JOURNAL OF MATERIALS SCIENCE 14 (1979) 619–625

An X-ray topographic assessment of cadmium mercury telluride

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This paper describes an X-ray topographic (Berg–Barrett) assessment of cadmium mercury telluride grown by the Bridgman and cast-recrystallize-anneal (CRA) methods. Reflection topographs reveal that the Bridgman material studied consists of large numbers of small grains (0.05 to 0.6 mm) with misorientations from 1 to 9 minutes per grain. In contrast, the CRA material studied had only a few grain boundaries and features consistent with a strained lattice, possibly caused by compositional variations.

1. Introduction

Cadmium mercury telluride (CMT), $Cd_xHg_{1-x}Te$, with $x = 0.2$, is used for the detection of infra-red radiation with a wavelength of approximately 10 μm [1, 2]. Mullard, Southampton have used two melt-growth techniques for preparing crystals, namely the Bridgman method [3] and the cast-recrystallize-anneal (CRA) methods [4, 5]. In order to assist in the choice of a suitable crystal growth technique, an X-ray topographic assessment of CMT prepared by these two methods has been undertaken. Particular attention has been paid to differences in orientation, grain structure and sub-grain structures.

The Berg–Barrett [6] reflection technique has been the principal method for assessment since it has a relatively low sensitivity and is therefore particularly well suited for the delineation of grains and sub-grain structures present in CMT. The experimental details of this technique are described in Section 2. Since the methods of crystal growth will clearly be a significant factor in determining the final structure of the crystals, details of the growth conditions of the Bridgman and cast, recrystallize, anneal methods are also given in Section 2.

Results of the X-ray topographic assessment are described in Section 3. It is shown that the material grown by the Bridgman method has a grain structure, with grain size in the range 0.2 to 0.6 mm near to the centre of a slice of 0.05 to 0.2 mm nearer the circumference. Misorientations per grain are also shown to vary from 1 to 3′ near the centre, to 6 to 9′ near the circumference. In contrast, the material grown by the cast-recrystallize-anneal method shows none of the large number of small grains found in the Bridgman samples. Some grain boundaries are evident but topographs are closer to those expected of single crystals. Also present on CRA grown CMT are features consistent with a strained lattice, possibly caused by variations in composition. These features were not as prominent in the Bridgman material.

2. Experimental details

The Berg–Barrett [6] technique was chosen for these studies because it has low sensitivity (cf. double crystal technique [7, 8]) and is therefore more suitable for the delineation of grains and sub-grain structures present in CMT. Moreover, it is readily assembled (cf. Lang Camera [9]), the broad reflection is easily found, and exposure times can be restricted to a few minutes. A beam of Cu$K\alpha$ radiation from a normal focus tube (1 mm × 1 mm) was incident on the sample set to the Bragg angle. The source to specimen distance was set at 65 cm for these experiments. An adjustable slit was used so that the sample was just covered with the beam. The resolution, which is limited by the vertical divergence of the incident beam [6], was calculated to be 15 μm for a 1 mm slit and 10 mm sample-to-film distance. Penetration of the beam was very small, generally

less than 1 μm. Exposure times of 5 min were used with Ilford Industrial G film.

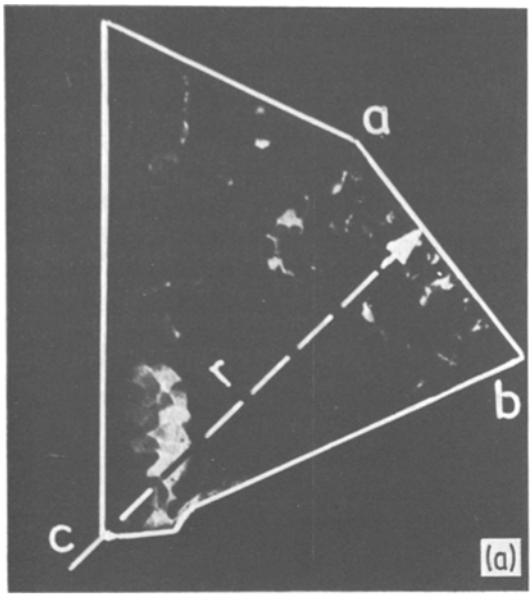
The Bridgman material was prepared in the usual way [10]. An appropriate mixture of cadmium, mercury and tellurium was sealed in a thick-walled silica tube and placed inside the furnace. At the furnace temperature of approximately 825° C the constituents formed a melt. The silica tube was rotated at about 2 rpm and lowered at approximately 0.5 mm h^{-1}. After 2 to 3 weeks a high purity *n*-type boule was obtained.

