A comparison between the fabric camera and texture goniometer methods of fabric analysis by X-ray diffraction

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With 6 figures

Zusammenfassung

Der Einfluß der Korngröße auf die Textur-Analyse durch Röntgen-Diffraktion wird mit Hilfe des Einsatzes der Textur-Kamera (fabric camera) und des Textur-Goniometers diskutiert. Einfache Berechnungen zeigen die wechselseitige Abhängigkeit zwischen dem mittleren Korndurchmesser und der Anzahl der Kristalle, die durch jedes Gerät vermessen wurden. Die Einflüsse der Korngröße und der modalen Zusammensetzung der Probe werden durch vergleichende Analysen dargestellt. Sowohl die Textur-Kamera als auch das Textur-Goniometer sind geeignet für die Analyse von Proben, deren mittlere Korngröße zwischen ca. 10 und 100 Mikron liegt. Für Korngrößen unterhalb dieser Werte ist das Textur-Goniometer besser geeignet, für gröbere Körner dagegen die Textur-Kamera. besonders dann, wenn die Probe nicht monomineralisch zusammengesetzt ist.

Abstract

The effects of grain size on the analysis of fabrics by X-ray diffraction using both the fabric camera and the texture goniometer are discussed. Simple calculations show the interdependance between the mean grain diameter and the number of crystals which are measured by each instrument. The effects of both grain size and modal composition of the specimen are illustrated by comparative analyses.

Both the fabric camera and texture goniometer are well suited to the analysis of specimens in which the mean grain diameter is between approximately 10 and 100 microns. The texture goniometer is more suitable when the grain size is less than this, while the fabric camera is better suited when the grain size is coarser, especially when the specimen is not monomineralic.

Résumé

Les effets de la dimension des grains dans l'analyse des structures par diffraction des R. X. pour lesquelles on utilise des chambres photographiques pour textures et des goniamètres texturaux font ici l'objet d'une discussion. Des calculs simples montrent l'interdépendance entre le diamètre moyen des grains et le nombre de cristaux mesurés par chaque instrument. Les effets de chaque dimension de grain comme aussi de la composition modale de l'échantillon sont mis en relief par des analyses comparatives.

Ces chambres photographiques et goniamètres sont tous deux bien adaptés pour des analyses d'échantillons dans lesquels le diamètre moyen des grains varie approximativement entre 10 et 100 microns. Le goniomètre textural est mieux adapté quand la dimension du grain est plus faible, tandis que la chambre photographique l'est davantage pour les granularités plus grossières, spécialement quand l'échantillon n'est pas monominéralique.

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Краткое содержание

С помощью камеры (fabric camera) и гониометра (Textur-Goniometer) изучали влияние размерности минералов на результаты исследования текстуры методом диффракции рентгеновских лучей. Простые рассчеты показывают, что имеется взаимосвязь между средним поперечником зерна и количеством кристаллов, которые измеряли этими методами. Сравнили влияние величины зерна и модального состава проб на результаты анализа. Как камера, так и гониометр применимы для анализа проб, средняя зернистость которых лежит в проделах от 10 до 100 микрон. Для зернистости ниже этих величин подходит анализ с помощью гониометра, а для более грубых-камера, в особенности, если проба состоит не из мономинеральной фракции.

Introduction

The determination of crystal orientations in polycrystalline aggregates by X-ray diffraction has become well established since the pioneering work by Sander and Sachs in 1930. Two techniques are available. The fabric camera has been described by Starkey (1964 a, 1964 b, 1977) while methods based on the texture goniometer have been described by Decker, Asp and Harker (1948), Schulz (1949 a, 1949 b) and Gehlen (1960). Reference should be made to these authors for details of the techniques. The following discussion is concerned mainly with the constraints imposed by the specimen on the application of the two methods.

The Fabric Camera

The fabric camera permits the determination of the orientation of selected crystal planes in a standard petrographic thin section. The specimen can therefore be examined first with the optical microscope to select an area for study. It is also possible to prepare the specimen by either mechanically or chemically removing undesired parts of it (Kerrich and Starkey, 1979); this can be of importance in eliminating interfering diffraction from adjacent Bragg reflections.

