Quantitative texture analysis on D19 using the new 120° curved area PSD

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Quantitative texture analysis has recently been developed on the D19 diffractometer at the ILL. The newly developed detector ranks this instrument as nearly the quickest world-wide for this kind of experiment, with potentialities in dynamic texture measurements.

Quantitative Texture Analysis (QTA) relies on the measurements of many pole figures, each being acquired for a typical scan of around 1400 measurement points when using point detectors, giving rise to prohibitive acquisition times for most materials at low-flux neutron centres. Neutron QTA has been developed for several decades at many neutron centres [1]. Only in the 1980's time measurement could be reduced to typically one day on D1B, then to a few hours around a decade later when intense beam became available and curved PSD developed, like on D20 [2]. We show here that this measuring time can still be reduced by increasing the solid-angle range spanned by the detector, as recently became possible on the D19 diffractometer. In order to characterise the upgraded instrument in terms of QTA, we measured a Belemnite rostrum from the cretaceous, which can be considered as a standard sample. The rostrum is the fossilised calcitic part of this ancient species from the Cephalopoda, which exhibits a planar texture (one crystallographic direction is aligned at random in a macroscopic plane) with c-axes randomly oriented around the rostrum axis. The crystal orientation typically culminates at around 8 times the powder value along this axis.

A typical texture experiment requires the acquisition of 1368 diagrams measured in as many sample orientations using a 5°x5° grid in φ and χ. In such samples, statistically reliable diagrams are obtained in 10 seconds of integration, giving a total of around 4h of acquisition time per pole figure (using a 1D detector) or per sample (using a PSD), not including dead times due to motor increments and data storage. By using the D19 position-sensitive curved area detector, we measured Debye-Scherrer diagrams of the textured Belemnite rostrum (**figure 1a**), in approximately one hour.

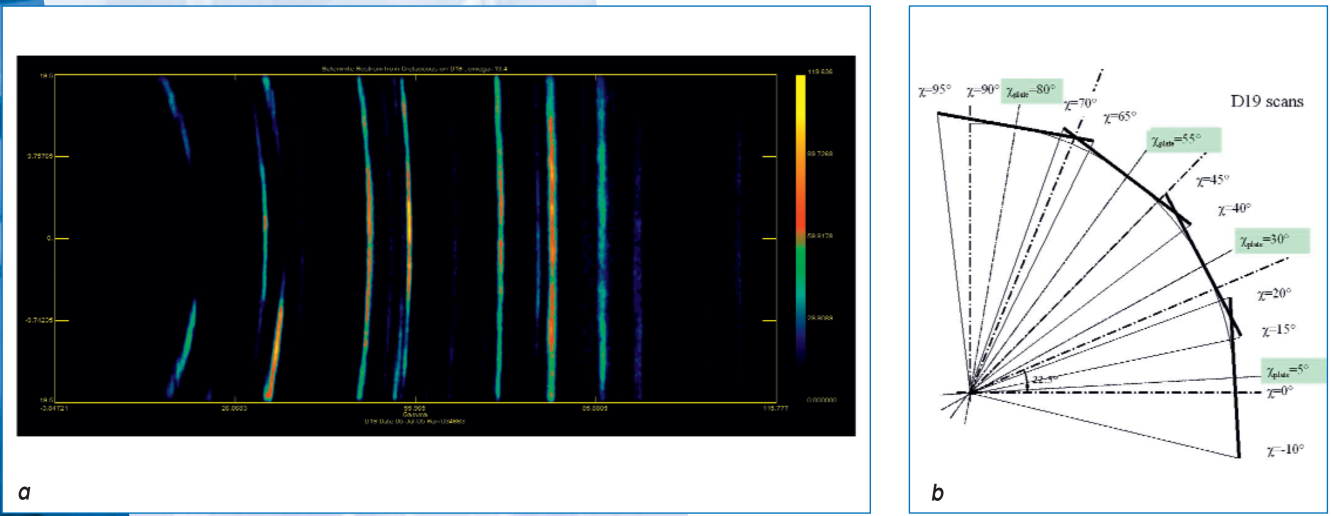

Figure 1a: *Debye-Scherrer raw diagram for one orientation (χ=0°, φ=0°) of the cretaceous Belemnite rostrum and* ***b****) corresponding required χ-scan.*

Such diagrams clearly reveal the presence of texture, with the strongly discontinuous rings of calcite (see for instance the two first rings from the left). One large advantage of such diagram is that its vertical axis simultaneously spans 30° in χ roughly. This correspondingly diminishes the number of diagrams to be measured in χ from 19 to 4 (**figure 1b**), *i.e.* a theoretical gain in acquisition time of nearly 5. Furthermore, less motor positioning (less dead time) is required. Data reduction is operated in the following sequence. First, a flat field correction is applied on each Debye-Scherrer diagram, in order to take account of cells efficiency, absorption and apparent surface irradiation. Then the Debye rings are developed to straight lines, in order to calculate equivalent 2θ diagrams. This latter step has been operated on a 5° binning scheme, in order to generate 19 diagrams (equivalent to 19 χ positions) out of the 4 detector positions, for each φ value. Data binning could have been operated to any desired resolution grid (*e.g.* with larger resolutions into 1° cells) using the same data set, for instance to analyse narrower crystallite dispersions. It is another advantage of this detecting approach which is only limited to the intrinsic cell size of the detector, and is less conditioned by the texture strength.