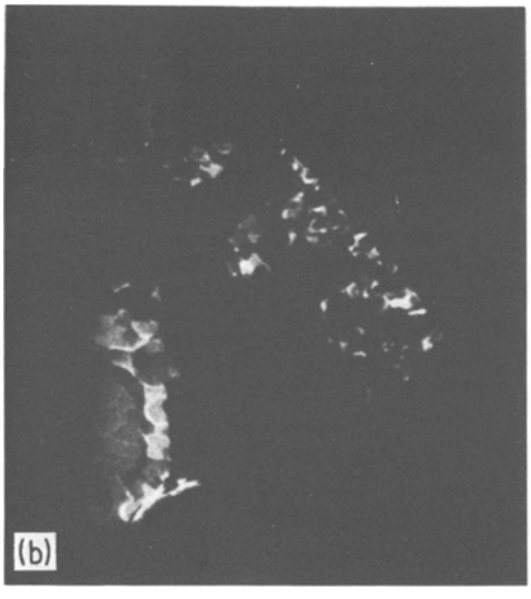
The first step in the cast-recrystallize-anneal process [4] was to prepare a fine grain polycrystalline boule of CMT of the appropriate composition by quenching rapidly from the melt. By adjusting experimental parameters, a polycrystalline boule of approximately constant composition may be prepared. The molten boule was lowered from the furnace into the air at approximately 300 cm h^{-1} The second step in this process involved the growth of a *p*-type single crystal by heating the polycrystalline boule to just below the solidus temperature of 700° C. At this temperature, grain growth occurs, so that after about 10 days the crystals are of reasonable quality. Finally, the *p*-type crystals were converted to *n*-type by heating at 250 to 300° C in mercury vapour for several days.

3. Topography

3.1. Bridgman

The orientation of the Bridgman slice (cut perpendicular to the long axis) was found to be constant to within 1° at nine different positions across the slice using the Läue back-reflection technique. The orientation was approximately 30° from [1 1 1] and 27° from [1 1 0]. The sample had been etch-polished using a 5% bromine–methanol solution. Only a section of this slice was used since the use of too large a slit on the complete slice reduced the contrast of the topographs. Even so it was not found possible to image the section com-





