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The Compression Textures of Polycrystalline Materials of Rocksalt 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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Compacted specimens prepared by compressing powders of LiF, NaF, NaCl, KCl, KBr and MgO at 75 tons/in^2 or more, are investigated by electron diffraction and X-ray diffraction. The compression textures are found to be ⟨100⟩ for all these materials except MgO, which developed a ⟨111⟩ preferred orientation. The ⟨100⟩ texture is shown to be expected on the basis of the usual slip systems, i.e. {110}, ⟨110⟩; or {100}, ⟨110⟩; but the reason for the ⟨111⟩ texture of the MgO is not clear.

Introduction

The re-orientation textures produced in metals by plastic flow in compression have been extensively studied long ago (e.g. see Schmid & Boas, 1935; Barrett, 1953). It appears, however, that no similar data have as yet been obtained for the compression textures of inorganic salts, even those of simple structure such as the rocksalt type. This is surprising, because translational slip in such crystals has long been studied and the slip systems defined; and moreover the movement of dislocations in such crystals has recently been much studied. Although these materials are brittle and readily cleaved, it is well known that they undergo easy plastic flow in compression when suitably stressed.

The results described below now show clearly the nature of the compression textures developed in LiF, NaF, NaCl, KCl, KBr, and MgO. These results were obtained particularly for comparison with our observations on the surface re-orientation of these materials by unidirectional abrasion (Dobson & Wilman, 1961), in connection with friction and wear studies.

Experimental

The initial materials used were in the form of powders. The purity was as follows: LiF, 98%; NaF, 99·0%; NaCl, 99·5%, KCl, 99·5%; KBr, 99·0%; MgO, 97%. These materials were ground further by hand with a pestle and mortar, and the mean particle diameter after grinding was of the order of 140 μ for NaCl, KCl, and KBr, and less than 5 μ for LiF, NaF and MgO. The powders were compressed in a steel die of 0·5 in. diameter, using mainly pressures of 75 tons/in.^{2} (NaCl and MgO at 96 tons/in.^{2}) at the Laboratories of the Atomic Power Division, The English Electric Co., Ltd., Whetstone, nr. Leicester.

On removal from the die, the NaCl, KCl, KBr and MgO had formed well-compacted specimens about 8 mm. thick and had densities of 0·99, 0·99, 0·98, 0·60, of the single-crystal densities respectively. The LiF and NaF specimens, on removal from the die, consisted of a number of laminations about 1 mm. thick, but the outermost surfaces which had been in contact with the die faces were smooth and compact, and suitable for examination.

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. Since the electron-diffraction patterns from NaCl and KBr showed no well-defined orientation (although abrasion caused a pronounced preferred orientation), all the specimen surfaces were also investigated by X-ray diffraction, using Mo $K\alpha$ radiation incident at about 10° to the surface, and a flat photographic film distant 4 cm. from the specimen and normal to the beam. The form of the crystal grains in the NaCl specimens was examined optically with a Vickers projection microscope, after etching the surface in a stream of water, followed by immediate dehydration in acetone.

Results

Figs. 1(*a*) and 1(*b*) are representative of the results for NaCl, KCl and KBr, which all showed relatively strong orientation with a ⟨100⟩ axis parallel to the compression axis. Fig. 2 shows the roughly rectangular shape of many of the grain boundaries at the compressed surface after etching; and this is compatible with the observed ⟨100⟩ orientation.

LiF and NaF showed less strong orientation, but this was of the same ⟨100⟩ type, as the electron diffraction photographs Figs. 3 and 4 show. The X-ray diffraction photographs showed no preferred orientation, indicating that the plastic deformation was here practically confined to a very thin surface layer.

