Pergamon

Journal of Structural Geology, Vol. 17, No. 6, pp. 923 to 924, 1995

0191–8141/95 $9.50 + 0.00

0191–8141(95)00019–4

High-resolution X-ray texture goniometry: Discussion

MANUEL SINTUBIN

Laboratorium voor Algemene Geologie, Katholieke Universiteit Leuven, Redingenstraat 16, B-3000 Leuven, Belgium

(*Received 21 September 1994; accepted 30 January 1995*)

In their short note in the *Journal of Structural Geology* van der Pluijm *et al.* (1994) claim that for high-resolution X-ray texture goniometry the use of a single-crystal diffractometer has to be preferred to that of a standard diffractometer. For their demonstration these authors use texture measurements on a graded slate (Martinsburg Formation, Pennsylvania). Their argumentation is primarily based on instrumental characteristics of a single-crystal diffractometer. Both the narrow collimation and the low absorption Mo X-ray source allow the analysis of small sampling areas (<1 mm^2). The Mo-radiation moreover enables the analysis of 200 μm-thick sample sections, increasing the irradiated volume over which is measured. The possibility of fully automated, computer-controlled data collection is also considered characteristic for a single-crystal diffractometer. Furthermore, in processing the raw texture data these authors chose the application of a calculated absorption correction. Finally, they consider that incomplete pole figures sufficiently represent the phyllosilicate orientation distribution to be applied in the subsequent kinematic interpretation, based on the March (1932) model.

In our laboratory at Leuven, Belgium, a standard X-ray goniometer has been gradually optimized since 1986 to perform texture analysis in transmission geometry, specifically on shales, slates and other argillaceous rocks (Sintubin 1992, 1994a,b,c). This optimization is primarily the result of an integration of the expertise of Prof. P. Van Houtte (K. U. Leuven), Prof. H.-R. Wenk (UC Berkeley), and Prof. G. Oertel (UC Los Angeles), communicated to me personally or through their publications (e.g. Oertel 1983, Wenk 1985).

In this Discussion I would like to demonstrate, by comparing our equipment with that of van der Pluijm *et al.* (1994), that high-resolution can very well be achieved with a standard X-ray texture goniometer. On the other hand a comparison of the processing procedures and a discussion on the pole figures, presented by van der Pluijm *et al.* (1994), will show that not the instrumentation but rather the processing procedure is crucial for the proper application of phyllosilicate textures in a quantitative strain analysis. The achievement of high-resolution is in this respect of lesser importance.

The standard X-ray texture goniometer used in our laboratory has been gradually optimized for texture measurements in transmission geometry. The application of separate θ–2θ motors allows a fully automated, computer-controlled data collection. The intensity data are collected over a tilt of 40° on nine separate circles, each 5° apart. On each circle the intensity is measured over circle segments of 3.75°, resulting in 96 measurements per circle. For every sample section 864 intensity data are thus obtained. The collimation has furthermore been modified, so that the irradiated surface does not exceeds 1 mm^2. Sample sections with a thickness of 100 μm (cf. Oertel 1983) are used. Texture measurements are performed using Ni-filtered Co-radiation (40 kV × 30 mA). The Co-radiation is preferred to the commonly used Cu-radiation, first because of limited Fe-fluorescence, which can be a nuisance in the analysis of argillaceous rocks, and secondly because of a higher wavelength, giving rise to higher 2θ-angles and less overlap between neighbouring diffraction peaks.

This optimized, fully automated, standard X-ray texture goniometer has identical advantages as the equipment, described by van der Pluijm *et al.* (1994), except for the low absorption Mo-radiation, enabling measurements over larger sample volumes. However, this increased irradiated volume seems to be in contradiction with the effort to achieve high resolution by minimizing the irradiated surface. Thicker sample sections indeed imply an increased chance to encounter heterogeneities, which may have a negative effect on the resolution. A supplementary disadvantage of Mo-radiation, especially in the case of phyllosilicates, is its low wavelength, which implies that diffraction peaks of phyllosilicates are at very low 2θ-angles (<5°), and that the chance of overlapping peaks increases significantly. Mo-radiation could definitively not be used in our standard X-ray texture goniometer because of the high background intensity at low angles.

