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A method for extraction of crystallography-related information from a data cube of very-low-energy electron micrographs

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ABSTRACT

Scanning Low Energy Electron Microscopy (SLEEM) is an imaging technique which uses low energy electrons while providing a very good image resolution. Reflectivity of very slow electrons in the range 0–30 eV can be correlated with the electronic structure of the material, aiming at the determination of the local crystallographic orientation. Since SLEEM is a 2D imaging method, a suitable algorithm is needed to pre-process the image data depending on the beam energy as the third dimension. The crucial task is to detect grain boundaries in polycrystals and evaluate the image signal in connection to the energy of electron impact. Recent algorithms performing the task for the traditional EBSD method are not suitable as they do not address the side-effects of the SLEEM technique. We propose a method that detects the grain boundaries while correcting for image distortion caused by the variation of cathode lens strength, and for several other issues.

1. Introduction

Scanning Low Energy Electron Microscopy (SLEEM) is an electron microscopy technique in which a focused beam of electrons of arbitrarily low incident energy is scanned over the sample, and the resulting signal is used for imaging [1]. This technique boasts excellent lateral resolution, close to the values acquired at the usual high primary beam energies of 10–30 keV. Image aberrations that would be impractically large in a low energy beam are kept low here owing to the fact that the primary beam is kept at high energy throughout the entire electron optical column, and is decelerated only shortly before it hits the sample. This is done by the insertion of an electrostatic field (a *cathode lens*) spanning the few millimetres between a negatively biased sample and a grounded electrode above it. This setup allows setting the incident electron energy to an arbitrary value, going down to units or even fractions of electron volts. The field accelerates and collimates the signal electrons towards the earthed detector above the sample, assuring a high collection efficiency and high amplification of the image signal. The image signal consists both of elastically and inelastically scattered electrons, as there is no energy filtration. Under the energy of the plasmon excitation threshold however, the elastic mean free path dominates over the inelastic one [2]. One important feature is the ability to detect the entire emission of the backscattered electrons, including those emitted at high angles with respect to the surface normal. At low energies and with their complete angular distribution acquired, the backscattered electron images offer enhanced information about crystalline and electronic structures thanks to contrast mechanisms that are otherwise unavailable [3]. Effects not available at traditional electron energies are connected with the electron wavelength approaching interatomic distances so that diffraction and interference phenomena can appear even in the reflected electron flux, as is the case in the low-energy electron diffraction (LEED) experiment.

True low-energy electron diffraction beams besides the specular (00) beam do not appear below a certain energy threshold which is around 30 eV for most materials and normal incidence [4].

At low energies, the crystalline information is enhanced, as for example, the grain contrast in polycrystalline materials. The reasons for this phenomenon include the dependence of the generation and absorption of SE on crystallographic orientation, which goes also for electron backscattering. In ordinary vacuum conditions, it is also the increased influence of surface layers, such as oxides, the thickness of which is also orientation dependent.

High-angle BSE bears significantly enhanced crystallographic information. When immersing the sample in a strong electric field, we can easily control the acquisition of the high-angle BSE signal; this is true even in the case of only slight deceleration.

Reflectivity of electrons in the very low energy range of 0–30 eV can be correlated with the electronic structure of the material [5], since that is unique to the local crystallographic

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orientation [6]. Although the correspondence is far from straightforward, the main reflectivity $R(E)$ features represent the peculiarities of DOS [7,8]. In this energy range, each crystal face exhibits a unique reflectivity curve (see Fig. 8b) which is indicative of its particular crystallographic orientation.

2. Experimental setup

Polycrystalline high purity copper with a highly varying distribution of crystallographic orientations of its grains was chosen to prove this concept. The sample was cleaned by solvents and inserted into an ultra-high vacuum SLEEM microscope. Subsequently, the sample was *in situ* cleaned by multiple cycles of argon ion sputtering and by flashing to 400–450 °C. The surface cleanliness was evaluated using Auger Electron Spectroscopy (AES). The ultra-high vacuum conditions and meticulous in situ sample cleaning allowed us to observe a pristine surface of the sample, free of any oxide layers or hydrocarbon contamination. For verification, copper single crystal samples of a defined orientation were included in the experiment as well. The samples were imaged at very low electron energies in the range from 0 to 30 eV with a step of 0.3 eV, yielding 100 micrographs that form a data cube. Each single 8-bit micrograph is actually a 2D distribution map of low energy electron reflectivity which varies within the image due to the different crystallographic orientations of each grain (Fig. 1). The input data of the algorithm can be considered a 3D matrix (data cube). Indices x and y are coordinates in the two-dimensional image function $f(x, y)$, which is acquired for a range of beam energies (usually 0–30 eV) that represents the third dimension of the matrix.

