JOURNAL OF MATERIALS SCIENCE 3 (1968) 120-126

Deformation Behaviour of Aluminium Macrocrystals in the Grain Boundary Region

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Received 19 May 1967, and in revised form 9 October

Lattice distortions of deformed aluminium macrocrystals were observed by Electron-microprobe-Kossel techniques. Grain boundary effects due to deformation appeared in various ways such as extraordinary separation and branching of Kossel lines, and as intergranular rotation of the grains in pseudo-Kossel X-ray diffraction patterns obtained from the grain boundary regions of the specimens. Such distortions of the patterns peculiar to the grain boundary regions were attributed to the effect of local slipping induced by the active slip systems of the neighbouring crystals. By the interpretation of these patterns and the orientation relationships between neighbouring grains, it was found that the local slipping was induced towards the slip direction of the active slip plane in the neighbouring crystals, or induced by a type of local stress along the slip direction of the active slip planes in the neighbouring crystals. It was also recognised that at an early stage of stressing, deformation bands were produced in the grain boundary region as a result of a grain boundary effect.

1. Introduction

The slip behaviour of aluminium single crystals caused by external tensile stresses can be analysed by considering their Schmid factors, but the behaviour of macrocrystals is much more complicated and difficult to analyse. These complications occur mainly at the grain boundary regions, where the slip systems of neighbouring grains often operate simultaneously with the slip systems of the grain itself. The conditions for two neighbouring grains interacting at the boundary region depend on many factors, such as the relative orientation differences, the shape of the grain boundary, the direction of the applied stresses, etc. Because of these many factors the deformation mechanisms of macrocrystals are not well understood. It was very difficult with ordinary X-ray methods to observe slip modes at a restricted position of such a small area as the grain boundary region, until the advent of the Electron-microprobe-Kossel technique [1].

When a finely focused electron beam irradiates a metal crystal, a characteristic X-ray point source is produced on the crystal surface. The divergent X-rays emitted from the source toward the directions satisfying the Bragg condition are diffracted, and form diffraction cones characteristic to the crystal planes, and they produce absorption and diffraction lines on a photographic film [2]. The pattern thus produced by a monochromatic and divergent X-ray source is a type of projection of the crystal planes, and is called a Kossel pattern if the X-ray source is inside the crystal, or a pseudo-Kossel pattern if it is outside the crystal [3]. Using capillary tubes or other fine focus X-ray methods, pseudo-Kossel patterns can be obtained [4, 5], but observations by these methods are rather macroscopic. When the Electron-microprobe-Kossel technique was developed, the resolution of the Kossel method was greatly improved, and patterns from very small areas on the specimen became obtainable. This enabled the observation of deformation modes of macrocrystals on a microscopic scale.

2. Experimental

The specimen macrocrystals used were grown in 99.99 % aluminium plates by the strain-annealing method, producing a grain size larger than 1 cm. Tensile tests were performed, and at the various stages of deformation Kossel patterns were obtained from the observation points shown in fig. 1. The crystallographic directions of tensile stress and crystal surface of every grain are shown in fig. 2. As the wavelength of the AlK_α radiation is too long to satisfy the Bragg condition, the pseudo-Kossel method using molybdenum radiation was employed. At each observation point a molybdenum target foil, 50 μm in thickness, was fixed using a conductive adhesive agent [6, 7]; also, a scale was ruled from which the amounts of local deformation at the point, due to inhomogeneous deformation, were measured. The Kossel camera used in this experiment was attached to a JEOL JXA-3-microprobe. The distance from film to specimen was about 60 mm, and the diameter of the X-ray film cassette 55 mm; with this film cassette five patterns can be taken successively.

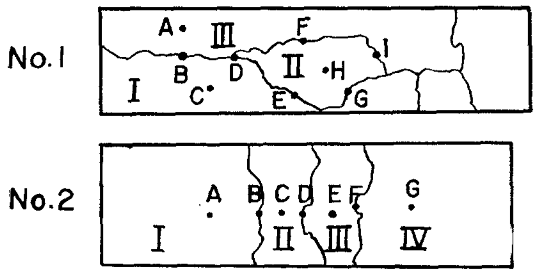


Figure 1 Shapes of specimen macrocrystals; A, B, C, D, E, F, G, and I denote the observation points.

