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Exploring transmission Kikuchi diffraction using a Timepix detector

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ABSTRACT: Electron backscatter diffraction (EBSD) is a well-established scanning electron microscope (SEM)-based technique [1]. It allows the non-destructive mapping of the crystal structure, texture, crystal phase and strain with a spatial resolution of tens of nanometers. Conventionally this is performed by placing an electron sensitive screen, typically consisting of a phosphor screen combined with a charge coupled device (CCD) camera, in front of a specimen, usually tilted 70° to the normal of the exciting electron beam. Recently, a number of authors have shown that a significant increase in spatial resolution is achievable when Kikuchi diffraction patterns are acquired in transmission geometry; that is when diffraction patterns are generated by electrons transmitted through an electron-transparent, usually thinned, specimen. The resolution of this technique, called transmission Kikuchi diffraction (TKD), has been demonstrated to be better than 10 nm [2, 3]. We have recently demonstrated the advantages of a direct electron detector, Timepix [4, 5], for the acquisition of standard EBSD patterns [5]. In this article we will discuss the advantages of Timepix to perform TKD and for acquiring spot diffraction patterns and more generally for acquiring scanning transmission electron microscopy micrographs in the SEM. Particularly relevant for TKD, is its very compact size, which allows much more flexibility in the positioning of the detector in the SEM chamber. We will furthermore show recent results using Timepix as a virtual forward scatter

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2017 JINST 12 C02075

detector, and will illustrate the information derivable on producing images through processing of data acquired from different areas of the detector. We will show results from samples ranging from gold nanoparticles to nitride semiconductor nanorods.

Keywords: Detection of defects; Gamma detectors (scintillators, CZT, HPG, HgI etc); X-ray detectors

Contents

1 Introduction

Direct particle detectors are becoming an essential tool for the development of non destructive material characterization techniques. Pixelated particle counting detectors such as Timepix/Medipix2 [4, 6] have been proven to be extremely useful for electron microscopy applications [7]. The ability to set an energy threshold, which allow to counting only particles within a selected energy window, has been proved to increase the signal to noise ratio and the spatial frequencies in the recorded data [8].

One of the techniques where the use of direct detectors has brought clear advantages over the conventional detection systems is electron backscatter diffraction (EBSD), a very powerful technique used in scanning electron microscopy for the characterization of crystalline materials. EBSD allows the measurement of crystal orientation, crystal phase identification, texture and strain over a large range of length scale, from nanometres to millimetres, with a very high spatial resolution, of the order of few tens of nanometres [9].

EBSD patterns are recorded in a scanning electron microscope (SEM), by placing an EBSD detector in front (with the sensitive screen nearly parallel to the incident electron beam) of a tilted specimen to image electrons that have been backscattered from the specimen when a beam of electrons is focused at a point of the specimen's surface. As consequence of the elastic and inelastic interaction of the electrons with the specimen's structure, electron diffraction patterns are manifested as a number of bright bands in the pattern, called Kikuchi bands. The geometrical arrangement and the intensity of the Kikuchi bands in the EBSD pattern provide the crystallographic information in an EBSD analysis.

A number of authors [2, 3] have recently shown that is possible to increase the spatial resolution of the EBSD technique by acquiring Kikuchi diffraction patterns using a transmission geometry, where the diffraction pattern is no longer generated by the backscattered electrons, but from the electrons transmitted through an electron transparent specimen. This variant of EBSD is typically called transmission EBSD or transmission Kikuchi diffraction (TKD). The spatial resolution of TKD has been demonstrated to be better than 10 nm, making possible the characterization of materials having a truly nanoscale texture [2, 3].

An EBSD/TKD detector typically consists of an electron-sensitive screen such as a phosphor or a scintillator. When electrons impinge on the screen they produce a fluorescence which is then recorded using a combination of lenses and CCD cameras. Such detectors limit the performance of EBSD system, for example by intrinsic light scattering and optical absorption within the phosphor which may degrade the quality of the recorded diffraction patterns.

