## Investigations on the deformation of experimentally shock-loaded biotites using X-ray single crystal diffraction techniques

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SUMMARY. The deformation of biotite single crystals submitted to experimental shock-pressures of about 200 and 400 kb with direction of the shock wave parallel to [001] was investigated by X-ray precession and Weissenberg techniques. The X-ray analysis reveals a 'fracturing' of the biotite crystals upon pressure release into relatively large lattice blocks (> 1000 Å), which increases with the degree of shock compression. Deformation is inferred to take place by gliding and/or microfracturing processes parallel to the (010) and (110) lattice planes. Chains of tetrahedra running parallel to [100] and [110] probably exert a controlling influence on shock deformation mechanisms.

BIOTITE is an important rock-forming mineral, and is found in many rocks of terrestrial impact craters. Investigations of the behaviour of micas under shock-compression have consequently been carried out by several mineralogists (see, for example Schneider, 1972, 1974; review on the behaviour of rock-forming minerals under shock compression, Stöffler, 1972). These investigations dealt essentially with the identification of microscopic deformation features, such as kink bands and planar elements, in naturally shock-loaded biotites from the Ries-crater (Germany).

Experimental work on the deformation of biotite caused by high dynamic pressures has been reported by Hörz and Ahrens (1969), Hörz (1970), and Schneider and Hornemann (1974). The papers of Hörz and co-workers describe the geometrical relationships of shock-produced kink bands and changes of optical lattice angles with increasing dynamic pressures as revealed by microscope investigations. Previous studies by Schneider and Hornemann of experimentally shock-loaded biotite samples were performed by infra-red spectroscopy and using X-ray single crystal oscillation and rotation photography. At that time we believed that moderately shock-loaded biotites (P < 400 kb)

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with direction of the shock wave parallel to the  $c^*$ axis show weak deformation produced 'by various gliding processes within different lattice planes' (Schneider and Hornemann, 1974, p. 154). Based on recent, more detailed X-ray work, we can now give some more results on the shock-loading behaviour of micas in the pressure range up to 400 kb.

*Experimental.* Thin discs of biotite single crystals (thickness approximately  $35 \ \mu$ m, diameter 10 mm, MgO/FeO 0.92) from a pegmatite of Miask, Ural, U.S.S.R., were experimentally shock-loaded by subjecting them to dynamic pressures of about 200 and 400 kb. Shock-recovery experiments were carried out by the reflection technique. The shock-wave direction was parallel to the c\*-axis (for more details see Schneider and Hornemann, 1974, pp. 150-3).

The deformation mode of shock-loaded mica crystals was investigated by X-ray single-crystal diffraction methods using filtered Cu- and Moradiation. X-ray diffraction photographs were taken of the reciprocal (hol), (okl), (hko), and (hkI) planes by the precession technique and of the reciprocal (hol), (h1l), (okl), and (1kl) planes by the Weissenberg method. Starting from the suggestion that structural deformation is documented by the shape of individual X-ray reflection spots streaking and broadening of reflections obtained from precession photographs was carefully measured optically with a standard measuring device to an accuracy of approximately  $\pm 0.1$  mm.

Results. Strong line broadening was observed in many of our X-ray patterns with 020 reflections being especially sharp (fig. 1a-c). Streaking measurements of the 200, 020, and 003 reflections within the reciprocal (hol), (okl), and (hko) planes are given in fig. 2. Some characteristic features valid for both the 200 kb and the 400 kb run are: X-ray diffraction spots are smeared out most strongly within the reciprocal (hol) plane with streaking maxima of the hoo reflections. X-ray reflections of the reciprocal (hko) plane show least streaking, with

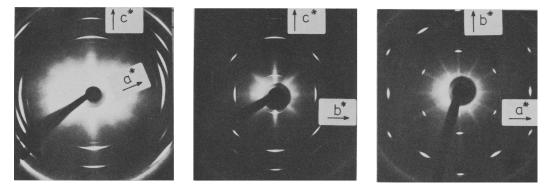


FIG. I. Precession photographs, taken from a biotite single crystal submitted to a dynamic pressure of about 400 kb: a, reciprocal (hol) plane; b, reciprocal (okl) plane; c, reciprocal (hko) plane. Ni-filtered Cu-Kα-radiation.

oko being half as elongated as hoo reflections. The degree of arcing of the reciprocal lattice spots of the (okl) plane lies between those of the reciprocal (hko) and (hol) planes (see figs. 1a-c and 2).

