



QUANTITATIVE TEXTURE ANALYSIS AT THE D19-ILL BEAM LINE USING A 120° CURVED AREA PSD

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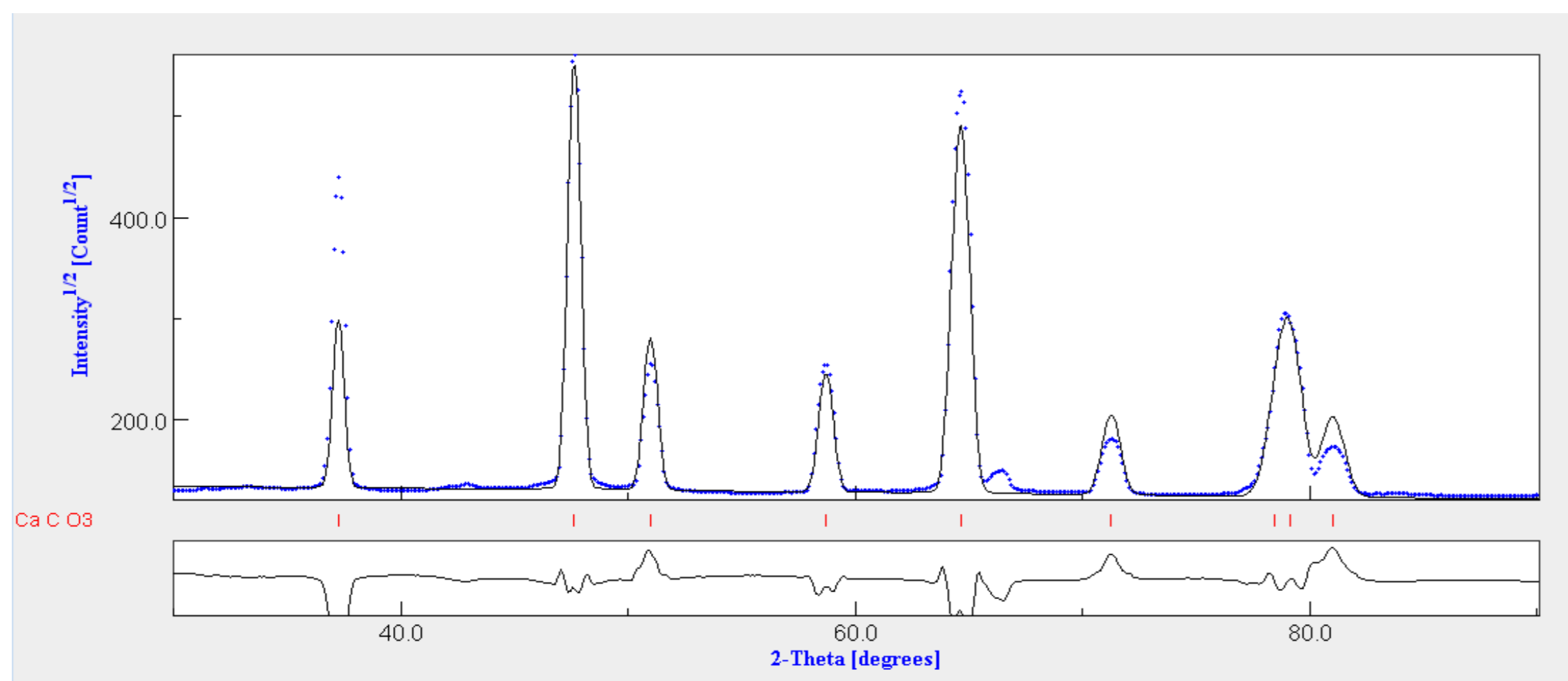
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Quantitative Texture Analysis (QTA) [1] has been developed for several decades at many neutron centres worldwide. Time measurement can be reduced at a few hours when intense beam got available like on D20 at the steady-state source of ILL [2]. We show here that this time can still be reduced by increasing the solid-angle range spanned by the detector, as recently became available at the D19 beamline of ILL.

Results from the D20 instrument.

