Explorer

Texture analysis of polycrystalline materials

Introduction

Polycrystalline materials are constituted by a large amount of crystallites which, in the ideal case of powders, have random orientation with respect to a reference frame. Quite often, there is a departure from the ideal behavior, due to the presence of a preferred orientation of crystallites or texture: this results into an X-Ray diffraction pattern characterized by modified relative intensities with respect to the reference powder one. Thus, in order to perform structural and quantitative phase analysis of polycrystalline samples, texture modelling is required.

Texture can influence the macroscopic properties of materials, e.g. stress, strain and elastic constants, sound propagation, piezoelectric and pyroelectric coefficients and magnetic anisotropy. As a consequence, for several technological materials texture characterization is of paramount importance in order to tailor their functional properties and tune the corresponding manufacturing process[1].

Texture is described first by associating an orthogonal coordinate system to both the macroscopic sample and each crystallite. Then, three Euler angles (φ_1, ϕ_0, φ_2) define the rotations required to make the sample coordinate system match the crystallite one. Performing this operation for all crystallites results into an orientation distribution function (ODF), which describes how crystallite orientations are distributed in Euler space, i.e. the space of all possible Euler angle triads[2].

Unfortunately, what can be really measured by X-Ray Diffraction is a 2D projection of ODF, called Pole Figure (PF), which defines the probability that the normal to a diffracting plane (hkl) in crystallites is parallel to an arbitrary sample direction, representing the scattering vector in sample coordinate system. Usually, three or more (hkl) PFs are required in order to derive the ODF by means of mathematical methods, such as the Fourier analysis or the WIMV one[3].

In this note, texture analysis of orthorhombic and triclinic symmetry samples from Bonet company[4] is performed with the Schulz reflection method by using a Eulerian cradle mounted on Explorer diffractometer and Beartex software[5]. Results are compared with reference values provided by the sample manufacturer.

Summary

Polycrystalline materials are often characterized by a different degree of departure from the ideal random orientation of its constituent crystallites, i.e. texture. Beyond its role for correct quantitative phase analysis, texture also affects the functional properties of materials: thus, its estimation is of paramount importance in several technological fields. Explorer diffractometer, thanks to the new Eulerian cradle and Mythen Hybrid Photon Counting linear detector, allows to perform texture analysis in an accurate and straightforward way.

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[1] Nicolae C. Popa, Microstructural Properties: Texture and Macrostress Effects in *Powder Diffraction, Theory and Practice*, edited by R. E. Dinnebier S.J.L. Billinge, RSC Publishing.

[2] H.J. Bunge, Texture Analysis in Materials Science, Helga and Hans-Peter Bunge, Wolfrathausen 2015, free digital edition

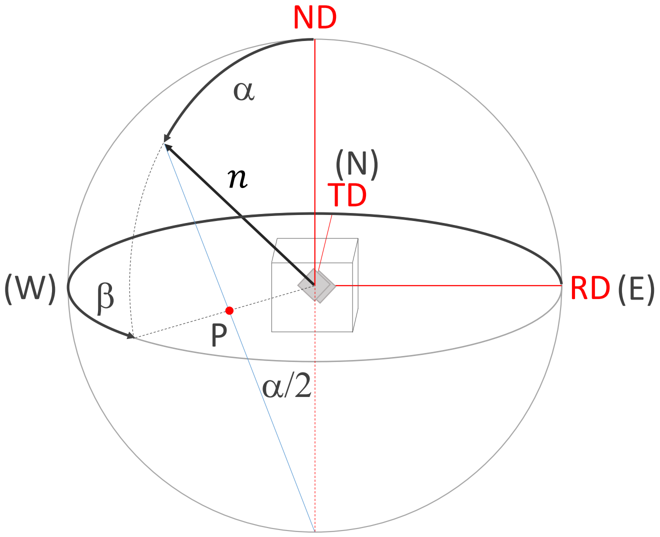
[3] Matthies, S., Wenk, H.-R. & Vinel, G. W. (1988). J. Appl. Cryst. 21, 285-304.

