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The Compression and Rolling Textures of AgCl and AgBr.

BY J. N. KING AND H. WILMAN

Applied Physics & Chemistry of Surfaces Laboratory, Chemical Engineering Department, Imperial College, London, S. W. 7, England

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X-ray and electron-diffraction methods show that the rolling texture of AgCl and AgBr is {110}⟨1$\bar{1}$2⟩ with, however, generally very prominent twinning on those {111} planes which are least inclined to the rolling plane and the rolling direction. In some cases the ⟨110⟩ orientation axis was tilted away from the specimen normal by up to ~ 30°.

When compression is between flat surfaces permitting extensive lateral flow, as in previous studies of the compression of metals, the compression texture is ⟨110⟩ with additional {111} twinning, as for the rolling texture. A different texture, ⟨100⟩ (with some oblique ⟨110⟩ texture also present), is developed when the initial granular material is compacted in a die at 32 tons/in.², so that lateral flow is minimized.

1. Introduction

Our recent electron-diffraction results have shown that extensive surface re-orientation is caused by unidirectional abrasion on metals (Scott & Wilman, 1958; Harker, 1958; Goddard, 1960; Wilman, 1957, 1960; Goddard & Wilman, 1962), graphite (Porgess & Wilman, 1960; King, 1960), AgCl and AgBr (King, 1960; King & Wilman, 1961) and on alkali halides (Dobson & Wilman, 1962; Wilman, 1960). In all these cases except Zn and Cd (cf. Avient 1961) the common axis of orientation is tilted backward away from the specimen normal towards the direction from which the abrasive particles came.

In order to elucidate the nature of this re-orientation on the AgCl and AgBr, which are examples of particularly plastically deformable non-metals, the compression and rolling textures of these materials were determined by X-ray and electron-diffraction methods, as described below. These textures do not appear to have been determined and reported previously, nor indeed any similar data for non-metals generally.

These are particularly interesting examples of deformation textures, since we find that the nature of the compression texture depends on the conditions of compression; i.e. it is ⟨100⟩ with some oblique ⟨110⟩, when the granular material is compressed in a die, but is ⟨110⟩ with additional {111} twinning when extensive lateral flow is permitted by compression between parallel plates. Furthermore, in the rolled material pronounced {111} twinning also occurs, modifying the main {110} ⟨1$\bar{1}$2⟩ texture, but the twinning is only on the {111} planes that are least steeply inclined to the rolling plane; and the ⟨110⟩ axis of the main texture is often inclined away from the rolling plane.

These results lead to the conclusion that the 'abrasion texture' is of the same type as, and closely related to, both the rolling texture and the compression texture that is observed when extensive lateral flow is permitted.

2. Experimental

The AgCl and AgBr were obtained from Messrs. Hopkin & Williams Ltd., and the purity was stated to be 99·8%. The granular material (0·1 to 2 mm. diam. granules) was compressed at 32 tons/in.², in a die of stainless steel of internal diameter 0·5 in. The specimens obtained were 0·2 to 0·5 cm. thick.

Specimens about 1 cm. diam. and 0·3 cm. height were also prepared by melting and resolidifying the halide on a glass slide, and were used as starting material for rolling, and also for compression between oil-lubricated polished steel flats in a vice. In these cases the specimens were reduced to <20% of the initial thickness. The rolling was done in about 20 stages in the same direction, without reversal, and without lubrication between the highly polished steel rollers, which were 2½ in. in diameter.

X-ray diffraction examination of the flat specimen surfaces was made using Mo $K\alpha$ radiation (with Zr filter) at ~ 10° grazing incidence, with photographic recording on a flat film 4 cm. from the specimen. Patterns were also obtained at normal transmission from a thin rolled sheet of AgCl. Electron-diffraction patterns at grazing incidence were recorded in a Finch camera at 50–70 kV. and 47 cm. camera length.

To investigate the structure below the compressed surface, the specimens were etched in a 0·2% aqueous KCN solution, and examined by X-ray and electron diffraction at grazing incidence on the exposed surface.

