

the domain wall width, only if  $t > 1000 \text{ \AA}$ . A similar effect has been observed in crystalline foils of h c p Co [6], with angle  $\theta$  near  $90^\circ$ .

Near 500 K, the width of the cross-tie wall is minimal and approaches the values typical of thin crystals of h c p Co. Changes in domain wall width are caused by a change in energy which in this case mainly comprises the exchange energy and anisotropy energy. Magnetostriction energy, which is much lower, is neglected because the first measurements pointed to a very low value of  $\lambda_s$  in the amorphous alloy  $\text{Co}_{90}\text{Zr}_{10}$ ; it is assumed that the magnetostatic energy,  $E_{\text{me}} = 0$  for a  $180^\circ$  Bloch wall.

To summarize, for the amorphous ferromagnetic alloy  $\text{Co}_{90}\text{Zr}_{10}$  the localization of vector  $\bar{M}_s$  which proved to be consistent with the easy axis was determined, and the fluctuation,  $\Delta$ , of the exchange integral was evaluated to be 0.2. The effect of temperature on the cross-tie domain wall width,  $\delta_w$ , which has a minimum at about 500 K was demonstrated.

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A. J. JANICKI  
P. G. ZIELIŃSKI  
H. MATYJA

*Institute of Materials Science and Engineering,  
Warsaw Technical University,  
02-524 Warsaw,  
Narbutta 85,  
Poland*

M. JURCZYK  
*Institute of Molecular Physics,  
Polish Academy of Science,  
60-179 Poznań,  
Smoluchowskiego 17,  
Poland*

### *Formations of rhombohedral boron nitride, as revealed by TEM—electron energy loss spectroscopy*

Boron nitride crystallizes at atmospheric pressure in a graphite-like layered structure called hexagonal (or h-) BN [1], but the form of stacking of successive layers is different for h-BN compared with graphite; i.e. the hexagons in the honeycomb network lie parallel to the  $c$ -axis in h-BN but are oblique in graphite. From these packing characteristics, the presence of rhombohedral  $\beta$ -graphite is naturally expected but that of h-BN not because, as Pease [1] first pointed out, in order to take the rhombohedral (or r-) BN form, the spatial relations between the two nearest layers must be changed

from those in h-BN. In fact, reliable experimental data on r-BN have not so far been presented; it has only been reported that in powder X-ray diffraction of a fusion product of KCN and  $\text{Na}_2\text{B}_4\text{O}_7$  some peaks can be indexed as r-BN [2]. In the present note, however, we present more reliable experimental data on the existence of r-BN, by the combination of transmission electron microscopy (TEM) and qualitative microanalyses by electron energy loss spectroscopy (ELS).

The samples were prepared by deposition at  $1500^\circ\text{C}$  from h-BN vapour originally formed at  $2100^\circ\text{C}$  in a graphite-resistance tube-furnace [3]. The products were collected on a pitted carbon film and examined by an ordinal TEM (H-500 type,

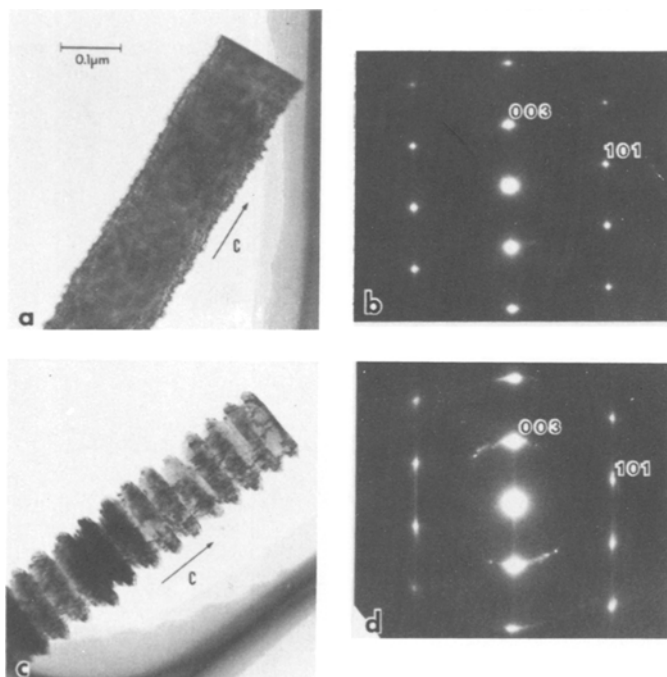


Figure 1 (a) Electron micrograph of well ordered crystal of r-BN and (b) its electron diffraction pattern. A less ordered case is shown in (c) and (d).