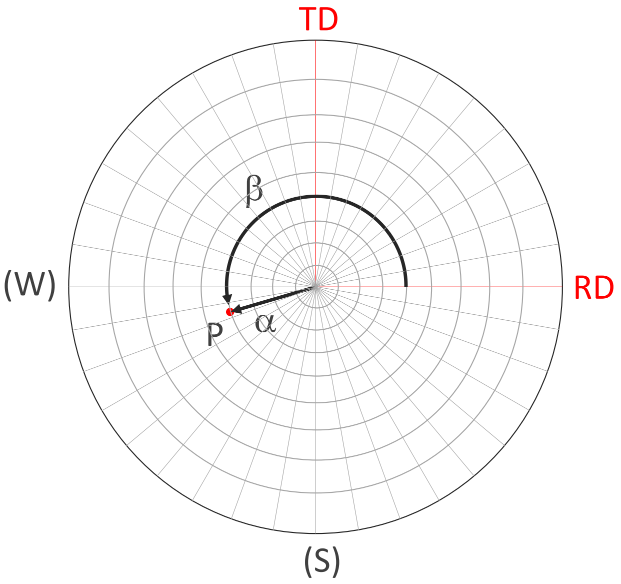
[4] http://www.labosoft.com.pl/texture_standards.html

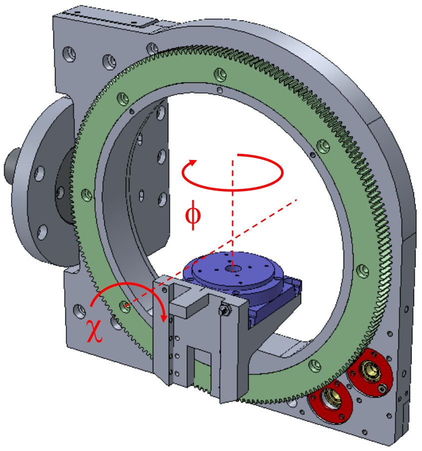
[5] http://eps.berkeley.edu/~wenk/TexturePage/beartex.htm#Introduction%20BEARTEX

Theoretical overview

Pole figures can be determined for a (hkl) diffracting plane by varying the orientation of the sample with respect to the X-Ray incident and diffracted beam, i.e. the scattering plane, so that crystallites having a certain orientation are set in diffracting condition for that hkl on the diffractometer. The top picture below shows the diffracting plane normal n (pole) of a crystallite with an orientation described by spherical coordinates α and β in the sample reference system. Usually, if there is a rolling direction (RD), that is taken as the zero value for β, while the normal direction (ND) for α. The picture at the bottom is the corresponding 2D stereographic projection, the PF.







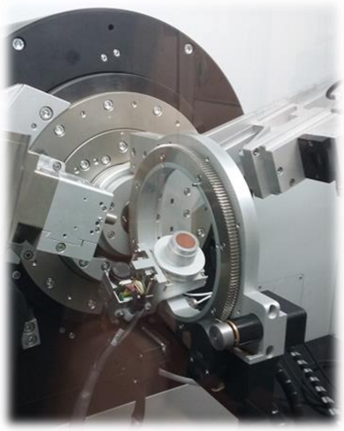
A Eulerian cradle allows to perform the required rotations to explore different α and β angles thanks to its ϕ and χ rotation axes. In order to minimize defocusing during χ tilting, a point focus X-Ray source with limited projection on the sample surface is required.

Product Specifications

Explorer is a Multi-Purpose Theta/Theta high resolution diffractometer which, thanks to its direct drive torque motors, offers top performances in many analytical areas, ranging from phase analysis to determination of microstructural properties on bulk or thin film materials.

Thanks to its modularity and the wide range of accessories and attachments available, Explorer allows to perform measurements in different configurations: traditional X-Ray Powder Diffraction (XRPD), Reflectometry (XRR), Grazing Incidence Diffraction (GID), High Resolution X-Ray Diffraction (HRXRD), Total Reflection X-Ray Fluorescence (TXRF), Residual Stress and Texture X-Ray Diffraction.

Explorer high resolution diffraction system incorporates the high efficiency of the direct drive torque motors controlled by optical encoders, allowing to reach an angular accuracy of 0.00001°.