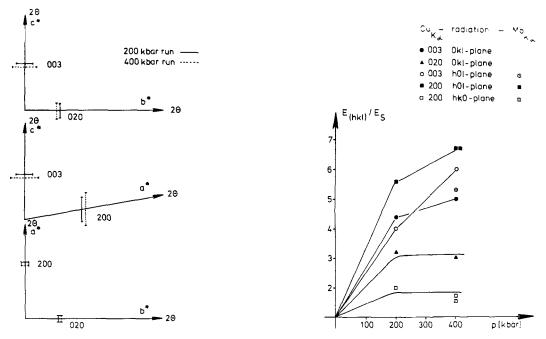
It is an interesting fact that the degree of streaking of individual diffraction spots within the different reciprocal lattice planes does not change with the order of the reflection. Though shockloading experiments at 200 kb and 400 kb yield quite similar results, some characteristic differences in the degree of the deformation become evident if relative rather than absolute streaking amounts are taken into account. Fig. 3 presents relative streaking amounts, which were obtained by dividing the degree of streaking (arcing) of the 200, 020, and 003 reflections of the reciprocal (hko), (hol), and (okl) planes by that of 020 of the reciprocal (hko) plane. While the degree of arcing of the majority of X-ray reflections is nearly constant for both high-pressure runs, 200 and 003 diffraction spots of the reciprocal (hol) plane are more extensively smeared for the 400 kb biotite than for the 200 kb sample.

Discussion. The interpretation of recent investigations of shock-loaded minerals is based on the assumption that the shape of an X-ray reflection reveals the mode and degree of lattice distortion. and that X-ray diffraction spot-width can be related to the variation of corresponding lattice spacings and to the shock-induced crystallite or domain size. The X-ray patterns of biotite crystals shock-loaded up to 200 and 400 kb show only slight broadening of diffraction spots, especially of hoo and ool. We conclude that shock compression and stress relaxation behind the elastic wave produced lattice blocks approximately 1000 Å in size. Furthermore, there exists no shock-induced stacking disorder of individual mica sheets in the direction of the  $c^*$ -axis, though  $d_{001}$ spacings may vary within certain limits. This is

reasonable, because the shock waves propagated parallel to this lattice direction.

Orientation relationships of the mosaic lattice blocks produced by the shock event are derived from figs. I to 3. It is evident that the  $a^*$ - and c\*-axes show a much greater pressure-controlled deviation from their original positions as compared to the  $b^*$ -axis. This should be interpreted in terms of a structural deformation of biotite that takes place most favourably within the (010) lattice planes. The deformation is not only due to simple rotational distortions, therefore, but must have taken place by various rotational and translational movements within the (010) (and (110), see below) planes. Because of the large size of the lattice blocks the deformation does not effect a change in the periodicity along the  $b^*$ -axis, which would be expected if deformation took place on an atomic scale. Due to the trigonal symmetry of the  $[AlSi_3O_{10}]^{5-}$  tetrahedral nets gliding and microfracturing processes, which are responsible for the shock-induced lattice deformation, may also take place parallel to the (110) planes. Under the stereoelectron-microscope the plastic lattice deformation systems appear as very thin planar discontinuities that could not be resolved further (see fig. 4a and b).