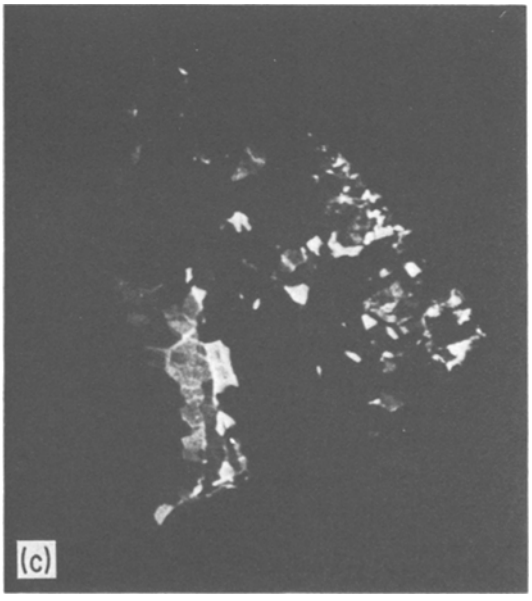
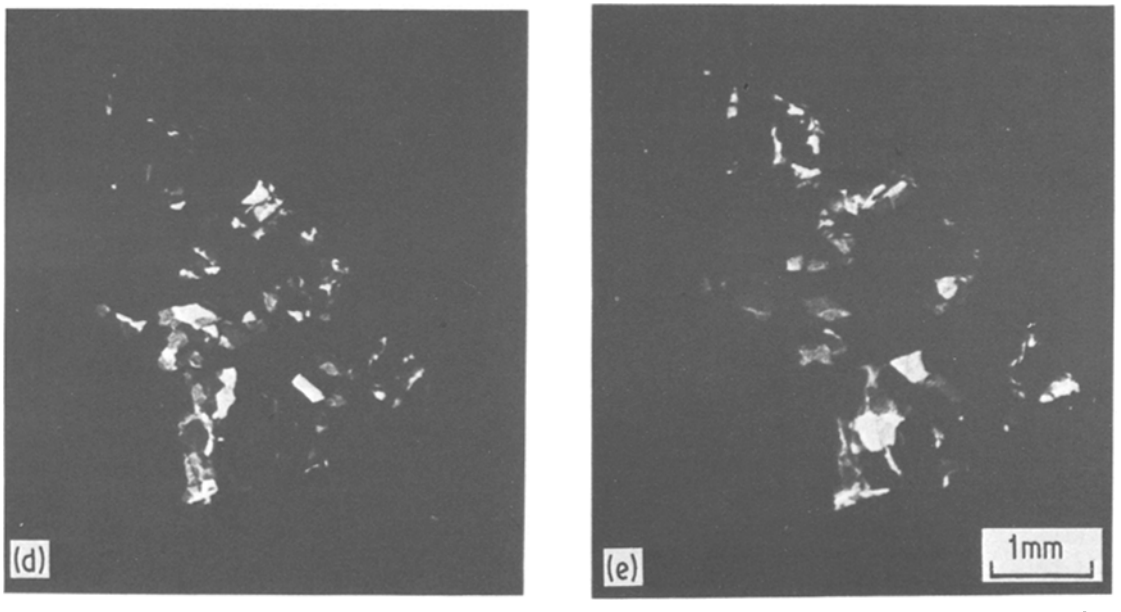


Figure 1 Topographs of part of a Bridgman slice as the incident angle (θ_I) is increased in steps of 10′ from θ_I. See text for details.

pletely with a single topograph, even with the relatively low sensitivity of the Berg–Barrett technique. Fig. 1a to e illustrates the topographs taken as the incident angle θ_I was increased in steps of 10′ from θ_I. The shape of the section itself is shown as an outline on Fig. 1a. The centre of the complete circular slice is at c, r represents a radius of the slice and ab is an arc on the outer circumference of the slice. The (4 4 0) reflection was used for which $\theta_B = 42.4°$ and $\theta_I = 15°$ and the scale is as shown. The most prominent feature is the presence of large numbers of small grains which come in and out of contrast as θ_I is varied. Near to the centre of the slice, c, the grains are larger, in the range 0.3 to 0.5 mm while close to the circumference, ab, they are smaller, in the range 0.05 to 0.2 mm. The misorientations from grain to grain must be smaller around the centre, c, since groups of approximately 20 adjacent grains are imaged at one particular value of θ_I. Moreover, since the positions of these groups gradually moves out from the centre, c, as the sample is rotated (θ_I increased) it follows that the change in orientation of these grains on a radius, r, from the centre, c, is continuous. However, as the circumference (ab) is approached a more random orientation is evident with a single grain being in contrast while its neighbour is not. The misorientations from grain to grain are, therefore, much larger nearer the circumference than nearer the centre. For these larger grains near to the centre, c, the misorientation per grain is approximately 2 to 3′. Nearer the circumference this is increased by at least a factor of 3.

