### Preparation of Aluminium Single Crystals with a Definite Orientation

By T. J. TIEDEMA\*

Laboratorium voor Physische Chemie, Julianalaan 136, Delft, Holland

### (Received 28 March 1949)

A method is described for the preparation of aluminium single-crystal wires and plates with any desired orientation to within  $1^{\circ}$ .

### 1. Introduction

It is of value to have available aluminium single crystals with any desired orientation, for example, in studying the recrystallization of deformed single crystals or in experiments on creep. A method has been worked out which allows the preparation of single crystals with a definite orientation within  $1^{\circ}$ . Fujiwara (1939, 1941) has already described a method for the preparation of such single crystals, but, owing to the war, it is impossible to consult his papers.

# 2. The preparation of single-crystal wires with a definite crystallographic direction parallel to the wire axis

Hard-drawn wire is used of a purity of 99.5 % and a diameter of 2 mm. This wire has a more or less pronounced [111] texture in the wire axis. To convert this polycrystalline wire into a single crystal, Lebbink has worked out the following method, which has so far not been published.

The wire is first annealed by passing it through a furnace at a rate of 6.7 cm./min. at a temperature of  $550^{\circ}$  C. The diameter of the tube of the furnace is 2 cm. After stretching the wire 2 %, it is passed again through the furnace, now at a rate of 4 cm./hr. and at a temperature of  $630^{\circ}$  C. This produces very long single crystals, up to a length of 150 cm. and beyond. These single crystals always possess a [210] direction parallel within a few degree to the wire axis (Burgers & Sandee, 1942).

To prepare single crystals with a different direction parallel to the wire axis the following method is used: The recrystallization is interrupted when the crystal has grown to a length of some centimetres. The wire is then mounted in a bending apparatus (see Fig. 1) and fixed by a drop of molten wax. The figure shows that the crystal sticks out freely. Care must be taken not to deform the single crystal, otherwise any further growth is impossible as was shown by van Arkel & Ploos van Amstel (1930). A Laue transmission photograph of the single crystal is taken, and from this photograph the orientation of the crystal is determined by the method of Schiebold & Sachs (1926). During the exposure the

 $\ast$  Associate worker of the Foundation for Fundamental Research in Holland.

wire axis is vertical and the direction of irradiation is perpendicular to the wire axis. The wire axis is therefore parallel to the plane of the stereographic projection. The pole of the plane, which is chosen to be perpendicular to the wire axis, must be brought on to the circumference of the stereographic projection. The angle through which the wire must be rotated around its axis to realize this can be read from a stereographic chart. After rotating the wire, the polycrystalline part is bent in such a way that the wire axis becomes parallel to the desired crystallographic direction. Fig. 2(a, b)illustrates this for a wire in which the cube direction is brought parallel to the wire axis.



Fig. 1. Bending apparatus for aluminium wire. The single crystal wire (c) of length some centimetres is fixed in the capillary (a) by a drop of molten wax (b). The wire can be rotated about its axis by means of the disc (d) and bent by means of the slide (e).

Fig. 2 (a) gives the projection of the cube poles of the crystal which has grown for some centimetres into the polycrystalline wire. By rotating the wire  $\alpha^{\circ}$ around its axis, indicated in Fig. 2 (a) by the arrows, the cube pole (p) comes on the circumference of the stereographic projection. Of course that cube pole is chosen, the direction of which deviates least from the (vertical) wire axis, so that the curvature of the wire becomes as small as possible. By bending the polycrystalline part through an angle of  $\beta^{\circ}$ , indicated in Fig. 2 (b), the wire axis becomes parallel to the cube direction.

After these two manipulations the wire is removed from the bending apparatus and the recrystallization continued. This time the wire passes on a nickel-silver plate through a furnace with a 'tube' of rectangular cross-section at a rate of 2 cm./hr. and at a temperature of 630° C. The dimensions of the 'tube' of the furnace used by us are: length 60 cm., width 6 cm. and height 1.2 cm.