Sample preparation and positioning is identical in both our procedure and that of van der Pluijm *et al.* (1994). In processing the raw intensity data a different procedure is however followed, although apparently the same software package (provided by Prof. H.-R. Wenk) is used. Contrary to van der Pluijm *et al.* (1994), the corrections applied in our case are empirically determined using a method similar to that described by O'Brien *et al.* (1987) (Sintubin 1992). First, a tilt-corrected background subtraction, which is absent in the procedure of van der Pluijm *et al.* (1994), is performed. In our case this corrected background subtraction is even sufficient to take into account the effects of increasing absorption and irradiated volume. Any further correction, applying the empirically determined correction curves, leads to an overcorrection, resulting in distorted pole figures. Such an overcorrection can be found in the pole figures, obtained by van der Pluijm *et al.* (1994). This overcorrection is expressed by the girdle aspect of the pole figures with a mirror plane at 0° tilt (= vertical plane in fig. 2). This is especially obvious in the chlorite textures. Such a distortion of the pole figures also has its consequences for the subsequent application of the March (1932) model. The exaggerated ellipticity of the pole figure, which is due to the overcorrection, results in incorrect March strains, and subsequently incorrect k_M- and d_M-values. The resulting Flinn plot, and subsequent kinematic interpretation, is therefore very contestable.

A second difference in processing procedure concerns the question whether or not incomplete pole figures are sufficiently representative for the phyllosilicate orientation distribution. In our case we do aim at obtaining complete pole figures by combining measurements on two perpendicular sample sections. Such a combination allows on the one hand a supplementary removal of any remaining tilting effect. On the other hand, a normalization only seems feasible on a complete pole figure. The zero-intensity regions in an incomplete pole figure distort the normalization, giving rise to overestimated texture peak intensities.

Summarizing, it seems that the only possible advantage of the equipment of van der Pluijm *et al.* (1994) is the application of a low absorption Mo X-ray source, enabling the analysis of 200 μm-thick sample sections. Whether or not increasing the irradiated volume by means of thicker samples and decreasing the irradiated surface by means of a finer collimation has contrasting effects on the resolution remains however a matter of confusion. In this respect the effort of van der Pluijm *et al.* (1994) to minimize the irradiated surface to achieve high resolution has definitively to be evaluated with regard to the effort of Lipshie *et al.* (1976) to integrate over a larger area by translating the sample to improve counting statistics. Furthermore, computer-controlled data collection and the achievement of high-resolution are equally feasible with an optimized standard X-ray texture goniometer. Finally, processing and subsequent interpretation of the texture data by van der Pluijm *et al.* (1994) show some imperfections. The absorption correction applied leads to an overcorrection. This overcorrection, the absence of any background subtraction and the use of incomplete pole figures make their application of the March model rather contestable. The resulting pole figures and subsequent kinematic interpretation clearly show that the way in which texture data are obtained, with or without high-resolution equipment, is not as crucial in the evaluation of phyllosilicate textures as the way the texture data are processed. The latter has definitively to be emphasized the most when applying phyllosilicate textures in a quantitative strain analysis.

REFERENCES

Lipshie, S. R., Oertel, G. & Christie, J. M. 1976. Measurement of preferred orientation of phyllosilicates in schists. *Tectonophysics* **34**, 91–99.

March, A. 1932. Mathematische Theorie der Regelung nach der Korngestalt bei affiner Deformation. *Z. Kristallogr.* **81**, 285–297.

O'Brien, D., Wenk, H.-R., Ratschbacher, L. & You, Z. 1987. Preferred orientation of phyllosilicates in phyllonites and ultramylonites. *J. Struct. Geol.* **9**, 719–730.

Oertel, G. 1983. The relationship of strain and preferred orientation of phyllosilicate grains in rocks — a review. *Tectonophysics* **100**, 413–447.

Sintubin, M. 1992. De kristallografische voorkeursoriëntatie van phyllosilicaten als vervormingselement in de structurele geologie. Unpublished Ph.D. Thesis, Katholieke Universiteit Leuven.

Sintubin, M. 1994a. Structural and paleogeographic inferences from a texture analysis on Ordovician and Silurian pelites of the Wépion borehole (Ardennes, Belgium). *Geologie Mijnb.* **92**, 305–310.

Sintubin, M. 1994b. Texture types in shales and slates. In: *Textures of Geological Materials* (edited by Bunge, H. J., Siegesmund, S., Skrotski, W. & Weber, K.). DGM Informationsgesellschaft Verlag, 221–229.

Sintubin, M. 1994c. Clay textures in relation to the burial history of shales. *Sedimentology* **41**, 1161–1169.

van der Pluijm, B. A., Ho, N. C. & Peacor, D. R. 1994. High-resolution X-ray texture goniometry. *J. Struct. Geol.* **16**, 1029–1032.

Wenk, H.-R. 1985. *Preferred Orientation in Deformed Metals and Rocks: An Introduction to Modern Texture Analysis.* Academic Press, Orlando.