The plank (cut parallel with the long axis of the boule) investigated came from a different boule to the previous slice. The orientation of this plank was approximately 33° from [1 1 1] and 2° from [1 1 0]. A (3 3 1) reflection was used for which $\theta_B = 31.5°$ and $\theta_I = 18.2°$. Topographs of this plank are shown in Fig. 2a to d, with θ_I varying by 20′ in 5′ increments. Superimposed on the first topograph (a) is the outline of the slice, while the dotted lines represent the positions of surface features revealed after the slice had been polish/etched with 5% bromine–methanol solution. It is clear that the line ab and part of the line cd coincide with regions where there is a large change in orientation of the lattice. However, a back-reflection Läue at A, revealed that this misorientation must be less than 1°. This is consistent with the fact that small grains in these areas are just beginning to be imaged in Fig. 2d (see, for example, areas B and C). Grain sizes, which are best observed in the area D, are in the range 0.2 to 0.6 mm, which is comparable to the larger grains in the slice described above. Although grains near the edge of area D are comparable in size to those nearer the centre, there is a line of smaller grains evident in Fig. 2b (see area E). However, these are not as small as some of those

Figure 1 continued.



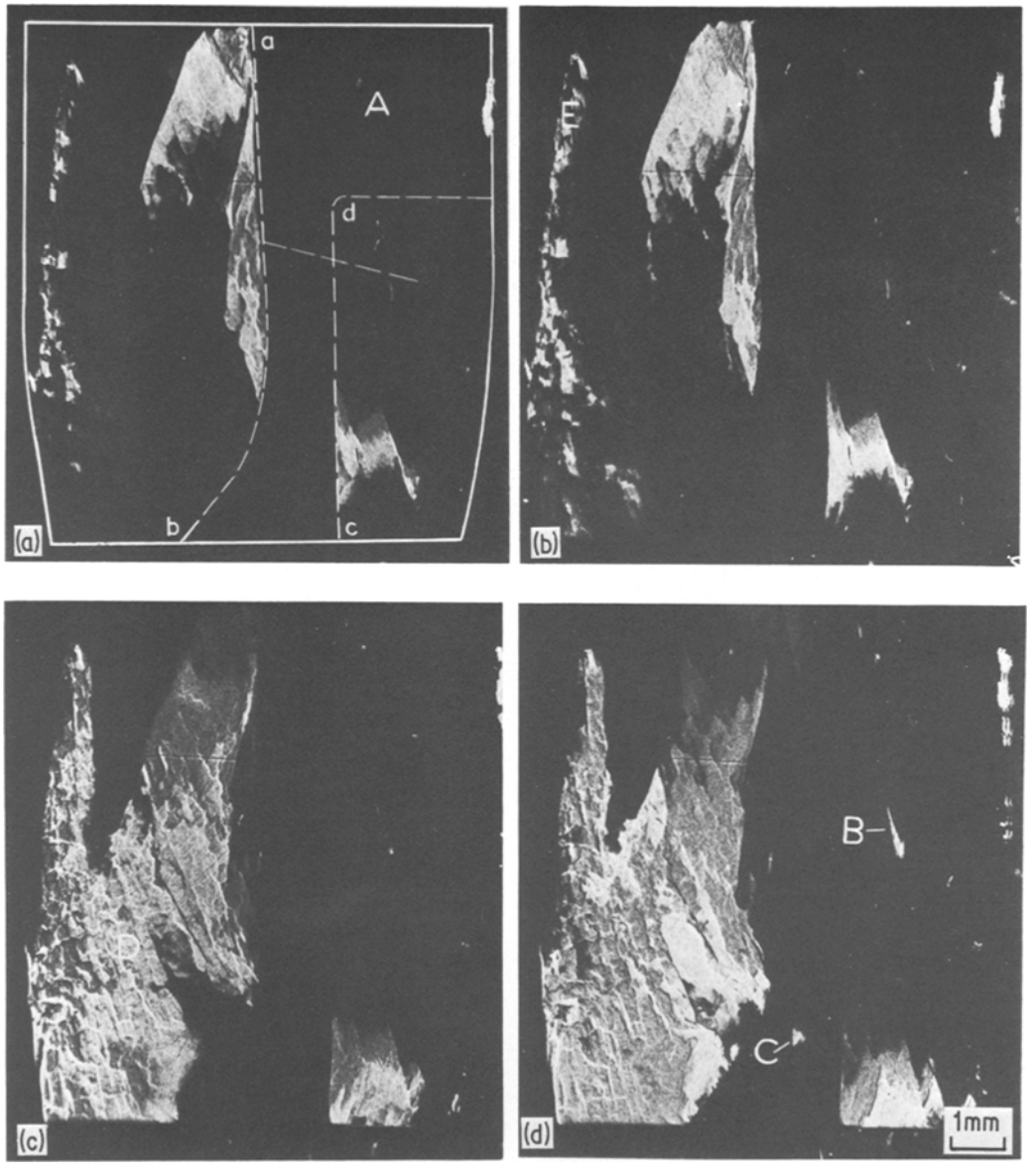


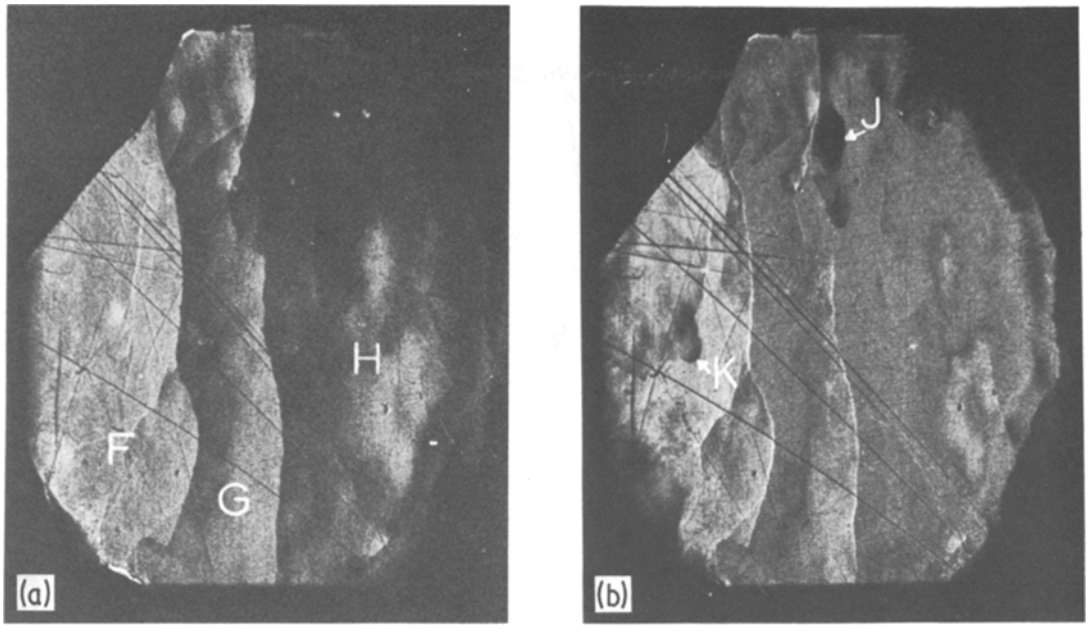
Figure 2 Topographs of a Bridgman plank as the incident angle (θ_I) is changed in steps of 5′ from θ_I. See text for details.

observed near the circumference of the slice. The average misorientation per grain in the area D (Fig. 2c) is approximately 1 to 2′, a little smaller than that for the slice (2 to 3′ per grain).

The reason for the formation of small grains in these particular Bridgman materials may be related to the fairly large temperature gradients present during growth [11]. These temperature gradients would be greater nearer the circumference of the boule, thus accounting for smaller grains with larger misorientations (cf. CR and CRA materials in the following sections).