3. Results

3·1. *The orientation of rolled* AgCl

Five different specimens were rolled and examined by X-ray diffraction with the beam normal to the rolling direction. The direction of motion of the strip during rolling was from left to right in Figs. 1 to 4. Fig. 1 from a specimen rolled to 60% reduction, shows the basic orientation to be such that a {110} plane is parallel to the rolled surface or nearly so. This plane gives rise, by Bragg reflection of the primary beam, to the 220 arc centred on the plane of incidence. The other strong arcs in the pattern lie on layer lines (horizontal in Fig. 1) corresponding to a range of lattice rotation azimuthally round this {110} plane normal, i.e. round the common ⟨110⟩ axis of orientation of the crystal grains of the rolled sheet, as may be seen by comparing Fig. 1 with Fig. 10 (but neglecting the triangular symbols there). Some less strong arcs are also present which correspond to twinning of the above main orientation on {111} planes (cf. Fig. 1 and the triangular symbols in Fig. 10). Fig. 2, from a specimen rolled to 90% reduction, shows the same basic {110} orientation still more strongly developed, and with very strong {111} twinning.

The positions of the short diffraction arcs in Fig. 2 are displaced appreciably round the ring positions as compared with those of the corresponding reciprocal-lattice points shown in Fig. 10; and the horizontal layer lines become slightly curved instead of being straight. This is of course due to the relatively large Bragg angle at which reflection of the primary beam occurs, together with the fact that the beam is incident on the specimen at about 10° grazing angle. For example, the radii to the two 111 diffractions in Fig. 2 enclose an angle of approximately 65° (as compared with 70° 32′ between the corresponding points in Fig. 10) and this agrees with the angle we estimate for this diffraction in the present case. These 111 diffractions would only occur at an azimuth $\sim 35°$ from the main lattice orientation having $\langle 1\bar{1}2\rangle$ along the rolling direction (see below).

The 200 diffraction arcs lying above these 111 arcs (and the 400 arcs also) are clearly due to {111} twinning and correspond in their positions and their relatively strong intensity, to a cube face of the twins concerned being very close to the position required for Bragg reflection, already at an azimuthal orientation of the initial lattice near the main $\{110\}\langle 1\bar{1}2\rangle$ orientation of the rolled strip, especially in view of the observed $\pm 10-15°$ spread of orientation round the rolling direction (see below relative to the transmission pattern, Fig. 5). The combination of this rotation round the rolling direction, and the $\sim 35°$ azimuthal rotation round the normal of the {110} rolling plane, is also seen to be responsible for the drawing out of these 200 diffractions towards the plane of incidence, and their coalescence there. The 111 arcs, however, are not elongated in this way because the corresponding {111} planes are much more inclined (at 35° 16′) to the rolling plane, and the rotational spread round the rolling direction is small compared with this angle. Similar considerations account for the relatively strong 311 arc which is centred on the plane of incidence in Fig. 2, and is also due to the {111} twins.

Some photographs showed the above type of orientation except that the ⟨110⟩ axis was inclined away from the specimen normal towards the leading edge of the rolled strip. For example, in Fig. 3 this tilt was about 10°, and in Fig. 4 it was about 30°, although otherwise these patterns are closely analogous to Fig. 2.

In order to determine the preferred lattice direction aligned along the rolling direction, transmission photographs such as Fig. 5 were prepared with the beam normal to the rolled strip. These showed extremely strong azimuthal orientation of the crystal grains, with a {110} plane parallel to the rolled strip and a $\langle 1\bar{1}2\rangle$ lattice row along the rolling direction. The two equivalent lattice orientations of this type are symmetrically disposed and occur with equal probability. The strongest spots in Fig. 5 are indeed seen to correspond to reciprocal-lattice points forming two centred-$\surd 2$-rectangle arrays, in the {110}* planes (parallel to the plane of the rolled strip) of these two orientations, as is shown in Fig. 11. The simultaneous presence of all these diffractions indicates a range of rotation of the order of $\pm 10°$ about the rolling direction. The additional less strong diffractions in Fig. 5 lie on intermediate horizontal layer lines and indicate a similar range of about ± 10–$15°$ of lattice rotation about the rolling direction, away from the mean. They correspond to reciprocal-lattice points lying on the intermediate planes of $\{1\bar{1}2\}$* type which are normal to the rolling direction, and these are indicated in Fig. 11 in the positions where they would cross the above {110}* plane.