Data analysis is then carried out through Rietveld refinement of the whole dataset. In this step the previously extracted 2θ diagrams are directly interpreted using the combined analysis [2] implemented in the MAUD software. The angular transformations from the diffractometer space to the pole figure space are obtained using classical relations for CPS detectors [2]. The refinement of the Orientation Distribution Function allows subsequently recalculating any pole figures of interest. **Figure 2** compares the pole figures for the main axes of the belemnite rostrum as measured using the previously validated D20 (**figure 2a**) [3], and the D19 (**figure 2b**) diffractometers. In this figure the rostrum axis is the perpendicular to the pole figures. Using both datasets the same texture is obtained, with calcite c-axes ({006} pole figure) strongly aligned around the rostrum axis, and at random around it forming the predicted planar texture. Correspondingly, the calcite

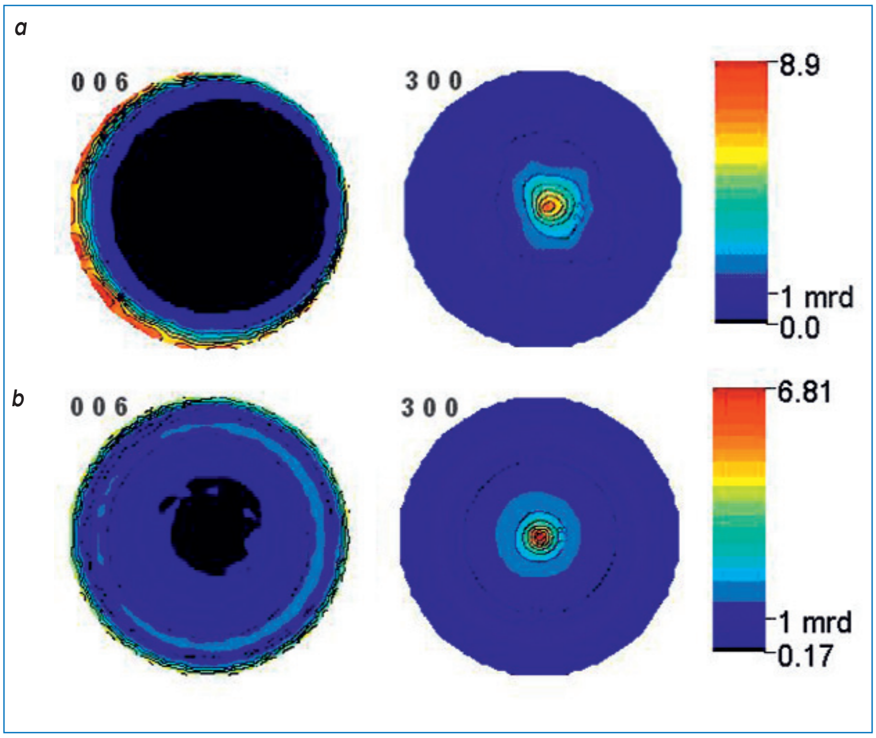

Figure 2: *{006} and {300} pole figures for the Belemnite rostrum recalculated from the ODF refined on **a**) D20 and **b**) D19 data.*

a-axes are distributed throughout the {300} pole figure with a gradual and slight reinforcement towards the centre due to the progressive lack of c-axes in this region. The weak tilt of the {006} planes observed on D20 data is only due to a slight misalignment on the diffractometer. The maximum orientation density is around 9 and 7 times the random orientation (9 and 7 m.r.d.) for the D20 and D19 instruments respectively. The observed difference of 2 m.r.d. is related in a worse reliability factor in the latter case (Rwp = 27 % and 44 %, respectively) and is attributed to small artefacts introduced during data reduction, which still need improvements. For instance, the blind zone at the left extremity of the detector together with the large curvature of the rings in this region makes a small bias in the actual correction, though the refined cell parameters are identical.

This study aimed at checking the quality of the approach developed for quantitative texture analysis using two-dimensional position-sensitive detectors. Although the level of the data processing is still to be optimised, already good results are obtained, with the shortest acquisition times world-wide for this grid resolution using neutrons.

References

(1) *H.-J. Bunge and C. Esling, Quantitative Texture Analysis, DGM, Germany (1982) 450p*

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(3) *D. Chateigner, L. Lutterotti and T. Hansen: ILL Annual Report 97 (1998), 28-29*