Journal of Scientific Instruments (Journal of Physics E) 1969 Series 2 Volume 2

Microbeam back-reflection x-ray camera with arrangement for positioning a pre-selected area

F J ter Avest and J A Klostermann
University of Technology Twente, Enschede, The Netherlands

MS *received 18 April 1969, in revised form 16 July 1969*

Abstract A high precision back-reflection x-ray microbeam camera is described which uses a very accurate translation of a slide to move a point or a specimen selected by the cross-wires of a microscope into the centre of a very narrow x-ray beam. This positioning can be performed with an accuracy to better than 2 μm. The use of a lead glass capillary collimator, diameter 10 μm, is shown to give an intense x-ray beam and also to change the x-ray spectrum by selection of longer wavelengths. The microbeam camera has been used to study grain boundary migration during creep in aluminium by a Laue technique.

1 Introduction

A microbeam x-ray camera is an important tool in the study of phase transformations, deformation phenomena (e.g. in the case of creep) and many other subjects in metallurgy.

A description is given of an instrument used in conjunction with a Philips fine focus tungsten tube. The instrument consists of a camera, a microscope and an arrangement for positioning a pre-selected area very quickly and accurately.

2 Positioning of a pre-selected area of the sample

The problem is to move a point seen in the cross wires of a microscope to the centre of the x-ray beam of a camera, and various methods have been devised to solve it, of which the following are well known.

(i) The method of Otte and Cahn (1959): the distance from the area to be irradiated to a set of tungsten cross wires – 130 μm in diameter – is measured with two micrometer screws calibrated in units of 5 μm. With the aid of a Geiger–Muller counter the cross wires are brought into the x-ray beam and then the pre-selected area is moved along the measured distance into the beam. A drawback of this method is that to locate the cross-hairs initially is time consuming (10–15 min). (ii) Microscope and camera with joint axis: the disadvantages of this method are that the magnification of the microscope is limited, and that the distance from x-ray tube to sample is large, which results in a loss of intensity. (iii) Michels and Wayman (1962) have used a thin optical flat with an etched reticule on one of its surfaces. A light beam is made to follow the same path as the x-ray beam. A reference mark on the reticule is aligned in front of the collimator in the light beam. The relative positions of the desired area of the specimen and the reference mark are determined by means of an auxiliary microscope, and the desired area is brought in front of the collimator with an accurate XY positioner. This method is fast, but rather inaccurate (the maximum error was 10 μm).

The method described in this paper makes use of a very accurate translation of a slide to cover the distance between two fixed points. One of these points is determined by the cross-wires of a microscope. (In fact it is the centre of the virtual image of the cross-wire.) The other point is the centre of the narrow x-ray beam, which is obtained by a lead glass collimator.

The camera and the microscope (magnification 200×) are attached to the same stage. The microscope may be adjusted vertically (figures 1 and 2). The sample is moved from microscope to camera with the aid of a slide with roller bearings without backlash. The whole unit is mounted on the existing adjustable bracket in front of the x-ray tube. With this bracket

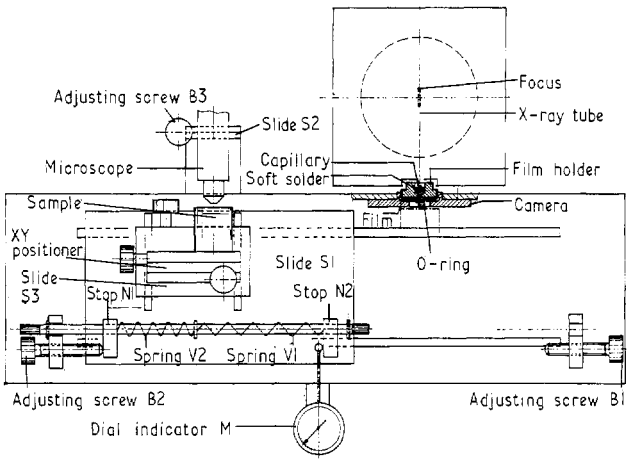

Figure 1 Schematic top view of the camera and arrangement for positioning a pre-selected area. Slide S1 moves the sample from the microscope towards the camera. The microscope is brought at the same level as the camera by means of adjusting screw B3. The position of the slide S1 can be adjusted by the adjusting screws B1 and B2 and read from dial indicator M

the collimator can be easily directed to the focus. A proportional tube – or flowcounter – is used to find the maximum intensity. A device made of two platinum foils is used to determine the centre of the x-ray beam (figure 3). Instead of the sample, the beam locator is attached to the XY stage (cross-table) K.

