INVESTIGATION OF SiC CRYSTALS BY MEANS OF SYNCHROTRON TOPOGRAPHY

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Abstract: The crystallographic quality of monopolytypic 6H SiC crystals grown by Physical Vapour Transport in graphite crucible was studied. The diameter of crystals was increased up to 65 mm. The crystals were investigated using several methods of characterisation including white and monochromatic beam synchrotron diffraction topography and scanning electron microscopy. Particularly useful results were obtained using back reflection white beam synchrotron section topography, which provided the intersection of the large thickness of the sample investigated. The topographs revealed a great part of macro and micropipes present in the samples, reproduced as white areas. The additional possibility offered the section topographs taken using a fine grid with the distance between the wires equal to 0.7 mm, which enabled evaluation of the lattice deformation. The scanning electron microscopy was also very useful in studying the micropipes and voids as well as in observation of the selective etching pattern.

Badania kryształów SiC za pomocą topografii synchrotronowej
Streszczenie: Badano doskonałość krystalograficzną monopolitypowych kryształów SiC 6H o średnicy do 65 mm otrzymywanych metodą PVT. Kryształy były badane za pomocą synchrotronowej topografii rentgenowskiej w wiązce białej i monochromatycznej oraz skaningowej mikroskopii elektronowej. Szczególnie cenne rezultaty uzyskano przy odbiciowej topografii przekrojowej, które dostarczały obrazów z dużej grubości próbki wzdłuż przecięcia wąską wiązką. Topogramy ujawniły większą część makro- i mikro- rurek w badanych kryształach. Dodatkową możliwość ujawniania deformacji sieci stworzyło rejestrowanie topogramów synchrotronowych przez metalową siateczkę o wymiarach oka 0.7 mm.

1. Introduction

The technology concerning SiC including both the production of substrate crystals and epitaxial layers is widely developed in the world in view of the applications in technology of high temperature electronic devices and blue light optoelectronic elements. In the letter case mono/polytypic SiC is already widely applied as substrate material for semiconducting nitrides providing a much better lattice fitting than formerly applied sapphire [1-3].

The investigation of structural defects in SiC is important both in practical and cognitive aspects. The presence of structural defects can significantly influence the physical properties and introduce serious problems in further technological steps and unwanted values of the technical parameters of the ready electronic devices. X-ray topographic investigations were described in [4-10].

The technology of SiC started more than hundred years ago but the monopolytypic crystals became available not much more than ten years ago. The other very important problem is the reduction of the defect concentration. The usually applied technology of SiC bulk crystals, modified Lely method, consists of growing the crystals on a large plate formed seed which makes the subsequent improving of the crystal quality very difficult. The greatest problem in SiC based technology are pipe- formed voids of different size: so called nano-, micro- and macro-pipes. The nature and formation mechanism of these defects is not yet quite clear. The problem was intensively studied by Dudley and coworkers, basing on X-ray conventional and synchrotron experiments supported by numerical simulation of the topographic images obtained with several methods [6-9]. The results support the interpretation of micro and nano pipes at super screw dislocations formed by joining of many screw dislocations. According to the theory developed by Frank, such dislocation with large Burgers vector should became a hollow core dislocation. Moreover some authors proposed another interpretation of pipe formed defects [10-12].

In the present paper, the synchrotron topographic method was used for examination of crystallographic imperfection of monopolitypic 6H SiC obtained with sublimation method.

A special attention was paid for application of section topography in Bragg case and revealing the lattice deformation with application of the fine mesh. Some additional investigations with SEM were also performed.

2. Experimental

The investigated crystals were grown at the Institute of Electronic Materials Technology in Warsaw by Physical Vapour Transport in graphite crucible, using large plate-formed seed of (00·1) orientation. The diameter of crystals was set up to 65 mm. The good structural quality of the central part of the crystal was gradually spread on the peripheral part of the crystal. The crystals were investigated using several methods of characterisation including white and monochromatic beam synchrotron topography and scanning electron microscopy. Particularly useful results were obtained using back reflection white beam synchrotron section topography, which provided the image from the large thickness of the samples along the intersection of the narrow beam.

