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STEM tomography and CBED polarity analysis of crystallographic pores in InP

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The sophisticated 3D architecture of crystallographic pores produced by electrochemical etching in III-V compound semiconductors, like GaAs, GaP and InP, has attracted considerable research interest [1,2]. During formation of such pores the crystal polarity plays a key role in selecting the pore growth directions and pore branching which together determine the 3D architecture. In a previous work the morphology of crystallographic pores in n-type InP(001) was analysed by conventional transmission electron microscopy (TEM) [3]. By a detailed study of thin TEM lamella prepared in different crystal orientations combined with convergent-beam electron diffraction (CBED) for polarity analysis a qualitative model of the pore branching and 3D pore morphology could be derived. A few results of this earlier study are summarized in Figure 1. More details can be found in reference [3].

Here we present results of an electron tomographic study of these crystallographic pores. Electron tomography (ET) is an advanced microscopy technique which allows obtaining 3D information from the reconstruction of a tilt series consisting of two-dimensional images [4]. For the investigation a (101) TEM lamella inclined from the (001) surface by 45° has been chosen since it contains two <111> pore growth directions enabling the study of extended pores and of pore branching.

A STEM image acquired with a FEI Titan^3 80-300 at 200 keV using a camera length of 46 mm (Figure 2a) reveals the distribution of pores parallel and inclined to the surface. However, the real 3D morphology (faces and branches of the pores) cannot be clearly distinguished. Therefore, ADF-STEM electron tomography has been performed at the same microscope working at 300 keV, using a microprobe mode with a convergence semi-angle of 3 mrad to obtain enough depth of focus [5]. A camera length of 29.5 mm was used during the acquisition of the tilt series which was recorded from -64° to +59°, with 1° tilt increments. For 3D-reconstruction the Simultaneous Iterative Reconstruction Technique (SIRT) has been applied with 50 iterations. From these reconstructions the 3D-morphology of the pores and of pore branches can be studied (Figure 2b). The pores possess a triangular cross-section with oscillating pore diameter. For some pores, like the one shown in Figure 2b), the oscillation is small resulting in a more or less uniform pore channel. Other pores show much stronger oscillation and appear to consist of interconnected tetrahedrons. From the CBED analysis (Figure 1) it can be concluded that the inner surfaces of such pores are dominated by polar In-terminated {111}A facets.

A detailed 3D analysis of crystallographic pores in InP will be presented. In particular, by combining electron tomography and CBED polarity determination the relation between the crystal polarity and the three-dimensional pore distribution and pore shape will be clarified.

