

The Re-Orientation Caused by Unidirectional Abrasion on Materials of CsCl Structure Type

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The surface and subsurface structure of unidirectionally abraded polycrystalline TiCl, CsCl, NH₄Cl and NH₄Br has been studied by electron diffraction. For comparison with the abrasion results the rolling texture of TiCl is investigated by X-ray diffraction.

All the abraded materials developed a backwardly-tilted $\langle 110 \rangle$ orientation which was azimuthally strongly limited to an orientation with a $\langle 001 \rangle$ cube edge direction normal to the abrasion direction. Additional arcs indicate more than $\pm 35^\circ$ rotation range around the abrasion direction, away from this $\{110\} \langle 1\bar{1}0 \rangle$ main orientation, which was concluded to be similar to the rolling texture.

1. Introduction

The surface re-orientation caused by abrasion on materials of NaCl structure type has been investigated recently by Dobson & Wilman, 1961 and King & Wilman, 1961. In the experiments described below, the structure of the surface and sub-surface regions of four typical salts (CsCl, TiCl, NH₄Cl and NH₄Br) of CsCl structure type, abraded on 4/0 emery paper (of particle diameter 5 microns), have been investigated by electron diffraction.

2. Experimental

The polycrystalline specimens were formed by compressing the powders of each material at a pressure of 75 tons/in.², in a steel die 0.5 in. in diameter. The purity of the powders was stated by the suppliers to be: CsCl > 99%; TiCl (unspecified); NH₄Cl > 99%; NH₄Br > 99.5%; and the mean particle diameter after grinding was of the order of 150 μ for the CsCl, NH₄Cl and NH₄Br, and 60 μ for the TiCl.

The average density of all the compressed specimens was found to be 95–99% of the true density. The structure of the plane faces was studied by electron diffraction, and in all these materials a compression texture with a $\langle 111 \rangle$ axis perpendicular to the surface was found to have been developed (Dobson & Wilman, 1962).

The unidirectionally abraded surfaces were examined at grazing incidence by electron diffraction, using a camera length of 47 cm., and electrons accelerated through 60–70 kV. The TiCl and NH₄Cl specimens were etched so that the structure at various depths below the surface could be examined. The etchants used were a mixture of water and propyl alcohol, and the concentrations of water, and corresponding etch rates, were: TiCl, 100%, 200 Å/sec.; NH₄Cl, 5%, 250 Å/sec. After being etched, the specimens were dehydrated immediately by washing in propyl alcohol, and were then dried in a desiccator

and transferred to the electron-diffraction camera and examined in vacuo.

For comparison with the abrasion texture, the compacted TiCl specimens were rolled between two stainless-steel rollers of 2.5 in. diameter. The initial specimens were about 3 mm. thick and 1.27 cm. diameter, and after rolling the thin strips formed were about 0.2 mm. thick. The rolled surfaces were examined by diffraction of Mo K α X-rays at about 10° grazing incidence, the pattern being recorded on a flat film normal to the beam and 4 cm. from the specimen.

3. Results

1. The electron-diffraction examination of the abraded surfaces

The TiCl, CsCl, NH₄Cl and NH₄Br surfaces were unidirectionally abraded on dry 4/0 emery paper at 200 g. load. The results described below were obtained on two different specimens of each material, and were repeated several times on each specimen. All the materials showed a closely similar form of structure, which is represented by Fig. 1, from the TiCl with the beam normal to the abrasion direction. This shows particularly strong orientation, and the strongest arcs in the pattern are seen to be in a square array, the strongest 110 arc lying on a radius which is tilted back by about 25° from the plane of incidence towards the direction from which the abrasive particles came. This pattern evidently corresponds to a backwardly-tilted $\{110\}$ orientation which is strongly limited azimuthally to an orientation such that a $\langle 001 \rangle$ cube edge is normal to the abrasion direction, or nearly so.

The remaining arcs in the pattern seem at first sight to be due to a one-degree $\{111\}$ orientation with an azimuthal spread of >30°, and therefore effectively azimuthally random. A striking feature common to the patterns from all the abraded materials (cf. Fig. 1) is that the 111 arc is practically vertically above the strong 110 arc, and Fig. 3 shows the theoretical posi-

tions of the diffractions from such a one-degree $\langle 111 \rangle$ orientation in this relationship to the $\langle 110 \rangle$ orientation. Although there is a close similarity of practically all the diffraction positions with those observed in the pattern (Fig. 1), there is a noticeable asymmetry of the positions of pairs of arcs such as the strong 211 diffractions near the plane of incidence (cf. Fig. 1 and Fig. 3) relative to the radius on which lie the centres of the 111 and 222 arcs. There is furthermore a 221 diffraction arc between the 220 arc and the 222 arc vertically above it, and this is not expected from the postulated $\langle 111 \rangle$ orientation. These discrepancies show clearly that the arcs additional to the square $hk0$ array cannot be attributed to a one-degree $\langle 111 \rangle$ orientation.