100 kV). Fibrous micro-crystals elongated in the  $c$  direction were observed, as shown in Fig. 1a and c. Electron diffraction experiments revealed that some of them have a rhombohedral symmetry not a hexagonal one as shown in Fig. 1b and d. The unit cell dimensions were found to be  $a = 2.5 \text{ \AA}$  and  $c = 10.0 \text{ \AA}$ , indicating that  $c$  is about 1.5 times larger than that of h-BN ( $a = 2.504 \text{ \AA}$  and  $c = 6.661 \text{ \AA}$ ) [1]. One of the possible and attractive explanations of these observations is that micro-crystals as shown in Fig. 1a and c are rhombohedral boron nitride r-BN. Experiments, however, showed that such rhombohedral crystals are observed only when the preparations are made in a furnace containing graphite as a main component [3]. There is then the second possibility that the crystals of rhombohedral symmetry are no more than carbon from the furnace which was accidentally crystallized in the  $\beta$ -graphite structure. It is impossible to distinguish between these two possibilities by electron diffraction in an ordinal TEM because the differences in the lattice constants of r-BN and  $\beta$ -graphite are very small.

Therefore direct elemental analyses of the microcrystals of rhombohedral symmetry is needed in order to determine whether they are r-BN or  $\beta$ -graphite. For this purpose, TEM-ELS

experiments were performed because the method is considered to be effective for qualitative analyses of light elements such as B, C, N or O [4, 5]. In order to prevent complications caused by carbon contaminations during ELS measurements, a new type of TEM (H-600 type, 100 kV) improved in the evacuating system was used with a double focusing type electron spectrometer, below the camera chamber. The resolution of the spectrometer is about 3 eV, sufficient for distinguishing the energy loss by K-shell excitations of B (188 eV), C (284 eV) and N (402 eV) [6]. The electron probe diameter was about  $100 \text{ \AA}$  using a triple condenser lens system.

The micro-crystals of rhombohedral symmetry were selected using a side-entry goniometer and were then analysed by ELS. The well ordered crystals, as shown in Fig. 1a, are unfortunately too thick to obtain sharp ELS spectrographs because the K-shell excitation peaks are obscured by the superposition of large plasma excitation peaks. Therefore, the measurements were made mainly on the thinner crystals, as shown in Fig. 1c, although they are slightly disordered compared with a rhombohedral lattice. An ELS spectrograph, displayed on the cathode ray tube, is shown in Fig. 2. The K-shell excitation peaks from B and

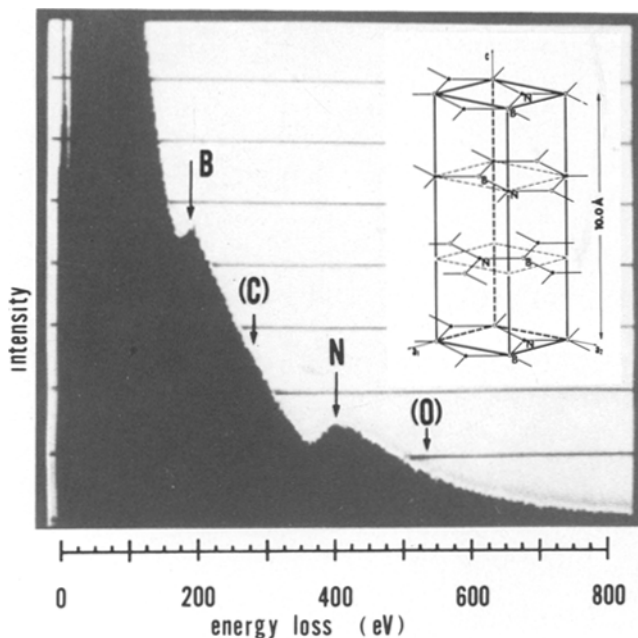


Figure 2 Electron energy loss spectrograph taken from the particle of the type shown in Fig. 1c. Crystal structure of r-BN is inserted.

N are clearly observed, while those from C (or O) are not. The peaks corresponding to other elements are also not observed in the spectrograph. It can therefore be concluded that the crystals of rhombohedral symmetry are composed only of boron and nitrogen. Although quantitative estimates of the B/N ratio are impossible at the present stage, the crystals may be reasonably assigned as r-BN because the lattice constants are well explained as the polytypic form of h-BN.

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Y. MATSUI  
Y. SEKIKAWA  
T. SATO  
T. ISHII

National Institute for Researches in  
Inorganic Materials,  
Sakura-mura,  
Niihari-gun,  
Ibaraki 305, Japan

S. ISAKOSAWA  
K. SHII  
Hitachi Co.,  
Katsuta,  
Ibaraki 312,  
Japan