Using a one millimeter diameter collimator, the irradiated area of the specimen is approximately three square millimeters. The area varies only slightly with the Bragg angle of the reflection being measured so that all the measurements are made essentially on the same sample of crystals. The selected Bragg reflections from all the crystals within the irradiated area are recorded, thus a sample of 100 crystals is measured if the mean grain diameter is approximately 170 microns, assuming that the specimen is monomineralic. However, the specimen can be scanned over a distance of ten millimeters and this results in a ten-fold increase in the irradiated area. Furthermore, the Bragg reflections from several scans, or even from several different thin sections, can be accumulated on a single film. There is, therefore, no upper limit to the size of crystals which can be measured. Even if a thin section contains only one crystal its orientation can be determined, since all the selected Bragg reflections are recorded on the film.

The use of film does, however, have a serious disadvantage in that the inherently low sensitivity of X-ray film places limits on the finest grained specimens which can be studied. This will be demonstrated below. However, the use of film does produce photographs which can be interpreted immediately on inspection as pole figures on a cylindrical projection. For quantitative analysis the films are digitized using an X-Y recording microdensitometer and a computer is used to generate pole figures based on the Lambert Equal Area spherical projection.

The Texture Goniometer

Most researchers who use the texture goniometer examine slabs of the specimen which can be considered infinitely thick for the purpose of applying X-ray absorption corrections. A second, thinner slab is used to obtain data by transmission of the X-rays through the specimen. Such an analysis therefore combines data from two completely different samples of the specimen. Using the fully automated texture goniometer at the E. T. H. Zurich it has been possible to obtain satisfactory results from a single, standard petrographic thin section, thus the investigated volume of the specimen remains more nearly constant. The use of a thin section is also desirable from the point of view of being able to observe and prepare the sample as described above.

With a one millimeter collimator the irradiated area of the specimen varies from approximately three square millimeters to a little more than one square millimeter depending on whether reflected or transmitted data are being recorded. The precise area also depends on the Bragg angle of the reflection being measured. Hence, even if a single thin section is used in the analysis the samples of crystals investigated in reflection and transmission are significantly different. Assuming an average value of two square millimeters, a sample of 100 crystals will be irradiated if the mean grain diameter is approximately 140 microns, for a monomineralic specimen. The specimen can be oscillated over ten millimeters; this increases the irradiated area to about twenty square millimeters. Thus, even if the mean grain diameter is approximately 450 microns, 100 crystals will be irradiated. This represents the upper limit because the texture goniometer collects data synchronously and therefore data cannot be accumulated over several samples of the specimen as with the fabric camera. Any increase in the grain size results in fewer crystals being irradiated.

The number of crystals measured is considerably less than the number irradiated. This is because the detector is moved incrementally from one measuring position to the next and the intervening data are lost. Where the increments are 5°, even if the acceptance angle of the detector is set unusually wide at 4°, more than half of the data are lost and therefore the effective mean diameter which would yield a sample of 100 measured crystals from an area of 20 square millimeters is approximately 300 microns. The finest grained sample which can be analysed is limited only by the sensitivity of available detectors.

The final pole figure is derived by combining the reflected and transmitted data using a computer. It is therefore a composite of data obtained from different samples of crystals as noted above.

Comparison of Analyses

Specimen 7816 is a calcite mylonite from the Morcles nappe in the Swiss Alps. Figure la is a fabric camera photograph of the r = 10.4 Bragg reflection, the indices used here are Miller-Bravais indices based on a hexagonal unit cell with c = 17 Å and a = 5 Å. Figure 1 b shows the pole figure derived from the photograph and figure 1 c shows the pole figure calculated from texture goniometer data. The two pole figures are very similar.

Pole figures for c = {00.1}, a = {11.0}, e = {01.8}, f = {01.2}, h = {20.2}, {11.6} and {11.3} were also determined satisfactorily using the texture goniometer

