

**14.4-07 INTERGROWTH STRUCTURES AND NON-RANDOM DISORDER IN SILICON CARBIDE CRYSTALS.** By S.R.Singh, M.Dubey\* and G.Singh, Department of Physics, Banaras Hindu University, Varanasi-221005, India. \*Presently at the Research Triangle Institute, 807 Bacon Street, Durham, NC 27703, U.S.A.

Lattice imaging, by virtue of its capacity to reveal local structural variations, has been found to give new information on the tendency of repetition of planar disorder in crystals like SiC which was hitherto not possible with the help of X-ray diffraction alone. The present investigation on SiC crystals deals with: (1) the local variations of structures in terms of the stackings of small period unit cells, (2) the statistical occurrence of small period unit cells and their tendency to create long period ordered structures. The failure to create long range order and hence periodic structures have been found to be due to non-random disorder.

On the basis of our observations on intergrowth structures of SiC, it was found that crystals having coalesced polytype structures in parallel orientation contain superperiod structures in the transition region between coalesced structures which are built up by unit cells of the adjoining structures. Therefore, the transition from one structure to another is not abrupt. Instead, there seems to be a boundary region of substantial extent where unit cells of the two adjoining structures stack in a definite fashion to create a large period structure.

A number of SiC crystals showing diffuse streaks in their X-ray diffraction patterns have been examined by lattice imaging technique. In most of the cases, it has been observed that there is some memory effect which prompts structural blocks to repeat at regular intervals in space, thereby creating an average lattice. As a consequence, diffuse intensity shows modulations in the form of weak spots corresponding to the average lattice. In some cases a distinct structural entity occurs at strictly regular intervals thereby defining a lattice periodicity. Each such periodic block is built of sequences of the same number and type of unit cells of common polytypes but are not identical in their distribution in each superperiod block. The implications of these observations on the formation of polytype structures in SiC are discussed.

**14.4-08 DIRECT OBSERVATION OF POINT DEFECTS IN NON-STOICHIOMETRIC  $Nb_2O_5-x$  BY 1 MV HIGH-RESOLUTION TEM.** By S.Horiuchi and S.Kimura, National Institute for Researches in Inorganic Materials, Sakura-mura, Niihari-gun, Ibaraki, Japan.

It has so far been considered for a homologous series of  $Nb_2O_5-x$  crystals that a small fraction of Nb moves from the tetrahedral to octahedral sites in order to accommodate the non-stoichiometry. We tried to directly detect the transition by means of 1 MV high-resolution transmission electron microscopy, which enables us to resolve individual cations when observed along the short b axis. At the very thin part of the crystal fragment the image contrast was almost the same as that of the stoichiometric one. This means that the fraction of the transition is very small. At the rather thick part, on the other hand, some evidence of the transition was attained and this was confirmed with aid of the computer simulation of contrast. A further evidence was obtained as follows; a  $Nb_{22}O_{54-x}$  crystal gradually changes the colour from black to white when heated at 200°C and at the same time it increases the weight because of oxidation. The partially oxidized, gray crystal gives the diffraction pattern unchanged. On the structure image, however, some local displacements of cations between neighbouring octahedral sites were clearly found. They may be related to the beginning of the phase transformation to the higher oxide, i.e.  $Nb_2O_5$ . Since they do not contribute to change the composition, there must have been the reduced state due to the transitions of cations.