did not generally differ from the average breadths by more than a factor of ~ 2, with the possible exception of some spots of the recovered material. In general, therefore, the asymmetry of the interference function does not seem to be very pronounced (Hirsch, 1950).

11. Background

A characteristic feature of the micro-beam photographs of rolled aluminium is the background between spots. For slightly deformed material the background and spots form an arc of continuously varying radius. It follows that the background between spots is due to boundary regions between particles (see Hirsch & Kellar, 1952). For the heavily rolled material neighbouring spots are sometimes joined continuously by background, thus indicating that the particles are neighbours and that the background is due to the boundary region between them. After recovery, this background largely disappears. This diffuse nature of the background suggests that the boundary region consists of highly distorted material.

12. Conclusions

The method described in this paper enables the determination of approximate values (to perhaps within a factor of 2) of the breadths of the interference function in reciprocal space from the shapes of the spots. It should be emphasised that this method can be applied to any spotty-ring photograph from stationary specimens (not only to micro-beam photographs), provided that the spots on the photographs are large compared with the dimensions of the diffracting crystals. This condition can usually be satisfied by choosing a sufficiently large specimen-film distance. If necessary, however, it is possible to correct for the finite cross-section of the crystal (Hirsch, 1950). By using this method it is possible to obtain quantitative estimates of the perfection of the diffracting particles inside a polycrystalline matrix from simple back-reflexion photographs. The results on polycrystalline aluminium reported here will be interpreted, in terms of the distortion and shape of the particles, in a later paper.

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A Study of Cold-Worked Aluminium by an X-ray Micro-Beam Technique. III. The Structure of Cold-Worked Aluminium

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The results reported in two previous papers are discussed in the light of present theories of plastic deformation of metals. It is estimated that there are $\sim 10^{10}$ dislocations/cm.^{2} in the boundaries between particles in cold-worked aluminium. The physical broadenings of the X-ray reflexions from the particles are discussed, and it is concluded that the particles are distorted. Some deductions are made about the possible types of distortion in the particles. The formation of particles is considered to be due to polygonization. The changes which occur during recovery and those induced by impurities are interpreted on this basis.

Introduction

Theories of the strength of cold-worked metals depend critically on the assumed distribution of dislocations inside the metal, but so far it has not been possible to determine this distribution experimentally. The results of the micro-beam experiments on cold-worked aluminium, however, lead to some conclusions about the possible distribution of dislocations in this metal.

In the first part of this paper the physical broadenings of the X-ray reflexions from the particles in cold-worked aluminium are interpreted. (For a determination of the physical broadenings see Hirsch, 1952*a*). It is shown that the broadenings are mainly due to distortion of the particles, although some contribution may be due to their shapes. The types of distortion possible are discussed, and an attempt is made to determine the number of dislocations inside the particles.

The second part of this paper contains a summary and discussion of all the experimental results (Hirsch & Kellar, 1952; Hirsch, 1952). The formation of distinct particles is interpreted as a segregation of dislocations into walls forming the particle boundaries. An estimate of the density of dislocations in the boundaries is made from the size of the particles and the angles between them (Hirsch & Kellar, 1952).

I. INTERPRETATION OF PHYSICAL BROADENINGS

It is convenient to consider two possible limiting hypotheses: (*a*) the broadenings may be due to the shape of the particles, the particles being perfect; or (*b*) the broadenings may be due entirely to distortion of the particles.

It is expected that both factors contribute to the broadenings, but if the effects predicted on the basis of each of these assumptions are examined in turn, it is possible to determine the importance of the two types of broadening.

1. Particle-shape broadening

The broadening is conveniently described in terms of the interference function associated with each reciprocal point. The function depends on the exact shape of the particle (e.g. James, 1948, chap. 10). Orders of magnitude of the broadening may be obtained by considering a rectangular parallelepiped crystal with edges in directions parallel to dr_1, dr_2, dr_3 (for notation see Hirsch, 1952). For such a crystal the interference function will be appreciable only over distances

$$dr_1 = 1/X, \quad dr_2 = 1/Y, \quad dr_3 = 1/Z ,$$

where X, Y, Z are the lengths of the edges of the crystal.

