 100, route de Versailles, 78161 Marly le Roi, France

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ABSTRACT

Collection of selected area channeling patterns (SACPs) on a high resolution FEG-SEM is essential to carry out quantitative electron channeling contrast imaging (ECCI) studies, as it facilitates accurate determination of the crystal plane normal with respect to the incident beam direction and thus allows control the electron channeling conditions. Unfortunately commercial SACP modes developed in the past were limited in spatial resolution and are often no longer offered. In this contribution we present a novel approach for collecting high resolution SACPs (HR-SACPs) developed on a Gemini column. This HR-SACP technique combines the first demonstrated sub-micron spatial resolution with high angular accuracy of about 0.1°, at a convenient working distance of 10 mm. This innovative approach integrates the use of aperture alignment coils to rock the beam with a digitally calibrated beam shift procedure to ensure the rocking beam is maintained on a point of interest. Moreover a new methodology to accurately measure SACP spatial resolution is proposed. While column considerations limit the rocking angle to 4°, this range is adequate to index the HR-SACP in conjunction with the pattern simulated from the approximate orientation deduced by EBSD. This new technique facilitates Accurate ECCI (A-ECCI) studies from very fine grained and/or highly strained materials. It offers also new insights for developing HR-SACP modes on new generation high-resolution electron columns.

1. Introduction

Since they were first identified by Coates [1] and subsequently interpreted by Booker et al. [2] using a block wave formulation, electron channeling patterns (ECPs) formed from backscattered electrons have been recognized as an effective tools for determining crystal orientations in a scanning electron microscope. In their general form, ECPs are collected at low magnification from single crystals or from very large grains of a polycrystalline material, where the scanning motion of the electron beam leads to a variation in the angle between the incoming electron beam and the crystal lattice planes as a function of position on the sample. These ECPs are then simply collected by imaging with the backscattered electrons. Depending on the microscope, the angular range of ECPs is between 3° and 25°. However, the spatial resolution is quite poor (~1 mm). Consequently, in this configuration, ECPs cannot be used to determine crystal orientations from most polycrystalline materials, which would typically have much finer grain sizes [3].

To overcome this spatial resolution limitation, the selected area electron channeling pattern (SACP) technique was developed, which reduces the area from which a pattern is collected by nominally rocking the electron beam at a point. Different procedures have been proposed to rock the beam in TEM[1][4–6] or to obtain SACPs in SEM [7–9]. The basic approach is to use two coils that are driven separately to rock the beam on an area of interest [10]. In the past, to produce SACPs several SEM microscope configurations have been developed, and due to the configuration, the area from which the pattern is collected can vary significantly in size. Practical issues encountered include: (1) the rocking point is not coincident with the surface of the sample and (2) spherical aberration in the final objective lens deflects the electron beam from the rocking point. Some microscopes have made corrections to improve the spatial resolution of the SACP (including the

* Corresponding author at: Laboratoire d'Étude des Microstructures et de Mécanique des Matériaux (LEM3), UMR CNRS 7239, Université de Lorraine, 57045 Metz, France.
E-mail address: nabila.maloufi@univ-lorraine.fr (N. Maloufi).

[1] Rocking beam diffraction has been proposed in TEM by Vincent and Midgley [4] using the dark field scan coils in a circular hollow cone above the sample. This technique, termed “Double conical beam-rocking system”, allows the measurement of integrated electron diffraction intensities with reduced dynamical effects and opens the way to crystal structure and symmetry determination [5,6].

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CamScan 44 and more recently the Tescan Mira instruments [11], leading to the ability to collect SACPs from relatively small areas, with claims in the range of 5–10 μm for an angular range of $\sim 20°$). However, the spatial resolutions have not been effectively demonstrated in the literature.

Since the early 1990s the ECP/SACP technique has been overshadowed by the development of electron backscattered diffraction (EBSD) pattern analysis. This is because EBSD offers a number of advantages over ECP, primarily good spatial resolution ($\sim$30 nm) and very high data acquisition rates ($>$500 orientations per second) [12–16]. Because of the advantages of EBSD, the use of the electron channeling phenomenon for determining crystal orientation has become uncommon. Despite this, there is one application where SACP offers significant advantages over EBSD, that being in relation to the imaging of dislocations and stacking faults in the near surface region of bulk samples using electron channeling contrast imaging (ECCI). In recent years, the use of ECCI has become more common, with a significant number of groups carrying out studies on a variety of materials [11,17–27]. The reason SACPs have an advantage over EBSDs for conducting ECCI studies is that in order to carry out quantitative studies, it is necessary to control the electron channeling conditions (in the same manner that specific diffracting "two-beam" conditions are used in diffraction contrast TEM analysis). The angular accuracy of EBSD is typically limited to about 1–2° [28–31], and does not readily give the orientation with respect to the incident beam direction (optic axis). In contrast, SACPs directly display patterns with respect to the incident beam direction and does so with accuracies in the range of 0.1°, suitable for determining not only the specific set of planes used for imaging (g-vector), but also for quantifying the deviation from the Bragg condition (s or ω).

