Angular sensitivity of crystallographic orientation analysis by scanning nanobeam electron diffraction

H Kirmse, I Haeusler

Humboldt University of Berlin, Institute of Physics, TEM group, Newtonstr. 15, 12489 Berlin, Germany.

holm.kirmse@physik.hu-berlin.de
scanning nano-beam diffraction, crystallographic orientation mapping, ZnO

Scanning nano-beam transmission electron diffraction (SNBD) offers the opportunity to gain valuable information on the crystallographic orientation of various kinds of nano-sized structures e.g. isolated nano-crystals, textured nano-materials, ferecrystals and others [1]. Moreover, the distribution of various nano-sized phases can be analysed basing on the input of crystallographic data of expected crystalline phases.

The structural perfection of a single crystalline phase might be reduced when growth is performed at comparably low temperatures. As an example, hybrid materials combining inorganic and organic components have to be deposited at low temperatures since the organics being weakly-attached to the substrate have to be prevented from desorption, cracking and reorganisation during overgrowth. Recently, almost perfect homoepitaxial growth of ZnO at 100 °C by molecular beam epitaxy was reported [2]. In particular, the high structural perfection of the ZnO cap layer is the basis for successful deposition of a multilayer hybrid inorganic/organic system. Hence, the structural perfection of the low-temperature (LT) grown ZnO cap layer has to be studied in detail.

First, established methods of transmission electron microscopy (TEM) were applied for structural characterization (Fig. 1). In Fig. 1a) a sketch of the sample structure is given. Cross-sectional diffraction contrast dark-field imaging of a homoepitaxial area reveals platelet-like contrast features in the LT-grown ZnO cap hinting to local disorientation of the $10\underline{1}0$ lattice planes (see Fig. 1b). The areas of homogeneous intensity have a width of about 30 nm and a height up to 10 nm. The angle of disorientation is hard to estimate from such kind of an image. Both, high temperature (HT) grown (300°C) and LT-grown ZnO (100°C) look almost perfect beside a few artefacts originating from the conventional preparation for TEM. The high-resolution TEM image of Fig. 1c) exclusively shows the LT-grown ZnO cap layer. The 0002 lattice planes exhibit some deviation from horizontal alignment. For a quantification of the lattice distortion geometric phase method [3] will be applied.

In order to analyse the crystallographic orientation of ZnO in more detail, the ASTAR system of NanoMegas installed at the TEM/STEM JEOL 2200 FS (FEG, 200 kV) was used. The analysis bases on comparison of experimental transmission electron diffraction patterns recorded in nano-beam mode and a set of calculated diffraction patterns. The angular resolution of the data set is defined by the number of calculated patterns. In case of a hexagonal crystal an angular resolution of 0.05° is realized by about 5000 patterns.

Figs. 2 a), b) and c) give orientation maps for the three spatial directions x, y and z, respectively. The indices of the identified lattice planes are encoded as shown in the colour zone selector of Fig. 2d). The colour coding using the full angular range limits the sensitivity to 10°. Thus in the three orientation maps no disorientations can be detected.

Adapting the angular range to the small orientation variations certainly present in the map, as done in Fig. 3a), the colour zone selector covers a solid angle of ± 4° around the [21.0] zone axis. The orientation map of Fig. 2a) is again represented in Fig. 3b) with the adapted angular range. Some disorientations are visible but still a correspondence to the contrast features seen in Fig. 1b) for the LT ZnO cap layer is missing. In order to gain quantitative data, the disorientation of the [21.0] zone axis was extracted along two lines defined in the HT ZnO and in the LT ZnO cap layer. As concluded from Fig. 3c) the angular orientation interval of HT ZnO amounts to 0.8° (cf. the cross markers), whereas the orientation interval for the LT ZnO cap layer is 1.6°. This tendency corresponds to the findings of diffraction contrast imaging (see Fig. 1b). The size of the homogeneously oriented domains ranges from 5 to 15 nm.

In our study we will discuss potential sources for the disorientation.

References

[1] P. Moeck *et al*., Cryst. Res. Technol. **46** (2011), p. 589.
[2] S. Blumstengel *et al*., Appl. Phys. Lett. **98** (2011), p. 031907.
[3] M. Hÿtch *et al*., Nature **423** (2003), p. 270.
[4] The authors gratefully acknowledge the provision of the sample by F. Hennberger and S. Blumstengel (HU Berlin, Photonics group) and TEM preparation by E. Oehlschlegel. Work was financially supported by German Research Foundation (DFG) in the framework of the SFB 951.