Hence

$$d\varphi_1 = \lambda/2X \cos\theta , \qquad (1)$$

$$d\varphi_2 = \lambda/2Y \sin\theta , \qquad (2)$$

$$d\varphi_3 = \lambda/2Z \sin\theta . \qquad (3)$$

(For definition of $d\varphi_1$, $d\varphi_2$, $d\varphi_3$, see Hirsch, 1952.)

2. Distortion broadening

The broadening will depend on the kind of distortion present in the crystal. The types of distortion expected in a cold-worked metal may be divided into two limiting classes:

(*a*) The distortion may be purely elastic, in the form of elastic stresses which are continuous over the crystal. If the distortion is due to elastic bending, the arcs of bending are given approximately by $d\varphi_2$ and $d\varphi_3$, while the range of strains is obtained from $d\varphi_1 = -\tan\theta\delta d/d$ ("strain" broadening).

(*b*) The distortion may be localised around dislocations. In this case the broadening may be of the "strain" or "particle size" type according to the distribution of dislocations. For example, if the dislocations are arranged in walls inside the particle, the particle contains a mosaic structure. The broadening may then be due to the small size of the mosaics, to the curvature of the particle as a whole, and to the elastic stresses around the dislocations.

3. Shape of particles

For a specimen of heavily rolled spectroscopically pure aluminium examined immediately after rolling, the physical broadenings averaged around the ring were found to be

$$d\varphi_2 = 1{\cdot}5\times10^{-3} \text{ radians} ,$$

$$d\varphi_3 = 1{\cdot}9\times10^{-3} \text{ radians} .$$

It was also found that although the broadenings of individual spots assumed a range of values about this mean value, the width of the distribution curve was small. The order of magnitude of the broadening of the reflexion from a single particle is therefore given by the values quoted above.

Suppose first that the whole of the observed breadths are due to particle-shape effects. Applying formulae (2) and (3), it is found that $Y = 5{\cdot}5\times10^{-6}$ cm., $Z = 4{\cdot}3\times10^{-6}$ cm. If it is further assumed that the particles are approximately equiaxed, the mean particle size is $\sim 5\times10^{-6}$ cm. From the method of counting spots, however, the particle size was found to be $\sim 2\times10^{-4}$ cm. Hence this model leads to inconsistencies, and if the broadenings are due to particle shape, non-equiaxed particles, such as rods or lamellae, must be considered.

Now, from the broadenings, two dimensions at right angles in the crystal, Y, Z, are known. Since the volume is known to be $\sim (2\times10^{-4})^3$ cm.^{3} the third dimension, X, must be $\sim 3{\cdot}4$ mm. The assumption that the observed breadths are due to particle shape effects thus leads to the conclusion that the particles are in the form of long thin rods; the length of the rods is much greater than the mean diameter of the original grains ($\sim 20\mu$). Hence, this model, too, must be rejected as physically unreal, and it is concluded that part of the broadening must be due to distortion.

Suppose now that at least part of the observed broadening is due to distortion. Then, if the particles are rods, their maximum length may be taken as $X \sim 20\mu$; hence $Y = Z \sim 6\times10^{-5}$ cm. The shape broadening from such particles is negligible compared with the values found for $d\varphi_2$ and $d\varphi_3$. Therefore, in this case most of the observed broadening must be due to distortion. If the particles are in the form of thin lamellae of thickness $Y \sim 5 \times 10^{-6}$ cm., then, taking the maximum value of $X \sim 20\mu$, Z must be $\sim 8\mu$. The shape broadening due to Z is negligible, and therefore all the broadening $d\varphi_3$ must be due to distortion.

In conclusion, it appears that all the particles are distorted, but some may be in the form of thin lamellae, so that the broadening in some directions may be due to small particle size. In any case, however, the order of magnitude of the distortion is given by either $d\varphi_2$ or $d\varphi_3$ or both.