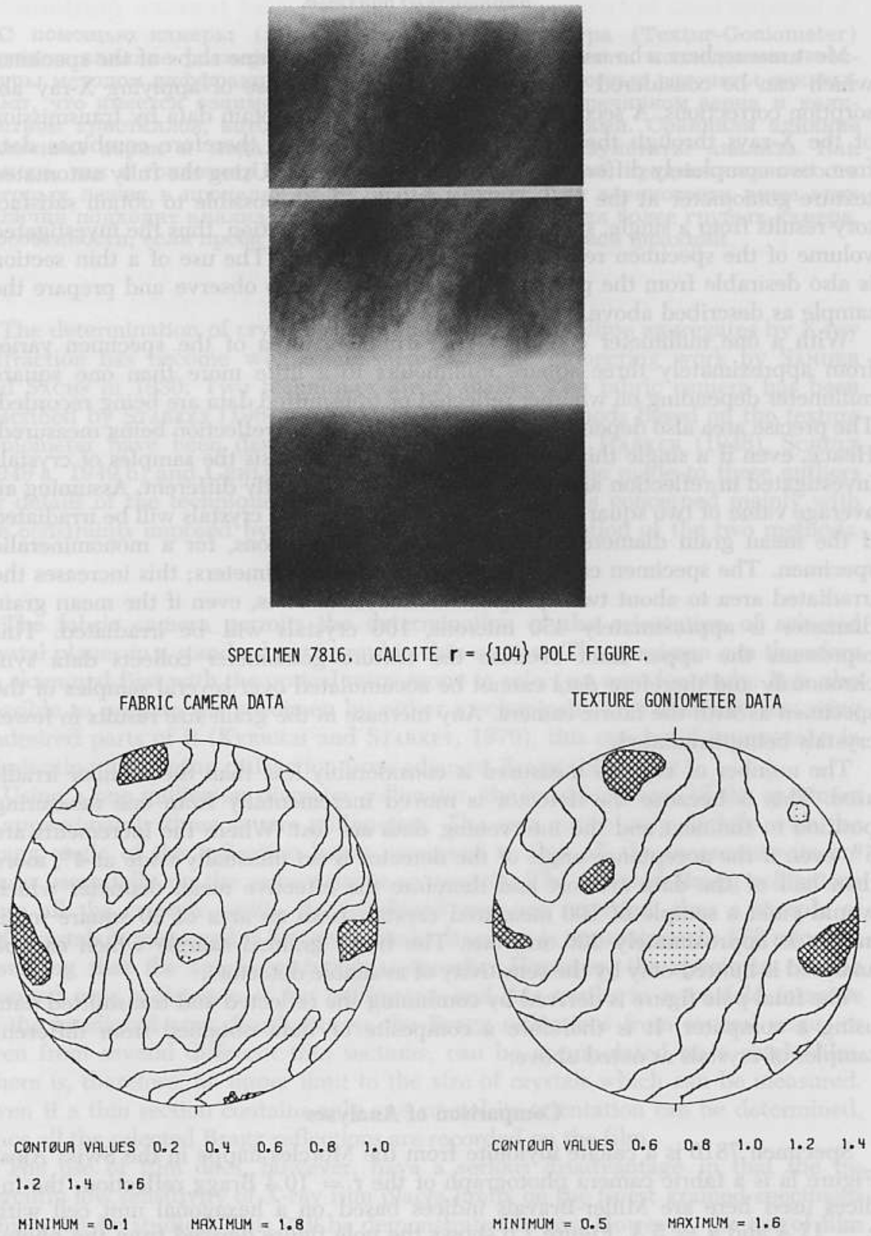

Fig. 1. The calcite r fabric in mylonite from the Morcles Nappe, Swiss Alps, specimen 7816. a) The fabric camera photograph. b) The pole figure derived from the fabric camera photograph. c) The pole figure derived from tecture goniometer data. In b and c the contour intervals are multiples of a uniform distribution of the data. The area below the lowest contour is stippled, the area above the highest contour is cross hatched.