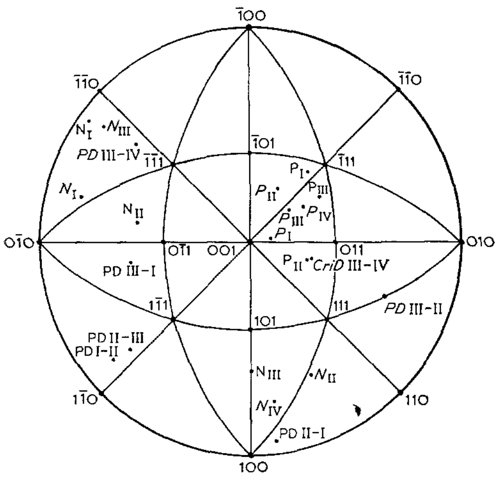


Figure 2 Crystallographic direction of surface normal of every crystal (P_i, P_i) and that of tensile stress for it (N_i, N_i). Italics denote specimen no. 2.

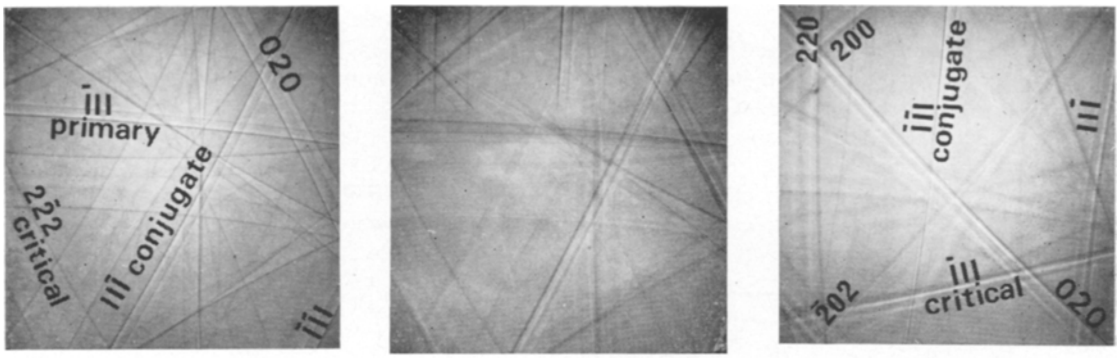
3. Experimental Results

Five patterns were obtained, in steps of approximately 150 μm, across each grain boundary region. Using an accelerating voltage of 40 kV and a specimen current of 1 μA, the exposure time was 8 min with a fine-grained X-ray film. Lattice distortion of the grain boundary region is generally larger than that in the inner part of the grain, and its behaviour depends on the orientation difference between neighbouring crystals, the shape of the boundary, the angle between the grain boundary and the external tensile stress, and on the direction of the stress. Crystals having a soft orientation to the tensile stress tend to be less affected by neighbouring crystals.

The distortion behaviour which is frequently observed at the grain boundary regions occurs in the following manner. As the deformation proceeds, some fine branches are generated near the tips of the Kossel lines; some of these are separated from the original lines, and some disappear. So, the Kossel lines obtained at certain positions of the grain boundary regions tend to become shorter, according to the amount of deformation. This means that minute crystallites are produced during the deformation at the grain boundary region. Fig. 3 is a typical example of these distorted Kossel patterns where (a) and (c) correspond to those of different crystals, and in (b) Kossel lines from both the neighbouring crystals overlap.

