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Introducing a non-pixelated and fast centre of mass detector for differential phase contrast microscopy

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ABSTRACT

With the advent of probe corrected STEM machines it became possible to probe specimens on a scale of less than 50 pm resolution. This opens completely new horizons for research, as it is e.g. possible to probe the electrostatic fields between individual rows of atoms, using differential phase contrast (DPC). However, in contrast to conventional DPC, where one deals with extended fields which can be assumed constant across the electron probe, this is not possible for sub-atomic probes in DPC. For the latter case it was shown [1,2], that the strongly inhomogeneous field distribution within the probe diameter, which usually is caused by the nuclear potentials of an atomic column, leads to a complicated intensity redistribution within the diffraction disk. The task is then to determine the intensity weighted centre of the diffraction disk pattern (frequently also called centre of mass, COM), which is proportional to the average lateral momentum gained by the average electron, transmitted through the probe diameter. In first reported measurements, the determination of this COM was achieved using a pixelated detector in combination with a software-based evaluation of the COM. This suffers from two disadvantages: first, the nowadays available pixelated detectors are still not very fast (approximately 1000 fps) and quite expensive, and second, the amount of data to be processed after acquisition is comparatively huge.

In this paper we report on an alternative to a pixelated detector, which is able to directly deliver the COM of a diffraction disk's intensity distribution with frequencies up to 200 kHz. We present measurements on the sensitivity of this detector as well as first results from DPC imaging. From these results we expect the detector also to serve well in sub-atomic DPC field sensing, possibly replacing today's segmented or pixelated detectors.

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1. Introduction

The development of probe-corrected scanning transmission electron microscopy (STEM) enabled mapping the potential of electric field distributions on the picometre scale [2,3], using differential phase contrast microscopy (atomic DPC). This gain in lateral resolution requires new measurement techniques for differential phase contrast. In conventional DPC measurements the deflection of the electron beam due to electric or magnetic fields inside the specimen is typically measured with a segmented annular ring detector which is based on the layout introduced by Chapman et al. in 1990 [4]. This is made possible due to the fact that the electron probe size (nanometre range) is much smaller than the extent of the deflecting fields (some tens of nanometres and above), and thus every electron experiences a homogeneous field

https://doi.org/10.1016/j.ultramic.2018.05.003 0304-3991/© 2018 Elsevier B.V. All rights reserved. which leads to a lateral momentum change. This, in turn, leads to a shift of the - in this case homogeneously lit - diffraction disk in the detector plane, not altering its intensity profile. This is also true, because the convergence angle of the illuminating beam is smaller than the usual Bragg angles, which prevents an overlap of diffracted beams with each other and the central beam cone. For atomic DPC the measured fields are no longer homogeneous across the beam diameter and therefore the top-hat like intensity profile of the diffraction disk is altered by diffractive effects to an inhomogeneous distribution. When performing atomic DPC measurements with an annular DPC detector such a redistribution of intensity can falsely be interpreted as a shift of the diffraction disk. For example, even without a displacement of the disk itself, an increase of intensity on one segment due to diffractive effects would be interpreted as a shift in the direction of this segment. This was discussed in more detail by MacLaren et al. [5] based on DPC measurements on grain boundaries in a polar material. Further, elastic and inelastic beam broadening can cause inaccuracies when evaluating DPC measurements as it affects the size of the diffraction disk which





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itself influences the field sensitivity of a annular DPC detector (for homogeneous and inhomogeneous intensity profiles) [6].

To circumvent the effects mentioned above, the annular detector is often replaced by a (pixelated) detector that is capable of measuring the whole intensity distribution in the diffraction disk. The intensity weighted centre or also frequently called centre of mass (COM) \vec{R}_{COM} is obtained by the summation over all *N* diffraction pattern coordinates \vec{r}_i (pixel positions) weighted by their corresponding local intensities j_i and normalized by the total intensity J_{tot} .

$$\vec{R}_{\text{COM}} = \frac{\sum_{i=1}^{N} \vec{r}_i \cdot j_i}{\sum_{i=1}^{N} j_i} = \frac{1}{J_{\text{tot}}} \sum_{i=1}^{N} \vec{r}_i \cdot j_i$$
(1)

Müller–Caspary and Krause showed that this COM can be related to the quantum mechanical expectation value of the lateral average momentum transfer on the electron ensemble interacting with an electric field of arbitrary shape inside the specimen. Thus the COM, independent of the diffraction disk's homogeneity, can directly be related to the field strength and direction [1,2] at a certain scanning position on the specimen.