The Eulerian cradle features motorized χ and ϕ rotation axes with a range from -90 to 90 deg and 0 to 360 deg, respectively. χ resolution is 0.02 deg, ϕ one better than 10^{-4} deg.

ϕ rotation stage is mounted on a motorized z stage in order to position correctly the sample surface with respect to incident beam. The z stage resolution is better than 0.005 mm.

The allowed maximum sample diameter is 80 mm, its height 20 mm while its max weight is up to 20 N (dedicated sample holder may be required).

Experimental

Configuration:	Theta-Theta	Detector:	MYTHEN2 R 1D
Goniometer radius [mm]:	240	Active area[°]:	4
X-Ray Source:	Cu FF, Point Focus	Acquisition mode:	single snapshot
Power settings[kV, mA]	40, 35	χ (range, step) [deg]:	0-80, 5
Monocapillary collimator		ϕ (range, step) [deg]:	0-355, 5
diameter[mm]:	0.7	Reflections:	111, 200, 220
length [mm]:	80.7	Single PF time [h]:	2.5
Detector slit [mm]	12		
Filter[mm]:	Nickel 0.02		

Bonet reference standard set consists of one powdered and two massive samples prepared from commercial purity copper in form of metal disks, 27 mm in diameter and about 6 mm thick.

The measurable surface is 20 mm in diameter.

The powder sample is provided for the defocusing correction calculation at different χ angles: this is then applied to intensities of the experimental pole figure of the massive samples.

First, the diffraction pattern was collected for all the samples, in order to check for interfering phases.

It is important that reflections of interest are well separated from other peaks: if there is overlap, then the contribution of the interfering peak must be estimated and subtracted.

The peak belonging to reflections of interest was collected for each χ and ϕ in a static mode, i.e. by taking a 4 degree frame with the linear detector.

Results

The diffraction patterns for the 3 samples are reported in figure 1: it is apparent that peak relative intensities are quite different among them, especially the orthorhombic one, where 220 peak is much larger. No interference between peaks is present.

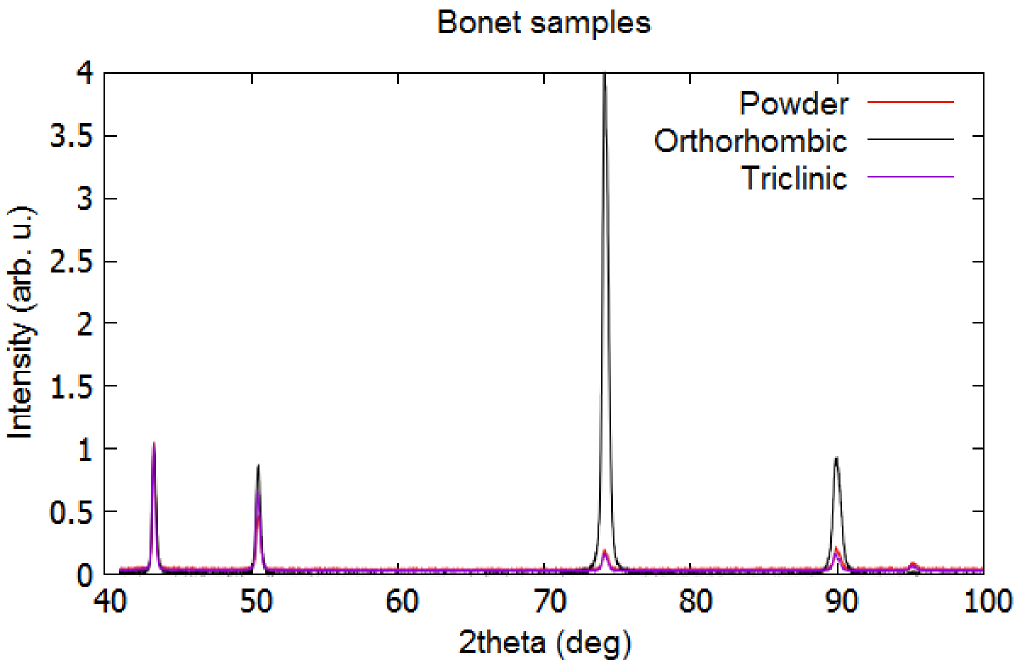


Figure 1. Diffraction patterns for Bonet reference samples at $\alpha=\beta=0$. Intensity is normalized to 111 peak for comparison.