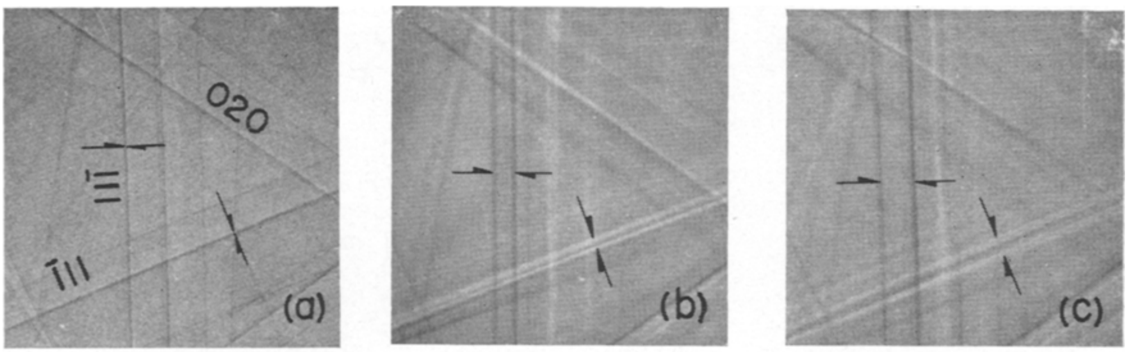
3.1. Anisotropy of Kossel Line Breadth

The breadth of a Kossel line is considered to be a measure of the crystalline state at the observation position, and in the case of deformation analyses the breadth shows the state of mosaic structure. An extraordinary separation of Kossel lines at a grain boundary region is shown in fig. 4. Three lines, i.e. $(11\bar{1})$, $(\bar{1}11)$, and (020), are separated into two lines, and their separation increases linearly with the amount of deformation. From the orientation analyses the $(11\bar{1})$ and $(\bar{1}11)$ lines correspond to the conjugate and critical slip planes respectively. Although the breadth of the $(\bar{1}11)$ line is larger than that of the $(11\bar{1})$ line in an inner region of crystal I,



(a) (b) (c)

Figure 3 Kossel patterns obtained from two neighbouring grains (a, c), and from their boundary region (b). (a), (b) and (c) correspond respectively to the observation points C, D, and E of specimen no. 2. Elongation 1.2%, 40 kV, 8 min.



(a) (b) (c)

Figure 4 Extraordinary Kossel line separation at the grain boundary region, at E of specimen no. 1. Elongation (a) 0%, (b) 1.0% (2.5%), (c) 2.0% (2.5%), where the figures in brackets denote local elongation at E. 40 kV, 8 min.

which is in agreement with the estimation from Schmid factors, this relationship is reversed near the grain boundary. Thus the separation of the $(11\bar{1})$ line is much larger than that of the $(\bar{1}11)$, as shown in fig. 4, and this implies that the neighbouring crystal affects the deformation of crystal I.

3.2. Distortions of $\{111\}$ Planes Parallel to Grain Boundaries

Lattice distortions of the same kind of crystal plane at the grain boundary regions depend on the angle between the plane and the grain boundary. Crystal planes parallel to the grain boundary are generally more distorted than those perpendicular to it. Kossel lines from the various $\{111\}$ planes are most sensitive to lattice distortions, so the $\{111\}$ lines were carefully examined for the case in which the $\{111\}$ planes were nearly parallel to the grain boundary. Two cases have to be considered, in which the tensile stress is either parallel or perpendicular to the grain boundary.

Fig. 5 shows typical distortion behaviour of the former case. The branching of the $(11\bar{1})$-III line means that the crystal is torn into blocks. From the separations of the branches, the angular misfit between all the crystal blocks is estimated to be about a maximum of $1^\circ\,40'$. The length of the branches, which is a measure of crystal block size, varies at every stage of deformation; that is, the tips of the branches once produced become diffuse and disappear as the deformation proceeds. This might mean that the crystal blocks produced by deformation are again polygonised into minute crystallites.

When the grain boundary is perpendicular to the tensile stress, the deformation proceeds in a different way compared to the former case. The $(\bar{1}11)$-II line (fig. 6) corresponds to the $(\bar{1}11)$ plane of crystal II, which is nearly parallel to the grain boundary, and perpendicular

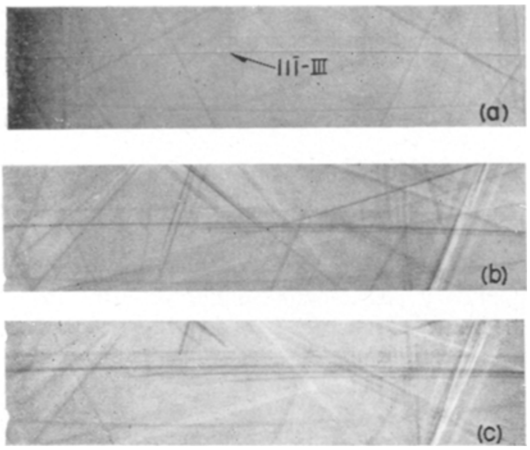


Figure 5 Distortion behaviour of a {1 1 1} plane parallel to both the grain boundary and the external tensile stress, at F in specimen no. 1. Elongation (a) 0%, (b) 0.5% (0.6%), (c) 1.0% (1.4%), where the figures in brackets denote local elongation at F. 40 kV, 8 min.

to the external tensile stress. The angular misfit appears to be more than 4° of arc in some cases; such large misfits and large block sizes are not observed in the other cases. Moreover the $(\bar{1}11)$ plane which is the primary slip plane for the external stress is not active in an inner part of crystal II. From another part of the Kossel pattern, the (1 1 1) plane whose Kossel line does not appear in fig. 6 is supposed also to be active at the grain boundary region of crystal II. Considering these circumstances the influences of neighbouring crystal III appear to be very large in this case.