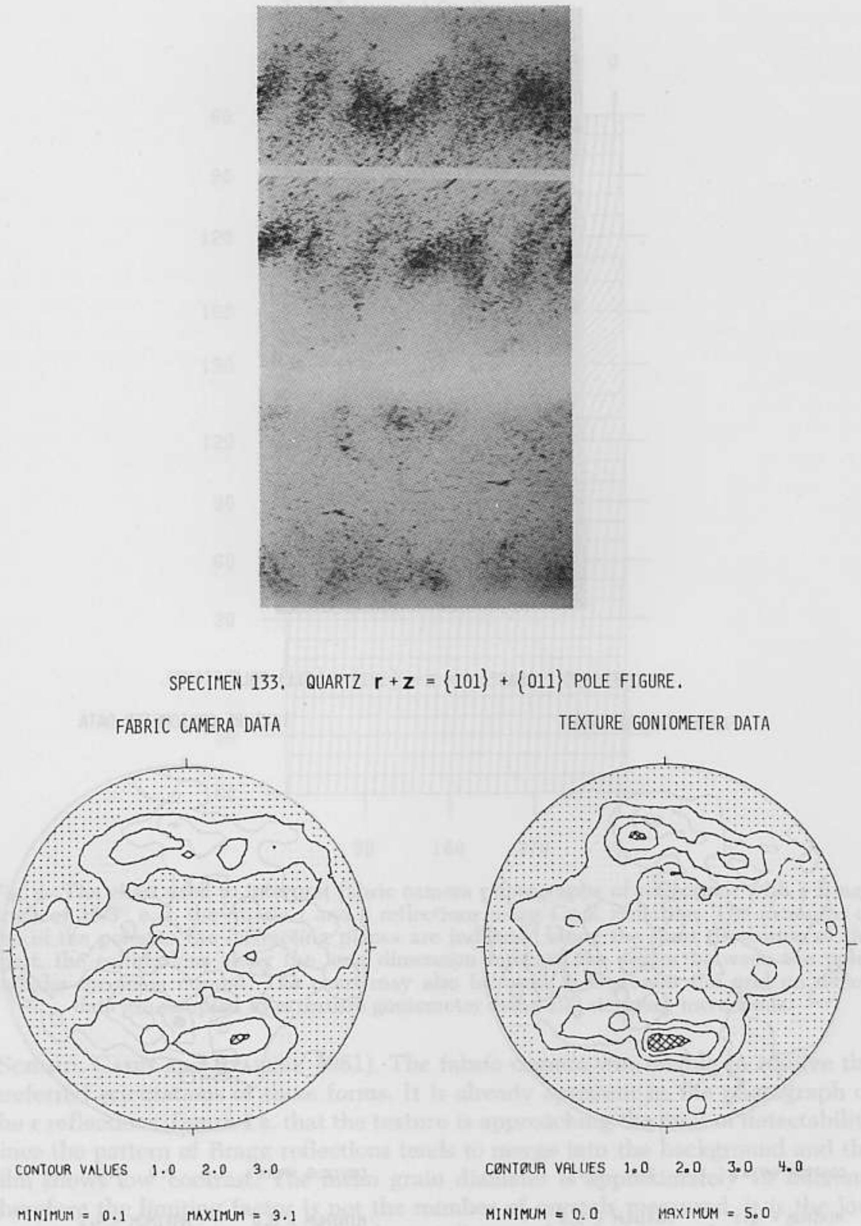

Fig. 2. The quartz r + z fabric in Saxony Granulite, specimen 133. a) The fabric camera photograph. b) The pole figure derived from the fabric camera photograph. c) The pole figure derived from texture goniometer data. In b and c the contour intervals are multiples of a uniform distribution of the data. The area below the lowest contour is stippled, the area above the highest contour is cross hatched.