For the material examined four months after rolling, the conclusions are similar, but the magnitude of the distortion is smaller. For the specimen examined a year after rolling, the broadenings averaged around the ring are due partly to some small remaining distortion in the metal; but some of the individual spots may be due entirely to non-equiaxed particles, e.g. lamellae of thickness $\sim 10^{-5}$ cm.

4. Distortion of particles

If the particles are bent elastically, the arc of bending is given approximately by $d\varphi_2, d\varphi_3$. For the material examined immediately after rolling, this arc is $\sim 1{\cdot}7 \times 10^{-3}$ radians; the corresponding range of strains $\delta d/d \sim 1{\cdot}7 \times 10^{-3}$. This is equivalent to a range of stresses of $\sim 12 \times 18^8$ dyne cm.$^{-2}$, which is of the same order as the measured yield stress of the bulk material, $7{\cdot}4 \times 10^8$ dyne cm.$^{-2}$. This agreement is expected, and it is likely that at least part of the distortion is due to elastic bending of the particles. The results show that the bending must decrease with time after rolling; a year after rolling the angle is $\sim 4 \times 10^{-4}$ radians. It should be noted here that the strains derived in this way are of the same order of magnitude as those deduced by previous workers from line-broadening measurements (Megaw & Stokes, 1945; Paterson, 1949; Hall, 1949). This shows that the line broadening is at least partly due to distortion of particles of diameter $\sim 2\mu$ (Hirsch & Kellar, 1952). It also indicates that the method of measurement used in the micro-beam experiments is essentially correct.

If the particles are distorted plastically, the number of possible edge dislocations in the particles is limited. This point will be discussed in §§ 6 and 7.

II. DISCUSSION

5. Density of dislocations in boundaries

The boundary between two particles may be considered as a row (or wall) of edge dislocations (Burgers, 1939). If s is the unit of slip, h the spacing of dislocations in the wall and α the angle between neighbouring particles, then $\alpha = s/h$. For a specimen of heavily cold-worked spectroscopically pure aluminium α was found to be $\sim 2°$ (Hirsch & Kellar, 1952). Taking $s = 2{\cdot}3 \times 10^{-8}$ cm. (the 110 spacing in aluminium), h is found to be $\sim 0{\cdot}5 \times 10^{-6}$ cm. If t is the particle size, the number of dislocations per boundary is t/h; since, on the average, there is one boundary wall of dislocations per side of particle, the density of dislocations in boundaries is $(t/h) \,.\, (1/t^2)$ or $1/ht$; t was found to be 2×10^{-4} cm. (Hirsch & Kellar, 1952), and therefore the density of dislocations in the boundaries is $\sim 10^{10}$ dislocations/cm.^{2}. This value does not change appreciably with time after rolling.

6. Density of excess dislocations inside particles

A similar argument may be applied to determine the maximum number of excess dislocations inside the particles. If h is the average spacing of the excess dislocations, the total arc of bending is $\sim (s/h) \,.\, (t/h)$; this must be less than the observed $d\varphi_2, d\varphi_3$. Thus for the heavily rolled material examined immediately after rolling $st/h^2 < \sim 1{\cdot}7 \times 10^{-3}$, i.e. $h > 5 \times 10^{-5}$ cm. This corresponds to $t^2/h^2 < 16$ dislocations per side of particle, or to a density of $< 4 \times 10^8$ dislocations/cm.^{2}. A year after rolling, $h > 10^{-4}$ cm. and the density is $< 10^8$ dislocations/cm.^{2}. Both these figures are small compared with the density of dislocations in the boundaries between particles. It appears, therefore, that even after cold working the maximum possible number of excess dislocations left inside the particle is very small, and after recovery the particles contain hardly any excess dislocations.

