A New Translational Sample Holder for Reflection Polefigure Measurements

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A new specimen holder is described which allows true area scanning of circular texture specimens. For large grain sized materials and geological samples this method can lead to a significant increase in the number of grains sampled and thus to more reliable texture measurements. The principle of operation is explained and a prototype apparatus described.

INTRODUCTION

The measurement of textures in crystalline materials is most usually carried out by the diffraction of some form of radiation from selected lattice planes. The most widespread technique is X-ray diffraction using a Texture Goniometer. A summary of the techniques and nomenclature used is given in Ref. 1. Texture measurements produce polefigures, which are the probability density functions for finding a given \( \{hkl\} \) reflection within an angular range on the pole-sphere defined by a specimen coordinate system. Thus polefigure measurements are statistical in nature and a minimum number of "crystallites" must be measured for statistical relevance. This minimum number depends on the sharpness of the texture, for weak textures more crystallites are necessary than for sharp ones.

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Many techniques have been developed to "scan" the specimen under the X-ray beam to increase the number of crystallites measured. This is necessary in the case of large grained materials and particularly important in the case of geological materials. Here each crystallite often has a high degree of perfection, constituting a single, sharp peak in the polefigure thus the chance of its not being measured is increased.

In the case of natural rock-salt (Halite) which has a grain size up to centimetre dimensions, conventional specimen translations have been unsuccessful in sampling enough grains to provide statistical relevance. Neutron diffraction measurements on these materials suggest that the textures are quite weak so that the problem becomes acute.

**EXISTING SPECIMEN MOVEMENTS**

Most commercial texture goniometers translate the specimen under the X-ray beam in a simple one-dimensional manner. These mechanisms can be divided into three groups:

"TRANS-ROT" where the phi rotation occurs after (or inside) the translational movement, i.e. the phi circle is bodily translated.

| INCIDENT BEAM 1 mm × 1 mm | INCIDENT BEAM 1 mm × 5 mm |
|--------------------------|--------------------------|
| **φ**        | 0°  | 45° | 90° | 0°  | 45° | 90° |
| ROT-TRANS (after φ) | | | | | | |
| TRANS-ROT (before φ) | | | | | | |
| CIRCULAR | | | | | | |

**FIGURE 1** The irradiated area of the specimen for the three one-dimensional translations as a function of the incident X-ray beam (spot, line) and the orientation of the specimen in phi.
"ROT-TRANS" where the translational movement occurs before (or outside) the phi rotation.

"CIRCULAR", after Morris, where a circular path on the specimen is described by the X-ray beam.

Figure 1 shows the sampled area on the specimen for each of these movements for two different shapes of focus. All of these cases represent one-dimensional scanning, and in general it is not possible to scan the entire specimen. One can also see in Figure 1 that the area scanned is not always the same for all values of phi.

A true surface scanning can be obtained with a combination of two linear movements such as illustrated in Figure 2. Such a specimen holder, realised in the RWTH Aachen, W. Germany also incorporates a cam mechanism to give a true linear speed in the X direction.

The present work describes a new method of combining a circular motion with a curvilinear motion to provide a spiral scanning of a circular texture specimen.

PRINCIPLES OF OPERATION

1 Circular motion

The circular component of the new holder can best be demonstrated by the combination of three gear-wheels such as used in the Morris specimen holder. Figure 3 shows these gear-wheels in perspective. Gear-wheels "B" and "C" are fixed together in a...
housing represented by triangle "abc" which can rotate about the centre of gear-wheel "A". Gear-wheel "C" is offset from the centre of "A" by a distance "X", wheels "C" and "A" are the same size. Wheel "A" is fixed not only in position (at the centre of rotation of the apparatus) but is also unable to rotate about its own axis. The specimen is fixed to gear-wheel "C" (and rotates with it). Gear-wheels "C" and "B" are free to rotate about their own axes. Gear-wheels "C" and "B" can thus roll bodily around the fixed wheel "A" as a unit. In the Morris holder this rotation is achieved using a motor driving a toothed ring on the outside of the housing "abc".

The movement of wheel "B" around wheel "A" imparts a rotation \(-S\) to wheel "B" which rotates wheel "C" in the opposite direction with rotation \(T-\). The change in orientation of wheel "C", i.e. the SPECIMEN, which would result from the bodily rotation \(-R\) alone is exactly nullified by this compensating rotation \(T-\) and the specimen remains in its original orientation.
The X-ray beam meets the specimen in the projected centre of wheel “A” (the central axis of the entire apparatus). This point is the distance “X” from the centre of the specimen. Rotating the housing “abc”, containing wheels “B” and “C”, around wheel “A” thus results in the X-ray beam tracing a circular path on the specimen of radius “X”, the specimen retaining its original orientation thereby. This path is shown in Figure 1.

