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Orientation of bismuth films on mica

During the growth of bismuth films on sodium chloride substrates, two orientations, (001) and (012), are reported to be developed with equal probabilities, and suitable changes in the deposition conditions result in a predominant (001) or (012) orientation [1–4]. The azimuthal orientation of the crystallites will be such that the [100] direction remains parallel to the [100] or [110] directions of the substrate [2, 3]. However, when deposited on mica cleavages, the (001) orientation is dominant over (011) and (012) orientations with the [100] direction making an angle of 30° with respect to the [010] direction of mica [1]. The different orientations mentioned are essentially of finished films and depend more on the kinetics of the development of the individual nuclei rather than the initial orientation of the crystallites. This seems reasonable as the conditions of condensation have a greater influence on the orientation of the deposits than the material of the substrate, and in spite of the difference in the planar symmetry of the substrate surfaces on which the deposits are formed, the character of bismuth epitaxy is basically the same on mica as well as on alkali halides [1]. In this letter we report the appearance and the elimination of the (012) orientation of the bismuth deposits on mica cleavages as an effect of the deposition parameters.

Observations were made on bismuth films, 600 to 700 Å thick, vapour-deposited onto mica cleavages heated to temperatures in the range 25 to 130° C. The rate of deposition of the vapour flux was maintained at 40 to 50 Å sec^{-1}. The films were examined by transmission electron microscopy after stripping from the substrates.

It is observed that all the films deposited at 25° C were polycrystalline but showed a clear (001) texture (Fig. 1a). At higher temperatures (50 to 100° C) the films were made up of single crystalline grains of (001), (011) and (012) orientations with various azimuthal orientations, but when the temperature was above 100° C, the (011) and (012) orientations were absent and the films were (001) oriented except for the mis-

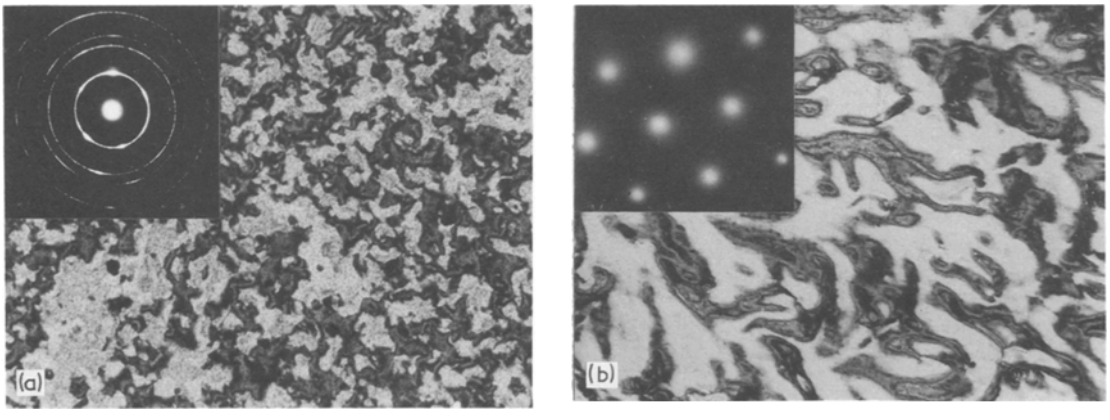

Figure 1 Bismuth films deposited onto mica substrates (a) at 25° C and (b) 130° C.

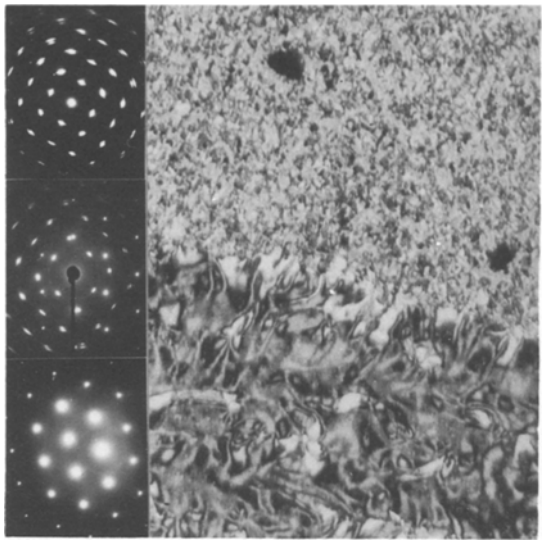

Figure 2 A boundary between (001) and (012) oriented grains.

orientation of the grains about the beam direction. The films deposited at 130° C were devoid of these large azimuthal mis-orientations and were purely (001) epitaxial (Fig. 1b).

Detailed investigations were carried out on films deposited on mica cleavages heated to 100° C in order to study the grain boundaries between

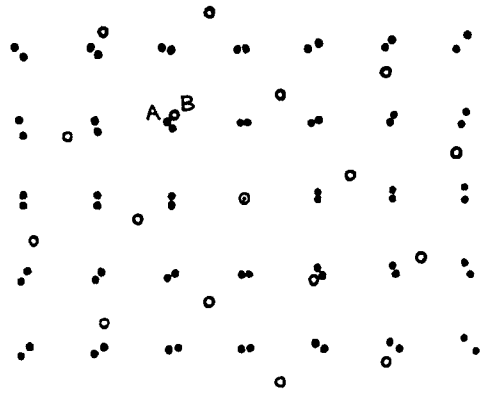

Figure 3 Reciprocal lattice construction for the composite diffraction pattern from the region including the boundary shown in Fig. 2.