Pole figures for 111, 200 and 220 reflections are reported in figure 2 and 3 for orthorhombic and triclinic sample, respectively: beside the experimental-defocusing corrected PFs, there are the recalculated ones, i.e. those corresponding to the ODF determined by WIMV algorithm. From a qualitative point of view, the more the experimental and recalculated PF look similar, the better the ODF computation is. As can be seen, the experimental PF has missing β circles, due to the impossibility of collecting data at $\chi(\alpha)$ close to 90 deg in the Schulz reflection geometry. Nevertheless, the recalculated one is complete.
From a quantitative point of view, according to Bunge, the texture index quantifies the texture strength:

$$J = \int \left(f(g)\right)^2 dg \qquad J \in [1, \infty[$$

where f is the ODF and g the orientation in Euler space. A value equal to 1 corresponds to powders (no texture) and ∞ to a perfect single crystal. In order to estimate the goodness of analysis, the compatibility between the analyzed sample texture and the declared one by the manufacturer has to be checked. According to the Tarasiuk et al.[6] linear regression can be used to compare different textures.
Experimental PFs of both samples were thus symmetrized to orthorhombic and the resulting ODFs compared with the original ones. The resulting linear regression correlation coefficient R is the measure of compatibility: if R>0.9 two textures are compatible, if R<0.9 they are significantly different. Results for the current analysis are reported in table 1 and in figure 4: the measured orthorhombic sample is compatible with the orthorhombic texture declared by the manufacturer, while the triclinic not, as expected.

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[6] Bonet reference manual and Tarasiuk, J., Wierzbanowski, K. (1996). Phil. Mag. A 73, 1083-1091

ODF can be visualized in Cartesian coordinates by means of one Euler angle sections: figure 5 shows some γ sections for orthorhombic sample (Roe/Matthies convention).

Table 1. Results for Bonet copper samples.

Sample	Texture index J	Calculated correlation coefficient –orthorhombic symmetrization
Orthorhombic	5.34	0.97
Triclinic	1.92	0.47