This paper describes the advantages of using the Timepix detector to perform TKD and transmission electron diffraction, in particular its compact size which allows much more flexibility in positioning the detector in the SEM chamber. This allow the acquisition of spot diffraction patterns and more generally the acquisition of scanning transmission electron microscopy micrographs in the SEM. The use of the Timepix chip as a virtual forward scatter detector [10] is also described, showing how dark field images can be produced exhibiting a variety of contrast mechanisms through processing of data acquired from different areas of the detector.

2 Experimental details

2.1 Timepix

The Timepix is a pixelated detector developed by an international collaboration (Medipix2) hosted at CERN [4]. The Timepix electronics offers 256 × 256 pixels having 55 μm pitch, covering a global surface of around 1.4 cm × 1.4 cm. Each pixel of the sensor has its own amplification pixel in the electronics chip. Each pixel of the detector can be individually programmed in order to work in one of the three possible configurations: Medipix, Time over threshold, and time of arrival mode [4]. In this paper we use the Timepix working in Medipix mode, where the device is used as a counter. For each pixel, an internal counter increments one unit every time an electron has an energy above the threshold value, this acts effectively as an high pass filter which allows the counting of electrons having an energy above a specific value. The global threshold set for the chip can be adjusted individually for each pixel, in order to compensate for small differences between pixels. In this regard the Timepix chip has been preferred to the Medipix2 [6] chip because it gives somewhat better threshold equalization when compared to the Medipix2 chip, due to the fact that the Timpix has 4 threshold adjustment bits for the threshold whereas the Medipix2 chip only has 3 per threshold.

The Timepix chip used in the experiments consisted of a 300 μm thick silicon sensor, biased with a voltage of 100 V.

The readout was serial and the read out operation and data acquisition were managed by a FitPix readout interface [11] and Pixelman software [12].

2.2 TKD set-up

TKD was performed in an FEI Sirion Schottky field emission scanning electron microscope. The geometrical set-up used for the experiments is sketched in figure 1(a). A Timepix detector is mounted on the SEM's motorized stage; a metal shield is used to protect the PCB Timepix board and to prevent damage to the electronics by the electron beam, as shown in figure 1(b).

The sample is kept above the Timepix chip by using a sample holder which is rigidly fixed on the Timepix metal shield. The SEM motorized stage is used to control the position of the specimen within the SEM chamber.

The SEM column generates a focused beam of electrons having an energy which can be varied between 0 and 30 keV, with a size of few nanometres, which can be positioned and/or scanned over the specimen's surface. In the geometrical set up used in the experiment, the electron beam direction is nearly perpendicular to the Timepix chip.

The electrons impinging on the top of the specimen surface are transmitted through the electron transparent sample and scattered in a forward direction toward the Timepix chip. By varying the specimen to the detector distance is possible to set the solid angle imaged by the detector. An example of a TKD pattern is shown in figure 1(c) where diffraction (Bragg) spots and Kikuchi bands are shown.

In order to perform TKD mapping, i.e. acquiring TKD patterns from a mash of points of the specimen's surface, software was developed to control both the SEM beam position on the surface of the specimen and the Timepix operations.

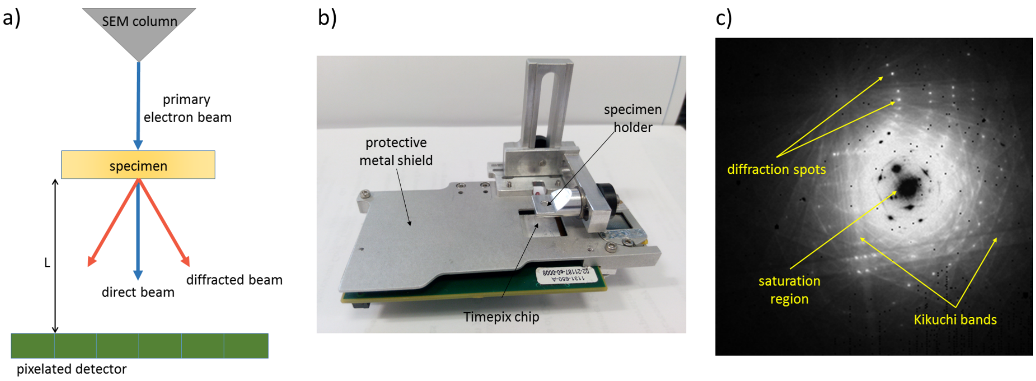

Figure 1. Description of the experimental set up used for transmission Kikuchi diffraction experiments, showing a) the schematic of the experimental geometry, b) the Timepix-specimen mechanical holder, and c) example of a TKD pattern obtained from a $\approx$ 100 nm thick AlN specimen showing the main features observed in a TKD pattern.

