Physics. — Influence of the texture of the original matrix on the number of inclusions in aluminium single crystals obtained by recrystallization. By W. G. BURGERS and V. CH. DALITZ. (Laboratorium voor Physische Scheikunde der Technische Hogeschool, Delft.) (Communicated by Prof. J. M. BURGERS.)

(Communicated at the meeting of May 28, 1949.)

1. Some years ago BURGERS and MAY (1945) observed that aluminium single crystals often contain a large number of small inclusions, which, judging from their identical reflection after etching, possess definite preferential orientations. These inclusions apparently cannot be removed, or in any case only with difficulty, even after prolonged heating at the highest possible temperature. So for example in crystals prepared by us, heating at 630° C during 1000 hours did not cause a noticeable decrease in number. CARPENTER and TAMURA (1927) and also SEUMEL (1936) observed a decrease only after very long periods of heating.

Both by etching and by X-rays it was found by TIEDEMA, MAY and BURGERS (1948; 1949) that in far the most cases the lattice orientation of the small included crystallites [called "cristaux insulaires" by LACOMBE and BERGHEZAN (1949)] was approximately, with deviations up to 5-10°, that of one of the four possible spinel twins with regard to the surrounding crystal. From the fact that the Laue-spots were slightly elongated, it could be deduced that the inclusions are grains of the original matrix, at the cost of which the large crystals have grown, and which were not consumed by these crystals during their growth. It seems obvious to ascribe this behaviour to the approximately symmetrical lattice position of growing and non-consumed crystal, which reduces the tendency of the boundary to displace itself in one or other direction. This conclusion seems to be supported by a recent paper by DUNN and LIONETTI (1949) on the effect of orientation difference on grain boundary energies, the result of which points to a low value for the relative surface energy between two lattices in either parallel 1) or in mutual twin orientations 2).

<sup>&</sup>lt;sup>1</sup>) In this connection it is important to mention that, as shown by X-rays, a crystal grown by recrystallization actually contains also inclusions which, within a few degrees, have the *same* orientation as that of the surrounding crystal [TIEDEMA, MAY and BURGERS (1949)]. Moreover it was found [TIEDEMA (1949)] that a large crystal cannot grow at the expense of a texture with approximately the same orientation as the growing crystal.

<sup>&</sup>lt;sup>2</sup>) SMITH (1948) pointed out that the extremely small energy of the twin boundary in annealed face-centered cubic metals in relation to the grain boundary can be deduced from the fact that a twin can meet a grain boundary at virtually any angle without much deviation of the latter.

Up till now we donot yet know whether a deviation of the exact twin position in an "arbitrary direction" is sufficient to impede boundary displacement or whether a deviation in one direction is more active in this respect than in others. We are inclined to believe that the latter supposition is the more probable and that the non-consumability of various grains is different, depending on their precise orientation relationship with regard to the growing crystal. We think this conclusion may be deduced from the experimental fact, illustrated in fig. 1<sup>3</sup>), that the number of inclusions at the boundary of a growing crystal is markedly greater than more inside the crystal. This suggests that of those grains which are left unabsorbed in the first moment of their contact with the growing crystal, a part is still consumed in the course of the annealing process.

If the conclusion put forward in the foregoing is correct, one may 2. expect it to be a necessary condition for the occurrence of inclusions in large crystals grown by recrystallization that the crystal growing at the expense of the fine-grained matrix should meet in the course of its growth grains in approximate twin position. If the grains of the original matrix exhibit none or in any case only a slightly pronounced preferential orientation, this possibility is always present, independant of the orientation of the new growing crystals. On the other hand, if the large crystals grow in a matrix with a very "narrow" range of orientations of its constituent grains, it may be anticipated that a new crystal either has practically no inclusions or on the contrary a very large number. The former would occur if the orientation of the new crystal is very much different from that of a spinel twin orientation with regard to the average position of the texture; the latter, if its orientation lays close to the twin orientation of the matrix.

In order to test this expectation, a matrix with a very narrow texture was prepared. To this end, starting from quasi-isotropic fine-grained material, large crystals (several  $cm^2$  in area) were prepared according to CARPENTER and ELAM's original method (CARPENTER and ELAM, 1921). Practically all of these crystals contained a considerable number of inclusions, on the average 30–100 per cm<sup>2</sup>. Figures 2 and 3 (crystal A) give examples. The inclusions were resistent against prolonged heating at 630° C.

When such crystals (we shall call them "mother-crystals") are subjected to an elongation of 10—15 %, they give rise to very pronounced textures (that of the deformed single crystals), which, on annealing, are transformed anew into large crystals. For example an extended single crystal plate of  $8 \times 2.5$  cm<sup>2</sup> (thickness 0.5 mm) was transformed, by recrystallization at 550° C during one hour, into 1—7 new crystals, the number in each case depending on the exact amount of stretching.