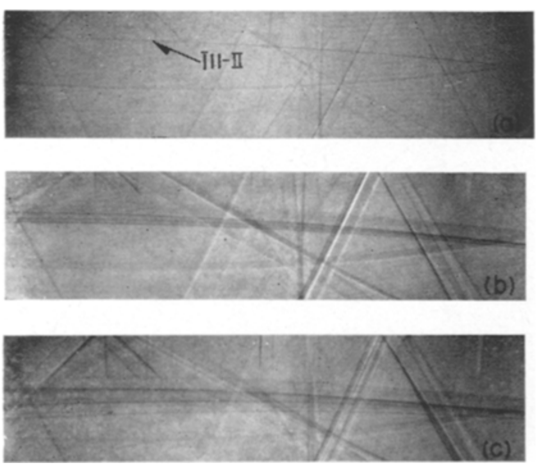


Figure 6 Distortion behaviour of a {1 1 1} plane parallel to the grain boundary and perpendicular to the external stress, at D in specimen no. 2. Elongation (a) 0%, (b) 0.5% (0.8%), (c) 1.0% (1.5%), where the figures in brackets enote local elongation at D. 40 kV, 8 min.

As for the distortions of the planes perpendicular to grain boundaries, no phenomena peculiar to the grain boundary can be seen. The deformation behaviour of such planes near the grain boundary is usually the same as that of those in the inner region of the grain. Therefore, it may be concluded that at a grain boundary region, fragmentation caused by the elongation occurs mainly on the {1 1 1} plane parallel to the grain boundary.

3.3. Intergranular Rotation

The Kossel lines shown in fig. 7 indicate that an

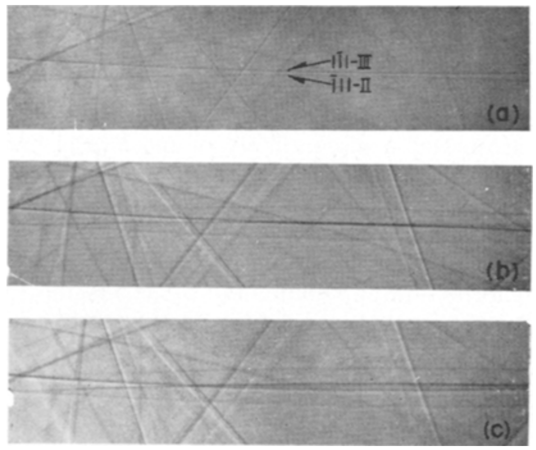


Figure 7 Intergranular rotation observed at I in specimen no. 1; $\bar{1}11$-II and $1\bar{1}1$-III lines correspond to grain II and III respectively. (a) 0%, (b) 1.0% (0%), (c) 2.0% (1.2%), where the figures in brackets denote local elongation at I. 40 kV, 8 min.

intergranular rotation is occurring between grain II and III. The $(\bar{1}11)$-II and $(1\bar{1}1)$-III lines in this figure gradually approach and finally intersect each other with increasing deformation. The amount of rotation around an axis taken parallel to the intersection of the $(\bar{1}11)$ and $(1\bar{1}1)$ planes of each grain is shown in fig. 8. Another type of rotation around an axis perpendicular to the specimen surface is also found by examining the whole area of Kossel patterns obtained at I and the maximum amount of this rotation is estimated to be about 30′.

3.4. Effect of Deformation Band on Kossel Patterns

With a certain amount of cold work, deformation

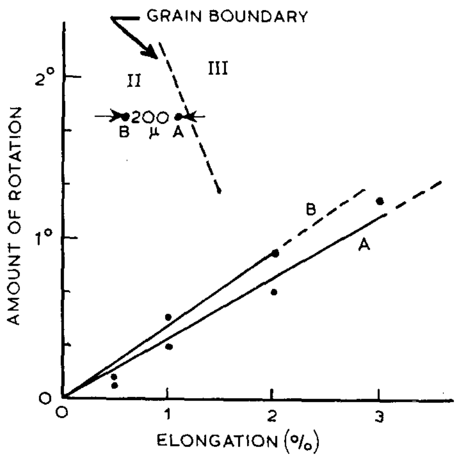


Figure 8 The amount of intergranular rotation shown in fig. 7, at two points shown by A and B.