The MgO surfaces also showed a well-marked preferred orientation in the electron-diffraction photograph, Fig. 5(*a*), in this case with a ⟨111⟩ axis normal to the surface, along the axis of compression. The X-ray diffraction photograph, Fig. 5(*b*), showed only

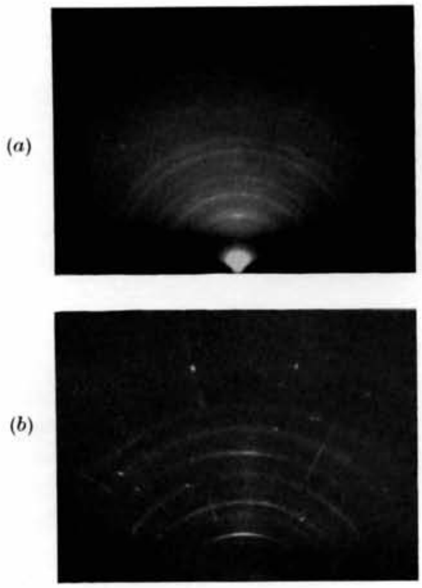

Fig. 1.

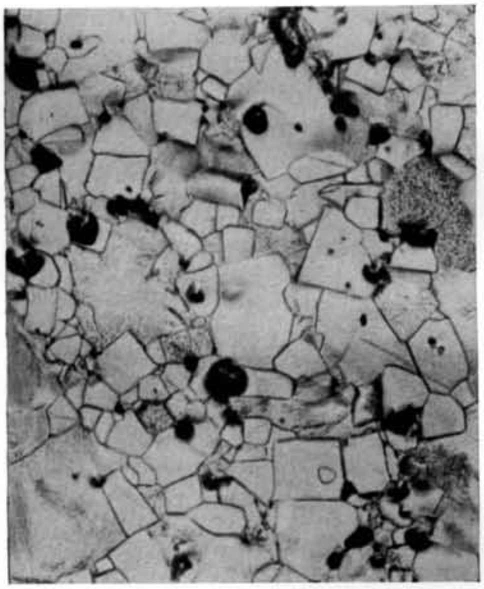

Fig. 2.

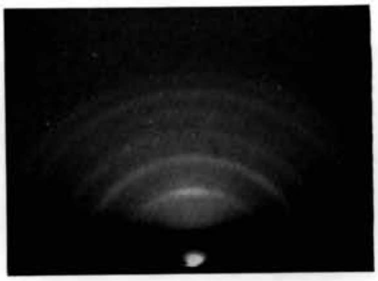

Fig. 3.

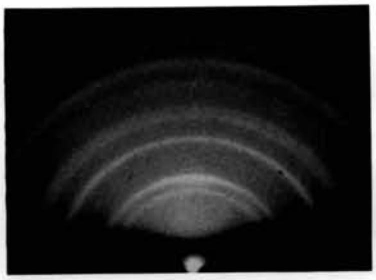

Fig. 4.

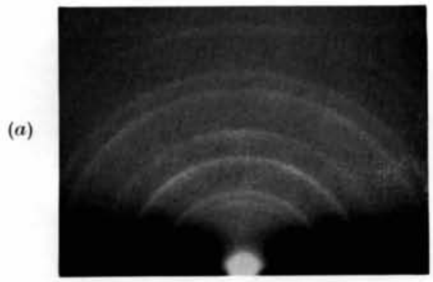

Fig. 5.

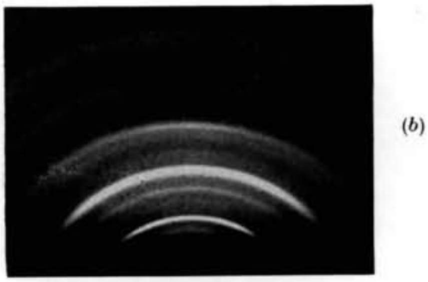

Fig. 5.

Fig. 1. KCl compressed at 75 tons/in^2; ⟨100⟩ orientation: (*a*) electron diffraction, (*b*) X-ray diffraction pattern.

Fig. 2. Photomicrograph from etched flat surface of NaCl compressed at 96 tons/in^2. × 140.

Fig. 3. NaF compressed at 75 tons/in^2; electron diffraction; ⟨100⟩ orientation.

Fig. 4. LiF compressed at 75 tons/in^2; electron diffraction; ⟨100⟩ orientation.