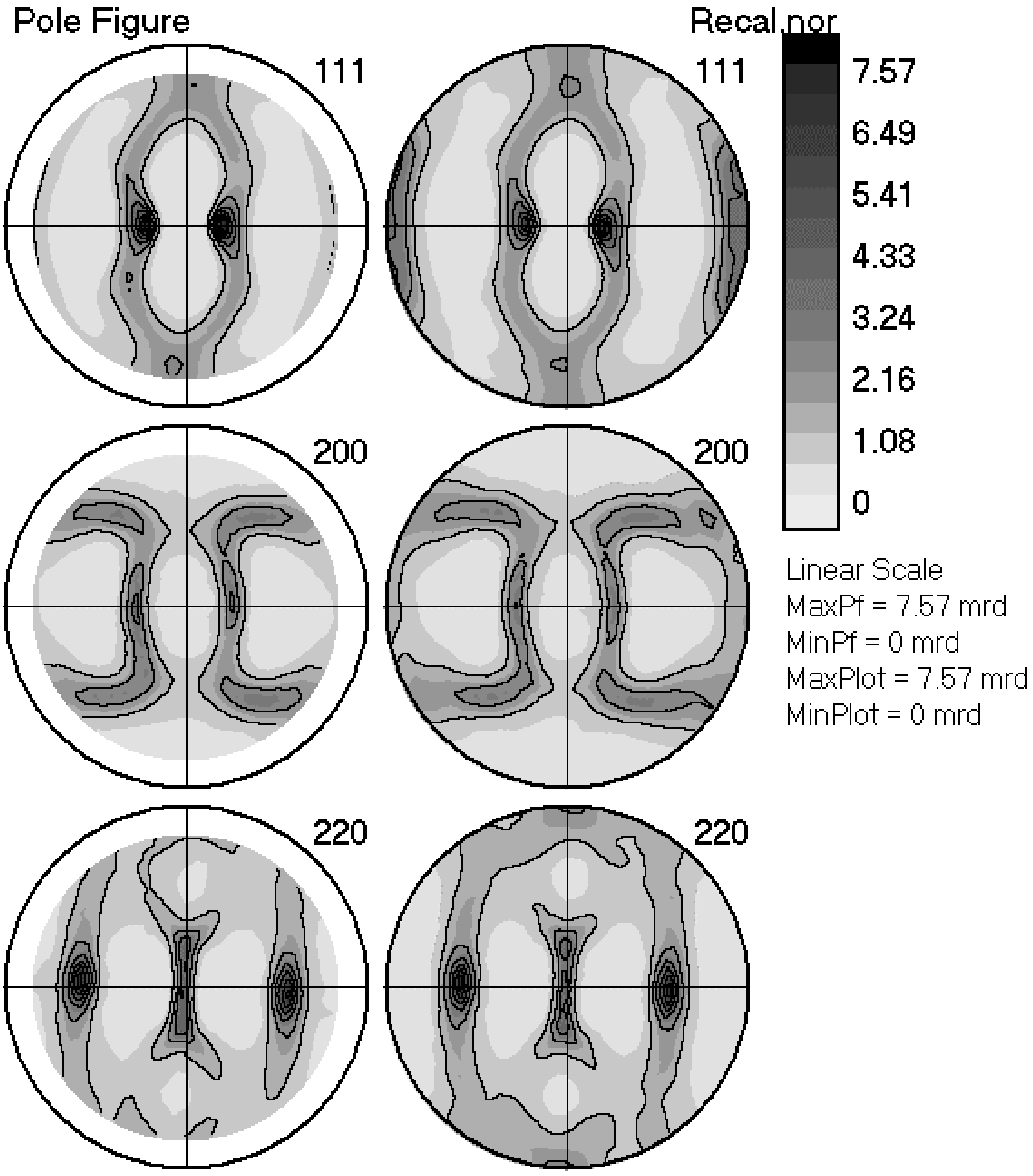

Figure 2. Orthorhombic experimental and recalculated PFs. RD is on the right side (East), while TD at the top (North). β runs counterclockwise. Note that the PF can be rotated, if necessary.