bands appear as well as the slip traces. As the crystal planes become discontinuous at these bands, the existence of a deformation band appears as a discontinuity of the Kossel lines, and such an example is shown in fig. 9. The deformation band appearing on the $(1\bar{1}\bar{1})$ line is considered as a kink band. Kink bands do not generally appear at such a low degree of deformation in aluminium single crystals, so this means that a local kink band was generated at the grain boundary region of grain II by the effect of pile-up of dislocations at the boundary. The breadth of the kink band is about 20 μm and the $(1\bar{1}\bar{1})$ planes separated by it are inclined to each other by about 1.5° of arc. By observing the growth of the band at each stage of the deformation, the band initiates as an inclination of the planes, and then grows into a kink band.

4. Discussion

In the previous section some examples of the lattice distortions which are peculiar to the grain boundary region are mentioned. These grain boundary phenomena should be attributed to the influence from the neighbouring crystals, but, in general, it is very difficult to analyse them because of the many factors concerning the deformation of such regions. However, from the orientation relationships between neighbouring grains, some of the grain boundary phenomena can be analysed.

Considering a type of tensile stress caused in a crystal by the active slip of the neighbouring crystal, it will operate locally in the grain boundary region of the crystal and might cause a lattice distortion there. When slip is occurring, the atoms on the slipping plane move toward the slip direction; and so the direction of the above local stress is considered to be the same as the slip direction in the first crystal. With this simple mechanism, the anisotropy of Kossel line breadth, shown in fig. 4, can be described as follows. By the orientation difference of crystals I and II, the primary slip direction of crystal II is transferred to the direction in

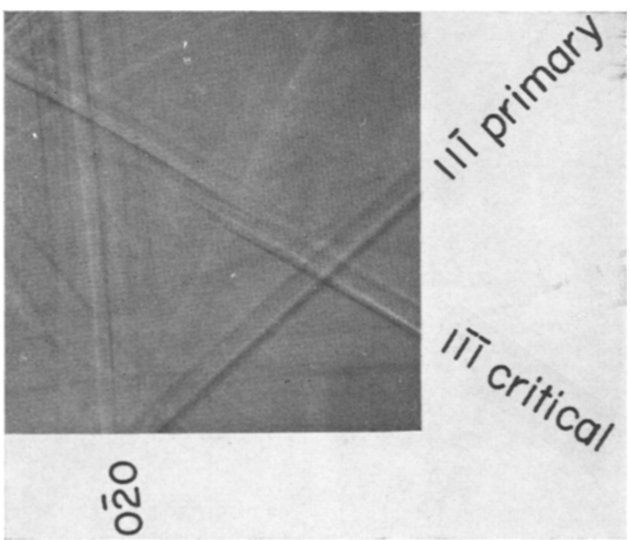


Figure 9 Effect of a kink band on a Kossel line, obtained at E in specimen no. 1. Elongation 2.5%.

crystal I, as shown by PDII-I in fig. 2. For a tensile stress of this direction, the following slip systems and their Schmid factors in crystal I may be considered.

$(11\bar{1})$ [101]	0.46
$(\bar{1}11)$ [101]	0.29
$(1\bar{1}1)$ [110]	0.42
(111) $[\bar{1}01]$	0.45

From this it should be noticed that the slip on the $(11\bar{1})$ plane is much more likely than that on the $(\bar{1}11)$ plane. The grain boundary effect appearing in fig. 4 is in agreement with this prediction. This interaction mechanism can also be applied to the phenomena shown in figs. 5 and 6. As for the distortions of the $(11\bar{1})$-III lines in fig. 5, the primary slip direction for crystal II (first crystal) becomes PDII-III direction for crystal III (second crystal), as shown in fig. 2. The local tensile stress of this direction will cause a slip on the $(11\bar{1})$ plane in crystal III, so the local distortion appearing in the $(11\bar{1})$-III line can be attributed to the effect of the active primary slip system in the neighbouring crystal II. In the case of fig. 6, the active slip systems in the first crystal, i.e. crystal III, are the primary and critical slip systems for crystal II, and the slip directions of these systems are denoted by PDIII-II and CriDIII-II respectively in fig. 2. The large distortion appearing in the $(\bar{1}11)$-II Kossel line in fig. 6, and also the local active slip on the (111) plane can be attributed to the local tensile stresses of these directions. In short, at the grain boundary region of crystal II, the dominant primary and critical slip systems of neighbouring crystal III have locally excited the following three slip systems: $(\bar{1}11)$ [101], (111) $[1\bar{1}0]$, and (111) $[\bar{1}01]$. Of these systems, the first was influenced by both of the active slip systems in crystal III, thus causing the large distortion as may be seen in fig. 6.