(001) and (012) oriented grains. Fig. 2 shows one such boundary the regions on either side of which revealed good single crystallinity as evident from the diffraction patterns. The selected-area diffraction pattern from the region including the boundary showed the composite of the (001) and (012) orientations which is schematically represented in Fig. 3. Spot A, corresponding to a (110) reflection from the (012) oreinted grain, and spot B, corresponding to a (110) reflection from the (001) oriented grain, were used to obtain dark-field

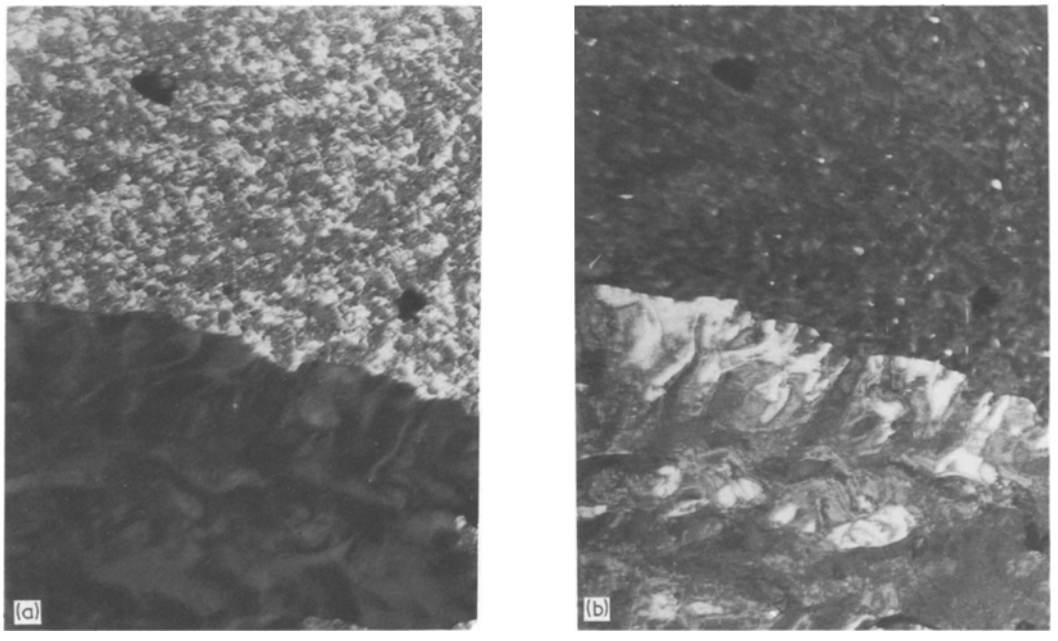

Figure 4 Dark-field micrographs of the region shown in Fig. 2 with the objective aperture on (a) spot A, and (b) spot B.

micrographs (Fig. 4) of the same region, which clearly show that the regions on either side of the boundary are purely (001) or (012) oriented.

A further examination of the boundaries revealed that in most of the cases the two orientations had the [110] reciprocal lattice direction common except for some rare observations such as the one mentioned above. It may be mentioned that irradiation of the film with the electron beam resulted in the re-orientation of the grains to have a common [110] reciprocal lattice direction. Similar results were observed when the films were aged in vacuum for a couple of days.

The appearance of (012) and (011) orientations in continuous bismuth films seems to be the result of the growth rates of the nuclei rather than their initial orientation. The (001) and (012) oriented grains usually have a common [110] reciprocal lattice direction or re-orient to have the common direction during observations under electron beam irradiation. Ageing of the films has a similar effect on the re-orientation of the films.

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Direct evidence for a brucite precipitate in a melt-grown MgO crystal

Since the studies of the infra-red absorption characteristics of MgO crystals by Kirklin *et al.* in 1965 [1] suggested that an absorption band found at 3700 cm^{-1} might be due to brucite ($Mg(OH)_2$) precipitates, other workers have accumulated further indirect evidence for the existence of such precipitates. Glass and Searle [2] also found a strong absorption peak at 3700 cm^{-1} which they attributed to the presence of brucite precipitates, whilst Sibley *et al.* [3] provided further confirmation by luminescence measurements. Briggs [4] reported the observation of a strong absorption at 3698 cm^{-1} in subsurface regions of MgO crystals containing relatively high levels of hydroxyl impurity. He also assumed that this absorption was due to brucite precipitates, on the basis of earlier work on $Mg(OH)_2$ crystals by Cabannes-Ott [5], who established the fundamental OH stretching frequency in $Mg(OH)_2$ at $3700 \pm 4\,cm^{-1}$, and Benesi [6], who found the brucite OH stretching frequency at $3698 \pm 2\,cm^{-1}$.

Buessem and Köberich [7] found that the transformation brucite to periclase (MgO) was crystallographically ordered, and that the brucite formed on rehydration of the periclase had the same orientation relations with the periclase as had the original brucite. These orientation relations for the transformation were examined in detail by Garrido [8, 9], who established that MgO crystallites formed from $Mg(OH)_2$ had two possible orientations in which the zone axes, with respect to those of the brucite, were as shown in Table I. That is, in the MgO resulting from thermal decomposition of brucite, the close-packed planes of oxygen ions lie parallel to the close-packed

TABLE I Orientation relations of periclase and brucite

Orientation	Periclase crystal direction	Brucite crystal direction
I	[1 1 0]	[1 0 $\bar{1}$ 0]
	[1 1 1]	[0 0 0 1]
	[1 0 0]	[1 0 $\bar{1}$ 1]
II	[1 1 0]	[1 0 $\bar{1}$ 0]
	[1 1 1]	[0 0 0 1]
	[1 0 0]	[1 $\bar{1}$ 0 1]