2.3 Virtual detectors

Often commercial EBSD/TKD systems are equipped with semiconductor diodes, typically placed at the top (back scatter direction) and the bottom (forward scatter direction) of the phosphor screen.

2017 JINST 12 C02075

These are used to obtain micrographs of the specimen by monitoring the intensity of the signal, e.g. number of electrons, as function of the electron beam position on the specimen. Similar information can be obtained by using the same dataset obtained from an EBSD/TKD map, as described in ref. [10], by using the phosphor screen as an imaging tool.

The use of a EBSD/TKD detector for imaging purposes has advantages. It makes it possible to select one or more areas of the screen and use them as a virtual diodes, making possible the generation of micrographs obtained by monitoring the intensity of scattered electrons in specific directions and the combination of them to obtain a variety of contrast mechanisms [10].

2.4 Sample detail

A number of specimens were used in this study in order to emphasize different aspects of the technique and illustrate possible applications:

- polycrystalline gold nano particles coated with amorphous silica, used to demonstrate the spatial resolution of TKD;
- GaN nanorods, used to show the effect of the thickness on the TKD pattern formation;
- AlGaN layer structure on laterally overgrown AlN/sapphire template, used to show the potential of the VD imaging technique.

3 Results and discussion

3.1 TKD on gold nanoparticles

A first example of TKD mapping was obtained from two gold nanoparticles deposited on a transmission electron microscopy (TEM) grid. The map was obtained by acquiring 50×38 points using a step size of approximately 6 nm. A dark field image extrapolated from the TKD map, using virtual detectors, is shown in figure 2(a). It is possible to distinguish three main regions: the dark background corresponding to the TEM grid composed of carbon; two gold nanoparticles showing the strongest intensity and the silica capsules showing intermediate intensity. The contrast observed in the image is a typical Z contrast type observed in dark field TEM images, giving in this case compositional information.

Diffraction patterns were observed only in the gold region. Figure 2(b) and 2(c) show two patterns obtained by the silica capsules and the gold particles respectively. By comparing patterns obtained within the same gold nanoparticles, we were furthermore able to determine its internal polycrystalline structure. The step size in this case has been chosen to give also an indication on the high spatial resolution of the technique. In this geometry the diffraction patterns show a strong background, i.e. anisotropic intensity on the screen not containing diffraction information. It is also possible to notice that the area on the Timepix chip hit by the direct electron beam shows a big dark spot, as shown also in figure 1(c). This effect is observed when a large flux of electrons hits a single pixel, creating a saturation effect. The explanation of this effect could be attributed to the combination of two causes, the exceeding of the maximum count rate in the pixel when a large flux of particles is hitting a single pixel, and the cumulative deposition of energy by a number of particles in one pixel, in the same measurement, preventing the detector signal to go below

2017 JINST 12 C02075

the threshold level, and therefore effectively only giving only one count every measurement. This saturation effect is clearly an artefact and it is contained in all the TKD patterns shown in this paper.

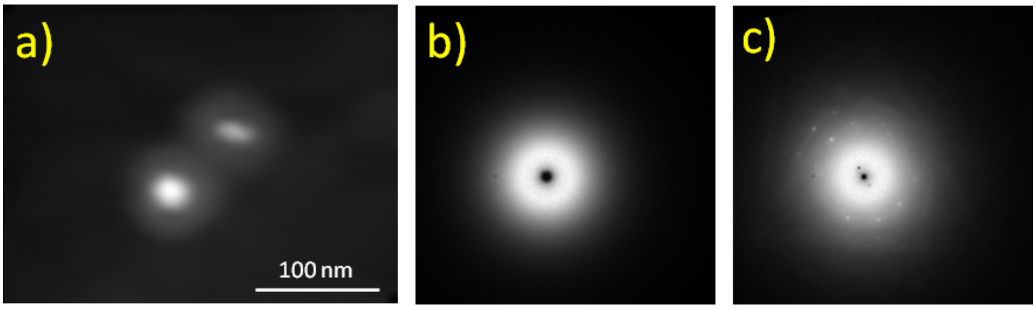

Figure 2. TKD map obtained from gold nanoparticles deposited on a TEM grid. a) Dark field image obtained by using virtual detectors, b) TKD pattern obtained from the amorphous silica capsule region and c) TKD pattern obtained from the gold region.