There are other mechanisms by which grain boundary effects can be introduced. When the $<110>$ direction coincides with the active slip direction of the neighbouring crystal, slipping would be enhanced on the {111} plane for the Schmid factor in the $<110>$ direction which is largest. This was observed as a cause of the extraordinary line broadening in crystal II by the effect of crystal I at E in specimen no. 1. This case might be considered as an example of a kind of a coherent grain boundary [8].

Grain boundary shear phenomena and lattice rotations resulting from active slip were considered as the cause of the intergranular rotation in fig. 7. Fig. 8 shows that the amount of rotation is larger at a point a little away from the grain boundary than at a position somewhat closer to it. From fig. 7, it is apparent that there is no dominant slipping in the grain boundary region of either crystal. These two facts suggest that the cause of intergranular rotation is due to neither of the above two phenomena. But it can be noted that the amount of elongation of crystal I at G is fairly large and the shape of the grain boundary near G has a curvature, so that a type of tension stress might act in such a way to stretch the grain boundary. This would induce the above intergranular rotation.

5. Conclusion

It was found from the Kossel patterns obtained at the grain boundary regions of deformed macrocrystals that the orientation relationship plays an important role in the occurrence of induced slip due to the effect of neighbouring crystals. The slip system of induced slip can be determined in an ideal case by considering Schmid factors for a tensile stress directed towards the slip direction of the first crystal. Another type of interference mechanism concerns the continuation of slip in which a grain boundary does not act as an arrest for the propagation of slip in the original direction.

Distortions of the Kossel lines are usually remarkable in the boundary region and their peculiar phenomena observed in these experiments are summarised as follows.

(i) Distortion phenomena of Kossel lines peculiar to the grain boundary region are extraordinary broadening or branching of lines, rotation of crystal planes, activation of the slip systems which are not active in the interior of the grain, and the appearance of local deformation bands. Some of these phenomena can be analysed by the interference mechanisms proposed above.

(ii) In the case of a {111} plane which is oriented nearly parallel to the grain boundary, mosaic blocks are generated on the plane by the deformation, but when the plane is perpendicular to the elongation stress, the generated blocks are much larger than those generated under other conditions; in the case parallel to the stress, many branches in Kossel lines are produced. Mosaic blocks are not usually generated on a plane normal to the grain boundary.

(iii) At a grain boundary region, the effect of the active slip system of the neighbouring crystal is large when the orientation of the neighbouring crystal is soft for the applied elongation stress. But sometimes, according to the shape of the grain boundary, and to the orientation relation to the neighbouring crystal, the effect of neighbouring grains cannot be seen.

(iv) When deformation proceeds, minute crystal blocks with small coherent domains are produced at the grain boundary region, and the width of the band formed by the minute crystal blocks increases with the deformation.

Acknowledgement

The Authors express their thanks to Dr H. Kawabe for his kind and appropriate suggestions during the work.

References

1. R. CASTAING, Thesis, University of Paris (1951). R. CASTAING and A. GUINIER, *Compt. rend.* **232** (1951) 1948.
2. K. LONSDALE, *Phil. Trans.* **240** (1947) 219.
3. P. GIELEN, H. YAKOWITZ, D. GANOW, and R. E. OGILVIE, *J. Appl. Phys.* **36** (1965) 773.
4. T. IMURA, *Bull. Naniwa Univ.* **2A** (1954) 51.
5. A. H. GEISLER, J. K. HILL, and J. B. NEWKIRK, *J. Appl. Phys.* **19** (1948) 1041.
6. M. UMENO, H. KAWABE, and G. SHINODA, "Advances in X-ray Analysis", Vol. 9, edited by G. R. Mallett, M. J. Fay, and W. M. Mueller (Plenum Press, New York, 1966) p. 25.
7. M. UMENO, H. KAWABE, and G. SHINODA, "Optique des Rayons X et Microanalyse", edited by R. Castaing, P. Deschamps, and J. Philibert (Hermann, Paris, 1966) p. 534.
8. K. G. DAVIS, E. T. TEGHTSOONIAN, and A. LU, *Acta Met.* **14** (1966) 1677.