The reflection patterns (Figs. 1 and 2) show an azimuthal spread of more than $\pm 35°$ away from this mean orientation, and this is evidently because the beam impinges (at $\sim 10°$ grazing incidence) on a much larger area of specimen than in the transmission case, and because the direction of plastic flow varies in different regions appreciably. Although twinning of the main $\{110\}\langle 1\bar{1}2\rangle$ orientation on {111} planes normal to the {110} rolling plane would introduce other azimuthal orientations, it is clear that this would not affect the above conclusion of about $\pm 35°$ azimuthal spread being present. If such twinning on these particular {111} planes were present, however, this would lead to 200 diffractions at the same level as the 111 diffractions in Figs. 1 and 2; but absence of 200 arcs at these positions indicates little such twinning. The {111} twinning is thus mainly on the {111} planes which are inclined to the rolling plane and rolling direction at only a relatively small angle, i.e. 35° 16′ to the rolling plane.

3·2. *The orientation of* AgCl *and* AgBr *compressed between lubricated flats*

Fig. 6 is typical of the X-ray diffraction patterns obtained from the compressed surfaces. This is clearly

Fig. 1.

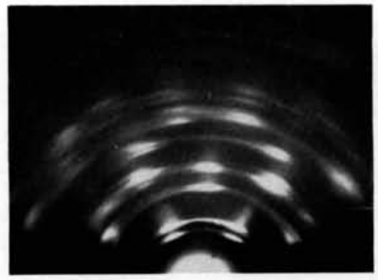

Fig. 2.

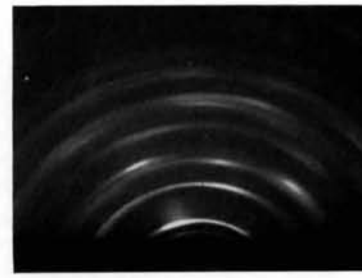

Fig. 3.

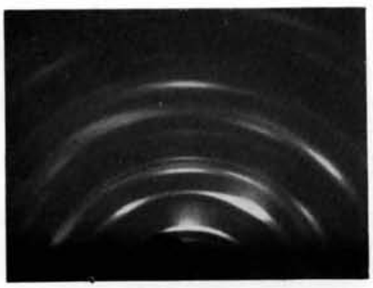

Fig. 4.

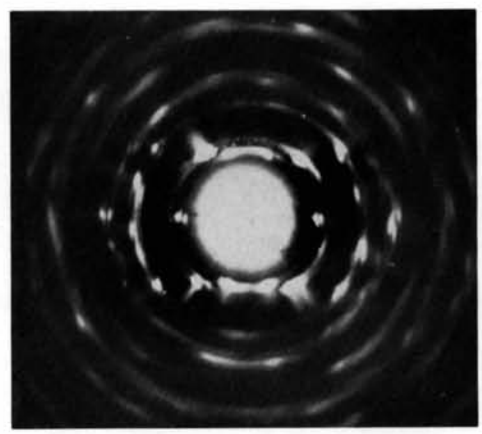

Fig. 5.

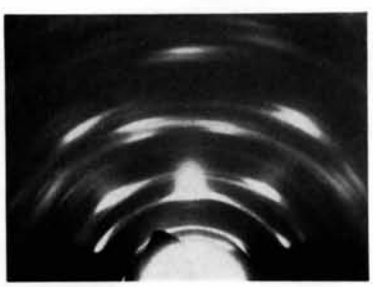

Fig. 6.

