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High-resolution X-ray texture goniometry: Reply

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We gladly use this opportunity offered by Sintubin's discussion to clarify some aspects of our work on high-resolution X-ray texture goniometry and respond to some misunderstandings. Four points can be identified in Sintubin's comment, which we address separately below. However, at the onset we wish to point out that our intention was and still is only to present a relatively simple, yet high-quality method to obtain accurately determined textures from small sample areas using a basic single-crystal X-ray goniometer fitted with an inexpensive and easy to manufacture stage.

Preference for a single crystal diffractometer

In our paper we discussed the advantages of a single-crystal diffractometer (SCD) over a *standard* powder diffractometer (SPD) for the purposes of analyzing small areas. Briefly, in a SCD a parallel, concentrated X-ray beam leaves the collimator ('exit point'), whereas in a SPD the beam is divergent. Thus, in order to restrict the radiated area of the sample using a SPD, a large proportion of the X-rays are blocked. This produces a much lower intensity and hence a low signal-to-background ratio (i.e. low precision), which creates the need to integrate over larger areas. In contrast, a SCD configuration produces high intensities for a small-diameter beam, and thus excellent counting statistics for small sample areas. Note that the Mo source provides a further advantage given its low absorption, but that a standard Cu source can also be used.

Computer-controlled operation

In our paper, we merely noted that the mechanics of a SCD make it easy to control the sample orientation with the three motors and company software that are standard with the equipment, essentially without modification. A SPD requires addition of extra motors (e.g. following the design of Oertel) and dedicated software to drive the stage.

Inferred overcorrection

For correction of the raw data we first subtract the background (as queried by Sintubin) and then apply absorption and volume corrections. Background correction is, of course, standard in all procedures, which we neglected to mention specifically. However, subsequent corrections do not lead to overcorrection as implied by Sintubin. To illustrate this point, we show X-ray pole figures for mica in a slate (Fig. 1a), the same pattern without rotation (Fig. 1b), and for a compacted mudstone (Fig. 1c). If overcorrection had occurred, the contours should follow symmetrical, small-circle trajectories in zero tilt, rather than show the elongate, but non-symmetrical pattern in Fig. 1(a), and the circular pattern in Fig. 1(c) (expected for a mudstone). We show the (circular) pattern from a mudstone sample because it would be particularly sensitive to overcorrection. Moreover, we calibrated our patterns by obtaining duplicate pole figures on several samples at UC Berkeley (Prof. H.-R. Wenk's laboratory; see Fig. 1d). Thus the inference that our patterns produce exaggerated March strains because of overcorrection is incorrect.

Incomplete pole figures

All pole figure methods from a single sample in a single orientation produce incomplete figures, whether they operate in transmission or reflection mode, or utilize a SCD or a SPD. Indeed, a reflection mode stage is available to us, but one reason for building a device that utilizes transmission geometry is that it provides an increase in the proportion of angular space for which measurements are made. Moreover, the Mo source allows us to cover a particularly large portion of the hemisphere by tilting of the sample. Complete pole figures can be obtained by analyzing more than one surface of a sample, but it is generally impossible to obtain patterns from exactly the same region when very small volumes are concerned. The two or more patterns are combined to produce a complete figure by superimposing these fabrics and equalizing the areas of overlap (this is a 'correction' that is not mentioned in Sintubin's comment, but probably is the reason that corrections beyond background are considered not necessary by Sintubin). We counsel against this approach because of the intrinsic heterogeneity of most geologic samples, as demonstrated for a slate in our original paper (van der

