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# Experiment Report Form

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## Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

| ESRF | **Experiment title:** | **Experiment number**: HS2390 |
|---|---|---|
| **Beamline**: ID13 | **Date of experiment**: from: 05.04.2004 to:09.04.2004 | **Date of report**: 25.02.2005 |
| **Shifts: 9** | **Local contact(s)**: M. Burghammer | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

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## Intoduction

Clinopyroxenes from the martian meteorite NWA856 were studied by means of single crystal diffractometry with synchrotron radiation at ID13/ESRF and D3/DESY. A structure refinement was performed and the cation distribution determined. In pyroxenes, the Mg and $Fe^{2+}$ cations fractionate between the non-equivalent M1 and M2 sites. The fractionation value $K_D$ ($Fe^{2+}$M1· MgM2) /($Fe^{2+}$M2 ·MgM1) is sensitive to temperature and thus serves as a recorder for the cooling rate of the rock.

## Sample material

Crystals with edge lengths of <1µm - to 10µm were examined and only smallest crystals exhibit reflection profiles suitable for data collection and, most importantly, extraction of integrated intensity. Due to the very similar lattice constants of the intergrown pigeonite and augite lamellae an overlap of reflections from both phases occurs and the separation of reflections belonging to the two phases is difficult, especially if the mosaicity of the crystals is high. Pyroxene samples from martian meteorites have previously been considered as a hybrid between single crystals and textured powder in literature [2]. The crystal size seems to be the most important factor for determining the crystal quality. In practice, powdered sample material is placed on a kapton foil, which was scanned for good quality single grains. Data was collected at l= 0.731Å and 0.976Å and varying sample-CCD distances. The best sample was a crystal (G1) of 7*3*5 µm, mainly pigeonite, with lattice constants a:9.73Å, b: 8.96 Å, c:5.26 Å, ß: 108.60°.

## Reconstructed reciprocal layers

The low degree of shock, as indicated by sharp reflections and absence of (mechanical) twinning is apparent in Fig.1a. No streaking along a* or c* directions was found. The FWHM of h+k = odd or even type reflections were determined by fitting with Gaussian profiles, results see Fig. 3b. The other crystals investigated showed a much higher degree of mosaicity (Fig. 1b,c) , streaking along a* and in one case (001) twinning affecting both augite and pigeonite (Fig. 1b).

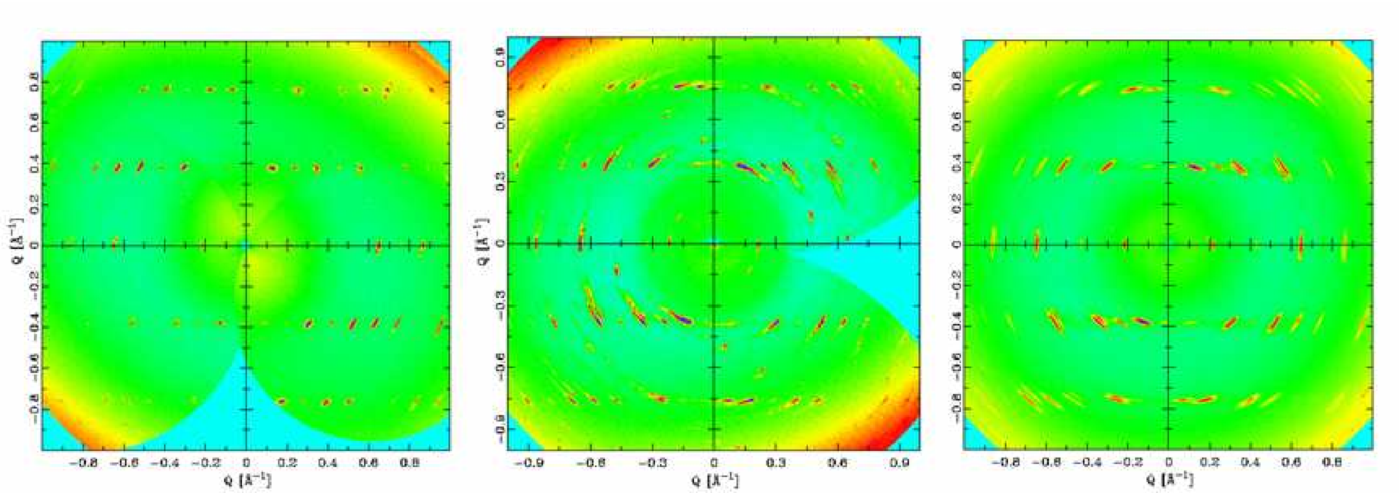

Fig 1a, b, c: Reconstructed (h0l) layer of three martian pyroxenes. a, left: low mosaic grain (G1); b, middle: high mosaic, twinned (G2) crystal; c, right: high mosaic (M2) crystal.