Fig. 5. MgO compressed at 96 tons/in^2; ⟨111⟩ orientation: (*a*) electron diffraction, (*b*) X-ray diffraction pattern.

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Table 1. *The most favourable slip systems* (*having highest resolved shear stress*) *in an* NaCl *type of crystal, as a function of stress direction, for slip on* {110} *along* ⟨110⟩

Stereographic projection of stress direction	Most favourable slip systems	No. of favourable systems
Within unit triangle	(101) [$10\bar{1}$]; ($10\bar{1}$) [101]	2
Boundary [100]/[110]	(101) [$10\bar{1}$]; ($10\bar{1}$) [101]	2
Boundary [100]/[111]	(110) [$1\bar{1}0$]; ($1\bar{1}0$) [110]; (101) [$10\bar{1}$]; ($10\bar{1}$) [101]	4
Boundary [110]/[111]	(101) [$10\bar{1}$]; ($10\bar{1}$) [101]; (011) [$01\bar{1}$]; ($01\bar{1}$) [011]	4
[110]	(101) [$10\bar{1}$]; ($10\bar{1}$) [101]; (110) [$1\bar{1}0$]; ($1\bar{1}0$) [110]	4
[111]	(101) [$10\bar{1}$]; ($10\bar{1}$) [101]; (110) [$1\bar{1}0$]; ($1\bar{1}0$) [110]; (011) [$01\bar{1}$]; ($01\bar{1}$) [011]	6
[100]	(101) [$10\bar{1}$]; ($10\bar{1}$) [101]; (011) [$01\bar{1}$]; ($01\bar{1}$) [011]	4

a slight suggestion of an intensified arc on the 222 ring position, centred on the plane of incidence, again indicating that the plastic deformation had been mainly confined to the outermost surface layer.

Discussion

Previous observations have shown that in suitable conditions of compression, crystals of rocksalt type undergo plastic deformation easily by slip on {110} planes along a ⟨110⟩ type of direction, whereas in tension these materials fail by brittle fracture or cleavage. The above results show that well-developed compression textures are developed in the softer materials, i.e. NaCl, KCl and KBr. For the other materials which were much harder and also had a smaller particle size, the compression led to less coherent specimens, but a well-defined compression texture was nevertheless developed in the surface region.

The observed compression textures were the same, ⟨100⟩, for LiF, NaF, NaCl, KCl and KBr; but for MgO it was ⟨111⟩, with no trace of ⟨100⟩.

The compression texture expected theoretically is indeed ⟨100⟩, if slip is on a {110} plane along a ⟨110⟩ slip direction, as is found predominantly in NaCl type of crystal structures. Of the six possible slip systems, those which are most favourable to operate in any one crystal when the polycrystalline specimen is subjected to unidirectional compressive stress, are the ones for which the resolved shear stress along the slip plane, in the slip direction, is highest. This resolved shear stress is proportional to $\cos\lambda \cos\psi$, where λ is the angle which the direction of the compressive stress on the crystal under consideration, makes with the slip-plane normal; and ψ is the angle between this compressive stress direction and the slip direction. If the operative direction of the compressive stress on the crystal is represented, as usual, by the corresponding point in the unit triangle in the stereographic projection, with vertices defined by the [100], [110], and [111] directions, then for the various types of stress direction the most favourable slip systems are as shown in Table 1.

The relative magnitudes of the resolved shear stress, represented by the values of $\cos\lambda \cos\psi$, are shown in Fig. 6 for the above slip systems, as a function of the stress direction, by a contour diagram such as Calnan & Clews (1950, 1951) used in considering the generation of such textures in face-centred cubic metals (slip on {111} along ⟨110⟩) and body-centred cubic metals (slip on {110} along ⟨111⟩). It is seen from Fig. 6 that the resolved shear stress has a maximum when the compressive stress on the crystal is along a cube edge, and the directions of steepest descent of the resolved shear stress (a locus orthogonal to the contours) diverge from the [100] pole throughout the unit stereographic triangle.