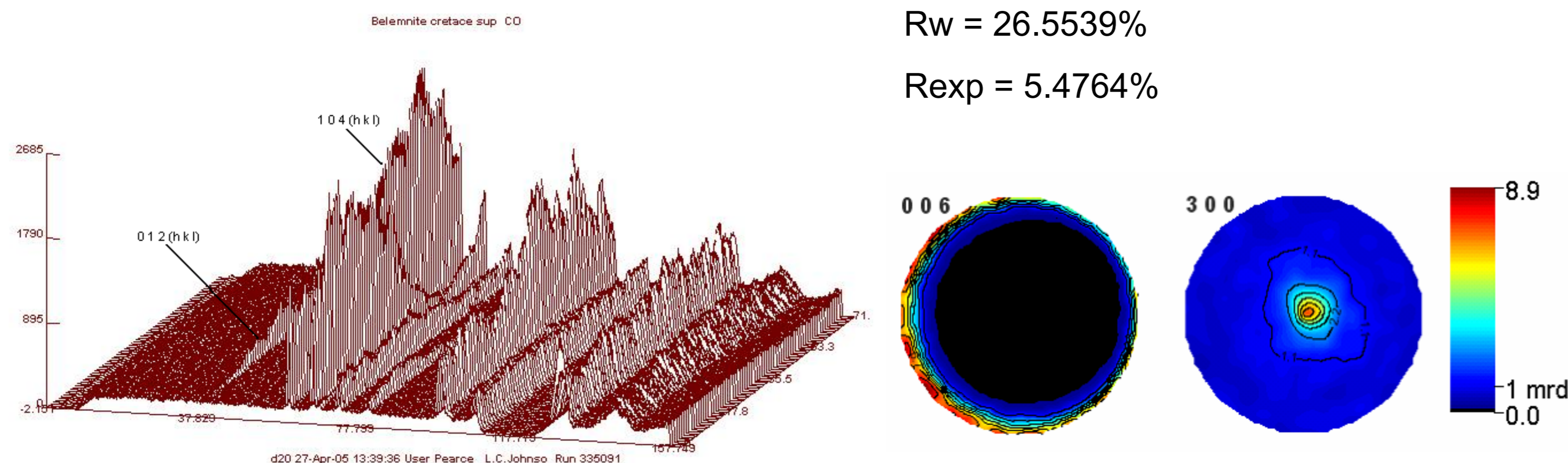
We choose two *Belemnite sp.* calcitic rostra from the Cretaceous and Jurassic to calibrate the instruments for the combined analysis.



Sum of the 1368 diagrams measured on the belemnite rostrum from Cretaceous ($\lambda=2.4\text{\AA}$).

The experiment consisted in 1368 diagrams measured in as many sample orientations using a $5^\circ \times 5^\circ$ grid in φ and χ , resulting in a 4h acquisition time. The refinement of the whole dataset using the Rietveld approach in the MAUD software and the E-WIMV algorithm to refine the ODF. Pole figures for the main a- and c-axes show a strong « planar » texture with c-axes of the calcite phase at random around the rostrum axis (perpendicular axis of the pole figures), and a-axes at random around c giving a slight reinforcement in the center of the {300} pole figures. The maximum density of orientation is around 9 m.r.d. for the cretaceous sp.