## Microstructure

Diffuse XRD is a rich source of information about microstructures of extraterrestrial crystals. XRD complements TEM investigations as the atomic structure and the microstructure on one and the same sample [1] may be studied and long-range coherency ("global") effects become obvious in contrast to "local" defect arrangements investigated by TEM.

Low (1-2°) mosaic grain: major pigeonite (P) and minor augite (A), no opx-phase (in agreement with TEM-observations [4]: interpretation by a relatively high primary cooling rate to 900-1000°C; P and A reflections belong to only one A-P pair. The strong "a-type" reflections are accompanied by diffuse intensity extended along c* (Fig. 2) corresponding to a (001) lamellae of only one A/P-exsolution-generation; reflection widths indicate average coherence lengths of ~ 220 Å, i.e. smaller than those reported in [4]; lamellae with thicknesses of several 1000 Å, or an A/P superorder with periods of the order of 1000 Å [8] could not be observed by XRD.

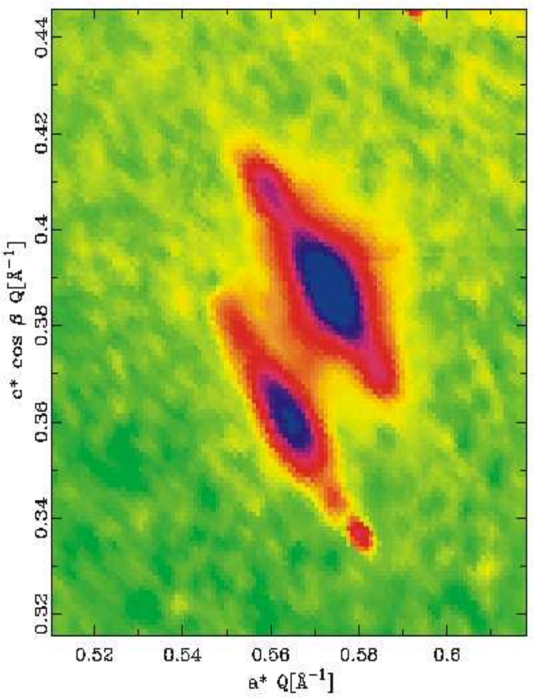

Fig. 2: G1 sample,(h0l) layer, 402 reflection

Faint diffuse streaks between A-P pairs may be explained either by domain walls between A and P with a gradient of the c-lattice constants or by additional small high-pigeonite lamellae which are exsolved due to an incomplete (spinodal) decomposition (?) of P within A lamellae (as reported in [2]).

Low-mosaic grain: no cpx-twinning on (001) as reported for a Shergotty meteorite [5]; missing (h00) streaks or (100) twinning - related to mechanical stacking faults as consequence of plastic deformation: no (strong) shock deformation. "Moderate-mosaic" (3-4°): generally same diffraction phenomena; twinning on (001), additional weak diffuse streaks occur along [h21] both in P- and A-phase.

"B-type" reflections (h+k=odd) are only slightly broadened along a* (corresponding to an antiphase domain size of 150Å, Fig. 3b), no diffuse contributions: absence of a significant amount of small pig-APD's, might be related to a slow cooling rate in the temperature range of the pigeonite inversion at T ~ 900°C. Pristine martian pyroxenes are almost free from mechanical deformation; a large mosaic spread distribution is, most likely, the signature of an impact event; (2) weak diffuse wings (“halos”) of the strong reflections might be related to point defects caused by radiation damage due to high energy particles after the impact: this is concluded from our results as the origin of same martian material is from the interior of solid lava (few meters below ground), which was well protected from external (solar) radiation.