Since all these additional arcs lie approximately vertically above arcs of the $hk0$ square pattern, they can all be accounted for as resulting from a disorientation from this main $\{110\}$ orientation, consisting of a range of rotation about the abrasion direction. The above-mentioned 220, 221, 222 vertical series for example corresponds to a vertical layer line which is perpendicular to the horizontal abrasion direction; and similarly the first-order corresponding diffractions, 110 and 111 above it, indicate the same range of rotation of $> \pm 35^\circ 16'$. The arc on the 110 ring position, above the short and strong 100 arc at the '2 o'clock' position, indicates that the rotation extends even beyond $\pm 55^\circ 44'$. Diffraction patterns were recorded with the beam at other azimuths relative to the abrasion direction, and these agreed with the above interpretation. With the beam along the abrasion direction, the pattern consisted of continuous rings as is indeed usually observed from abraded surfaces at this azimuth.

The TiCl was etched in stages down to a depth of 6,500 Å below the abraded surface, and the patterns from the surfaces so exposed were still closely similar to the patterns from the initial surface (cf. Fig. 1). The tilt δ of the main $\langle 110 \rangle$ axis was observed to be practically the same as that in the surface regions. μ of TiCl on 4/0 emery paper was 0.54, thus δ (25°) was nearly equal to $\tan^{-1} \mu$ (which was 28°).

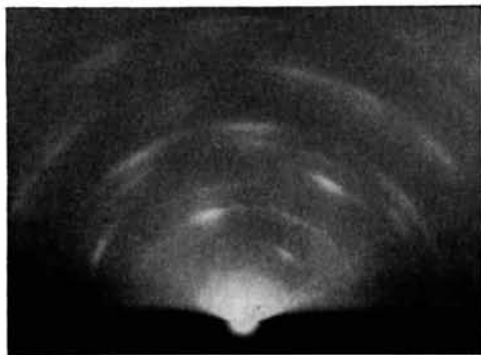


Fig. 1. Electron diffraction pattern ($\times 2$) from TiCl abraded (L to P') on 4/0 emery; \perp r. azimuth.

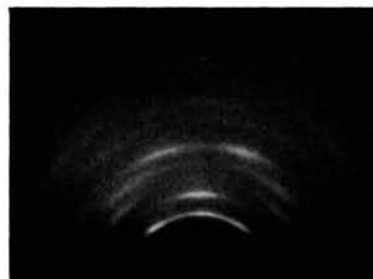


Fig. 2. X-ray diffraction reflection pattern from rolled TiCl; beam \perp r. to R direction.

2. The rolling texture of TiCl

Fig. 2 is an X-ray diffraction 'reflection' pattern from the rolled TiCl strip, with the beam normal to the rolling direction. The main orientation appears to be of one-degree $\{111\}$ type, i.e. with a $\{111\}$ plane parallel to the rolled surface, but with no clear azimuthal preference of orientation. The less-strong 110 arc also present in the plane of incidence indicates a proportion of material in $\{110\}$ orientation. Although the azimuthal preference, if any, of this $\{110\}$ orientation cannot be clearly determined from the pattern fully, it seems likely that it is such that a $\langle 001 \rangle$ direction is normal to the rolling direction, as for Fig. 1. This would then lead to $hk0$ arcs in a roughly square arrangement as in Fig. 1, and particularly to 210 arcs almost vertically below the strong 211 arcs as is seen in Fig. 2 (cf. Fig. 1). No such 210 arcs would be expected from the postulated main $\{111\}$ one-degree orientation (cf. Fig. 3, filled-in circles).

4. Discussion

The 'abrasion texture' of TiCl, CsCl, NH_4Cl and NH_4Br is shown conclusively to be $\{110\} \langle 1\bar{1}0 \rangle$, i.e. the plane which is perpendicular to the backwardly tilted main orientation axis is of $\{110\}$ type, and in this plane a $\langle 1\bar{1}0 \rangle$ direction is in the plane which is normal to the specimen surface and parallel to the abrasion direction. Since the slip system of these materials is known to be $\{110\} \langle 001 \rangle$, the abrasion texture is such that a slip plane is normal or nearly so to the resultant compressive force (of the normal load and the tangential force overcoming friction to cause motion) exerted by the abrasive particles on the material; and a slip direction is normal to the abrasion direction, and parallel to the surface. The close similarity in the surface orientation caused by the abrasion in these four materials emphasizes the dependence of the nature of the deformation on the type of crystalline structure involved.