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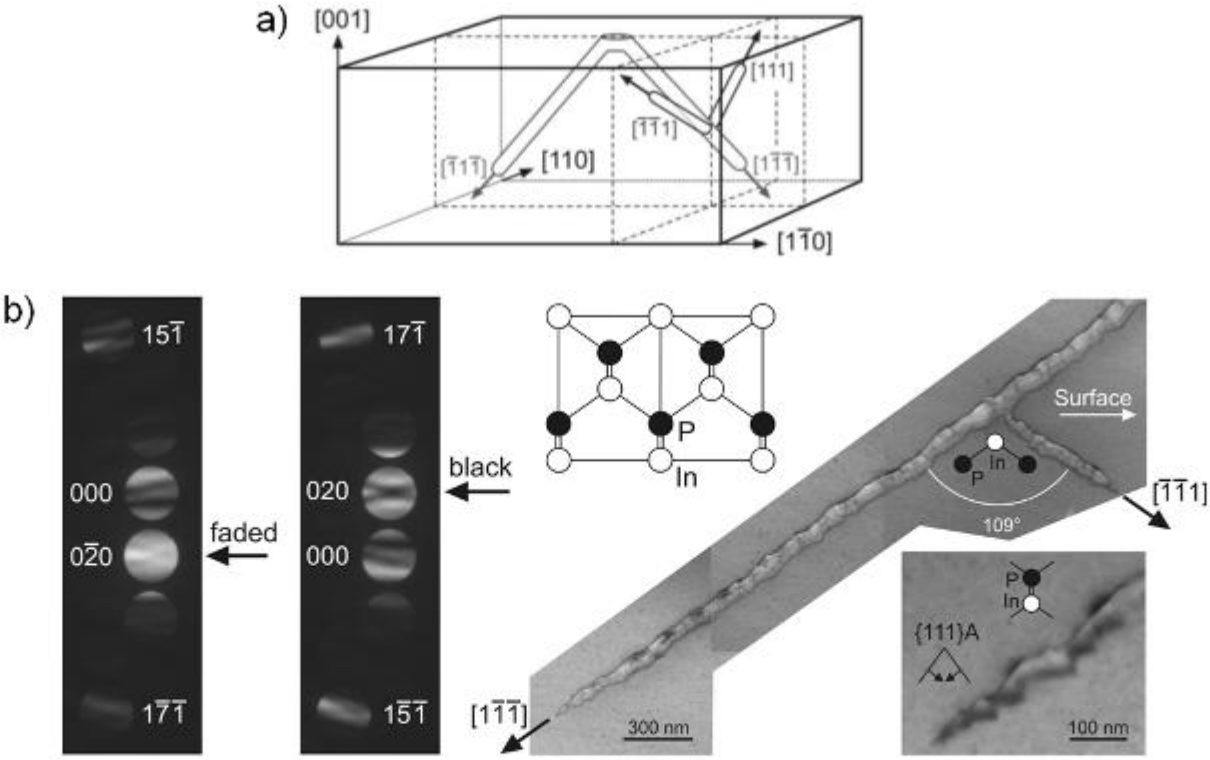

Figure 1. Crystallographic pores in InP obtained by electrochemical etching [3]: a) Schematic of pore branching along four <111> directions of equal polarity. b) Determination of crystal polarity by CBED and TEM bright-field imaging of a branching pore and a very pore tip showing oscillating contrast.

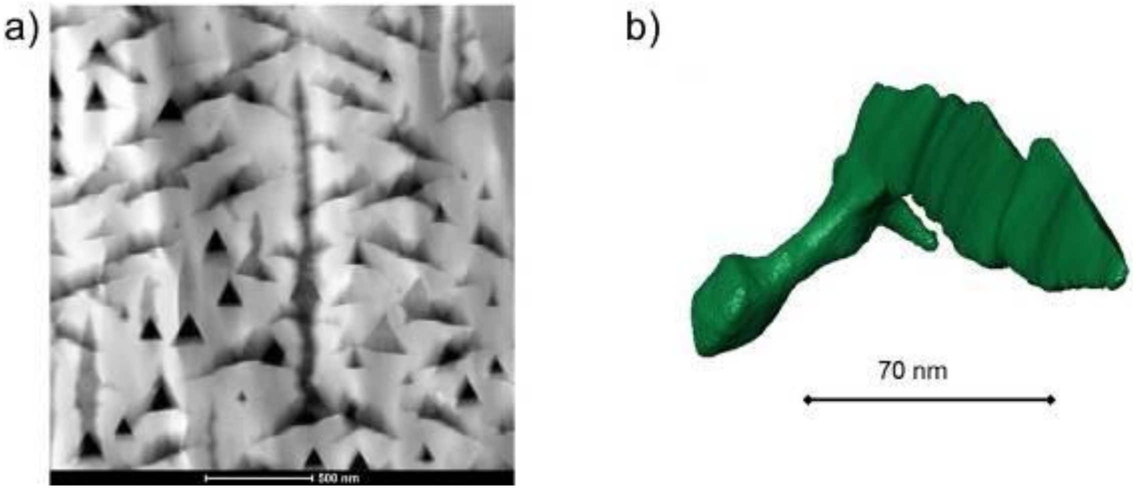

Figure 2. a) HAADF-STEM contrast used for acquisition of tomographic tilt series, b) Tomographic reconstruction of a larger pore showing triangular cross-section with oscillating pore size and sideward branching of a smaller pore.