

TRANSMISSION ELECTRON MICROSCOPY AND OPTICAL STUDIES OF THE DOMAIN STRUCTURE IN FERROIC ZINC HALOGEN BORACITES

A.G. Castellanos-Guzman¹ Michael Czank²

¹Universidad De Guadalajara Laboratorio De Investigacion En Materiales DIP Cucei Apdo.Postal 2-638 GUADALAJARA JALISCO 44281 MEXICO
²Universitaet Kiel. Institut fuer Geowissenschaften.

The domain structures of a ferroic triad of materials with the formula $Zn_3B_7O_{13}X$, where X=Cl, Br and I have been analyzed by transmission electron microscopy (TEM) and polarised light microscopy (PLM). $Zn_3B_7O_{13}X$ boracites exhibit a high temperature phase of cubic prototype symmetry F43c. The domain structure of $Zn_3B_7O_{13}Cl$ as observed by TEM and PLM indicates a trigonal symmetry at room temperature whilst those of $Zn_3B_7O_{13}Br$ and $Zn_3B_7O_{13}I$ indicate an orthorhombic symmetry at the same temperature. TEM work allowed to observe two different types of domains: First, domains due to the ferroic cubic to orthorhombic phase transition. These are in pseudo-merohedral twin relations, and therefore they show weak contrasts in bright-field as well as in dark-field images. Second, thin (10 to 20 nm) lamellae parallel to (100), which can be explained as microtwins due to the $Pca21$ to the ferroic phase transformation. In some crystal grains the 'reaction front' of the transformation is preserved at room temperature and irregular inter-grow of the twin domains is observed. *In situ* heating experiments point out that the reversible phase transitions have strong hysteresis and the domains show no memory effects. Occasionally, an incommensurable modulation ($q = 0.492(10)a^*$) at temperatures somewhat above the second transformation has been observed. This could represent a precursor of the micro-twinning. Support from Conacyt-Mexico (Project 34959E) and Deutsche Forschungsgemeinschaft is gratefully acknowledged.

Keywords: ELECTRON OPTICAL MICROSCOPY, FERROIC DOMAINS, PHASE TRANSFORMATIONS

MULTIPLE TWINS OF DIAMOND SYNTHESIZED BY ACETYLENE FLAME

S.I. Son S.J. Chung

Seoul National University School of Materials Science and Engineering College of Engineering San 56-1 ShillimDong KwankGu SEOUL 151-742 SOUTH KOREA

The icosahedral morphology of twinned cubic crystals is resulted from the multiple twinning by reticular merohedry with the twin element of 2-fold axis along $\langle 111 \rangle$ or (111) mirror plane. This twin element is naturally the symmetry element belong to the icosahedral point group: 235 or m35. The multiple twins could be frequently observed in the diamond particles synthesized by acetylene flame. One of the shapes of this multiple twin is almost perfect icosahedron in which all the trigonal faces are composed of {111} faces of octahedral crystal grown at a lower substrate temperature below 850°C. In the other shape, the trigonal faces of icosahedron altered with an indented negative trigonal pyramid formed by {100} faces of cube crystal grown at a substrate temperature of 980°C. Star-like 5-fold cyclic twins of octahedra also appeared. All of these shapes of twins are resulted from the twinning by reticular merohedry. The diffraction patterns of these diamond twins under the transmission electron microscope exhibited 5-fold symmetry. The relation between domains and domain boundaries of icosahedral twins of diamonds were studied by electron back scatter diffraction (EBSD) method. The result of EBSD clarified that the adjacent faces exhibit the twinning relation of 60° rotation along $\langle 111 \rangle$ axis and the domain boundaries are $\Sigma 3$ and the misfit angle after 5-fold cyclic twinning is 7.35°. This twinning by reticular merohedry is widely spread in the crystals of spinel, fluorite, galena, silicon, germanium, diamond, zinc blend and perovskite.

Keywords: DIAMOND, TWIN, DIFFRACTION

REVEALING DISLOCATION CORE STRUCTURES AT ATOMIC RESOLUTION BY IMAGE DECONVOLUTION

F.H. Li¹ D. Wang¹ W.Z. He¹ H. Chen¹ K. Kawasaki² T Oikawa²

¹Institute of Physics, Chinese Academy of Sciences Institute of Physics, P.O. Box 603 BEIJING 100080 CHINA ²JEOL-LTD, 1-2 Musashino 3-chome, Akishima, Tokyo 196, Japan

The field-emission gun (FEG) high-resolution electron microscopes are characterized with the high brightness and high information resolution limit. However, the deviation of Scherzer focus image from the projected structure is inevitable owing to the strong oscillation of contrast transfer function in high scattering angle region. The technique of image deconvolution in combination with the dynamical scattering effect correction has been developed to restore the projected structure from the FEG high-resolution electron microscope image. This leads to the possibility of determining the projected structure of resolution close to the information resolution limit of the electron microscope, at least for crystals with a small unit cell. The test of the technique with simulated images of Si crystals with and without defect indicates that atoms are resolved individually in the restored image. In the present work the technique has been employed to study the dislocation core structures in $Si_{0.76}Ge_{0.24}/Si$ epilayer. The cross-section sample was observed with a JEM 2010F high-resolution electron microscope. The incident electron beam is parallel to the [110] direction. A series of images were taken from the area close to the boundary between the epilayer and the Si substrate. The core structures of perfect 60 degree dislocation, extended 60 degree dislocation and Lomer dislocation have been revealed at atomic resolution.

Keywords: DISLOCATION CORE SIGE EPILAYER IMAGE DECONVOLUTION

REAL STRUCTURE OF IRON NIOBATE POLYMORPHS II AND III

R. Theissmann L. Erac H. Ehrenberg H. Weitzel H. Fuess

TU Darmstadt Institute for Materials Science Petersenstr. 23 DARMSTADT 64287 GERMANY

Possible applications for some types of ABO_4 oxides, e.g. $FeNbO_4$, $FeTaO_4$ and $FeWO_4$, as gas sensors and catalysts or in photodetector technology have given rise to an intensive study of these materials within the last decade. Special topics of interest are the electric and magnetic properties and new routes of synthesis. Of primary importance for the understanding of the physical properties of these materials is a detailed knowledge of the underlying ideal and real structures. Iron niobate ($FeNbO_4$) was synthesized by colloidal sol-gel technique. Several temperature programs were chosen to obtain different kinds of microstructures. Quenching of iron niobate II and subsequent annealing in the stability range of iron niobate III leads to nanostructured cation distribution that is described in terms of a domain size and degree of cation order. Samples quenched from the phase transition range (1050-1100°C) contain a defect stabilized new iron-niobium-oxide phase, that occurs between regions of the polymorphs II and III, depending on the annealing conditions. The microstructures were investigated on several length scales using dark field and high resolution transmission electron microscopy as well as high resolution synchrotron powder data analysis. These investigations show, that the influence of defects on $FeNbO_4$ II bulk material should not be underestimated. It doesn't seem to be possible, to produce $FeNbO_4$ with insignificant defect concentrations by standard synthesis methods (sol-gel, ceramic, etc.).

Keywords: DOMAIN STRUCTURE, PHASE TRANSITION, IRON NIOBATE