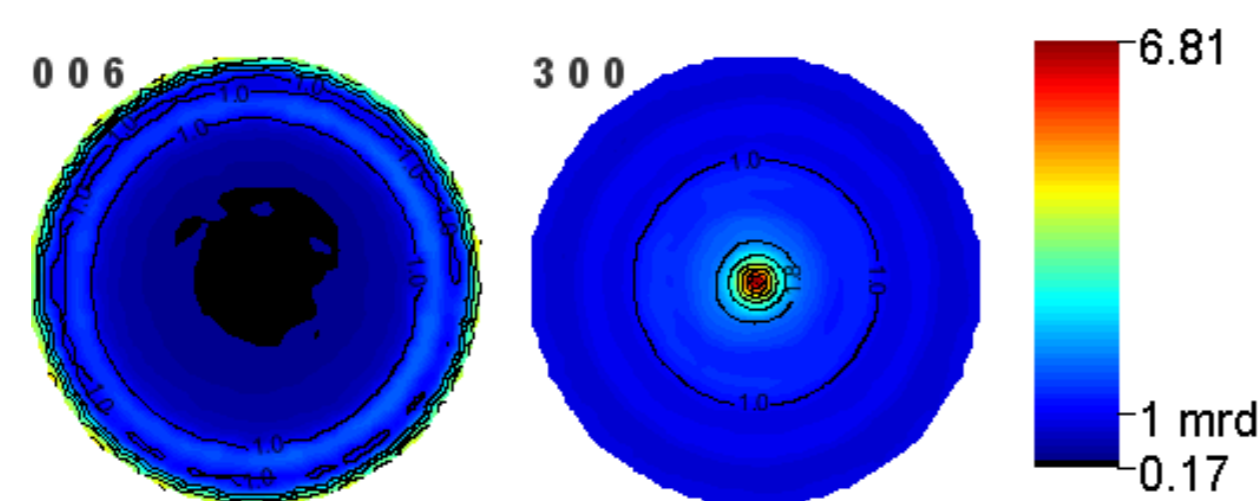
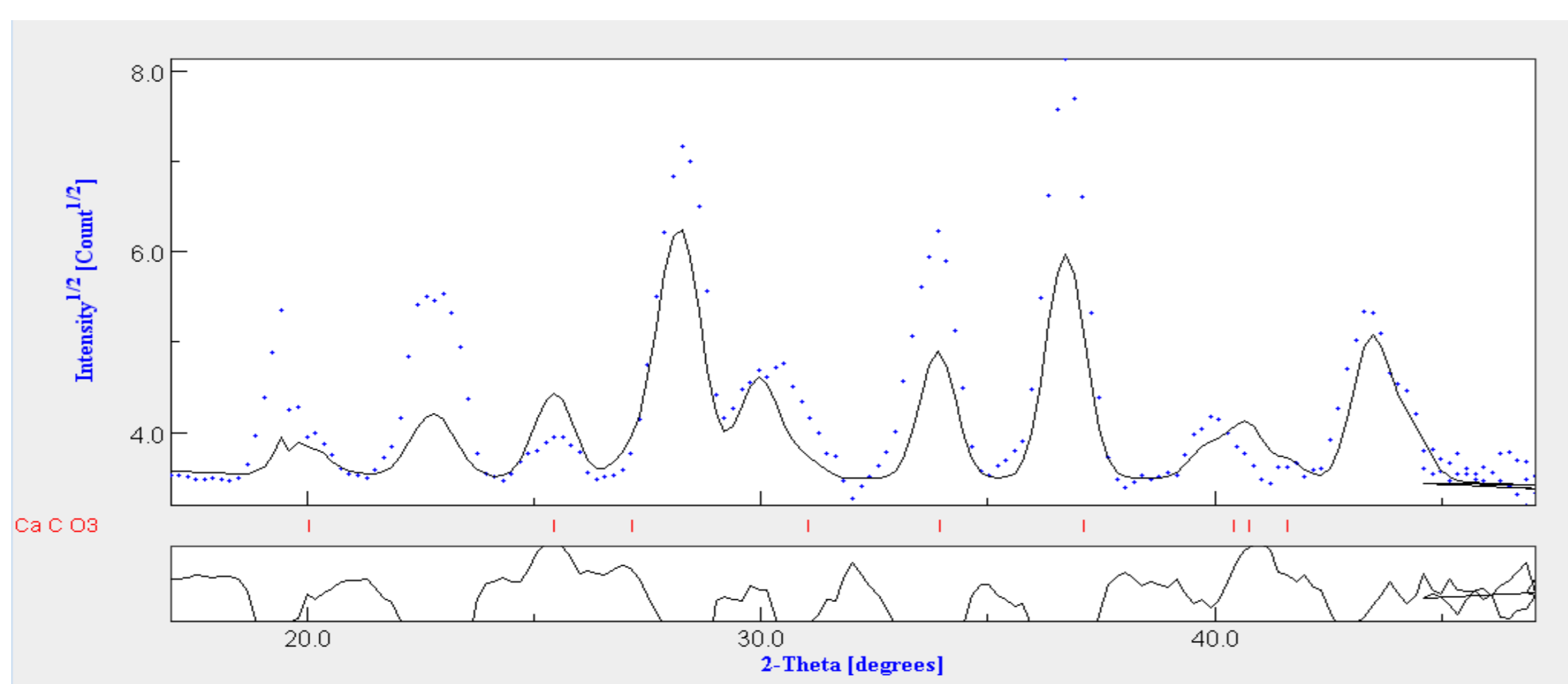
Reliability factors for the Rietveld and ODF refinement:



2D Representation of the Belemnite rostra from the cretaceous.

{006} and {300} pole figures for the Belemnite rostra from the cretaceous.