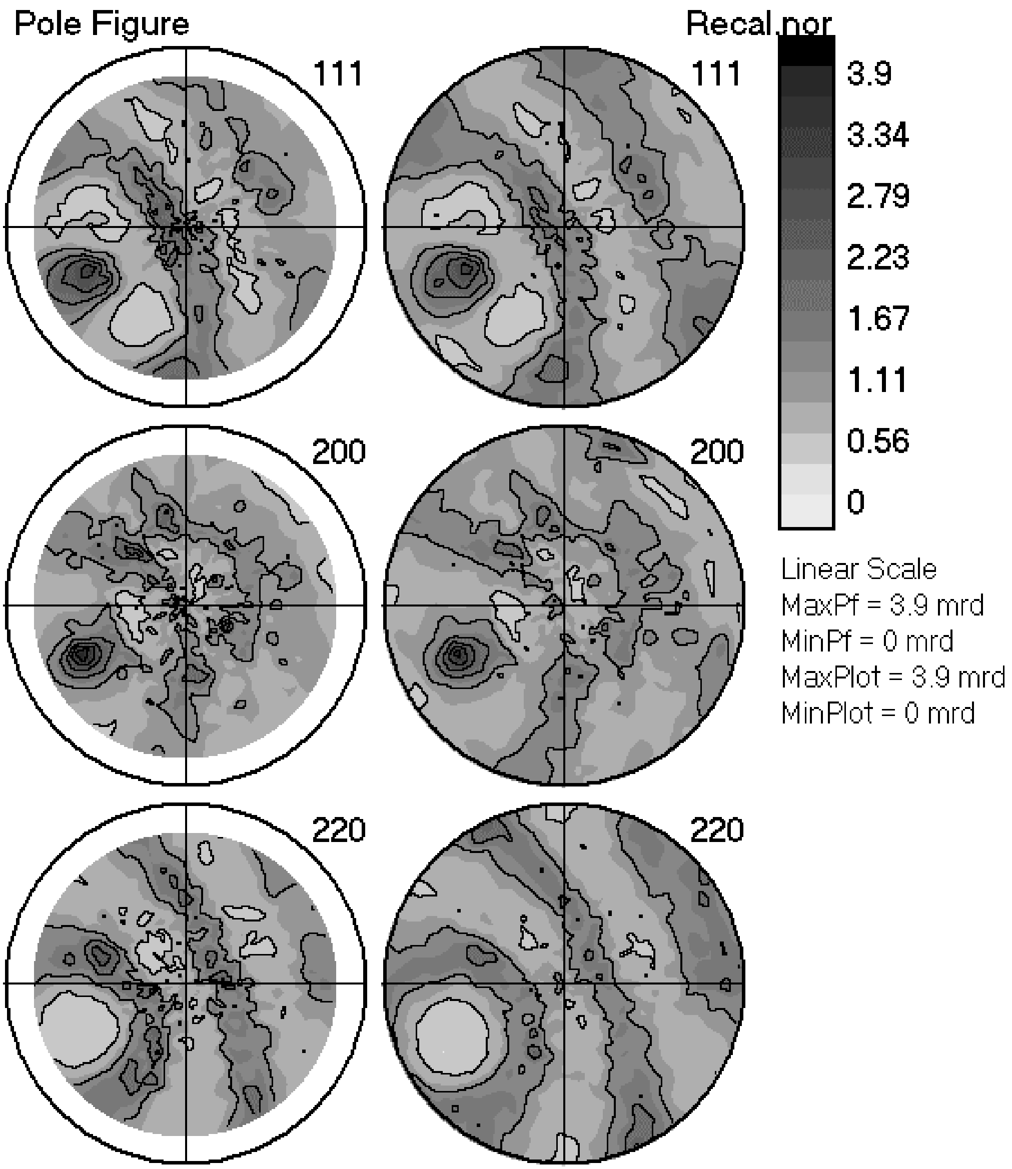

Figure 3. Orthorhombic experimental and recalculated PFs. RD is on the right side (East), while TD at the top (North). β runs counterclockwise. Note that the PF can be rotated, if necessary.

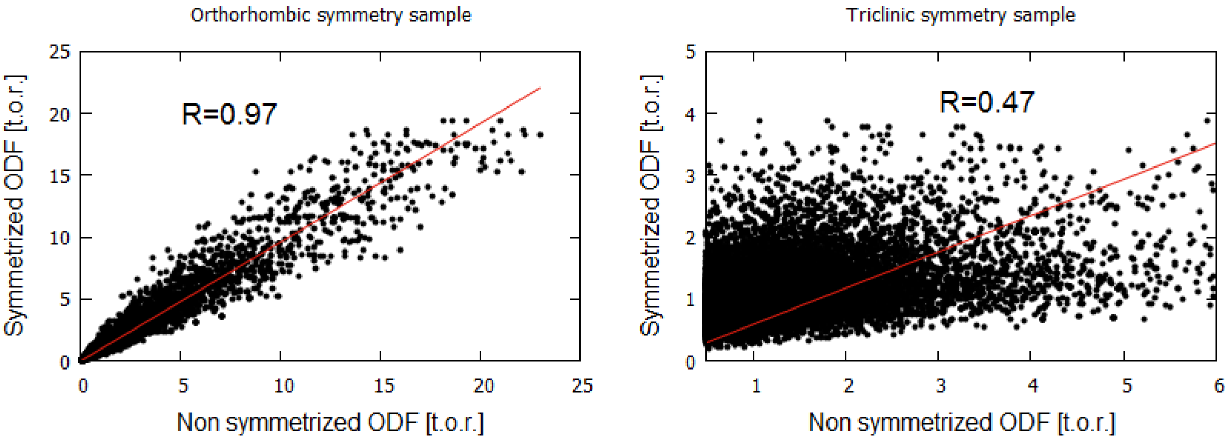

Figure 4.Linear regression of orthorhombic-symmetrized and not-symmetrized PF ODFs.