These topographs revealed a great part of macro and micropipes present in the samples, reproduced as white areas. The additional possibility offered taking the section topographs using a fine mesh with the distance between the wires equal to 0.7mm, which enabled evaluation of the lattice deformation. The scheme of the method is shown in Fig. 1.

The scanning electron microscopy was also very useful in studying of the micropipes and voids as well as in observation of the selective etching pattern.

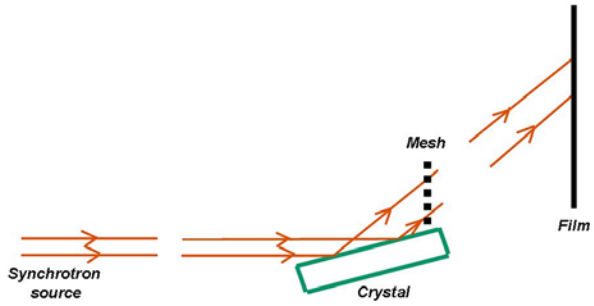
3. Results and discussion

As may be seen in Fgs. 1– 3 the projection topographs of studied crystals with relatively high dislocation density did not provide good visibility of all defects. Applying of the mesh allowed to reveal some local bandings of the lattice connected with non uniform distribution of the defects. The interesting and very useful results were obtained in section topographs also presented in Figs. 1– 3. Due to large defect concentration we did not observe the phenomena characteristic for ideal crystals when most of the intensity is reflected from the surface and some interference effects are formed close to the surface. Instead of that the intensity was reflected by the parts of the crystal located at the intersection of narrow beam with the crystal. Thanks to the very low absorption of synchrotron radiation providing the section images (10.3 nm) we obtained almost uniform images of defects located in the layer penetrated by the beam of thickness reaching single millimetres. It was also possible to take the section topographs using the mesh which revealed the lattice bending inside the crystal as may be seen in Figs 2b and 3c. Our simple evaluation based on the shift of the mesh shadows gives the value of lattice bending on the level of single minutes. It may be noticed that the lattice bending corresponds to a kind of grain (mosaic) structure, being especially well visible in Fig. 3.

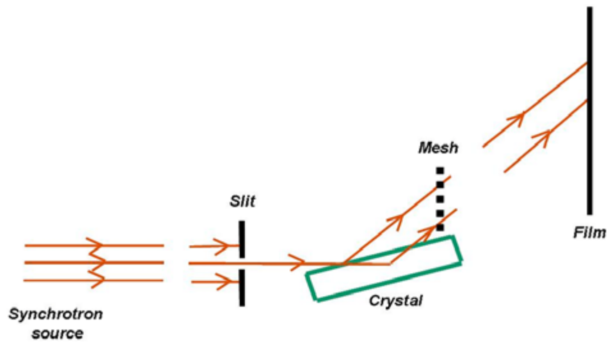
The back reflection projection topographs revealed a great amount of small white (brighter) contrasts which may correspond to micro and nano pipes. Apart of that in some samples we observed larger voids in form of hexagonal platelets extended at few tenths of micrometer formed at the end of single pipes. The example images of platelet voids are visible in Fig. 4b.

Contrary to white beam topographs the monochromatic beam topographs revealed only the small regions of the deformed sample and the examination of larger area of the sample required taking a number of exposures with different angular setting. The series of such topographs recorded at single film provided so called "zebra pattern" shown representatively in Fig. 5.

The observation with SEM revealed only a relatively large voids (Fig. 6) with the diameter exceeding few micrometers, of number distinctly smaller then concentration

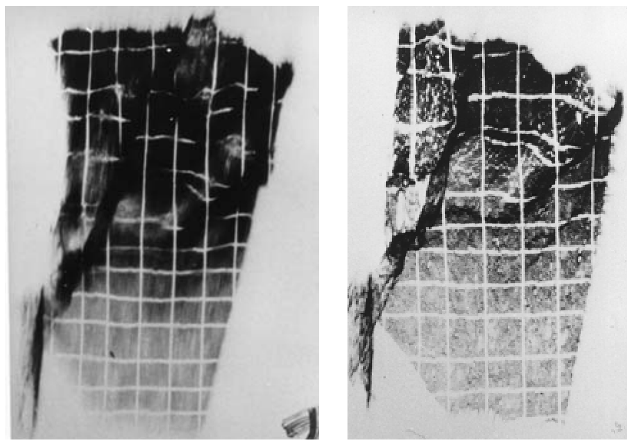


a



b

Fig. 1. Geometry of white beam synchrotron back reflection topography with use of fine wire mesh: (a) projection, (b) section (effective due to low absorption of radiation).