The large rotational spread of orientation about the abrasion direction is presumably in part associated with the abrasion grooves having surfaces which are largely inclined away from the mean plane of the specimen surface. A similar rotational spread about the rolling direction is often found in rolled metals, however.

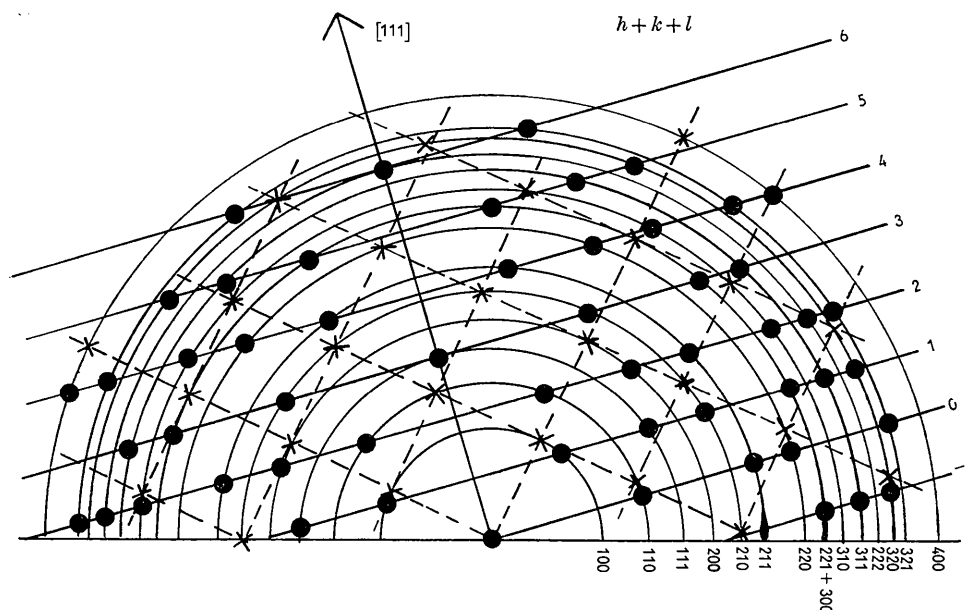


Fig. 3. Theoretical diffraction positions (denoted by \times) from a cubic crystal with beam along a cube edge, $[001]$, and with a $\{110\}$ plane normal tilted back to the left by $\delta \sim 21^\circ$; and also (filled-in circles) diffractions expected from an azimuthally random one-degree $\langle 111 \rangle$ orientation with axis slightly less tilted, so that the 111 diffraction is vertically above the 110 diffraction. (Not to same scale as Fig. 1.)

The rolling texture of TiCl was indeed found to be at least in part with a $\{110\}$ plane parallel to the rolling plane, and probably with an azimuthal preference such that a $\langle 1\bar{1}0 \rangle$ direction was along the rolling direction as for the 'abrasion texture'. If there were a spread of orientation from $\{110\}$ $\langle 1\bar{1}0 \rangle$ by a range of rotation about the rolling direction, this would have resulted in diffraction arcs which in the region of the central spot would coincide (or nearly so) with those from the one-degree $\{111\}$ orientation, as for the abrasion texture. In the X-ray photograph, Fig. 2, the arcs in the outer part of the pattern which distinguished between the two possibilities in the abrasion experiments are too faint and coalescent to be observed clearly.

The prominent $\{111\}$ orientation present in the rolled TiCl is of the same type as that of the initial compacted TiCl used as starting point for the rolling experiments, which may have tended to persist notwithstanding the more than 90% reduction during the rolling. Apart from this $\{111\}$ component orientation, the rolling texture appears to be $\{110\}$ like the abrasion texture. Such correspondence occurs also in f.c. cubic and b.c. cubic metals (Goddard, Harker & Wilman, 1962), the hexagonal metals Zn and Cd (Avient & Wilman, 1962), AgCl and AgBr (King & Wilman, 1961) and KCl (Dobson & Wilman, 1962).

The absence of appreciable change in the angle of tilt δ of the $\langle 110 \rangle$ orientation axis in the abraded TiCl in the subsurface regions to several thousand \AA depth, is similar to the observations with AgCl and AgBr (King & Wilman, 1961) and graphite (Porgess

& Wilman, 1960). In the present case of TiCl abraded on 4/0 emery paper at 200 g. load, the observed tilt δ was about 25° and was thus not far from $\tan^{-1} \mu$, which was $\tan^{-1} 0.54$, i.e. 28° . The orientation axis was thus along the resultant compressive force exerted by the abrasive particles on the material. Both μ and δ estimated here are the effective resultant values for the specimen as a whole, since they will be different for the two parts associated with the two kinds of contacts—emery/TiCl, and TiCl/TiCl which is due to the partial clogging of the abrasive surface by the worn-off material (cf. Porgess & Wilman, 1960).

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