A Goniometer for the Measurement of Stresses in Single Crystals and Coarse-Grained Specimens

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Abstract

A ψ–φ goniometer with two axes of rotation, driven by stepping motors, was designed as an attachment for a horizontal θ/2θ goniometer. By means of this arrangement it is possible to measure strains and compute stresses in single crystals and in separate crystals of a bicrystal or a coarse-grained polycrystalline specimen. The available rotations also make it possible to investigate residual macrostresses and textures in polycrystalline specimens. Results of the test measurements on a tensile specimen of a molybdenum single crystal are given.

1. Introduction

To determine the strain tensor for a triaxial stress condition in a single crystal, at least six strain components must be known (Taira, 1974). If the strains in more than six different directions are measured, a least-squares method can be used to improve the accuracy (Imura, Weissmann & Slade, 1962). From the strain tensor and the elastic constants of the crystal, the stress tensor can be calculated.

By means of X-ray back-reflection methods, it is possible to determine strains in different crystallographic directions from lattice distances in the strained and unstrained conditions. For measuring lattice distances (Bragg angles) in single crystals, bicrystals or crystallites of coarse-grained specimens on a horizontal θ/2θ goniometer like the Philips PW 1380, it is necessary to make an attachment for orientating the crystal in such a way that the normal to the planes concerned bisects the angle between the incident and reflected beams. For that purpose, an attachment with two axes of rotation, a ψ axis and, perpendicular to it, a φ axis, has been designed for the horizontal goniometer.

This method is used in our laboratory (because of its advantages with respect to the Kossel method in this field of investigation) particularly for measurements on plastically deformed specimens with broadened diffraction lines.

The attachment is also used as a ‘ψ goniometer’ (Wolffstieg, 1976), for measuring residual macrostresses in polycrystalline materials. The attachment as well as the results of some test measurements on a tensile specimen of a molybdenum single crystal are described.

2. Description of the ψ–φ goniometer attachment

Fig. 1 shows the attachment in section. The ψ(6) and φ(11) shafts are driven by stepping motors (3, 14) with a 1.8° step. Because of a 1:180 transmission by means of a combination of precision worm gears and pinned drive belts (4, 9), Δψ(min.) and Δφ(min.) are 0.01°. The hollow ψ shaft (6) is supported on one side only by means of a slide bearing (5). This construction offers some advantage with respect to the alignment procedure.

In this way, it is possible to rotate through θ/2θ = 0° with the collimator mounted with its end at a distance of about 10 mm from the specimen surface or a pinhole, both placed in the centre of the goniometer. For the alignment procedures, X-ray intensities at 2θ < 3° can be measured through the hollow ψ shaft. The bearing-house construction limits the measurable Bragg angle θ to a minimum value of 24°, which was not considered a real problem in stress measurements. Unlike the standard Eulerian cradle in combination with coupled θ–2θ rotations, there is no limitation with
this construction at high Bragg angles, which is very important for stress measurements, for which angles above 80° may be used (Wilson, 1970).

On the \( \varphi \) shaft, an \( X-Y \) positioning table (10) with a small tensile loading device is mounted.

The attachment offers different possibilities for adjusting the position of the \( \varphi \) and \( \psi \) shafts:

(a) An adjustable cleft (1) for adjusting the \( \psi \) axis normal to the \( \theta/2\theta \) axis.

(b) A shaft (2) for adjusting the \( \psi \) axis intersecting the \( \theta/2\theta \) axis.

(c) A threepoint adjustment in plate (7) to adjust the plane in which the \( \varphi \) shathouse (15) can move linearly along a ball bearing (8), parallel to the \( \theta/2\theta \) axes and perpendicular to the \( \psi \) shaft.

(d) A spherical bearing (13) for adjusting the \( \psi \) and \( \varphi \) axes to a perpendicular setting.

(e) A threepoint adjustment of the \( \varphi \) shathouse to adjust the \( \varphi \) shaft parallel to the movement of the \( \varphi \) shathouse along the linear bearing.