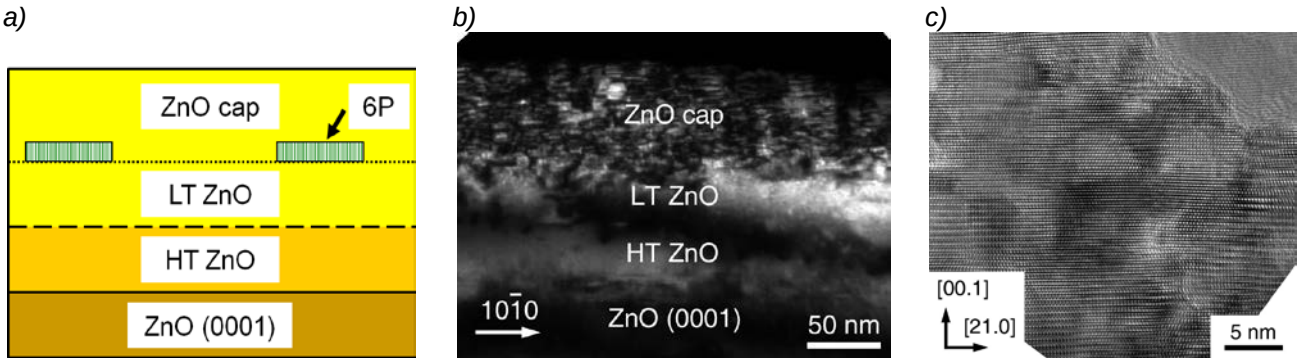

Figure 1. *TEM analysis of homoepitaxial ZnO growth (without organic sexiphenyl (6P)) comprising high-temperature (HT), low-temperature (LT) grown ZnO, and LT-grown ZnO cap: a) sample structure, b) diffraction contrast image (dark field), c) high-resolution TEM image of ZnO cap layer.*

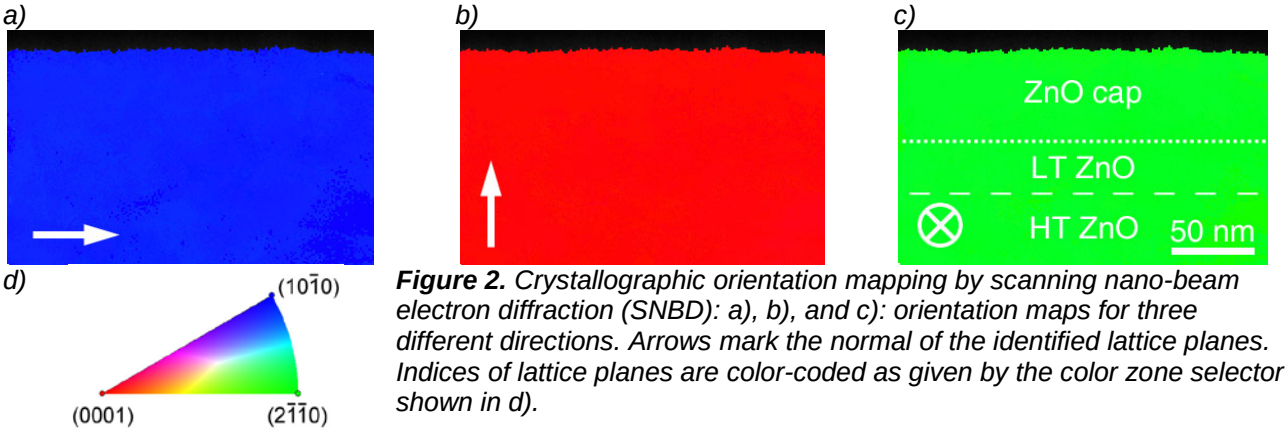

Figure 2. *Crystallographic orientation mapping by scanning nano-beam electron diffraction (SNBD): a), b), and c): orientation maps for three different directions. Arrows mark the normal of the identified lattice planes. Indices of lattice planes are color-coded as given by the color zone selector shown in d).*

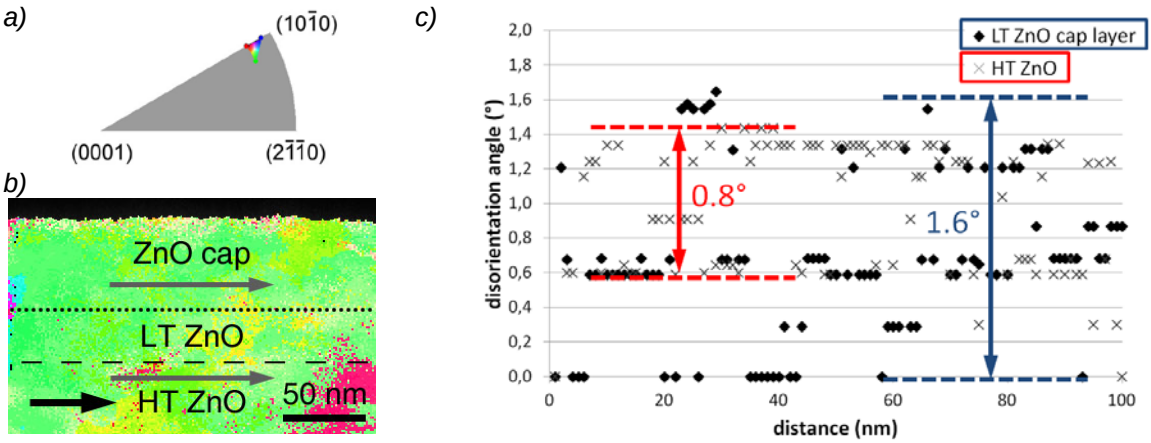

Figure 3. *Analysis of crystallographic orientation as identified by SNBD with optimized angular sensitivity: a) color zone selector with adapted angular range of color coding, b) map of lattice planes aligned normal to the black arrow marker (same data set as in Fig. 2a was used), c) disorientation analysis of the [21.0] zone axis of HT ZnO and LT ZnO cap layer. The gray arrows seen in Fig. b) mark the two paths of analysis.*