Results from the D19 instrument.



Sum of the diagrams measured on the belemnite rostrum from Cretaceous (top, $\lambda=1.315\text{\AA}$), and corresponding pole figures (bottom).

For this acquisition only 5 scans (vs 19 for D20) in χ become necessary thanks to the χ -spanning of the detector. This reduces the acquisition time to less than 1h. Very similar pole figures are obtained using the D19 instrument, though the maximum distribution density is lower than on D20. This is attributed to a relatively worse fit (as denoted by the largest reliability factors) very probably due to artifacts introduced during data reduction, which still need improvements.

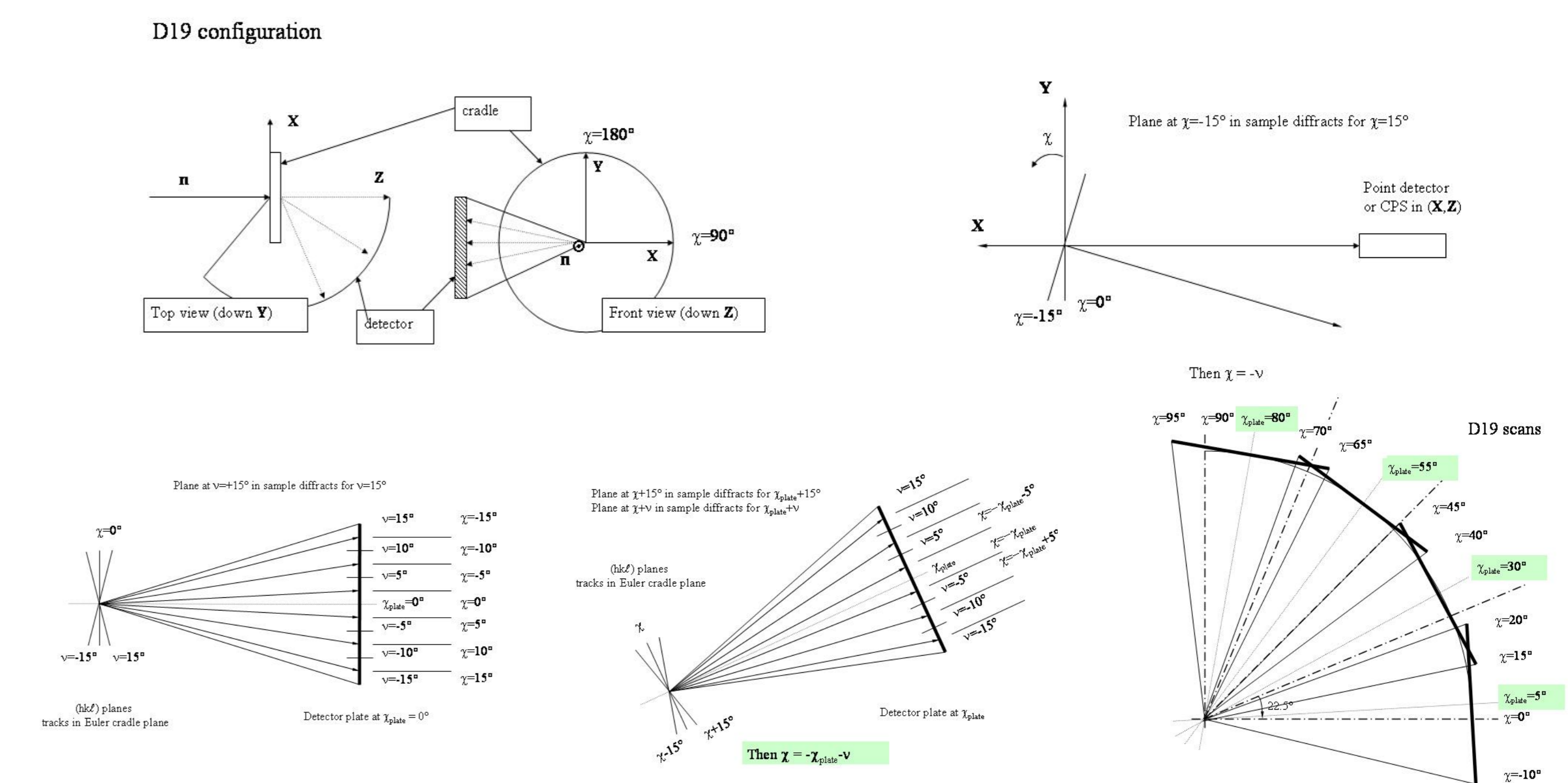
Reliability factors for the Rietveld and ODF refinement:

Rw = 44.0130 %

Rexp = 5.9349 %

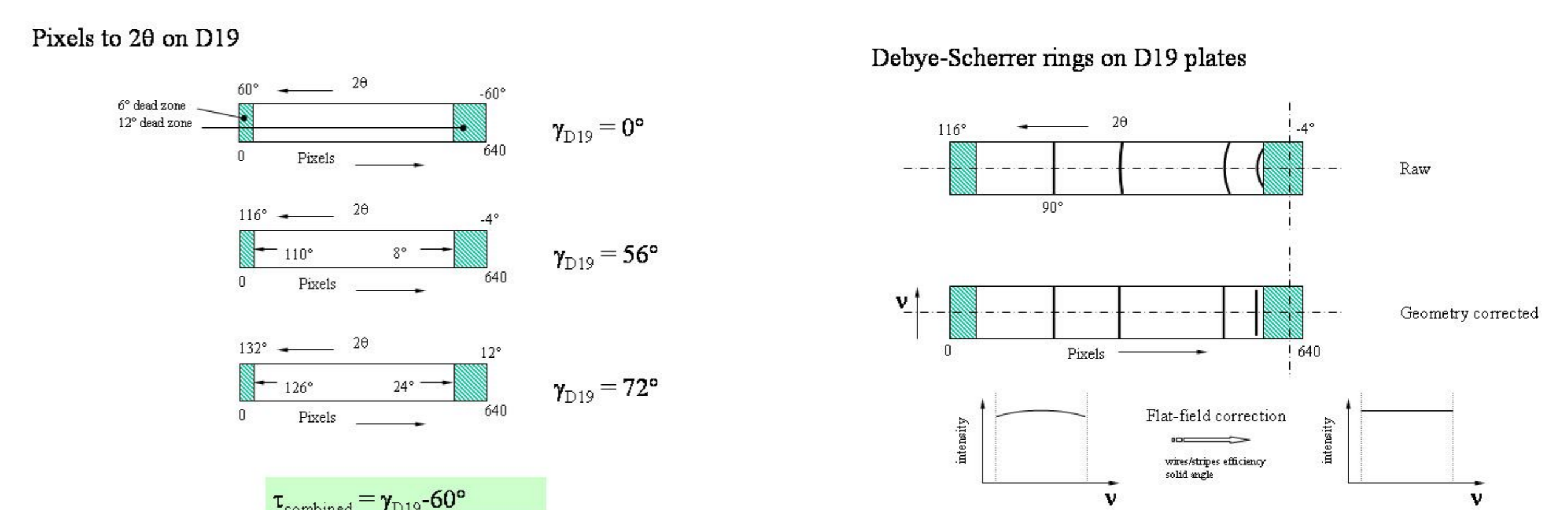
New scanning strategy on D19

The D19 instrument is equipped with a 120° curved position sensitive detector that encompasses 30° along the tilt angle (cf. Fig. Below), reducing the (χ, φ) texture scans nearly by a factor of 5. The φ rings (72 sample positions using a 5° resolution grid) are measured completely, but only 4 detector positions are needed to span the 90° in χ , resulting in only 288 measured points (vs 1368 on regular instruments) for the same resolution grid. Furthermore the grid resolution is a priori only limited by data binning.