3.2. Cast-recrystallized (CR)

The orientation of a slice of cast-recrystallized CMT was approximately 31° from [1 1 1]. The (3 3 1) reflection was used for topography for which $\theta_B = 31.5°$ and $\theta_I = 11°$. Three topographs taken in increments of 5′ are shown in Fig. 3a to c. There is no evidence of the large number of small grains present in the Bridgman material. Indeed it is just possible to image the complete slice (Fig. 3b) for a single fixed value of θ_I. Hence this particular CR slice is much nearer to being a single crystal than the Bridgman material. The straight



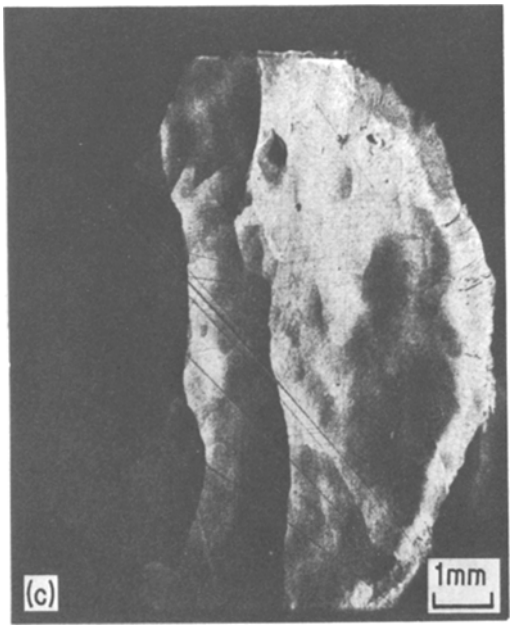


Figure 3 Topographs of a cast-recrystallized slice, taken as the incident angle (θ_I) is changed in steps of 5′ from θ_I.

lines running diagonally across the face are scratches produced by polishing. The two boundaries running from top to bottom are low-angle grain boundaries. This was deduced from observations on the positions at which the reflections from areas F, G and H peaked and a double crystal rocking curve from which it was found that the misorientation from area F to area G (Fig. 3a) was about 6′ while the misorientation from area G to area H was about 4′. Within these areas two additional features are evident. Firstly, a few additional grain boundaries are present (see areas J and K in Fig. 3b) with a fairly random distribution and direction. Secondly, a mottling effect characteristic of a strained crystal is also present in all three regions. Since it is very difficult to grow constant composition boules of CMT using either the Bridgman or the cast-recrystallize technique [4], it is possible that these mottled features may be associated with variations in composition (see also the effects of annealing in Section 3.3). This pattern and the presence of a few additional grain boundaries are even more clearly illustrated in Fig. 4 which shows double crystal reflection topographs [7, 8] of areas G and H (cf. Fig. 3a areas G and H). The difference in the value of θ_I for this pair of topographs is 4.5′. The fact that these two areas are imaged separately illustrates that the boundary between them is not a surface feature but a low-angle grain boundary.

3.3. Cast-recrystallized-annealed (CRA)

This slice was taken from a region close to the previous CR slice, consequently the orientation