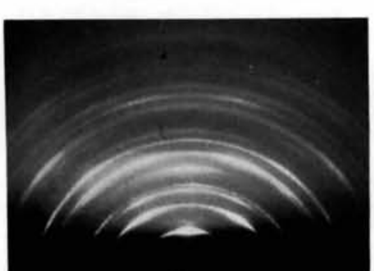

Fig. 7.

Fig. 8.

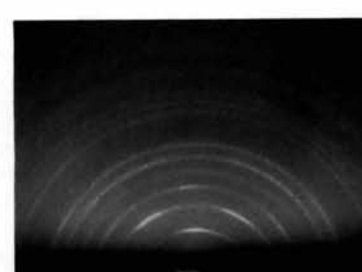

Fig. 9.

Fig. 1. X-ray reflection; AgCl rolled (*L* to *R*), 60% reduction; ⟨110⟩ orientation. — Fig. 2. As Fig. 1 but another specimen; 90% reduction; stronger {111} twinning. — Fig. 3. As Fig. 2 but 80% reduction; slightly inclined (to RHS) ⟨110⟩ orientation. — Fig. 4. As Fig. 3; ∼ 30°-inclined ⟨110⟩ orientation (axis in 1 o'clock direction). — Fig. 5. X-ray transmission; rolled AgCl, R.D. vertical. — Fig. 6. X-ray reflection; AgCl compressed between steel plates. — Fig. 7. Electron diffraction; AgCl compacted in a die; etched ∼ 1 μ. — Fig. 8. As Fig. 7 but X-ray reflection. — Fig. 9. As Fig. 7, but AgBr.

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similar to Fig. 1, showing the orientation to be of the same type, with a ⟨110⟩ direction parallel to the compression axis, i.e. normal to the surface. This ⟨110⟩ compression texture was observed after compression of specimens initially resolidified from the melt, and of specimens initially prepared by compacting the granular material in a die.

As in Figs. 1 and 2, Fig. 6 contains arcs which show presence of extensive {111} twinning of the main ⟨110⟩ orientation. The presence of considerable intensity of the 200 arc in Fig. 6, at the same horizontal level as the 111 arcs, is noticeable as compared with absence of such 200 diffractions in Figs. 1 and 2. The occurrence of these and other arcs on the layer lines shows complete azimuthal randomness of the lattice orientation, which is indeed to be expected in the present case. The radial outward flow of material past the compressing surfaces during compression is evidently closely similar in type to that occurring in the rolling direction when the material is rolled. Compression under these conditions allowing extensive lateral flow is thus effectively equivalent to rolling simultaneously in all directions radially.

3·3. *The orientation of granular* AgCl *and* AgBr *compressed in a die*

Figs. 7 and 9 from the AgCl and AgBr, respectively, are typical of the electron-diffraction patterns obtained from the surfaces exposed by etching the compressed specimens to about 1 μ depth. These are closely similar in type to the X-ray patterns such as Fig. 8, which was from the etched AgCl surface; and the X-ray patterns from the unetched specimens were similar to Fig. 8 except that the arcs were slightly weaker.

Comparison of Figs. 7 and 9 with Fig. 12 shows that most of the strong arcs correspond to a well-developed one-degree orientation with a ⟨100⟩ axis normal to the surface, i.e. along the compression axis. This ⟨100⟩ texture was also observed from alkali halides of NaCl structure type, compacted similarly (Dobson & Wilman, 1962).