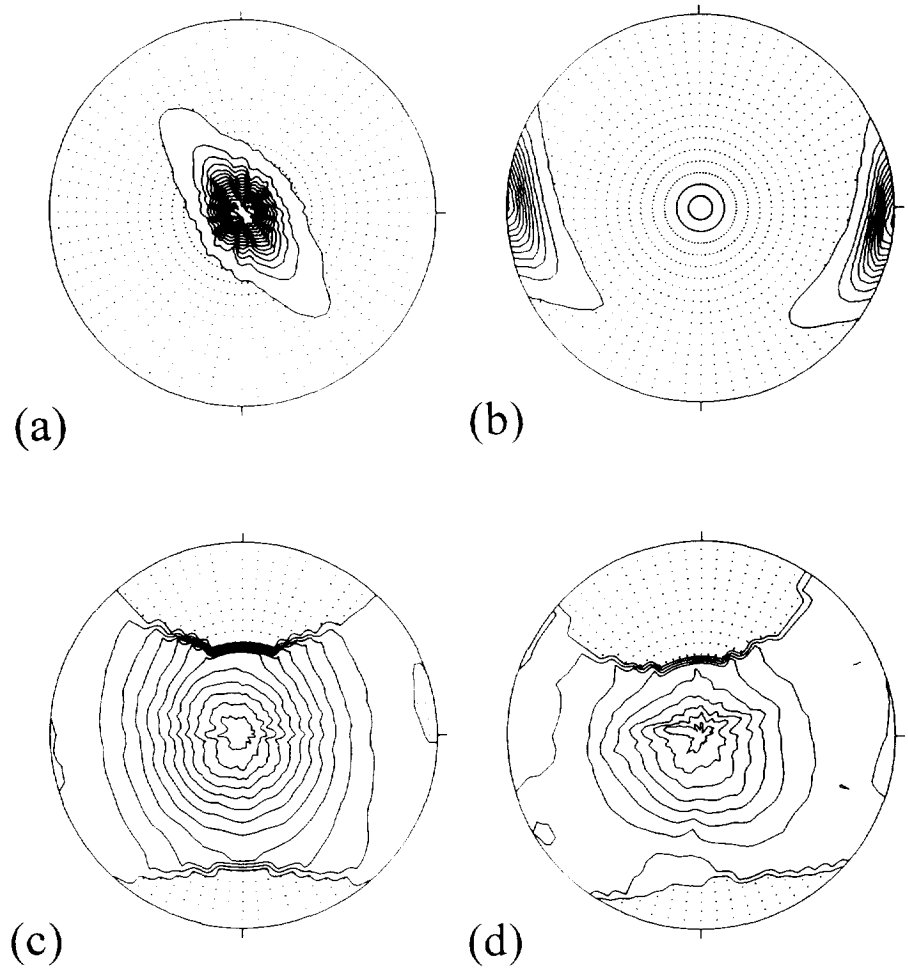

Fig. 1. X-ray pole figure for mica in a slate (a) and the same pattern relative to zero-tilt coordinates (b); the corresponding March strain, Z_M, is 0.31 (Quartenschiefer). In (c) the figure for a compacted mudstone is shown ($Z_M = 0.60$), which is indistinguishable from the pattern run at UC Berkeley (d; $Z_M = 0.63$) that was obtained for initial calibration of our set up (Gulf Coast sediment). The contour interval in (a) & (b) is 1.0 m.r.d., and 0.4 m.r.d. in (c) & (d).

Pluijm *et al.* 1994), and the empirical nature of the overlap correction.

Given the above relations, we remain somewhat nonplussed that Sintubin (1994) has chosen to criticize our approach by comparison with his own. Each of the various methods, SCD vs SPD, transmission mode vs reflection mode, has relative advantages and disadvantages. We again emphasize that our purpose was only to point out the advantages (high accuracy and small areas) of our newly designed method, and not to disparage alternative techniques, in the hope that others who have equivalent SCDs could make use of our improvements.

Finally, we could not have been aware of Sintubin's 1994 publications when writing our paper in 1993, and were also unaware of his 1992 dissertation, which may contain much pertinent information. (Even though one of us is somewhat familiar with the Dutch language, we do not have ready access to unpublished Ph.D. theses from Belgium.) In any case, it seems that the SPD procedures described in Sintubin's Discussion are not very different from those designed previously by Profs Oertel (UCLA) and Wenk (UC Berkeley), but they are quite different from our SCD system. Currently, we are in the process of completing a second-generation stage that will also allow us to measure *in situ* illite crystallinity of phyllosilicate fabrics in addition to quantifying these discrete preferred orientation fabrics.

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