a b

Fig. 2. Synchrotron white beam topographs of undoped SiC crystal with dominant 6H politype composition taken using fine mesh with wire distances 0.7mm: a) projection and b) section.

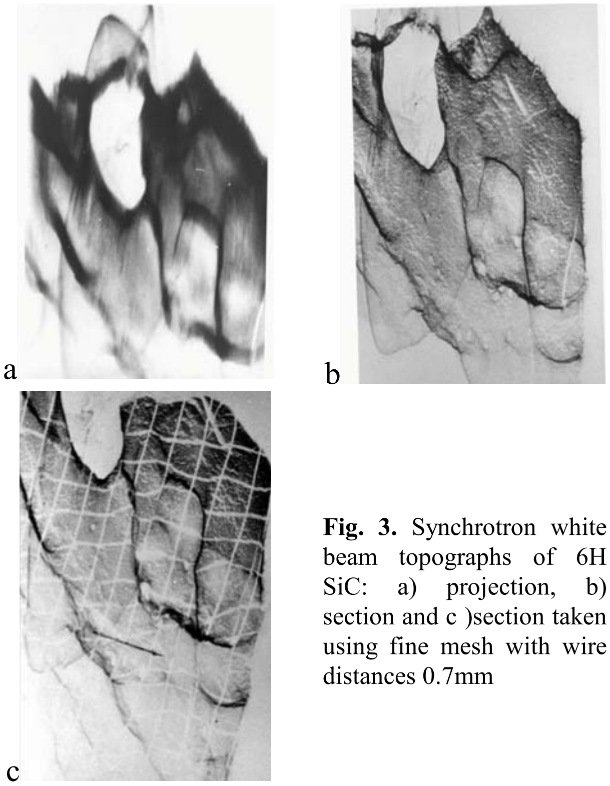


Fig. 3. Synchrotron white beam topographs of 6H SiC: a) projection, b) section and c)section taken using fine mesh with wire distances 0.7mm

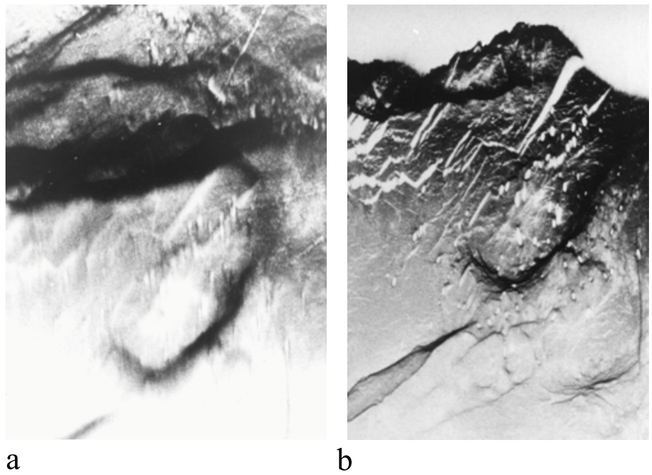


Fig. 4. Synchrotron white beam topographs of SiC sample with platelet voids terminating some of the micro-pipes: (a) projection, (b) section.