(f) The adjustable \( \varphi \) shaft support (12) to translate the \( \varphi \) shaft to the point of intersection of the \( \psi \) and \( \theta/2\theta \) axes.

The whole alignment procedure, as mentioned above, was carried out on a surface plate. An auxiliary tool, mounted on the \( X-Y \) table, is used to extend the \( \psi, \varphi \) and \( \theta/2\theta \) axes to the goniometer centre as shown in Fig. 2.

To determine whether or not the two cylindrical parts of this tool lie in the extension of the \( \psi, \varphi \) or \( \theta/2\theta \) axes, the movements of these parts during rotation of these axes may be measured. This can be done by a 'millitron' micrometer touching the surface of the two cylindrical parts at different places.

After instrumental alignment, the \( \varphi, \psi \) and \( \theta/2\theta \) axes lay within a distance of \( \pm 5 \, \mu m \) at the goniometer centre and the \( \psi \) axis was perpendicular to the \( \varphi \) and \( \theta/2\theta \) axes, within \( \pm 0.003^\circ \).

For the rough alignment of collimator, \( \psi \) axis and detector in the \( 2\theta \) zero position, a ruler placed in the hollow \( \psi \) shaft (Fig. 3) was used. The fine adjustment of the \( 2\theta \) zero setting was carried out by means of a pinhole (Parrish & Lowitzch, 1959). The pinhole, Fig. 4, has a diameter equal to that of the X-ray beam at the goniometer centre and can be chosen as 0.5, 0.3 or 0.2 mm.

The pinhole, which is fixed on the \( X-Y \) table, is placed optically in the centre of the goniometer by using a microscope with crosslines and rotating the \( \psi \) and \( \varphi \)
axes independently. Then, the primary beam can be centred by measuring the X-ray intensity through the pinhole and hollow $\psi$ shaft in the $2\theta$ zero position of the detector, found with the ruler, and maximizing this intensity by moving the adjustable collimator.

For the 2:1 setting of $2\theta$ and $\theta$, a flat plate on the specimen support perpendicular to the $\varphi$ shaft with its surface parallel to the $\theta/2\theta$ axis in the goniometer centre can be used (Parrish & Lowitzsch, 1959). It is important for high accuracy to position the specimen surface during each measurement at the same height in the goniometer centre (Wilson, 1970). Therefore, a removable part as shown in Fig. 5 fixed in the hollow $\psi$ shaft was used. The point of the pin was positioned optically in the centre, once again by means of a microscope and rotation of the $\psi$ and $\theta/2\theta$ axes. To minimize the load on the specimen during the positioning procedure, an electrical circuit with a voltmeter or lamp indication was used (Fig. 5).

To orientate the specimen in the right position for diffraction at an adjusted Bragg angle, as calculated from the approximate lattice parameter, firstly a Laue photograph was taken. From this photograph, the orientation procedure can be done roughly and after that it is possible to adjust more accurately by maximizing the measured intensity. This can be carried out by rotating $\varphi$, $\psi$ and $\theta/2\theta$ alternately. The receiving 'diaphragm', in combination with the 0.5 mm $\varnothing$ collimator used during the experiment described in the next section, had a diameter of 0.8 mm. The angle of rotation of the $\psi$ and $\varphi$ shafts can be read on a scale on the goniometer with an accuracy of 0.25° and on a digital electronic scale coupled on the stepping motors with an accuracy of 0.01°.

The position of the diffraction line is determined by calculating the position of the maximum of a least-squares quadratic through 31 measured points of the line above 85% of the maximum intensity (Kirk, 1971). The intensity measurements were done by means of a proportional counter. Fig. 6 shows the complete arrangement with a tensile loading device mounted on the X–Y positioning table.

### 3. Test measurement

For testing the possibilities of the apparatus, an investigation was carried out on a tensile specimen of a molybdenum single crystal. The cross-sectional area of the specimen was 1.9 x 0.5 mm with a measuring length of 8 mm and a total length of 25 mm.