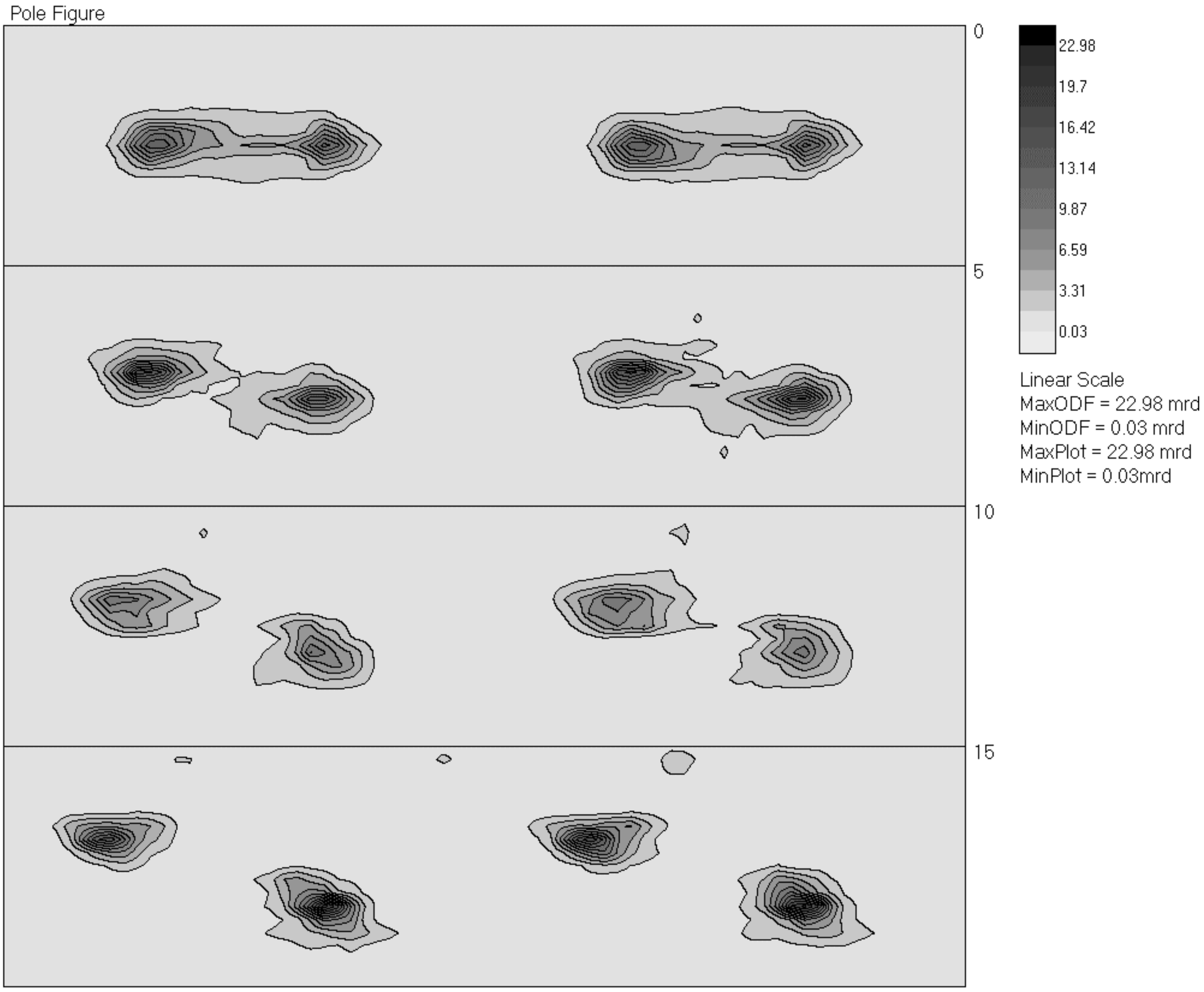

Figure 5. Example of calculated ODF γ sections for orthorhombic sample. Note that the Roe/Matthies convention for Euler angles is used in Beartex (α, β, γ).

Conclusions

Explorer equipped with a Eulerian cradle allowed to perform successfully texture analysis on reference samples from Bonet company.

The texture index and linear regression coefficient between symmetrized and non-symmetrized Pole figures are in good agreement with declared values, according to manufacturer established procedures.

Authors

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GNR is present on the main international markets through an efficient and motivated technical and commercial network, able to provide outstanding support for any customer requirements.