AC2

The lattice retains its original position when the crystal is growing through the curve, as is indicated in Fig. 4; it is thus possible to obtain single-crystal wires with any desired direction parallel to the wire axis. An exception, however, is the [111] direction. It has been found earlier that a growing crystal cannot consume grains which possess almost the same orientation as that crystal (Tiedema, May & Burgers, 1949). This case presents itself when a crystal with a [111] direction parallel to the wire axis grows into a [111] texture. A single-crystal wire with a [111] direction



Fig. 2. (a) Cube poles of the single crystal in their original position. (b) Position of the cube poles after rotating the wire about its axis through an angle  $\alpha^{\circ}$ .

Fig. 3. (a) Octahedral poles in their original position. (b) Position of the octahedral poles after torsion through  $\gamma^{\circ}$ . (c) Position of the octahedral poles after bending through  $\delta^{\circ}$ .

parallel to the wire axis, however, can be obtained by stretching a single-crystal wire with another crystallographic direction parallel to the wire axis, for example a [210] direction, after which it is recrystallized, rotated and bent in the way described above. If one starts from a single-crystal wire, the degree of stretching must be about 15 %.

Finally, it must be mentioned that too sharp curvatures must be avoided (maximum  $40^{\circ}$ ), otherwise the deformation in the curve becomes so large that new crystals start growing.

## 3. The preparation of single-crystal plates with a definite plane parallel to the surface of the plate

Rolled aluminium sheet of 1 mm. thickness and a purity of 99.5 % is used in our experiments. From this material a strip is sawn of length 30 cm. and width 2 cm. This strip is annealed by passing it on a nickel-silver plate

through the second furnace described in §2 (with the 'tube' of rectangular cross-section) at a rate of 9 cm./min. and at a temperature of 550°C. Thereafter the strip is stretched 2 % and passed again on a nickel-silver plate through the furnace now at a rate of 3 cm./hr. at a temperature of 630°C. The recrystallization is interrupted as soon as a crystal with a length of some centimetres is formed. A Laue transmission-photograph of this crystal is taken and from it the stereographic projection is determined. During the exposure the plate is parallel to the film, whereas the longitudinal direction is horizontal. Now the polycrystalline part of the plate is given such a position that, when recrystallization is continued, the chosen plane becomes parallel to the surface of the plate. This is done by a certain amount of torsion followed by a bending. The necessary angles can be read from a stereographic chart.



Fig. 4. Position of the lattice in a bent wire.

Fig. 3(a-c) gives an example for a plate in which an octahedral plane is brought parallel to the surface. Fig. 3(a) shows the projection of the four octahedral poles of the single crystal grown for some centimetres into the polycrystalline plate. Fig. 3(b) shows the positions of these poles after a torsion through an angle of  $\gamma^{\circ}$ , indicated by arrows in Fig. 3 (a), whereas Fig. 3 (c) shows the positions after bending through an angle of  $\delta^{\circ}$ , indicated by arrows in Fig. 3(b). In Fig. 3(b, c) the polycrystalline part is parallel to the plane of projection. Here, too, deformation of the single crystal must be avoided for the same reason as mentioned in §2. As the aluminium sheet used has no pronounced preferred orientations, no special difficulties are met at the further recrystallization, which is carried out in a furnace with a tube of rectangular cross-section. The dimensions of the tube are: length 66 cm., width 6 cm. and height 2.5 cm. The velocity is again 2 cm./hr. and the temperature 630° C.

The torsion should not exceed  $25^{\circ}$ , whereas the bending must be smaller than  $40^{\circ}$ . A Laue transmission-photograph of a single-crystal plate, obtained in the way described, shows no indication of asymmetry.

In conclusion, I find it a pleasure to thank Prof. Dr W. G. Burgers and Dr W. May for reading and criticizing the manuscript.

#### References

VAN ARKEL, A. E. & PLOOS VAN AMSTEL, J. J. A. (1930). Z. Phys. 62, 43.

BURGERS, W. G. & SANDEE, J. (1942). *Physica*, 's Grav. (N.S.), 9, 997.

FUJIWARA, T. (1939). J. Sci. Hiroshima Univ. A, 9, 227. FUJIWARA, T. (1941). J. Sci. Hiroshima Univ. A, 11, 89. SCHIEBOLD, E. & SACHS, G. (1926). Z. Krystallogr. 63, 34. TIEDEMA, T. J., MAY, W. & BURGERS, W. G. (1949).

Acta Cryst. 2, 151.