3.2 TKD on GaN nanorods

To investigate the effect of the specimen thickness on the TKD pattern formation we obtained a TKD map from a mono crystalline GaN nanorod. This sample is essentially an hexagonal pillar terminating with a pointed tip; in this region the thickness is reduced from approximately 700 nm to virtually zero at its tip, see the SEM image in figure 3(a). In order to highlight the most important aspects we acquired three TKD patterns from three regions of the GaN nanorod tip; these three regions have a thickness of less than 30 nm, between 30 nm and 300 nm, and larger than 300 nm respectively. The TKD patterns acquired from these regions are shown in figures 3(b)–3(d). To facilitate the visualization of the data and increase the visibility of the diffraction features, the patterns were flat fielded. The difference between the three patterns is significant. The first pattern obtained with a reduced specimen thickness is predominantly composed of diffraction spots [13] and the Kikuchi diffraction is almost invisible. As the thickness is increased, figure 3(c), both the Bragg diffraction and Kikuchi diffraction contribution are visible. When the thickness is too high, figure 3(d), a strong absorption effect occurs, due to the inelastic scattering, which is manifested by the almost complete disappearance of the diffraction spots and the reversed intensity observed in the Kikuchi bands which are no longer bright but dark. This effect is discussed in ref. [14]. For each material the choice of an optimum thickness is essential.

3.3 TKD on a AlGaN/AlN/Sapphire structure

A cross section of a AlGaN/AlN/Sapphire laterally overgrown structure (specimen detail can be found in ref. [15]), was focused ion beam thinned to approximately 100 nm. A TKD map obtained from the AlN region of the specimen is shown here as an example of the use of virtual detectors. In this case TKD patterns were acquired using an electron beam energy of 30 keV and a detector threshold of approximately 29 keV, to remove the contribution of low loss electrons. A comparison between two TKD patterns acquired from the same region but different threshold levels is shown in figure 4(a) and 4(b) using approximatively 5 keV and 29 keV threshold energy respectively.

2017 JINST 12 C02075

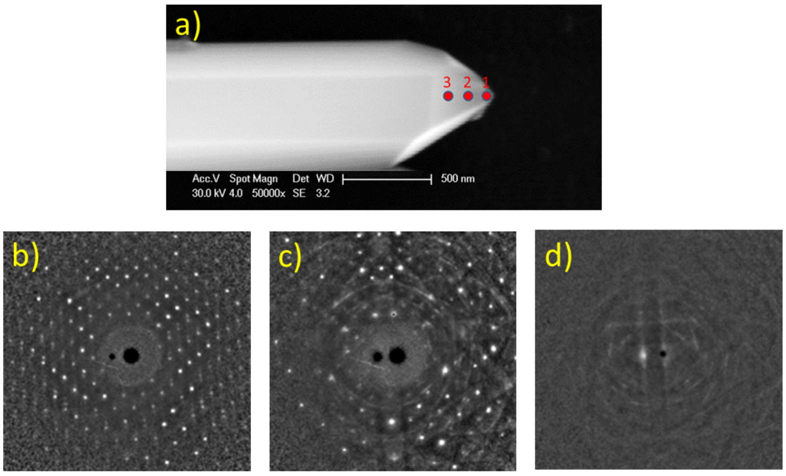

Figure 3. TKD map obtained from GaN nanorod scratched on to a TEM grid. a) SEM image of the nanorod showing three different areas from which TKD patterns were acquired. TKD patterns obtained from thicknesses of b) less than 30 nm, c) between 30 nm and 300 nm, d) more than 300 nm.