In Figs. 7 and 9, and all the similar photographs obtained, there is however a 222 diffraction arc slightly displaced away from the plane of incidence, and strong 220 arcs at a level much higher than the 220 arcs associated with the ⟨100⟩ orientation, cf. Fig. 12. At first sight these diffractions suggest presence of a moderate proportion of the halide oriented with a ⟨111⟩ axis along this slightly oblique radius of the 222 diffraction arc. This would lead to an angle of $70\frac{1}{2}°$ between the radii to the centres of these 220 arcs, however, whereas in all the patterns (e.g. Figs. 7 and 9) this angle is estimated to be nearer to 60° than 70°. This observation, together with the noticeable displacement of the 222 arc away from the

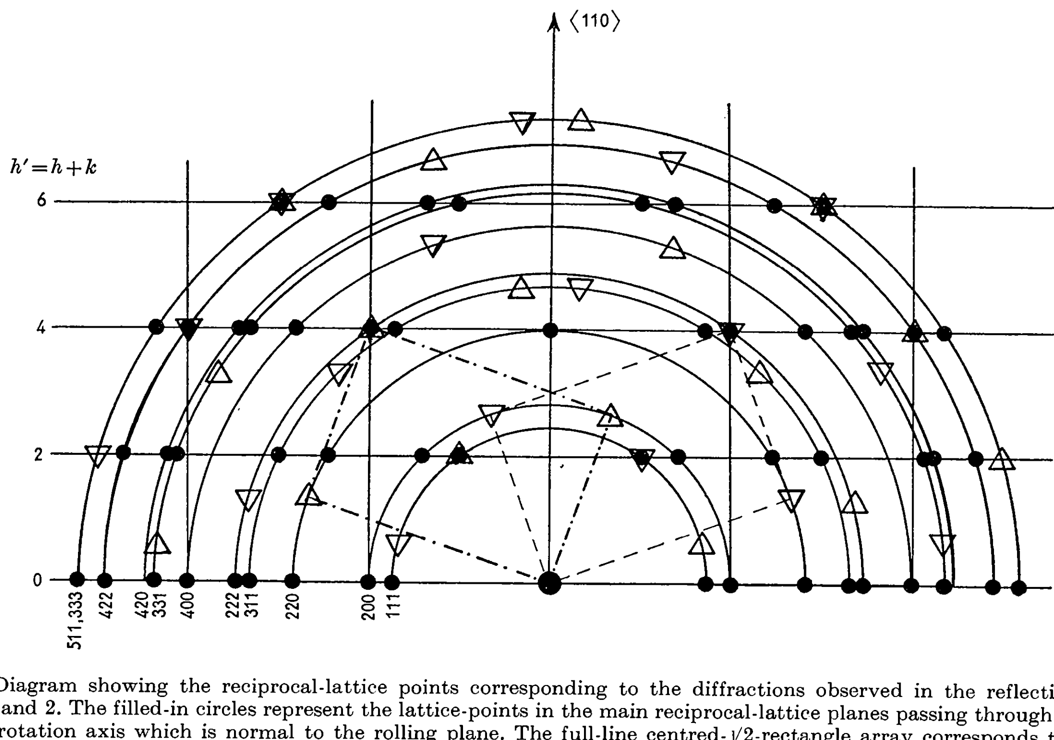

Fig. 10. Diagram showing the reciprocal-lattice points corresponding to the diffractions observed in the reflection patterns Figs. 1 and 2. The filled-in circles represent the lattice-points in the main reciprocal-lattice planes passing through the vertical ⟨110⟩ rotation axis which is normal to the rolling plane. The full-line centred-$\sqrt{2}$-rectangle array corresponds to a vertical {110}* type of plane. The triangles indicate the centred-$\sqrt{2}$-rectangular arrangement of the lattice points (shown by broken lines) in the corresponding {110}* planes of two octahedral twins relative to the initial lattice; these rectangles have one diagonal coinciding with a diagonal of the full-line rectangles.