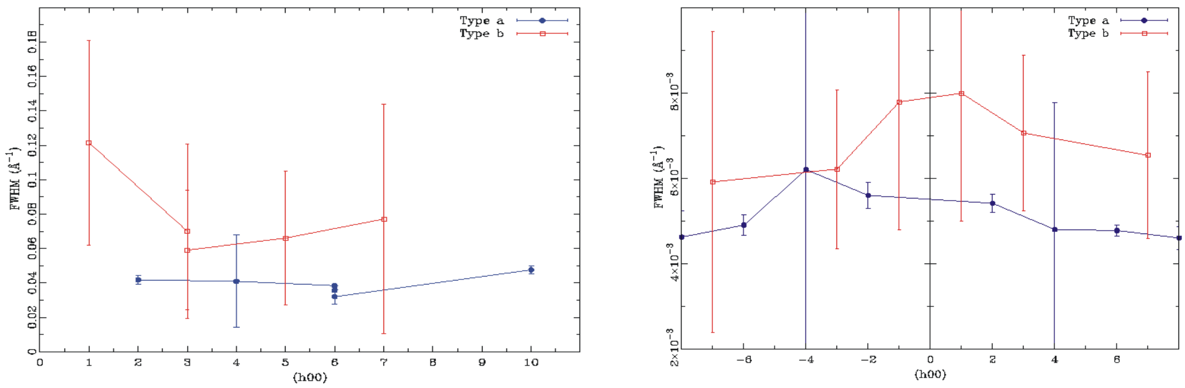


Fig. 3: (a, left) Lunar pyroxene, DESY/F1 (b, right) Martian meteorite NWA 856, FWHM of a- and b-type reflections in h-direction.

## Structure refinement and results

Integral intensities were derived from data processing with XDS[3]. The refinement was carried out in P21/c with Jana2000 [10]. About 800 independent reflections were used. Starting atomic parameters were taken from [8]. Due to the unknown starting composition, the En/Fs ratio was varied (Fig. 4). The best $R_{obs}$ value of 5.0 and a $GoF_{all}$ of 2.25 was achieved with $En_{52.5}Fs_{43.5}Wo_{04}$. The overlapped reflections at low 2θ were corrected, assuming a 15% volume of augite and structure parameters taken from [1].

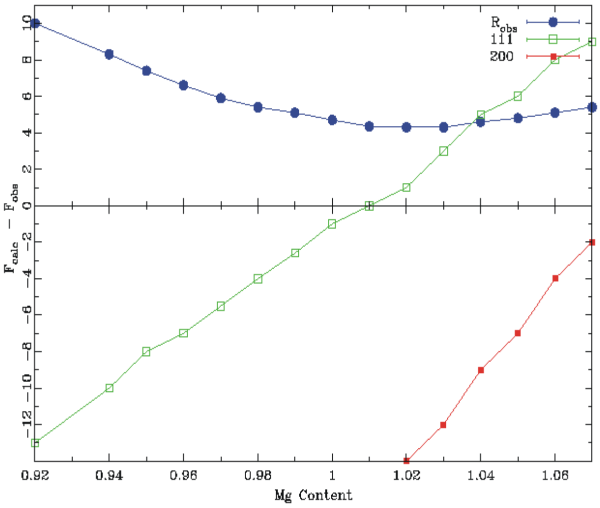


Fig. 4. $R_{obs}$ variation and $F_{calc}$ - $F_{obs}$ of high leverage M1occ and M2occ [6] (200) and (111) reflections with Mg content.

Integrated intensities and structure refinement may also be affected by diffuse contributions due to antiphase domains and exsolution textures, cf. [12]. The decrease in displacement parameters for O3A and B using only h+k = odd reflections was not remarkable, this is an indication that the sample consists of rather coarse antiphase domains.

The closure temperature of the exchange was calculated from the experimentally determined geo-thermometric equation [9]. A $K_D$ value of 0.05±0.0032 corresponds to a closure temperature of 557°C±13°C. The distribution coefficients of lunar pigeonites range from 0.07-0.12, model calculations based on Jaegers theory [see 11] suggest that the crystals derived from top and middle of lava flows of thicknesses 4-10m [11,13]. For NWA856 pyroxenes, Leroux et al. [4] (TEM methods) calculated cooling rates of 0.1°C/h in the temperature range above 700°C. Our data suggests that this cooling rate persisted down to 560 °C and that the crystals formed in the middle of a lava flow more than 6m across [5].

## References


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