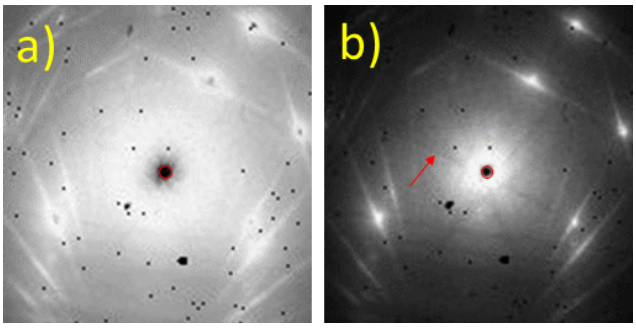

Figure 4. Comparison between two TKD patterns acquired with different threshold energies, corresponding to approximately a) 5 keV and b) 29 keV. The red circle indicates the position of the direct beam.

Due to the removal of high loss electrons, the increase in the contrast of the high filtered pattern is clearly visible, as discussed previously in ref. [5]; this is visible for example in figure 4(b), where the red arrow highlights the region around the direct beam where the contrast of the dark Kikuchi lines is much stronger when compared with the one observed in the less energy filtered pattern shown in figure 4(a).

The contrast observed in dark field images obtained using virtual diodes depends strongly on the position of the virtual diode. Figure 5(a) shows the two positions, labelled in red, of the two virtual diodes used to obtain the images shown in figure 5(b) and 5(c).

The difference between the two dark field images is substantial. Although the interpretation of the contrast shown in figure 5(b) and 5(c) is beyond the scope of this paper, it shows that is possible to highlight different aspects of the specimen structure by simply changing the virtual diode position. The use of images obtained by using virtual diodes, and their combination, offers an additional complementary tool for TKD.

2017 JINST 12 C02075

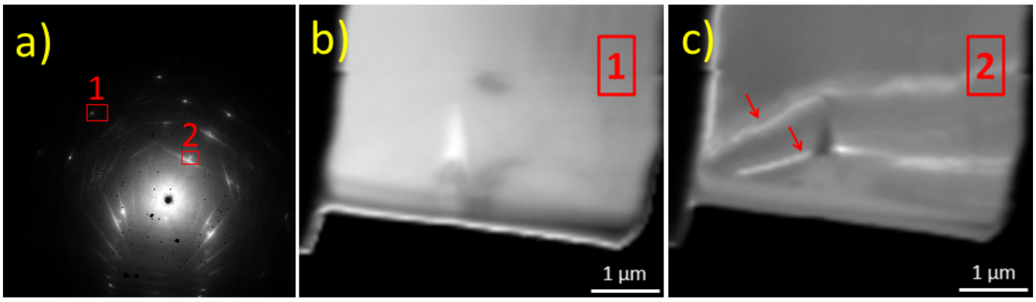

Figure 5. Comparison of the contrast obtained using virtual detectors as a function of their position. a) Position of the virtual detectors, labelled in red, b) dark field image obtained using the virtual detector labelled with “1” and c) dark field image obtained using the virtual detector labelled with “2”.

4 Final remarks

The Timepix chip has been used to demonstrate TKD. It allows much more flexibility in positioning the detector in the SEM chamber. In particular it allows easy placement of the detector on the SEM mechanical stage. The use of virtual detectors for the acquisition of dark field images in the SEM has been demonstrated, showing compositional contrast from gold nanoparticles, and the study of their internal structure. In addition, different types of contrast may be revealed through varying the position of the virtual diode used for the imaging. The contribution of diffraction spots to the TKD patterns depending on the specimen thickness has been shown and discussed. Although diffraction spots are not currently used in TKD analysis, they contain additional information which can be potentially used in TKD experiments, to extract diffraction condition information [16], which is of fundamental importance for the interpretation of dark field images. The energy discrimination still remains one of the most interesting capabilities of the Timepix, which allows low energy electrons to be removed from the TKD patterns, increasing the TKD pattern contrast. Combined with the decrease in the exposure doses and the decrease in the electron energy which can be used for the electron imaging [5], the Timepix chip proves to be a very suitable detector for TKD applications.

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2017 JINST 12 C02075

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2017 JINST 12 C02075