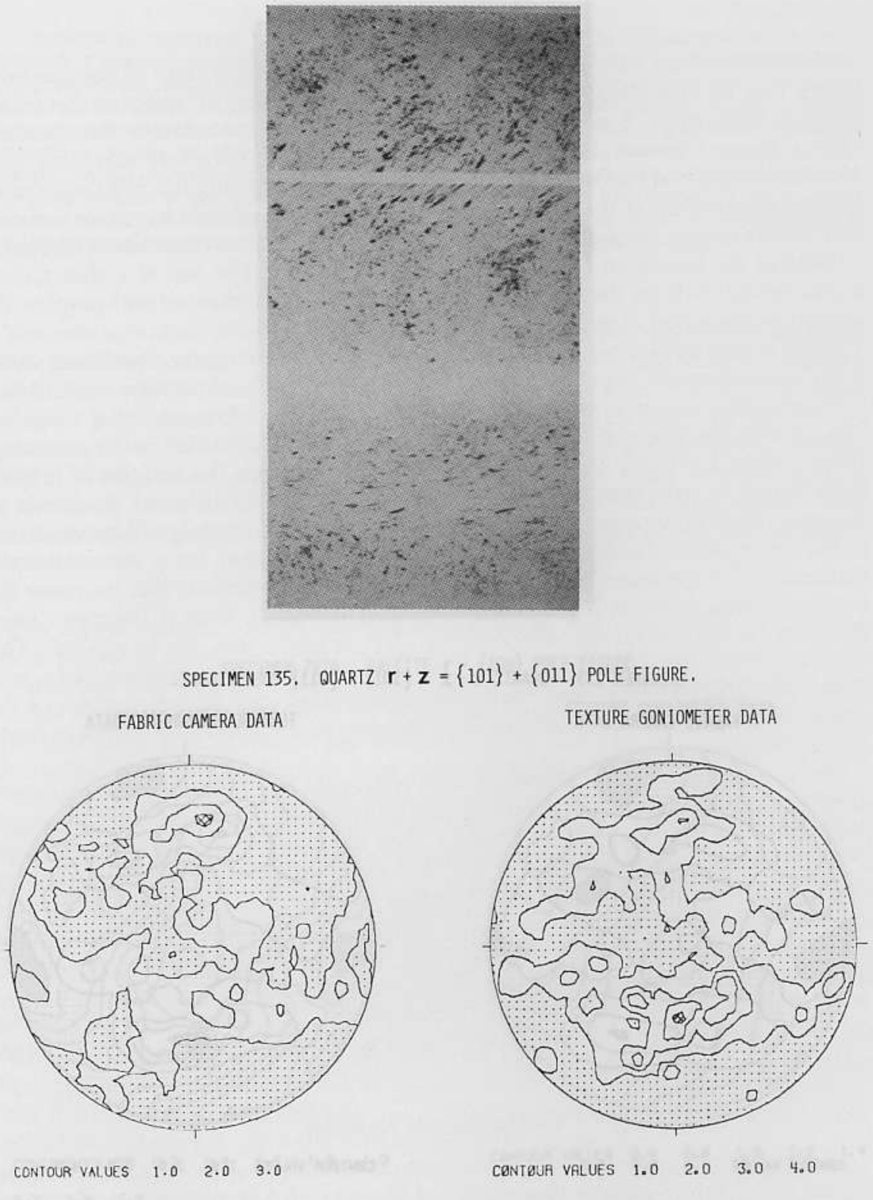

Fig. 3. The quartz r + z fabric in Saxony Granulite, specimen 135. a) The fabric camera photograph. b) The pole figure derived from the fabric camera photograph. c) The pole figure derived from texture goniometer data. In b and c the contour intervals are multiples of a uniform distribution of the data. The area below the lowest contour is stippled, the area above the highest contour is cross hatched.

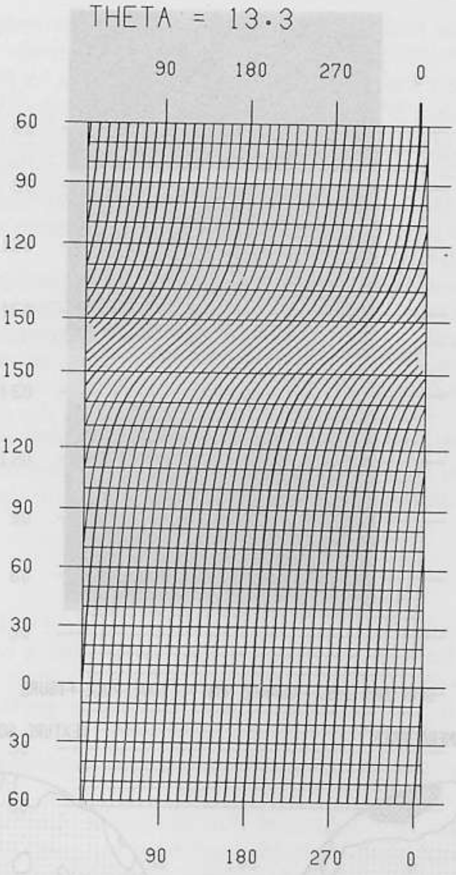

Fig. 4. The chart used to interpret fabric camera photographs of reflections with a Bragg angle of 13.3° e. g. the quartz r and z reflections using Cu K radiation. The azimuths of the of the poles to the diffracting planes are indicated along the short dimension of the chart, the calibrations along the long dimension indicate the angles between the poles and the specimen normal. The chart may also be used to represent the grid on which data are sampled by a texture goniometer using 10° stepping increments.

(Schmid, Casey and Starkey 1981). The fabric camera was unable to resolve the preferred orientations of these forms. It is already apparent in the photograph of the r reflections, figure 1 a, that the texture is approaching the limit of detectability since the pattern of Bragg reflections tends to merge into the background and the film shows low contrast. The mean grain diameter is approximately 10 microns, therefore the limiting factor is not the number of crystals measured, it is the low intensity of the X-rays diffracted by small crystals relative to the background level of radiation.