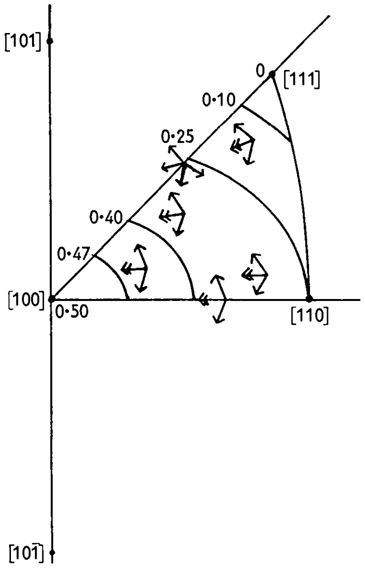

Fig. 6. Unit triangle of stereographic projection, showing contours of resolved-shear stress values for (101) and ($10\bar{1}$) slip planes, and typical directions of movement of the effective axis of compression for slip on (101) alone (arrows towards (101) pole), ($10\bar{1}$) alone (arrows towards ($10\bar{1}$) pole), and the resultant for slip on both (double arrows).

Taylor (1938) has shown that when a cubic polycrystalline aggregate is deformed, five slip systems must operate simultaneously if, as is found experimentally, no voids are to appear at the grain boundaries.

The effective resultant stress exerted on any one crystal grain by its neighbours during compression of the specimen as a whole does not in general coincide with the direction of the external applied compression. Calnan & Clews (1950) pointed out that the direction of this effective stress must tend (as the compression is increased) towards a direction such that slip can occur simultaneously on five (or more) slip systems, as is required for cohesion to be maintained. As they stated, however, the fact that lattice rotation is observed shows that some single or duplex slip must take place, because slip under a stress along such a symmetrical direction would not result in lattice rotation.

In compression, slip on a single plane causes a lattice rotation such that the slip-plane normal tends to rotate towards the axis of compression. If slip occurs on two slip planes equally extensively, the lattice rotation must presumably tend towards an orientation with the two slip planes roughly equally inclined to the compression axis, as steeply as possible. Fig. 6 indicates the general directions in which such rotations caused by slip, in this case of NaCl-type structures always double slip (except for still higher multiplicity of slip for the orientations of higher symmetry), alter the orientation of the effective compression direction on the crystal. In the case where the stereographic projection P of the compression axis falls on the [100]–[111] boundary there are then four equally favourable slip systems; thus if we assume the same amount of slip on each system, the resulting rotations do not cancel, but there is a resultant rotation, so that P moves down the boundary towards [100]. When P lies on the [110]–[111] boundary, however, there is no resultant rotation, since the rotations cancel.

It is seen clearly that for all crystal orientations the lattice rotates towards a final orientation having a cube edge along the compression axis of the specimen as a whole. This is therefore in agreement with the compression texture we have observed.

Similar considerations applied to the other form of slip system, {100}, ⟨110⟩, which has been observed for crystals of NaCl type, also indicate that this too would lead to expectation of a ⟨100⟩ compression texture. The reason for the development of a ⟨111⟩ texture in MgO is therefore not clear.

Many of the particles of NaCl were rectangular in form, presumably due to cleavage, and it is possible that in the particles which are in contact with or near to the die face, the flat {100} cleavage faces will tend to become parallel to the die face in the early stages of compression, simply because of the geometrical shape of the particles. Since this is a stable orientation, such crystals will tend to undergo little further change in orientation as the compression is increased. In the case of MgO, where there is an imperfect cleavage also on {111} (Palache, Berman & Frondel, 1944), it is perhaps possible that this process may be responsible for the observed ⟨111⟩ compression texture in the immediate surface regions, though the relatively perfect {100} type of cleavage is usually observed.

It may be noted that, also for a compressive force along [111], the resolved shear stress for all the slip systems of {110}, ⟨110⟩ type is zero, and consequently any particle originally in such an orientation cannot be deformed by slip, except presumably if kinking occurs.

The above results show additionally that when moderately hard materials, even after very heavy compression, do not yield clear evidence of a compression texture in the X-ray diffraction pattern, they may have nevertheless undergone sufficient plastic deformation in the immediate surface region to yield electron-diffraction patterns showing a well-defined preferred orientation.

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