Unfortunately nearly all recently developed microscope configurations, particularly those with the field emission guns necessary for ECCI, preclude the ability to readily collect SACPs. As a consequence, many ECCI studies can only be considered semi-quantitative as they do not consider the specific imaging conditions. As an example, high quality ECC images of dislocations using the Gemini column have been shown. However, because this column is not equipped with a dedicated SACP mode, the approximate channeling conditions have been estimated based on EBSD patterns [26]. Other ECCI studies, using FEI field emission gun columns, have been limited to single crystals [32–34].

In this paper, we present in detail a new approach for collecting SACPs on the Gemini column without further modifications. This approach results is the first report of sub-micron spatial resolution SACP, with demonstrated 500 nm resolution, which we term "High Resolution SACP". The resolution is demonstrated by a new, robust procedure for accurately determining the area that the SACP is collected from. This new approach to collecting SACP opens a new path towards improved SACP resolution and quantitative ECCI [35], not only with the Gemini electron column, but also with other new generation high-resolution electron columns.

2. Methodology

An innovative procedure has been developed to rock the electron beam on the high resolution Gemini electron column, which has no dedicated SACP mode. This procedure creates an SACP pivot point by combining control of the aperture alignment coils and the scanning coils, using them in a manner quite different from their primary functions. The beam is first deflected from and then brought back parallel to the optic axis by the aperture alignment coils; after the action of the scanning coils and the objective lens, the electron beam strikes the specimen surface at an angle. Because the off-axis beam is deflected in the scanning coils, the beam does not strike the sample at the optic axis. Thus, beam shift corrections controlled by the scanning coils are applied to ensure that the tilted beam strikes the sample at the intersection point of the sample surface and the optic axis; this point is the pivot point necessary for SACP. For a comprehensive understanding of this rocking beam procedure, it is important to review the specific configuration of the Gemini column and clarify the primary functions of the coils we used to rock the beam in this study.

2.1. Gemini column in conventional scanning mode

Simplified schemes of this column under different functionalities are given in Fig. 1. Unlike many scanning electron microscopes, the beam aperture in the Gemini column is not controlled by a mechanically selectable and aligned aperture, but instead by a series of coils that deflect the electron beam to the appropriate sized aperture hole in a multihole aperture (MA). That is, the beam is moved (in most cases off-axis) to the desired aperture, rather than the aperture being moved to and centered around the beam. This aperture control is accomplished by two sets of deflection coils. The first (upper) set deviates the beam to a selected hole (gun alignment coils, GC) and the second set (lower) brings the beam back to and parallel to the optic axis (aperture alignment coils, ApC). Both sets of coils have similar construction and arrangement, permitting displacement of the beam parallel to the optic axis after it passes through the aperture. Specifically, each assembly of coils consists of two sets of coils lying in different planes; in each plane, four co-planar Helmholtz coils allow the beam to be displaced in the X and Y axes of the coils. The currents applied to these coils are fully user controllable through the parameters Gun (and Aperture) Align X and Align Y. It should be noted that above these sets of coils there is a magnetic condenser lens that is activated only in high probe current mode to boost the electron density of the beam. The column also includes stigmator coils for classical corrections of the distortion of the electron beam. However, both of these components are working in the same manner in conventional scanning mode and in the HR-SACP rocking mode presented here, and will be ignored in this discussion.

Under normal imaging conditions (Fig. 1), the objective lens assembly (C3) focuses the beam at the desirable working distance. The objective lens consists of a combined electrostatic/electromagnetic lens triplet to minimize achromatic and spherical aberrations. A single-stage beam scanning system is integrated in the lens, just above the electromagnetic lens gap, resulting in minimization of transverse chromatic and other lenses aberrations. If no beam shift is superimposed on the scanning coils (SC), the beam is scanned symmetrically about the optic axis (Fig. 1a). Note that the focusing action of the objective lens inverts the direction of the beam deflection controlled by the SC. To scan over an area of interest not centered on the optic axis, without moving the sample, an additional beam shift can be applied, as illustrated in Fig. 1b. This beam shift applies an offset to the range of current variation of the SC. The X and Y beam shifts are also user controllable parameters.

2.2. Detailed principle of the rocking beam mode

Our rocking beam method is illustrated Fig. 2, starting from the basic concept of ECP collection in conventional scanning mode (Fig. 2a) and progressively showing the way the SACPs are collected in rocking beam mode (Fig. 2d). The scheme of the column shows the beam striking a large grain or single crystal in scanning mode at low magnification in Fig. 2a–c, but striking the sample at a single point in Fig. 2d. The resulting backscattered electron (BSE) images and SACP are represented schematically below each figure.