Specimen 133 is a granulite from Saxony. It contains approximately 40 % quartz and the mean grain diameter is approximately 50 microns. Figure 2 shows data for the r = 10.1 and z = 01.1 reflections, using Miller-Bravais indices based on

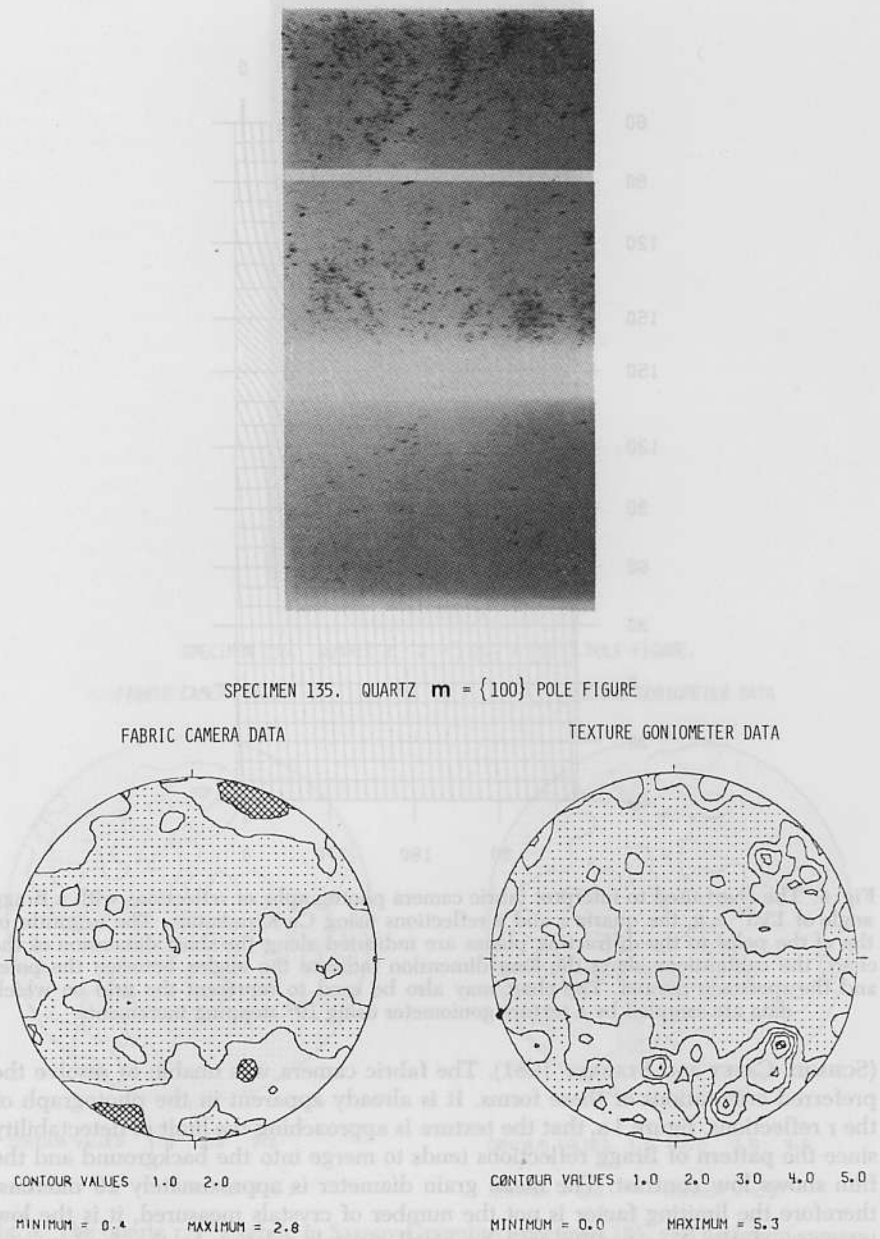

Fig. 5. The quartz m fabric in Saxony Granulite, specimen 135. a) The fabric camera photograph. b) The pole figure derived from the fabric camera photograph. c) The pole figure derived from texture goniometer data. In b and c the contour intervals are multiples of a uniform distribution of the data. The area below the lowest contour is stippled, the area above the highest contour is cross hatched.

