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Strain Analysis by Nano-Beam Electron Diffraction (SANBED): Present performance and future prospects

K. Müller[1], H. Ryll[2], I. Ordavo[2], D. Zillmann[1], M. Schowalter[1], J. Zweck[3], H. Soltau[2], S. Ihle[4], L. Strüder[4], K. Volz[5], P. Potapov[6], A. Rosenauer[1]

[1]Universität Bremen, Bremen, Germany
[2]PNSensor GmbH, München, Germany
[3]Universität, Regensburg, Germany
[4]MPI Halbleiterlabor, München, Germany
[5]Philipps Universität, Marburg, Germany
[6]GLOBALFOUNDRIES Dresden Module One, Dresden, Germany

mueller@ifp.uni-bremen.de
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Via metal-oxide semiconductor field effect transistors (MOSFET), light emitting or laser diodes, semiconductor nanostructures have developed to a central part of everyday economic and scientific life in industrial countries. Common to nearly all these structures are stoichiometric and strain variations at atomic scale which govern electronic and optical properties. Consequently, large effort on accurate and precise strain measurement in large areas with the high spatial resolution provided by transmission electron microscopy (TEM) is spent by the community since decades.

This study addresses strain analysis from diffraction pattern series acquired in scanning TEM (STEM) mode. In particular, three algorithms [1] have been developed to accurately determine the positions of discs in convergent beam electron diffraction (CBED) patterns originating from a focused STEM probe with a semi-convergence angle of 2.6mrad. From disc positions it is then straight forward to calculate strain via Bragg's equation. In comparison to traditional techniques which use parallel illumination [e.g. 2], we demonstrate that the spatial resolution is improved by a factor of 5 to 0.5nm. On the other hand, CBED disc recognition is more complex than Bragg spot detection because of the rich inner intensity structure as depicted in Figure 1a, which furthermore drastically changes throughout a series of CBED patterns due to variations of specimen thickness and -orientation.

As the most direct approach, we developed the selective edge detection and circle fitting algorithm (SE), in which a Prewitt-type edge detection is applied to raw CBED disc patches, leading to edge pixels shown in Figure 1a, too. Erroneous edges are ruled out by iteratively fitting circles to the edge pixels, whereas edges with the largest distance to a fit are ignored in subsequent iterations. In this way, we finally obtain the fit of the disc border on the right of Figure 1a. For a scan through the 5-fold stack of $In_xGa_{1-x}N_yAs_{1-y}$/GaAs visible in the high-angle annular dark field (HAADF) inset in Figure 1d, we obtain the black strain profile exhibiting an alternating tensile/compressive strain sequence over a distance of 600nm. Whereas SE evaluation takes 15min, a second method called radial gradient maximisation (RG) leads to the same profile as expressed by the blue curve in Figure 1d in 1min. Here, CBED disc position and radius are found by maximising the difference between the sum of rotational averages inside (coloured rings in Figure 1b) and the sum outside (blue rings) the CBED disc. A third method, cross-correlation with masks (CC), again yields equivalent strain as shown by the red profile in Figure 1d, now 75 times faster than SE. As an example, Figure 1c shows two masks which can be used for cross-correlation with the CBED discs. However, the lower one is preferable as it stresses the disc border and suppresses the inner disc structure. In contrast to SE, RG and CC perform best at log-scaled intensities.

While sub-nm resolution over a field of view of more than 500nm with a strain precision of 0.07% is very promising, strain analysis by nano-beam electron diffraction (SANBED) faces several challenges: To enable 2D strain mapping with a sampling of 200x200 scan points, detectors with high quantum efficiency, high signal-to-noise ratio and ultrafast readout are required to record large numbers of CBED patterns in finite time. In pilot works [3], we used a pnCCD [4,5] camera, which is a direct electron, scintillator-free CCD detector, to record CBED patterns with up to 1kHz rate using the same specimen as in Figure 1d. As shown in Figure 2a for a pnCCD frame time of 200ms, the result of Figure 1d (obtained with a conventional scintillator-based Gatan UltraScan2000 CCD with 500ms frame time) is reproduced accurately with respect to strain profile shape and -precision. As the insets in Figure 2a exhibit, this is not self-evident since direct electron detection at 300kV causes split events

an hence significant point spread. However, it has been shown [3] that this mainly causes an isotropic blurring of CBED discs and does not alter the position detection result.
In addition, Figure 2b shows the same strain profile again which has now been obtained from frame times of 1ms. Obviously, precision decreases slighty by a factor of 1.8 but on the other hand acquisition speeds up by a factor of 200, enabling 200x200 2D strain map acquisition in 40s.