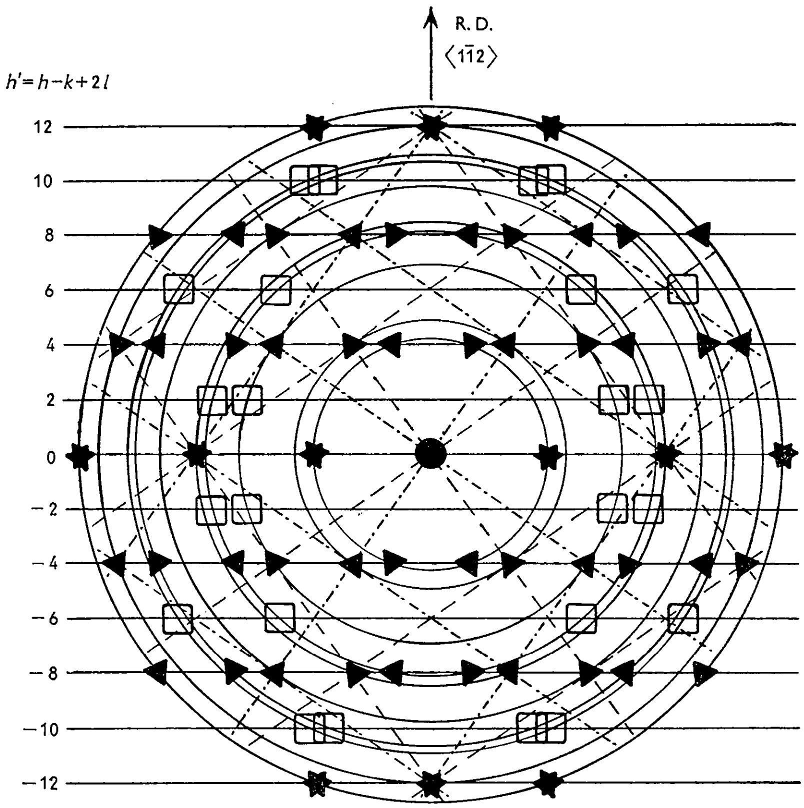

Fig. 11. Reciprocal lattice points corresponding to the diffractions in the transmission pattern of Fig. 5. The triangles correspond to the two centred-$\sqrt{2}$-rectangle arrangements (shown by the broken lines) of the points in the {110}* reciprocal-lattice planes parallel to the rolling plane, in the two symmetrically disposed {110} ⟨1$\bar{1}$2⟩ lattice orientations. The positions denoted by □ show where other reciprocal-lattice points cross this plane as a result of rotation of these lattices by ± 10–15° round the rolling direction, which is vertical. Laue indices for ring positions as in Fig. 10.

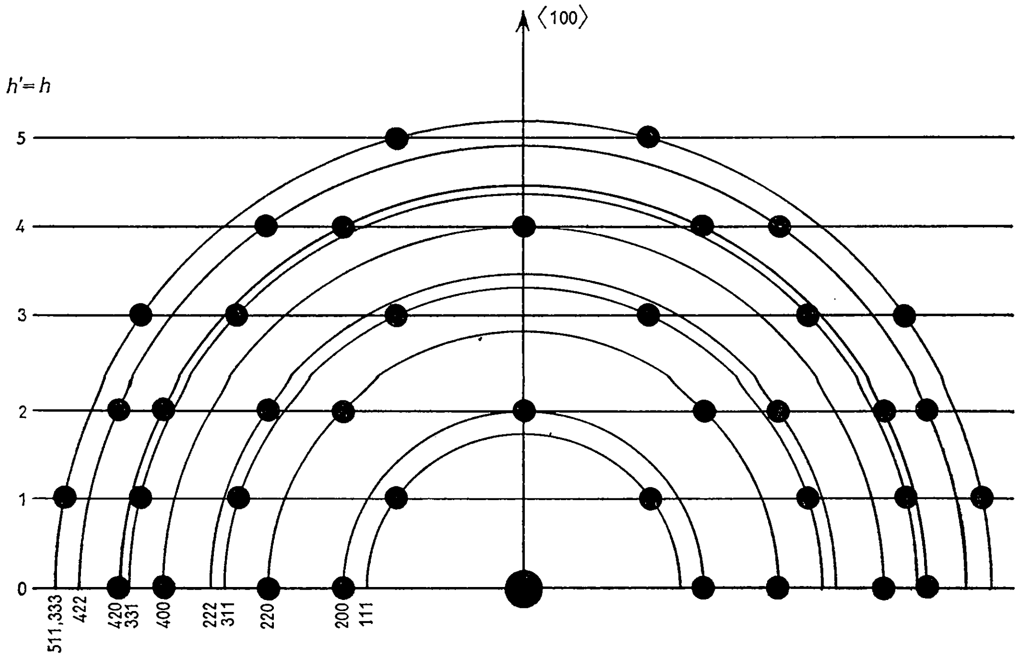

Fig. 12. Theoretical diffraction positions in electron diffraction reflection pattern from a one-degree ⟨100⟩-oriented f.c. cubic structure.