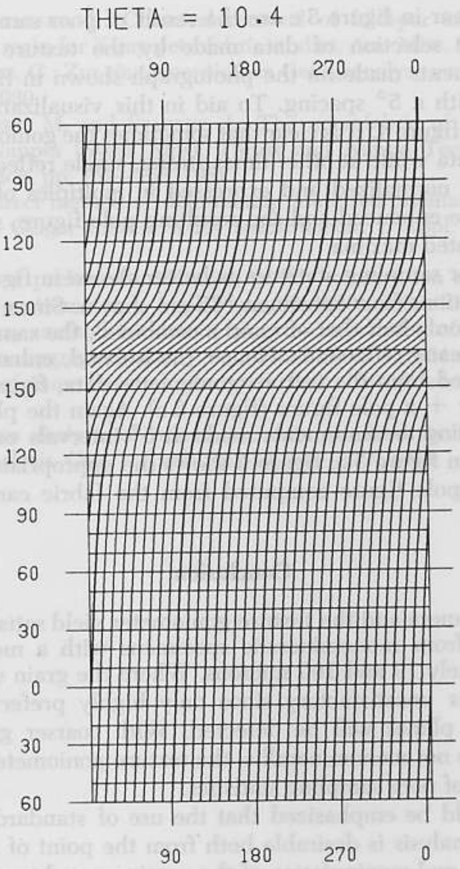

Fig. 6. The chart used to interpret fabric camera photographs of reflections with a Bragg angle of 10.2°, e. g. the quartz m reflection using Cu K radiation. The azimuths of the poles to the diffracting planes are indicated along the short dimension of the chart, the calibrations along the long dimension indicate the angles between the poles and the specimen normal. The chart may also be used to represent the grid on which data are sampled by a texture goniometer using 10° stepping increments.

a hexogenal unit cell. Since both forms have the same Bragg angle they cannot be isolated. The fabric camera photograph, figure 2 a, shows a well resolved pattern of preferred orientation. This is presented as a pole figure in figure 2 b. Figure 2 c shows the pole figure derived from texture goniometer data. The results of the fabric analyses by both fabric camera and texture goniometer are closely comparable. Agreement was also found in the measured fabrics of other crystallographic forms.

Specimen 135 is a Saxony granulite which contains approximately 70 % quartz with a mean grain diameter of 100 microns. The fabric camera photograph of the r + z reflections, figure 3 a, yields the pole figure shown in figure 3 b. Figure 3 c shows the pole figure derived from the texture goniometer data. The isolated maxima which appear in figure 3 c are the result of poor sampling statistics. One can appreciate the selection of data made by the texture goniometer if one considers measurements made on the photograph shown in figure 3 a at discrete points on a grid with a 5° spacing. To aid in this visualization a grid with 10° spacing is shown in figure 4. It is clear that sometimes the goniometer must measure only background data while at other times strong, single reflections are measured. When the data are normalized and expressed in multiples of a uniform density these differences are enhanced and the resulting pole figure, shown in figure 3 c, contains strong isolated maxima.

The effect of poor sampling statistics is better shown in figure 5 in which data for m = 10.0 reflections from specimen 135 are shown. Since the crystallographic multiplicity of m is only half that of r and z combined, the same sample of crystals yields only half as many reflections. Hence, the isolated, enhanced maxima in the m pole figure derived from the texture goniometer data, figure 5 c, are more apparent than in the r + z pole figure (figure 3 c). Again the phenomenon may be visualized by assuming measurements made at 5° intervals on the fabric camera photograph shown in figure 5 a, figure 6 shows the appropriate measuring grid at 10° intervals. The pole figure computed from the fabric camera photograph is shown in figure 5 b.

Conclusion

Both the fabric camera and the texture goniometer yield satisfactory data for the analysis of fabrics from monomineralic specimens with a mean grain diameter between approximately 10 and 100 microns. Where the grain size is less than this the fabric camera is unsatisfactory since only highly preferred orientations of strongly diffracting planes will be detected. With coarser grained samples, or specimens which are not monominerallic, the texture goniometer yields unsatisfactory results because of poor sampling statistics.

Finally, it is should be emphasized that the use of standard petrographic thin sections for fabric analysis is desirable both from the point of view of permitting optical investigation and manipulation of the specimen and to ensure that the data are collected from a single sample of crystals, as far as this is possible. This latter goal could be more nearly accomplished by a texture goniometer if it were designed with the same geometry as the fabric camera but such an instrument is not commercially available.

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