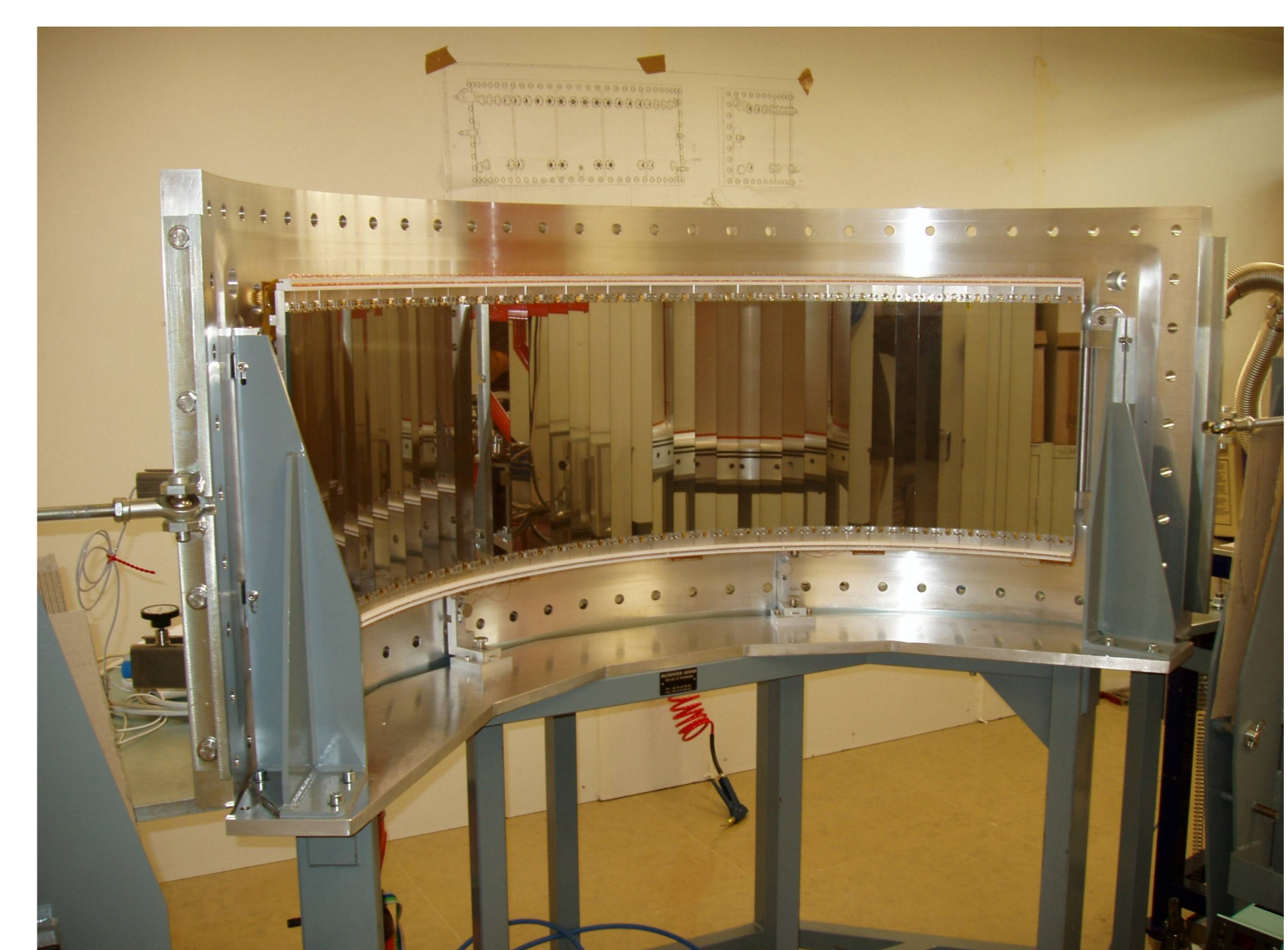


Data reduction

The Debye-Scherrer cones are corrected first for flatfield and solid-angle using a standard scatterer (V). The rings curvature are then developed, then binned to a desired resolution grid (5°). 2 θ - diagrams are obtained, equivalent to those of one dimensional CPS detectors. Simple angular relationships are obtained between coordinates of the D19 and other one dimensional detectors. Transformations from diffractometer space to pole figure space [3] are obtained using classical relations for CPS detectors. D-spacings obtained from the two instruments are in good agreement.



Dead zones and Debye-Scherrer rings.



D19 instrument 120° curved position sensitive detector that encompasses 30°.

Conclusions

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

References

- [1] H.-J. Bunge, C. Esling Ed.: Quantitative Texture Analysis, DGM, Germany, 1982, 450p.
- [2] D. Chateigner, L. Lutterotti, T. Hansen: Quantitative phase and texture analysis on ceramics-matrix composites using Rietveld texture analysis. ILL report 97 "Highlights": 1998, 28-29
- [3] D. Chateigner Ed.: Combined analysis: structure-texture-microstructure-phase-stresses-reflectivity analysis by x-ray and neutron scattering, 2004, 147p: <http://www.ecole.ensicaen.fr/~chateign/texture/combined.pdf>