The specimen was loaded in the tensile apparatus in such a way that we could assume a uniaxial tensile stress along its length. However, during the measurements and calculations of the stress tensor a triaxial stress condition was assumed and this meant that we needed, as mentioned before, at least six strain components for determining the strain and stress tensor. For the measurements, Cu $K\alpha_1$ and Cu $K\beta$ radiation was used.
The temperature of the sample was measured by means of a chromel–alumel thermocouple, and corrections were made for deviations from 295 K. Fig. 7 shows in stereographic projection the 13 directions in which we measured strains from the lattice spacings in the unloaded and loaded conditions. The measurements were made in the centre of the tensile specimen. The coordinate system of the specimen is also drawn, the load \( F \) was in the [100] direction of that system; the [001] direction was perpendicular to the surface of the specimen. The direction cosines relating the axes of the crystal and specimen systems are necessary for the calculations of the strain tensor.

Firstly, we measured strains in the [100] crystal direction on the 400 reflection as a function of the stress caused by the external tensile load. The results are given in Fig. 8; each point is the mean of eight measurements. The straight line gives the strains that were calculated, by means of the elastic constants \( C_{11} \), \( C_{12} \) and \( C_{44} \) (Landholt–Börnstein, 1966) and the orientation of the crystal, as a function of the applied stress (Taira, 1974).

One can see that the measured and calculated values agree well. The accuracy of the strain measurement in this case is about \( 5 \times 10^{-6} \). Subsequently, the tensile specimen was loaded to a stress of 156 MPa and the strains in 13 directions, given in Fig. 7, were measured (Table 1). The given strains are the mean of two measurements. From these measured strain components, the single-crystal elastic coefficients and the direction cosines of the crystal, the stress tensor and principal stresses were calculated by a least-squares method (Imura, Weissmann & Slade, 1962) (Table 2).

Non-uniaxial stress components up to about 4\% of 156 MPa have been measured. These may be due to, on the one hand, a non-uniaxial stress condition or, on the other hand, a deviation of the used elastic constants from the real values and/or errors in the measured strains and orientation of the crystal. The measured stress along the axial direction of the tensile specimen deviates no more than 1\% from the nominal calculated value of 156 MPa with an estimated error of about 2.5\%.

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### Table 1. Measured strains caused by an applied stress of 156 MPa in the [100] direction of the specimen

<table>
<thead>
<tr>
<th>Reflection</th>
<th>Strain ((\times 10^{-5}))</th>
<th>Reflection</th>
<th>Strain ((\times 10^{-5}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>400</td>
<td>-11.6</td>
<td>213</td>
<td>27.2</td>
</tr>
<tr>
<td>040</td>
<td>-11.3</td>
<td>123</td>
<td>28.1</td>
</tr>
<tr>
<td>420</td>
<td>-11.9</td>
<td>411</td>
<td>-7.8</td>
</tr>
<tr>
<td>240</td>
<td>-11.3</td>
<td>411</td>
<td>-10.3</td>
</tr>
<tr>
<td>330</td>
<td>-10.9</td>
<td>141</td>
<td>-9.9</td>
</tr>
<tr>
<td>213</td>
<td>18.8</td>
<td>141</td>
<td>-7.4</td>
</tr>
<tr>
<td>123</td>
<td>19.1</td>
<td></td>
<td></td>
</tr>
</tbody>
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### Table 2. Stresses and direction cosines of the principal axes calculated from the measured strains in Table 1

<table>
<thead>
<tr>
<th>Stress tensor in the coordinate system of the specimen (MPa)</th>
<th>Principal stresses (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>154.5  5.2  3.8</td>
<td>154.8</td>
</tr>
<tr>
<td>5.2  -1.8  -0.6</td>
<td>-0.6</td>
</tr>
<tr>
<td>3.8  -0.6  -0.9</td>
<td>-2.3</td>
</tr>
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<table>
<thead>
<tr>
<th>Direction cosines of the principal axes in the coordinate system of the specimen</th>
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<tbody>
<tr>
<td>0.999  -0.006  -0.041</td>
</tr>
<tr>
<td>0.033  -0.458  0.888</td>
</tr>
<tr>
<td>0.024  0.889  0.457</td>
</tr>
</tbody>
</table>

### References


