X-RAY DIFFRACTION LINE BROADENING IN EXPERIMENTALLY SHOCKED ORTHOPYROXENES

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Introduction. Orthopyroxene (opx) is an important mineral in several meteorite groups and lunar rocks. Although experimental calibration data are not available so far, shock effects in opx can be used on an empirical basis for shock classification [1,2]. These shock effects include mechanical twinning, mosaicism, the formation of planar elements, reduction of birefringence, and at higher pressures, the transformation of opx into majorite [3,4] which has a garnet structure. The onset of this transformation could possibly serve as shock wave barometer. In order to calibrate these shock effects, we have started a series of shock recovery experiments on single crystal enstatite ($(Mg_{0.73}Fe_{0.27}Al_{0.05})Al_{0.06}Si_{0.94}O_3$) sampled from the Egersund Anorthositic Complex in SW-Norway.

Experimental methods. *Shock recovery experiments* were performed at room temperature on 0.5 mm thick disks (Ø 15 mm) with the shock wave travelling parallel to the (100)-plane of the enstatite. The experimental set-up with an high-explosive driven flyer plate was similar to that described by [5]. Peak pressure of 15, 30, 48, 72.5 and 118 GPa was reached by reverberation of the shock wave [6]. Due to this technique, the duration of the peak-pressure was short, being 0.75 µs for 15 GPa, 0.5 µs for 30 GPa, 0.4 µs for 48 and 72.5 GPa, and > 0.1 µs for 118 GPa.

The recovered samples were investigated by *microscopic*, *spindle stage*, and *X-ray diffraction techniques*. *X-ray measurements* were performed with a Guinier-Jagodzinski powder camera operating with monochromatic Cu-$K_{\alpha 1}$ radiation at 40kV and 30 mA. X-ray line intensity profiles were recorded with an automatic photometer. To obtain the pure diffraction pattern from the measured raw intensity profile, a Gaussian shape for each diffraction line was assumed [7]. For the shocked samples, the pure half-maximum breadth, β, of each line was then calculated by substracting the half-maximum breadth of the respective line in the unshocked reference sample, *b*, from the corresponding value in the shocked sample, *B*, as follows: $\beta^2 = B^2 - b^2$. According to [8], β is related to the mean crystallite size, *L*, and the lattice strain, ε, by the equation:

$$\frac{\beta^2 \cos^2 \Theta}{\lambda^2} = \frac{1}{L^2} + 16\,\varepsilon^2\,\frac{\sin^2 \Theta}{\lambda^2}$$

Based on this relation, *L* and ε of the shocked sample can be evaluated in a plot of $\beta^2 \cos^2\Theta/\lambda^2$ vs. $\sin^2\Theta/\lambda^2$. In this type of diagram, the slope of a regression line through data points for several orders of one lattice plane equals $16\,\varepsilon^2$, and the intercept corresponds to $1/L^2$ (Fig. 1).

Usually, for this type of analysis, the β values from at least 3 orders of reflection for one (hkl) plane are used. In the case of Guinier-Jagodzinski analysis of orthorhombic enstatite, however, such higher orders of reflection are missing in the recorded range of Bragg angles ($2\,\Theta \leq 68°$). For this work, therefore, β-values of all measured (hkl) planes were included in the analysis as suggested by [9] for shocked quartz.

Results. Compared to the unshocked reference enstatite, the shocked samples show with increasing shock pressure, undulary extinction and increasing fragmentation. Decrease of birefingence or the beginning of amorphization, however, could not be substantiated on the microscopic scale. In contrast, X-ray patterns allow a more detailed characterization of the shocked enstatite samples. With increasing pressure, diffraction lines broaden and peak intensities decline but a shift of peak positions was not detected. All peaks could be indexed and no indications of the presence of majorite was found.

Using the procedure described above, the change in *L* and ε was determined. As shown in Fig. 1 for the 48 GPa experiment, regression lines ($16\,\varepsilon^2$) have distinct positive slopes for all shocked samples. With higher pressure, the value for ε increases from 1.5 ‰ at 15 GPa to 4.3 ‰ at 118 GPa (Fig. 2). This increase in lattice strain is accompanied by a reduction of the crystallite size *L*. Our data indicate that *L* decreases continuously from 4770 Å for the unshocked reference material to 590 Å at 72.5 GPa (Fig. 2). For the sample shock-loaded at 118 GPa, a cristallite size of 360 Å was calculated.

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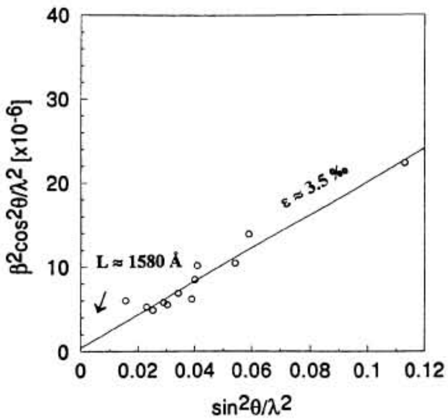

Fig. 1: Diagram for determination of the crystallite size L and the lattice strain ε. Points represent lattice planes (hkl); enstatite shocked experimentally at 48 GPa.

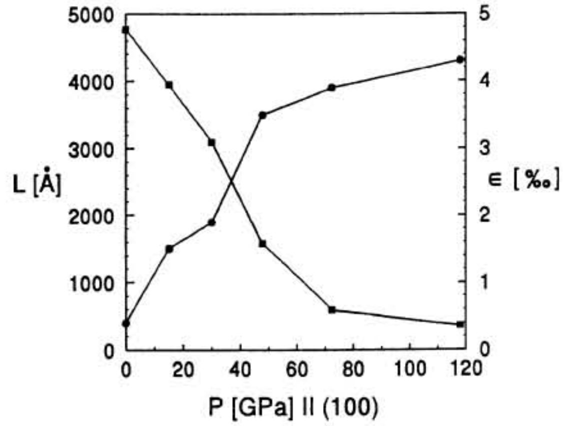

Fig. 2: Change of crystallite size L (squares) and lattice strain ε (circles) with shock pressure; experimentally shocked enstatite.

Discussion. Compared to the Debye-Scherrer method, as applied by [10], in the analysis of shocked silicates, the Guinier-Jagodzinski technique allows a quantitative analysis of line broadening. The new data for experimentally shocked enstatite suggest that a linear relation between internal grain size and shock pressure exists for opx in the range up to 72.5 GPa. Precise X-ray investigation of opx from natural impact lithologies may, therefore, yield a good estimate of the shock metamorphic overprint. As L and ε, however, are dependent of the pre-shock stage, such data can not serve as "absolute" shock barometer.

It is inherent in the reverberation method that the duration of the pressure pulse is short, in comparison to other experimental techniques, and that the post-shock temperature is low compared to the natural case. Therefore, shock recovery experiments carried out in this study are not directly applicable to defining the threshold pressure for the onset of shock-melting in opx, and for the shock-induced formation of majorite.

According to Hugoniot calculations by [11], the transition of opx into majorite should start at a shock pressure > 35 GPa. In static high pressure experiments, majorite begins to form at 15 GPa [4]. So far, the phase transition of enstatite into majorite has been reported only once [12] occuring in dynamic high pressure experiments between 35 and 50 GPa. On the basis of our X-ray analysis, however, this finding seems dubious, as the experiments were performed with the identical set-up, using shock wave reverberation. The short pressure impulse will hamper formation of high pressure polymorphs in all types of shock recovery experiments as long as total reorganization of the unit cell is required.

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