The shock-induced deformation of biotite can be explained by the crystal structure of the mineral. Fig. 5 is a simplified sketch of the mica structure in projection down the  $c^*$ -axis. Micas have perfect cleavage parallel to the basal (001) sheets; thus shock-induced deformation processes should take place preferably within these lattice planes. In our study this was not the case because the shock-wave direction was perpendicular to the basal sheets. In addition to the basal (001) planes tetrahedral chains running parallel to the crystallographic *a*-axis seem to be fundamentally important structural units (fig. 5). We infer from the present results that the



FIGS. 2 and 3: FIG. 2 (*left*). Streaking of 200, 020, and 003 X-ray reflection spots within the reciprocal (okl), (hol), and (hko) planes as obtained from precession photographs. Full line: peak pressure 200 kb; dotted line: peak pressure 400 kb. FIG. 3 (*right*). Relative streaking (arcing) of X-ray reflection spots.  $E_{hkl}$ : streaking of the 200, 020, and 003 reflections within the reciprocal (okl), (hol), and (hko) planes;  $E_s$ : streaking of the 020 reflection within the reciprocal (hko) plane.

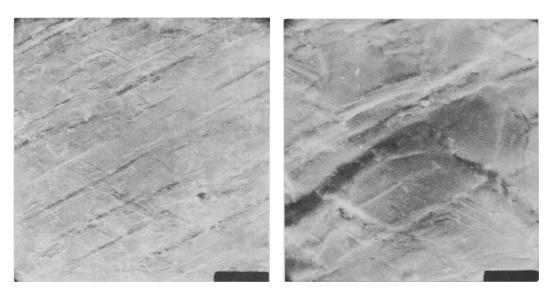


FIG. 4. Stereo electron-microscopic photograph (Siemens Autoscan U1) of a biotite single crystal, shock-loaded up to 200 kb with multiple sets of deformation structures, probably running parallel to (010) and (110). Scale:  $\times$  320 (a) and  $\times$  1600 (b).

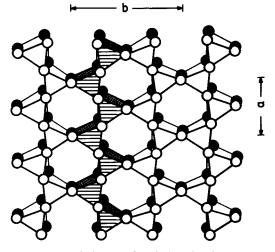


FIG. 5. Tetrahedral sheets of biotite in a view down the  $c^*$ -axis (after McCauley *et al.*, 1973). Dark oxygen atoms represent the lower, light ones the upper tetrahedral sheet. Shadowed area: structure controlling tetrahedral chains.

For simplicity octahedral layers are not drawn.

shock-induced deformation in mica is due to gliding or microfracturing processes or both parallel to these structural elements, although some tetrahedral bonds have to be broken and appropriate octahedral units are destroyed in these processes.

Slightly broadened hoo spots are interpreted in terms of a small variation of the *a* spacings, which can be explained by crumpling and stretching of the tetrahedra chains running parallel to [100] and [110] during shock compression and pressure release, respectively.

In the light of these facts we believe that the shock-induced deformation processes are produced in a similar way to percussion figures in mica, which are due to plastic lattice gliding parallel to  $\{010\}$  and  $\{110\}$  and subsequent microfracturing when the elastic limit is exceeded.

There are other plastic lattice deformations that

probably occur within the pressure range under consideration, but were not observed in our biotites; they are frequent, however, in naturally shock-loaded micas, which have been subjected to other shock-wave directions than [001]. These deformation features, also known as 'planar elements' (see Engelhardt and Bertsch, 1969), mostly occur in two systems crossing each other and are orientated parallel to (111), (111), (112), (112) (Schneider, 1972; Stöffler, 1972), and (111), (111) (Lethinen, 1976). Planar elements in biotite are assumed to be due to plastic lattice gliding. Probable gliding directions may lie parallel to the common zones of the different planar deformation systems occurring in the same crystal ([110], [110], and [101]; see Schneider, 1972). The advantage of this technique was applied for the present investigation, and further directions of favoured plastic lattice deformation in experimentally shocked biotite single crystals could be found parallel to [001] (common zone of (010) and (110)). Since [001] represents a short direction in the monoclinic basecentred lattice of biotite (10.1 Å), it is suitable for gliding and microfracturing processes.

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