plane of incidence, and the strong similarity of the positions of these arcs to those in Fig. 4, indicate that the best interpretation is that they are due to a ⟨110⟩ orientation with the ⟨110⟩ axis inclined away from the specimen normal by $\sim 30°$ as in Fig. 4. Such an orientation seems quite likely to be developed in part, in the present case, due to considerable lateral flow of the granules past the compressing end of the die, into the neighbouring voids between the granules. To test whether this auxiliary texture was also developed when finer particles were used in the die, a specimen was prepared from AgBr of mean particle diameter 0·3 mm., and a practically identical pattern was obtained.

4. Discussion

4·1. *The similarity of the rolling texture to that for f.c. cubic metals*

The above results show that the AgCl and AgBr develop strong preferred orientations when rolled or compressed, analogous to the well-known deformation textures observed in metals.

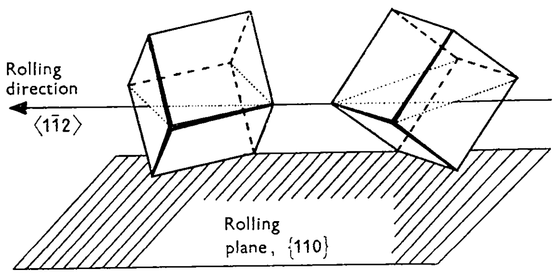

Fig. 13. Diagram showing the main orientation of the cubic lattice in the AgCl rolled strip, as shown by the transmission pattern, Fig. 5; there is a spread of orientation away from these main orientations, consisting of a rotation of up to about $\pm 15°$ about the rolling direction, and there is additionally {111} twinning. The reflection patterns, Figs. 1 and 2, are from larger regions of the strip surface, and indicate an azimuthal spread of more than $\pm 35°$ from the above main orientation, about the normal to the rolling plane, which is a ⟨110⟩ direction.

The {110} rolling texture is often very strongly defined in its azimuthal orientation, and this is clearly determined as ⟨1$\bar{1}$2⟩ aligned along the rolling direction (see Fig. 13). This texture is the same as the known rolling texture of f.c. cubic metals (Barrett, 1953; Hu & Cline, 1961). In these metals the normal slip system is of {111} ⟨1$\bar{1}$0⟩ type and in AgCl slip is along ⟨1$\bar{1}$0⟩ but of 'pencil glide' type (Nye, 1949). The observed twinning on the {111} planes which are not steeply inclined to the rolling plane seems to correspond to the greater resolved shear component of the stress on these planes than on the other two octahedral planes.

4·2. *The variation in tilt of the ⟨110⟩ axis away from the rolling-plane normal*

The tilt of the ⟨110⟩ orientation axis is interesting in that its magnitude was $\sim 30°$ in one case (Fig. 4), which is larger than the tilt ($\sim 20°$) observed in AgCl and AgBr abraded unidirectionally on 4/0 emery paper. Presumably it must be similarly due to tangential frictional forces on the halide, in the present case at its interface with the roller.

In abrasion of AgCl and AgBr (King & Wilman, 1961) the {110} orientation developed has a mean preferred azimuthal orientation which is ⟨1$\bar{1}$2⟩ (or a direction close to this) along the abrasion direction; thus the 'abrasion texture' is closely similar to the rolling texture and indicates a similar nature and direction of the plastic flow. The ⟨110⟩ axis of the abrasion texture is also in general inclined away from the specimen normal, back towards the direction from which the abrasive particles came. This tilt can be correlated approximately with the direction of the resultant compressive stress (normal load + tangential force overcoming friction) exerted by the indenting abrasive particles (King & Wilman, 1961).