2 Linear motion

The new holder combines this movement around the circle with radius “X” with a continuous change in this radius such that a spiral trace is obtained on the specimen. One can see from Figure 3 that by changing the distance “X”, the radius of the circular trace on the specimen is also changed. Unfortunately this cannot be achieved by the three gear-wheels shown here since changing the distance “X” also imparts a rotational component to wheel “C” and thus the specimen. This presents no problem in the Morris holder since the offset “X” is adjusted before the measurement and thereafter remains constant. If the offset “X” is to be changed continuously, e.g. by an excenter, then the imparted rotation must also be compensated for.

3 Combined linear and circular motions

Figure 4 shows the principal gear train involved in the new mechanism. This mechanism is a combination of the circular movement described earlier and a superimposed curvilinear movement which results in a true area scanning (without, however, the frequent changes in direction involved in the combination of two rectilinear movements).

Gear-wheel “A” can neither rotate nor change position and thus forms the reference orientation. Gear-wheel “B” can roll around “A” (at a fixed distance) and also rotate about its own axis. Gear-wheel “C” is fixed with respect to “B” in position but can rotate about its own axis. Gear-wheel “D” can roll around “C” (at a fixed distance) as well as rotate about its own axis. Gear-wheel “E” is fixed with respect to “D” in position but can rotate about its
The specimen is fixed to gear-wheel "E" with a certain angular position (which defines the angle phi).

Gear-wheels "B", "C", "D" and "E" are contained in a housing which maintains their position relative to each other. The housing can roll bodily around wheel "A" with wheel "B" maintaining contact with wheel "A" throughout—ROTATION OF SPECIMEN—This is shown by the solid lines in Figure 4. The centre of this rotation is the centre of wheel "A".

The bodily rotation of "B/C/D/E" about the fixed wheel "A" imparts a rotation of wheel "B" around its own axis which is passed on through wheels "C" and "D" to wheel "E". This rotates the specimen in the opposite direction to the original rotation and thus the specimen retains its angular position with respect to wheel "A". This function is similar to that of the Morris holder.

Wheels "E" and "D" are fixed with respect to each other in a sub-housing in the plane above wheels "A" and "B" that is pivoted about gear-wheel "C". Thus wheels "E" and "D" can roll around wheel "C" with respect to wheels "A" and "B"—LINEAR MOVEMENT OF SPECIMEN—This is shown in Figure 4 by a dashed line. The centre of the specimen moves along a circular arc centred on gear-wheel "C". On rolling the wheels "E/D" around "C", "D" rotates relative to "C" and rotates "E" in the opposite direction by the same amount. Thus the angular orientation of the
specimen with respect to "A" is now also maintained for the curvilinear movement (cf. Section 2).

Thus for both movements the orientation of the specimen is maintained with respect to the fixed reference point, wheel "A". The phi movement which changes the angular position of "A" (and thus "E", the Specimen) acts upon wheel "A" which is the reference orientation in the apparatus. In the prototype the phi drive acts upon the whole apparatus. The movement of the apparatus could be defined in the nomenclature from earlier as "ROT - CIRCULAR + TRANS". In the case of this true surface scanning the orientation of "A" and the specimen "E" is ALWAYS the same and so it is in fact irrelevant whether the phi motion comes before or after the apparatus.

The LINEAR movement can be provided by an excenter driven from the bodily rotation about gear-wheel "A". If the excenter is driven at a lower speed than the rotation a spiral path is achieved (Figure 5) and if the excenter rotates more quickly, a series of "petal" shapes is achieved with respect to the fixed point "A". "A" also represents in general the point at which the X-rays strike the specimen.

**THE WETEMAC SPECIMEN HOLDER**

Figure 6 shows the prototype apparatus built in the Institut für Metallkunde for the measurement of rock-salt specimens of up to
FIGURE 6 The apparatus built in the Institut für Metalkunde mounted on a Spindler and Hoyer 1/4 circle Eulerian cradle and phi drive. a) Null position with excenter at minimum; b) Excenter at maximum extension.

90 mm in diameter. Figure 7 shows a plan view of the apparatus with the maximum extension of the excenter arm. The various parts mentioned in the description are here labelled.