<sup>3)</sup> Also in fig. 9 in the paper by BURGERS and MAY (1945).

W. G. BURGERS and V. CH. DALITZ: Influence of the texture of the original matrix on the number of inclusions in aluminium single crystals obtained by recrystallization.



Fig. 1. Large aluminium crystals, growing into a fine-grained matrix. The large crystals show many inclusions ("insular grains"), which are non-absorbed grains of the original material. Their number is far greater close to the boundary of the growing crystal than in the centre of the crystal. The irregular shape of the boundary is very pronounced, due to the selective character of the growth process, as if the growing crystal were feeling its way in the fine-grained matrix. (<sup>1</sup>/<sub>2</sub> Natural Size).



Fig. 2. New crystal (B) grown at the cost of a deformed single crystal (A). Contrary to (A), (B) is practically free from inclusions. (Natural Size).



Fig. 3. The figure shows two parts of the same plate, which originally contained one large crystal (A), with many inclusions. The plate, after 20 % stretching, was cut into two parts, of which that shown at the right was recrystallized. One of the new crystals, (B), again contained a large number of inclusions. It was found that the orientation of (B) again contained to the first state of the state of th

(B) was approximately that of a spinel twin with regard to (A). (Natural Size).

625

Now it is obvious that in general neither the deformed texture of the "mother-crystal", nor the inclusions present in it, occupy a twin position with regard to the second generation of large crystals, so that we may expect these latter crystals to be free of inclusions. This proved actually to be the case for most crystals. Fig. 2 shows an example of such an "inclusion-free" crystal (B) grown into a mother-crystal (A) with many inclusions.

If, however, the particular case occurs that a crystal of the second generation happens to stand in (approximate) twin position with regard to the deformed mother-crystal, then the new crystal has a good chance to encounter, in the course of its growth, lattice regions in (approximate) twin position and the crystal may be expected to contain numerous inclusions. Actually such a case was found for the crystal shown in fig. 3. The two plates shown here formed originally one large plate, in which the "mother-crystal" (A) extended over the whole plate. After being cut into two halves, that shown at the right was stretched 20 % and recrystallized. The large new crystal (B) contains numerous inclusions. Laue-photographs of the (deformed) mother-crystal (A) and the new crystal (B) showed definitely that (B) occupied approximately a twin orientation with regard to (A).

From these experiments it may be concluded that actually the number of inclusions depends on the "width" of the texture of the original matrix in which the large crystals grow. In case this texture is sufficiently narrow, one obtains *either* (and presumably in most cases) crystals with none or only few inclusions or on the contrary (presumably rather seldom) crystals with very many inclusions. In case the original fine-grained matrix has no preferential orientation (is quasi-isotropic), the large crystals grown in it may be expected to have on the average the same number of inclusions (per unit of surface).

3. Finally the following remark may be made. As is well known, copper crystals, made by recrystallization of fine-grained material, in most cases contain numerous small twin crystals. It was found by TAKEYAMA (1930) that in some cases the number of twins could considerably be diminished by subjecting the primary crystals to a second deformation and recrystallizing anew. The same result was obtained by us in analogous experiments (although the elimination was far from complete). It seems justified to ascribe this behaviour to the same cause as valid in the case of aluminium.

## Summary.

Large crystals of aluminium grown by recrystallization in fine-grained material often contain many "inclusions". These inclusions are unabsorbed grains of the original matrix, which are standing approximately in twin position with regard to the surrounding crystal. Their presence is due to the fact that a growing crystal cannot, or, if so, with great difficulty absorb lattice regions in approximate twin position. It follows that the number of inclusions depends on the texture of the fine-grained material in which the crystals grow. If this texture is very narrow, crystals can grow which are practically free from inclusions.

## **REFERENCES.**

- W. G. BURGERS and W. MAY, Rec. Trav. Chim. Pays-Bas 64, 5 (1945).
- H. C. H. CARPENTER and C. F. ELAM, Proc. Roy. Soc. London (A) 100, 329 (1922).
- H. C. H. CARPENTER and S. TAMURA, Proc. Roy. Soc. London (A) 113, 161 (1927).
- C. G. DUNN and F. LIONETTI, Metals Transactions, February 1949, T. P. 2517.
- P. LACOMBE and A. BERGHEZAN, Métaux et Corrosion, January 1949.
- G. SEUMEL, Z. Krist. 93, 249 (1936).
- C. S. SMITH, Metals Technology 1948, T. P. 2387.
- S. TAKEYAMA, Mem. Coll. Sc. Kyoto Imp. Univ. (A) 13, 353 (1930).
- T. J. TIEDEMA, W. MAY and W. G. BURGERS, Nature 162, 740 (1948); Acta Cryst. 2, 151 (1949).
- T. J. TIEDEMA, Acta Cryst., in press.