4·3. *The relation of the rolling texture to the compression texture*

The correspondence in type between the {110} rolling texture and the {110} compression texture, observed when extensive lateral flow is permitted, is understandable, since the compression causes a radial flow of the material similar to that which would be obtained if the material were rolled simultaneously radially in all directions. Previous determinations of compression textures of metals have evidently been made using compression in this way between lubricated parallel plates; but the present results show that a different texture is in general expected when lateral flow is severely limited as in compacting materials in powder form in a die. Such compression of the AgCl and AgBr led to strong {100} orientation like that found (Dobson & Wilman, 1961) in similarly compacted alkali halides of NaCl structure type, which mainly slip on {110} along ⟨1$\bar{1}$0⟩. Similarly, these alkali halides behaved like the AgCl and AgBr in developing the same azimuthally limited ⟨110⟩ texture when abraded.

Compacted metal powders are now much used, e.g. for porous bearing materials to hold lubricating oil, and it is to be expected that, if the pressures used for compaction are high enough compared to the flow pressure of the metal, a preferred compression texture would similarly be developed. No results of this kind appear to have been reported hitherto.

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The Compression Textures of Polycrystalline Materials of Caesium Chloride Structure Type

BY P. S. DOBSON AND H. WILMAN

Applied Physics & Chemistry of Surfaces Laboratory, Chemical Engineering Department, Imperial College, London, S. W. 7, England

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Specimens prepared by compressing, in a steel die, powders of CsCl, TlCl and NH_4Cl, at up to 75 tons/in.^{2}, are investigated by electron diffraction. The compression textures under these conditions are found to be ⟨111⟩. This texture is shown to be expected on the basis of slip on the usual systems, i.e. {011}, ⟨100⟩.

1. Introduction

For comparison with our results in experiments on the surface deformation and re-orientation of non-metallic salts during unidirectional abrasion, we have studied also the textures developed by pure compression in these materials. We have recently described (Dobson & Wilman, 1961) the results for various alkali halides of rocksalt type. The compression textures found for some salts of CsCl structure type (CsCl, TlCl and NH_4Cl) are now described below.

2. Experimental

The initial materials used were in the form of powders. The purity quoted by the suppliers was:—CsCl (from B.D.H.) >99%; TlCl (B.D.H.) ?%; NH_4Cl ('Analar', Hopkin & Williams Ltd.) >99%. These materials were ground further by hand with a pestle and mortar, and the mean particle diameter was then of the order of 150μ for the CsCl and NH_4Cl, and 60μ for TlCl.

The powders were compressed in a steel die of 0·5 in. diameter, using pressures up to 75 tons/in.^{2}. On removal from the die, the specimens were seen to be well-compacted and had very smooth shining surfaces. They were about 8 mm. thick and had densities approximately 0·95, 0·98 and 0·99 of the true densities of CsCl, TlCl and NH_4Cl respectively.

The plane ends of the cylindrical specimens, normal to the compression axis, were examined at grazing incidence by electron diffraction, using a camera length of 47 cm., and electrons accelerated through 60–70 kV. Electron-diffraction patterns showing more clearly the nature of the preferred orientation were obtained from the TlCl and CsCl surfaces exposed by etching a few sec. in 10% HCl-water solution for the TlCl and 10% H_2O in propyl alcohol for the CsCl, respectively.

3. Results

Figs. 1 to 3 are typical of the results obtained, and in all cases show relatively strong orientation with a ⟨111⟩ axis parallel to the compression axis. This is seen not only from the presence of the arc on the 111 ring position centred on the plane of incidence but also because the positions of the intensified arcs which can be recognized on other ring positions agree with the diffraction positions which would be expected from the ⟨111⟩-oriented crystals, shown in Fig. 4.