Typically, the COM is measured with pixelated detectors like fast CCD or MOSFET based TEM cameras [7–15]. For each scanning position P(x', y') on the specimen the two-dimensional intensity distribution j(x, y) of the diffraction disk is acquired. Please note that the primed coordinates relate to the specimen's coordinate system, while the unprimed coordinates are those in the detector plane. Based on those four-dimensional data sets the COM for each pixel position *P* can be calculated (compare Section 2.2.3). Due to the required read-out of a complete image of the diffraction disk per pixel position, these cameras have a typical acquisition time of about 1 ms per diffraction pattern which is fairly slow compared to non-pixelated solid state detectors (1–10 µs per pixel).

For measurements where it is sufficient to know the exact location of the COM rather than having knowledge of the complete locally varying intensity distribution, we present a non-pixelated and fast COM-detector based on a commercially available duo-lateral position sensitive diode (PSD). The lateral-photo-effect upon which a PSD is based was first described by Wallmark in 1957 [16]. A PSD is a planar silicon pin-diode with a resistive layer and a pair of electrodes on top and bottom (see Fig. 1 and [17]). With this device, it is possible to obtain the position of the COM of the diffraction disk by measuring the ratio of the current through opposing electrodes. The advantages of this detector compared to pixelated detectors are its high acquisition speed (up to 200 kHz, or, respectively, 5 µs per pixel), its intrinsic ability to measure the absolute position of the COM, and its small dimensions of $(5 \times 5 \times 1.5)$ cm³ which allows a rather easy implementation into an existing microscope. Furthermore it is far less expensive compared to pixelated COM detectors. In its working principle a PSD is a practical realization of a so called first-moment detector which was theoretically described by Waddell et al. in 1977 [18] and Waddell and Chapman in 1979 [19] for the first time. They can show that a first-moment detector is the ideal system to image strong phase objects.

In the following, we will discuss the working principle of a PSD as a centre of mass detector in electron microscopy. We will present first tests with a PSD in our microscope showing sufficient signal strength and good responsiveness to movements of the electron beam and an actual measurement of magnetic domains.

2. Duo lateral PSD as COM detector in STEM

2.1. Why PSD?

In this study we investigated the applicability of a duo-lateral position sensitive diode (compare schematic drawing in Fig. 1(a)) as a COM detector for differential phase contrast microscopy. The main reason for experimenting with PSDs as detectors for electron differential phase contrast microscopy were their feature to directly measure the COM-position of an illuminating beam of radiation. Up until now, PSDs are widely used in the position measurement and alignment of laser beams (UV to IR range). In this area of application, they are a commonly used and well understood device which makes it easy to adapt this measurement technique for use in electron microscopy. As will be shown in Section 2.2 the measured position describes the COM of the intensity profile on the PSD. With regard to DPC measurements this means that we can measure the diffraction disk's absolute movement on the detector without need of an initial calibration to obtain absolute positions. Further, changes of the beam's COM-position caused by alterations of its intensity profile due to e.g. diffractive effects can be detected. The fairly simple layout of PSDs, based on semiconducting pin-diodes, makes them a reliable device. Furthermore PSD sensors with large active areas (edge lengths up to 45 mm) can be constructed that still posses a reasonably short rise times (below 10 μ s), still allowing up to 10⁵ measurements per second.



Fig. 1. (a) shows a schematic drawing of a silicon based duo-lateral PSD illuminated by an infinitesimally small focused parallel electron beam that is shifted along the x axis by x_{COM} . (b) shows a cross-section of the situation shown in **a** along the x-axis. Secondary electron hole pairs created at the incident position of the beam are separated by the potential gradient of the space charge region and further by the reverse bias voltage. The holes/electrons move on the shortest path from the intrinsic layer (high resistivity) to the p-/n-doped resistive layer (lower resistance). Then e.g. the hole current to the electrodes X1 and X2 (I_{X1} and I_{X2}) depends on their respective distance between the incident beam and the electrode [17].

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