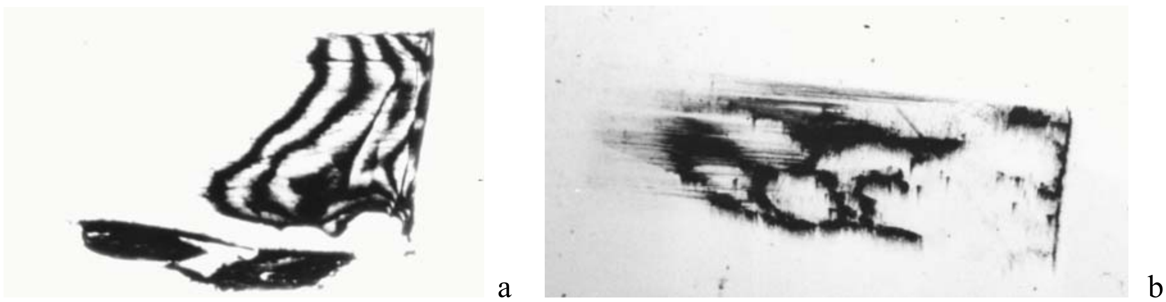


Fig. 5. Zebra-pattern topographs taken in synchrotron multicrystal arrangement selecting 0.115 nm radiation with angular setting changed stepwise 0.02º: (a) sample from SiC crystal grown without seed; (b) new 6H SiC sample grown using large seed.

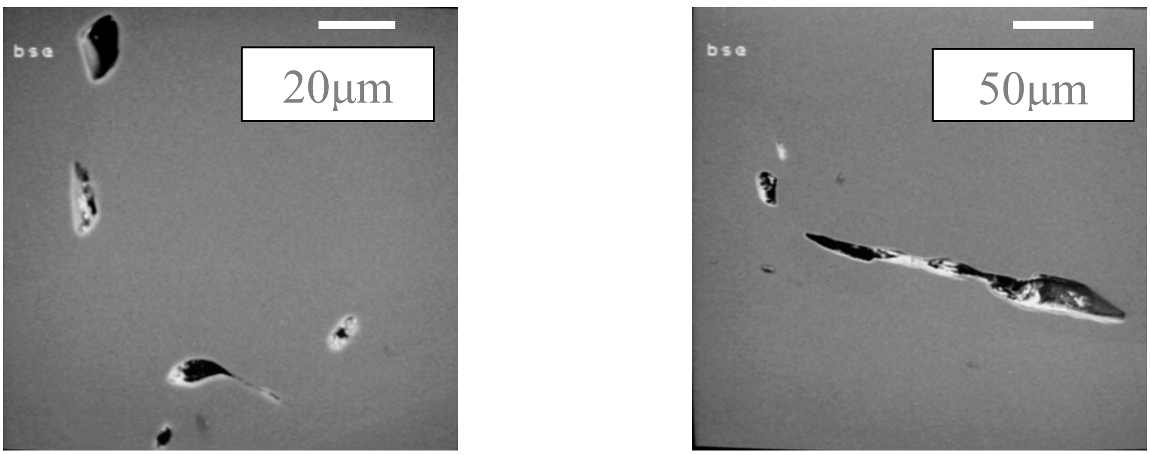


Fig. 6. SEM images of two diferent voids in 6H SiC sample (the picture size is identified in the figures).

of white contrasts in the topographs. A significantly larger number of pits was observed after selective etching in melted KOH. It is not yet quite clear which of those pits correspond to regular dislocations and which to pipe-formed defects.

4. ConclusionsA number of samples cut out from different SiC crystals grown with PVD method (the modified Lely method) included grown at ITME were studied. Different methods of characterization were used including X-ray topographic and diffractometric methods as well scanning electron microscopy and optical microscopy.An important part of the present work were synchrotron X-ray investigation in white and monochromatic beam. The white beam investigation included projection and section back reflection topography. Some white beam topographs were exposed through the fine wire mesh with the distance between the wires equal to 0.7 mm. The topographs taken using the mesh enabled revealing and evaluation of lattice deformation in the investigated crystals. It was found that the lattice bending was on the level of few minutes.The important novelty of the experiment was the use of wire mesh in case of back-reflection section topography, which due to a very low absorption of the radiation in SiC crystals provided a very good results.The white beam section topographs provided much better contrast and resolution of details than the projection topographs, in particular they revealed a certain number of "white" objects corresponding to voids, micro-pipes and regions of other polytypes.The diffractometric analysis confirmed mono-polytypic character of most crystals.The scanning electron-microscopic investigation confimed a good quality of sample polishing. It was also found of SEM images that the number of objects, which may be attributed to voids and micro-pipes was significantly lower than the number of white areas observed at synchrotron topographs.

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