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Study of Na_{0.44}MnO₂ by Manual Diffraction

Tomography using Beam Precession TEM method. <u>Athanassios S. Galanis</u>,^{ab} Mauro Gemmi,^d Fotini Karavassili,^a Alexandros Lappas,^c Irene Margiolaki,^a Stavros

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Precession Electron Diffraction (PED) in combination to

Automated / Manual Diffraction Tomography (ADT / MDT) is a

recently developed strategy which leads to improved diffraction

data both in terms of quantity and quality to study nm size

crystals in TEM (Transmission Electron Microscope),

impossible to study with conventional X-Ray techniques.

Diffraction tomography data are collected through a tilt of the

single crystal around an arbitrary crystallographic axis and

diffraction patterns are recorded in a tilt interval defined by the

operator, while the beam is precessing (PED) on a cone surface

[1]. Although automatic acquisition routines are mostly

efficient, this technique can be manually applied in any TEM. In

this work we present the study of the $Na_{0.44}MnO_2$ structure. This

structure has subsequently received attention as a potential

material for use in lithium-ion batteries, when the Na ions in the

pores are replaced with Li [2]. Although Na_{0.44}MnO₂ has been

structurally studied by X-ray techniques [2] we present herein

the first study of this material by means of electron diffraction

applying the MDT/PED method. MDT/PED analysis were

carried out on a Zeiss Libra 120 Plus transmission electron

microscope operating at 120 kV with an in-column Omega filter

and equipped with a Nanomegas Digistar P1000 device for

precession electron diffraction. In MDT, the sample was rotated

around an arbitrary axis and consequently 113 off-zone patterns

were taken every 1° from -60° to +52°, while the precession angle was kept at 1°. The patterns were energy filtered with a slit

of 10 eV centered on the zero loss peak in order to reduce the

background noise improving the integration of weak peaks. The

patterns were processed with the ADT3D software which provided the correct unit cell and integrated 733 reflections that

allowed to recover only the fully occupied Na site, while the

MS37-P1 Line Edge Roughness estimation in SEM images of lithographic nanostructures. <u>Konrad Bojar</u>, *Industrial Research Institute for Automation and Measurements, Poland,* E-mail: kbojar@piap.pl

We present a novel method for estimation of Line Edge Roughness (LER) in images acquired by the Scanning Electron Microscopy (SEM) method. Typical application of the LER is characterization of edge deviation from linearity in photo-etched semiconductor structures [1]. Wrong assessment of non-linearity and irregularity of edges in highly integrated circuits leads, for example, to parasitic couplings disturbing signal propagation [2]. In current literature, the LER is calculated as mean deviation of a curve from a straight line obtained by some fitting procedure, and the curve is understood as a chain of white pixels in the thresholded image, pre-filtered using some 2D wavelet transform [1,3,4]. This approach has this substantial drawback that edges in the SEM image are assumed to be crisp and thin, while they are inherently fuzzy and of width greater than one pixel, especially for lithographic masks and photoresists of lower quality. We propose an algorithm tailored to overcome this problem; its key property is fuzziness of edges. This algorithm is divided into three parts. In the first part straight line edges are estimated, in the second part true, curvilinear edges are calculated, and in the third part the LER is computed. In the first step a resolution pyramid is calculated by means of the 2D wavelet transformation using Haar wavelets. Then, straight, linear edges are fitted by fuzzy Hough transformation described in [5] (with some custom modifications), applied to the edge strength image computed from the pyramid level of lowest resolution. Each of the resulting edges is crisp in the longitudinal direction and fuzzy in the transversal direction. Subsequently, edges are updated in higher resolutions to obtain linear edge density map 1 for the original SEM image. The second part of the algorithm follows the multiscale edge detection method described in [6], except for the last step of thresholding. Instead of the original thresholding method, it is proposed to suppress values smaller than the threshold to 0, and to leave values higher than the threshold unchanged. In this manner we cut out noise and obtain edge intensity map e. Edge membership functions e and l are used in the third part of the algorithm to compute the LER. Our formula for the LER is root mean squared distance from the linear edge to true, curvilinear edge. This definition is inspired by the ó quantity widely used to characterize the LER [1,3]. The only difference between 6 and the quantity proposed here is located in the concept of distance between straight line and a curve. In the fuzzy framework this distance is defined as a respective integral of Euclidean distance function against a measure obtained naturally from the edge membership functions e and l. The proposed algorithm can be used to assess the LER in SEM images containing complex structures, like gates, without need to normalize intensities, pre-rotate image, or choose multiple regions of interest and manually perform numerous LER assessments.

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Keywords: Line Edge Roughness; fuzzy; multiresolution

to true, curvilinear ntity widely used to nce between ó and concept of distance zy framework this Euclidean distance



$$\label{eq:structural} \begin{split} Fig.1 & Na_{0.44}MnO_2 \ structural \\ model \ derived \ from \\ MDT/PED. \end{split}$$

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