Another challenge for SANBED is the compensation of varying crystallographic orientation and specimen thickness for applications where strain is to be mapped at large (15□m²) scale. To this end, electron precession experiments have been conducted via scripting at the COM-interface of an FEI Titan 80/300 machine, to correlate beam tilt and diffraction shift for recording a static CBED pattern while varying the beam tilt. As clearly visible in Figures 2c-d, a semi-opening angle of the precession cone of 1mrad already leads to a nearly homogeneous intensity distribution inside the discs which enhances large-scale SANBED analysis significantly.

Future prospects for SANBED are therefore essentially related to improvements of acquisition hardware as to speed and detection quantum efficiency, development of efficient scripts for automated, dedicated user applications such as a combination of electron precession and STEM, as well as implementing algorithms like CC to in-situ evaluation of CBED disc positions during SANBED scans. Finally, SANBED is very promising in combination with HAADF Z-contrast imaging to allow for simultaneous mapping of strain and composition as the CBED pattern passes the inner detector hole.

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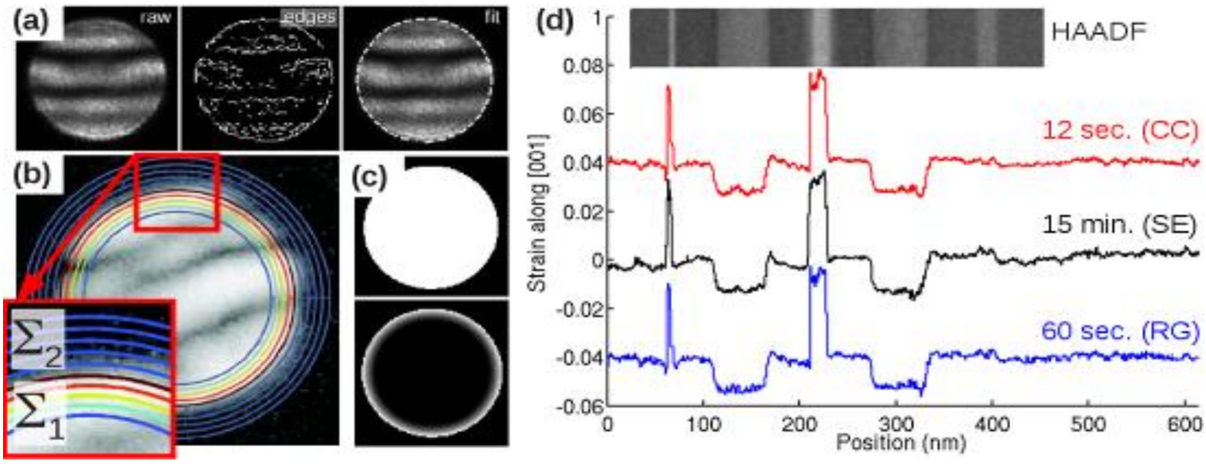

Figure 1. (a-c) Disc position and –radius recognition by **(a)** selective edge detection and circle fitting (SE), **(b)** radial gradient maximisation (RG) and **(c)** cross-correlation with masks (CC). **(d)** SANBED strain profiles for SE,RG,CC.

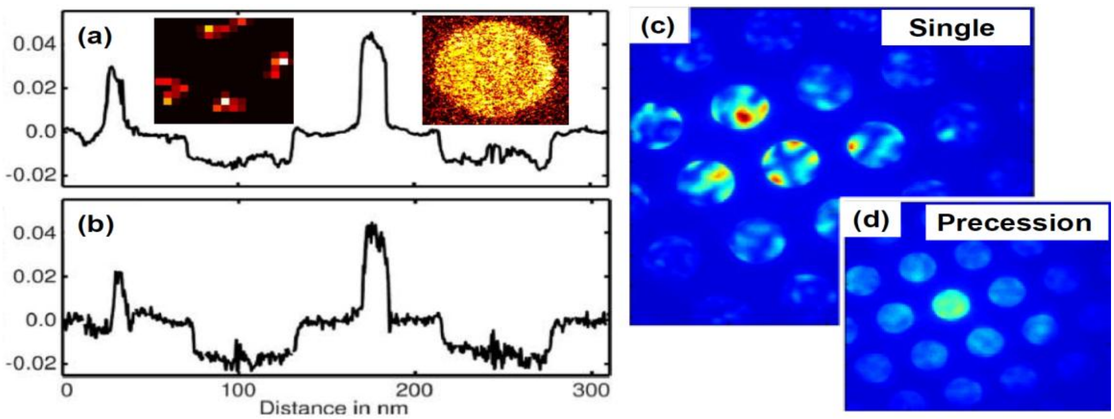

Figure 2.(a,b) Strain profiles as in Figure 1(d) but obtained with the direct electron detector pnCCD for 200 and 1ms frame time, respectively. Single events and a CBED reflection are shown as inset in (a). **(c)** Single CBED pattern. **(d)** CBED pattern obtained by precession and de-scan of the diffraction shift.