Our work aims at high-speed semi-automatic determination of crystallography-related information with a high lateral resolution and surface sensitivity.

3. Image processing

The whole stack of images is first processed one by one (*static mode*) to obtain grain boundaries in each image. Due to the interchanging contrast of the neighbouring grains, at no single energy are all boundaries to be seen distinctly, as there are always some grains and energies for which the contrast disappears and the boundary is not apparent (see Fig. 7a). In the second phase (*dynamic mode*), the obtained boundary maps are merged together and image transformations are performed to compensate for the distortions inherent to SLEEM optics. This is needed because as the incident electron energy is varied in the range of 0–30 eV, so does vary the strength of the cathode lens introducing a variety of undesired image effects [9]. In the third phase, for the merged image that provides a complete boundary representation, the region of each single grain is demarcated, which allows us to determine the brightness function within the area of the grain (see Fig. 8a).

3.1. Processing of a single micrograph, "Static mode"

The static mode is designed to evaluate a single micrograph and provide as much information as possible about the grain boundaries. A single micrograph represents reflectivity information at the particular incident beam energy; however, the information from a single micrograph cannot be used for obtaining the complete grain border map. For this reason, the "static mode" algorithm is used repeatedly (the so-called "dynamic mode") through the entire energy range of the data cube. For the static processing, six basic steps are performed.

Image acquisition may be considered as the *first step*; as it was mentioned before, the static mode is designed to evaluate a micrograph obtained at a single energy. Gain and Black Level parameters are set at the beginning of the measurement and kept constant for all energies.

Secondly, *image distortion* is being compensated. The image distortion is caused by the electron optical system of the SLEEM. For this reason, the image is rescaled using affine transformation. An affine transformation, such as translation, rotation, scaling or shear, is one in which parallel lines remain parallel even after being transformed. The transform attribute is defined by transformation matrices T and T', as illustrated in Fig. 2b. The transformation matrices T and T' are automatically obtained between two neighbouring energies so that the entire stack of images has the

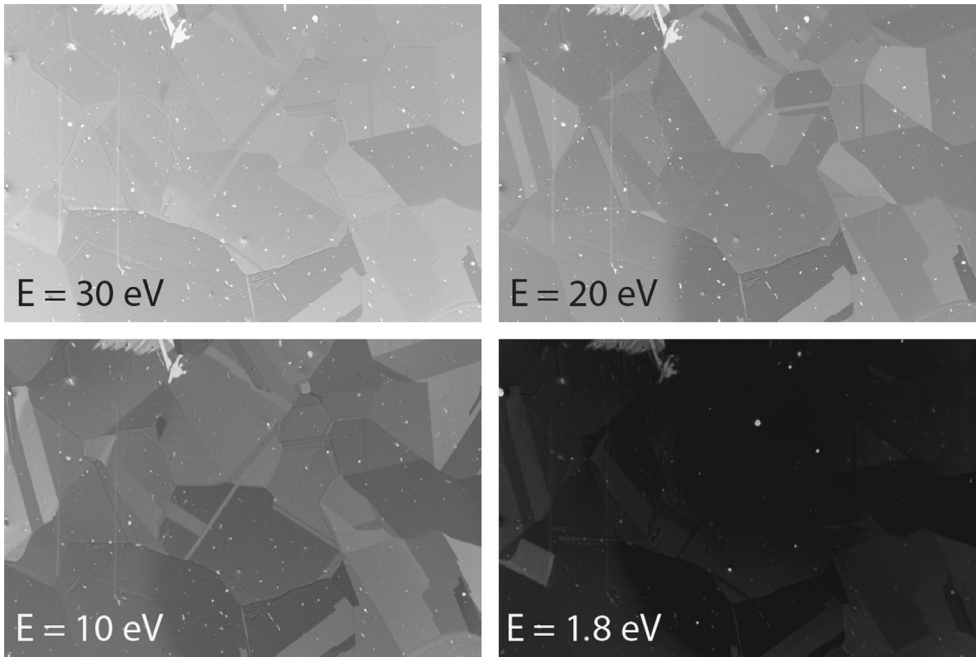

Fig. 1. Micrographs of polycrystalline Cu at incident energies 30 eV, 20 eV, 10 eV, and 1.8 eV showing surface oxide contaminations.