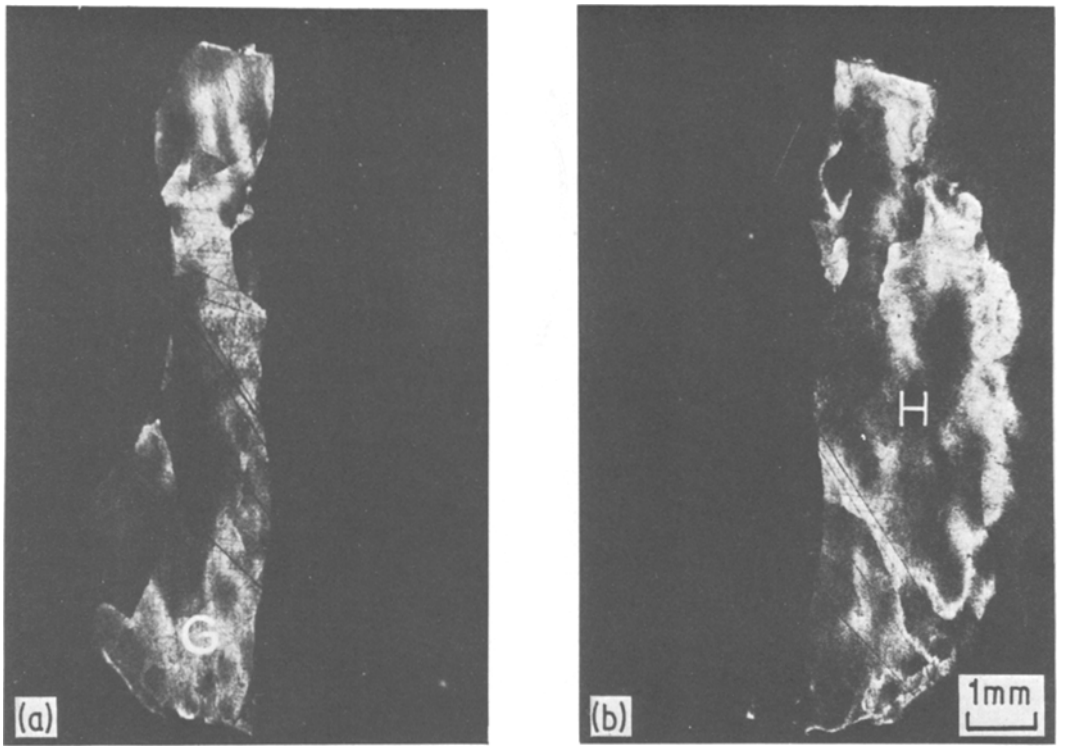


Figure 4 Double crystal reflection topographs of a cast-recrystallized slice, taken for two values of the incident angle. (a) θ_I and (b) $\theta_I + 4.5'$. The sample is identical to that shown in Fig. 3.

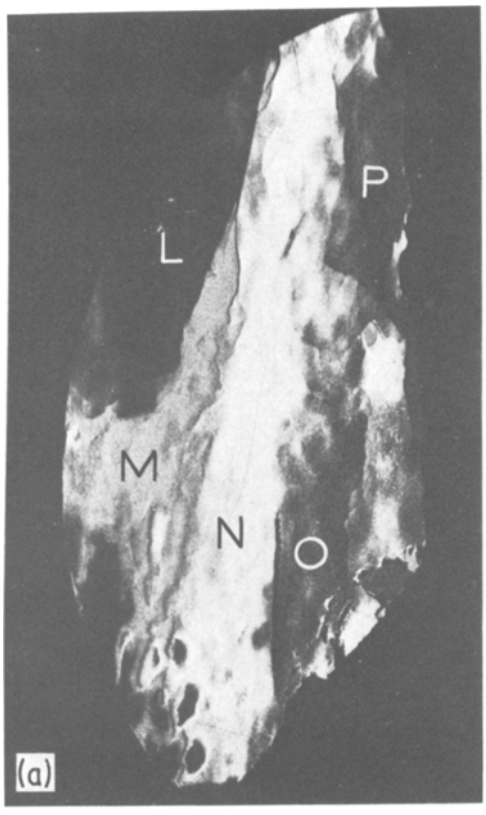
was approximately the same (31° from [1 1 1]). As before, the (3 3 1) reflection was used, with $\theta_B = 31.5°$ and $\theta_I = 11°$. This material has undergone an additional annealing treatment at 250 to 300° C in mercury vapour for several days. Topographs taken in 5′ increments are shown in Fig. 5a to c. The general features are similar to the CR material, namely fairly large sections within which the contrast is approximately the same (see for example areas L, M, N, O and P in Fig. 5b). The mottled features characteristic of a strained lattice are also present and have not been removed by the annealing. This supports the idea that they may be associated with compositional variations. The main difference is that there are rather more grain boundaries present in the CRA material than in the CR material.