The bodily rotation of the housing containing wheels "B", "C", "D" and "E" about "A" is provided by a DC motor acting through a reduction gearbox on the toothed wheel fixed to the outside of the (circular) housing. In Figure 7 the gearbox and motor have been
omitted for clarity. Figure 6 shows the gearbox attached to the Eulerian cradle. The linear motion is provided by a take-off gear acting on "A" through a further reduction gear train which drives the excenter coupled by a curved arm to the sub-housing containing wheels "E" and "D". This sub-housing can pivot around the axis of wheel "C", compare Figures 6a and 6b. The excenter is driven at 1/20th of the speed of rotation and thus a spiral is obtained of "X"/10 pitch. For the maximum movement of 45 mm this gives a pitch of 4.5 mm for the largest specimen. Figure 8 shows the trace of this spiral obtained by holding a pencil on the specimen holder.
FIGURE 7  Plan of new apparatus with excenter at maximum extension. 
Nomenclature for Figures 6 and 7:
  a) excenter arm 
  b) excenter 
  c) Pivot point of sub-housing about wheel “C” 
  d) Motor driving phi circle (underneath apparatus) 
  e) Specimen holder, coaxial with wheel “E” 
  f) Eulerian cradle (1/4 circle) 
  g) Gearbox housing, fixed to Eulerian cradle (before the phi circle) 
  h) Sub-housing pivots around wheel “C”.

FIGURE 8  Actual trace of X-ray beam obtained with the new specimen holder for 10 revolutions.
while rotating it. The pitch of the spiral is of comparable dimension to the size of the X-ray beam and thus the entire specimen is scanned with 10 rotations of the apparatus. The equipment can be driven at ca. 120 rpm and thus the specimen can be scanned in 5 seconds. For an apparatus of smaller dimensions the speed of rotation could be increased substantially.

Considering the case of the one-dimensional movements mentioned earlier, let us assume the X-ray beam incident on the specimen is firstly a square of 1 mm and secondly a line of width 5 mm and height 1 mm. The specimen is typically 30 mm in diameter and a maximum oscillation of 25 mm is allowed. The area scanned for the three specimen holders and three orientations of phi from Figure 1 is summarised in Table I. One can clearly see that the circular motion is in all cases superior in the surface scanned. For the spot, "ROT-TRANS" and "TRANS-ROT" are comparable, for a line the "TRANS-ROT" holder is superior.

In each case in Table I a further column is included, labelled "All". This is the surface which is common to each value of phi, i.e. the area scanned for every orientation of the specimen. The difference between this value and the area for individual values of phi gives a measure of the inhomogeneity of the scanning. This does not affect the statistical relevance directly but affects the internal self consistency of the polefigure. A grain could be detected in one phi position but missed in the symmetrically equivalent position. This problem also becomes more acute with larger grain sizes and higher perfection of the individual crystallites. All the one-dimensional scanning methods are not particularly satisfactory in

| Incident beam | Spot 1 mm² | Slit 5 mm × 1 mm |
|---------------|------------|-----------------|
| phi setting   | 0° | 45° | 90° | all | 0° | 45° | 90° | all |
| "ROT-TRANS"  | 26 | 26 | 26 | 26 | 128 | 90 | 30 | 30 |
| "TRANS-ROT"  | 26 | 26 | 26 | 1 | 128 | 128 | 128 | 20 |
| "CIRCULAR"   | 39 | 39 | 39 | 39 | 250 | 250 | 250 | 39 |
| NEW HOLDER    | 710 | 710 | 710 | 710 | 710 | 710 | 710 | 710 |
| "X" = 12.5 mm | 6360 | 6360 | 6360 | 6360 | 6360 | 6360 | 6360 | 6360 |
| "X" = 45 mm   | 6360 | 6360 | 6360 | 6360 | 6360 | 6360 | 6360 | 6360 |
this respect for the most common case of the line shaped incident beam.

A further problem is that the shape of the incident beam on the specimen is dependent on the Bragg angle. For small angles the beam is long and thin, for high angles it becomes shorter. In the case of the one-dimensional scans this can mean that each pole-figure is measured over a different area of the specimen.

The new holder accommodates specimens of up to 90 mm in diameter of which the entire surface can be scanned for every phi angle and every polefigure. If we consider a 25 mm movement ("X" = 12.5 mm) with the new holder for comparison we obtain for both the spot and the line an area of 710 mm\(^2\). This is scanned for every value of phi and every polefigure. The size of the irradiated area can be varied by varying the length of the excenter and thus specimens from 0 mm to 45 mm radius can be measured. Taking the maximum size of specimen we obtain a scanned area of 6360 mm\(^2\).

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