7. Total density of dislocations inside particles

The value of the total density of dislocations inside the particles depends on their distribution. A lower limit of the total density is given by the estimate of the excess dislocations of one sign, corresponding to a simple plastic bending of the particle. If the dislocations are arranged to form subsidiary mosaics (of length h), the upper limit may be estimated by assuming these to be randomly orientated about a mean position. Then the angle between neighbouring blocks ($> s/h$) must be $< d\varphi_3$. It follows that the densities of dislocations inside the particles must be $< 6 \times 10^9$, and $< 3 \times 10^8$ dislocations/cm.^{2} for the material examined immediately after rolling and after recovery, respectively. On this assumption of subsidiary block structure it appears that the total density of dislocations inside the particles is less than that of the boundary dislocations. If however the dislocations are arranged on a regular lattice (e.g. Taylor, 1934*a*, *b*) it is at present not possible to determine its parameters from the values of $d\varphi_2, d\varphi_3$. Such a distribution however seems physically unlikely.

8. Theories of strength of metals

The results show that most of the excess dislocations of one sign, and probably most of the dislocations of either sign, segregate into boundary walls. This is in agreement with Bragg's model of a cold-worked metal (Bragg, 1948). Further, on Bragg's theory the yield stress T must satisfy the inequality

$$T \geq \frac{\mu s}{t}\left(1{\cdot}46 \log_{10}\frac{t}{s}+0{\cdot}6\right),$$

where μ = modulus of rigidity. Substituting the experimentally determined value of t, $T \geq 1{\cdot}65 \times 10^8$ dyne cm.$^{-2}$. The experimental value of T is much greater than this value (see § 4), and therefore this condition is also satisfied.

The density of dislocations in the boundaries is found to be $\sim 10^{10}$ dislocations/cm.^{2}, and it is probable that the density of dislocations inside the particles is smaller. Taylor's theory (Taylor, 1934*a*, *b*) requires a density of 10^{12} dislocations/cm.^{2} inside the particles. This could be compatible with the experimental results only if the distribution of dislocations inside the particles were of the physically unlikely regular lattice type.

9. Summary of experimental results

After cold working a specimen of spectroscopically pure aluminium the material which constituted the original grain covers a large range of orientations, of the order of several degrees. The grain consists of small particles slightly misorientated relative to one another. The size of the particles decreases with deformation, reaching a lower limit of $\sim 2\mu$ after $\sim 10\%$ reduction. For a heavily rolled specimen ($\sim 60\%$ reduction) the angle between particles is $\sim 2°$. The particles are distorted, and probably elastically bent over a few minutes of arc. For the heavily rolled material the range of stresses inside the distorted particles is of the order of the yield stress of the bulk material. The heavily worked metal contains some heavily distorted material between particles (see Hirsch, 1952).

On recovery the particle size decreases slightly, and the distortion becomes considerably smaller. The heavily distorted material disappears completely.

Impurities lower the limiting particle size and the angles between particles. The total range of orientations in the original grain is of the same order as for the pure material.

10. Physical interpretation

During plastic flow the stresses around each grain will be non-uniform owing to the constraints imposed by neighbouring grains. Such non-uniform stresses will cause plastic bending (e.g. Barrett, 1943, chap. 15). It is thought that the grain recovers by polygonization (Cahn, 1949). Dislocations of the same sign segregate into walls, thus decreasing the elastic strain energy due to dislocations. The dislocation walls form the boundaries between the particles. This process must take place either during cold work or immediately after it. The recovery effect found in the experiments is considered to be due to a slow continuation of this process. The decrease in the distortion of the particles may be due partly to a migration to the walls of any of the dislocations left inside the particle. The small decrease of particle size is probably due to polygonization of the remaining very heavily distorted material; this process creates new particles and at the same time reduces distortion.

The effect of impurities in lowering the mean particle size and angles between particles can be understood if the dislocations do not tend to segregate so easily into boundaries. This may be due to an atmosphere of impurity atoms around dislocations, which decreases their strain energy, but at the same time hinders their movements (Cottrell, 1949).

Finally it should be noted here that the experiments show that polygonization occurs in polycrystalline aluminium at room temperature; this effect, however, would not have been apparent in experiments employing techniques of smaller resolution. It follows that great care must be taken in the interpretation of experiments in which plastic curvatures are investigated. The apparent stability of these curvatures may be due to polygonization on too fine a scale to be recorded in the experiments.

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