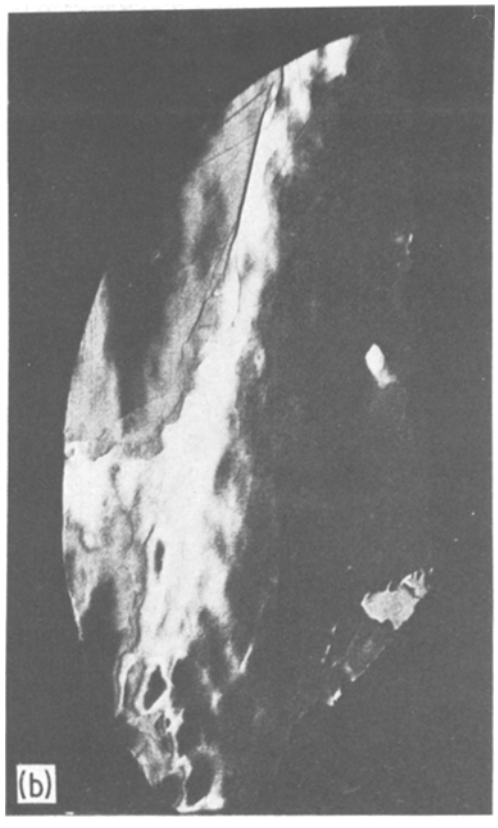
4. Conclusions

X-ray topography, in particular the Berg–Barrett technique, has proved to be extremely useful in the assessment of the grain structures of CMT prepared by the Bridgman and CRA methods. Topographs revealed that the Bridgman material studied consisted of large numbers of small grains (0.05 to 0.6 mm) with misorientations from 1 to 9′ per grain. In contrast, the CRA material studied had only a few grain boundaries and features consistent with a strained lattice, possibly caused by compositional variations.

Acknowledgements

I wish to thank Mullard Southampton for providing the CMT materials and Dr B. E. Bartlett for crystal preparation information. Also, thanks are due to Mrs R. S. Cosier for her invaluable assistance and for taking the double crystal reflection topographs. Finally, Dr P. T. Greene and my colleagues at PRL are thanked for many useful discussions, and the preparation of drawings and photographs by the PRL Photographic Department and Drawing Office is gratefully acknowledged. This work was carried out with the support of Procurement Executive, Ministry of Defence.





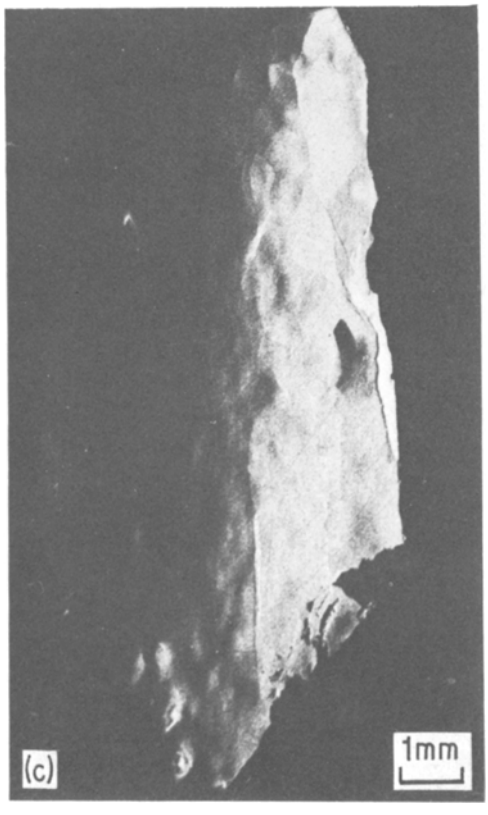


Figure 5 Topographs of a cast-recrystallized-annealed slice, taken as the incident angle (θ_I) is changed in steps of 5′ from θ_I.

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Received 28 April and accepted 22 August 1978.