Behind the camera is a flowcounter. Slide S1 (figure 1) is moved towards the camera and is pressed against adjusting-screw B1 by spring V1. The feeler of the dial indicator M

(calibrated in units of 2 μm) then presses against stop N1. The position of the slide is read from the dial indicator (position X). By means of the XY stage K the foils are placed successively in the positions A, B and C, as shown in figure 3 using the flowcounter. (These positions will result in a

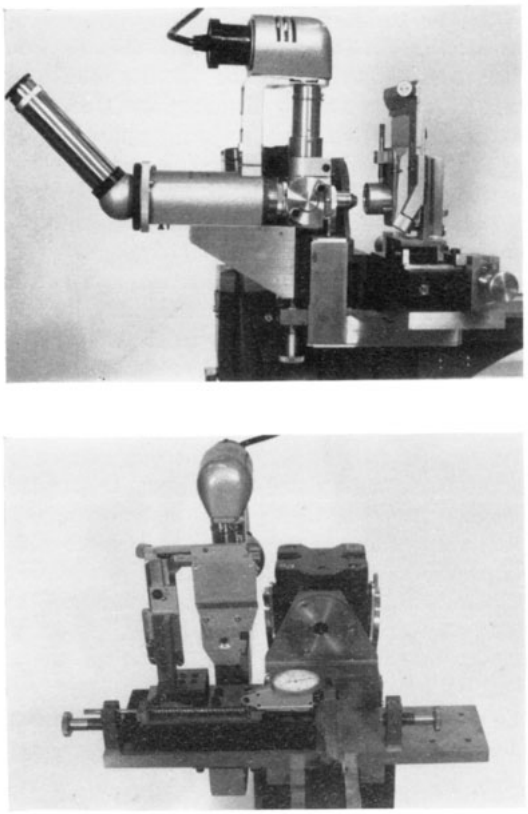

Figure 2 Photographs of the camera and the arrangement for positioning a pre-selected area

relative intensity of the measured x-rays of 1, $\frac{1}{2}$ and $\frac{1}{4}$). Slide S1 is then moved towards the microscope and pressed against adjusting screw B2 with the help of spring V2. The cross-wires of the microscope are then brought in the position of point T of figure 3 with the aid of the adjusting screws B2 and B3, the latter causing the vertical movement of the microscope slide S2. The feeler of the dial indicator then presses against stop N2 (position Y). Starting from this position a sample can be focused and adjusted in front of the microscope, by using slide S3 and XY stage K. Slide S1 is

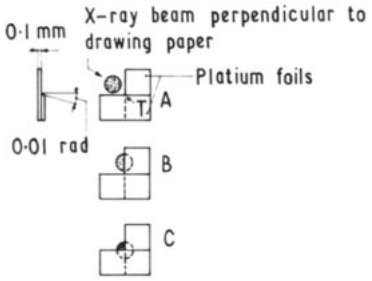

Figure 3 Two crossed platinum foils are used to calibrate the level of the microscope and the distance to be covered by the sample from microscope to camera. With the use of a flowcounter the centre of the x-ray beam is brought at point T. Relative count rates of the counter for A, B and C, 1, $\frac{1}{2}$ and $\frac{1}{4}$, respectively

now moved towards the camera (position X of the dial indicator). The error of positioning in this way has proved to be less than 2 μm.

If more than one photograph of a sample is made, the position of the microscope is checked by means of a reference-hole with a diameter of 20 μm (this is exactly the diameter of the divergent beam at 1 mm distance from the 10 μm capillary, see below) in a platinum foil next to the sample. The platinum foil is positioned to attain maximum count rate on the counter. A correction is necessary because the position of the camera may vary within an area of 5 μm after changing a film. If necessary, the level of the microscope screw B3 and the position of screw B2 (position Y) can be adjusted easily and quickly, so that the centre of the cross-hairs of the microscope is seen in the centre of the hole after translation with slide S1.

3 Camera

The construction of the camera is also shown in figure 1. The dimensions of the camera are kept small in order to get as high a radiation intensity as possible. The distance between collimator and sample is 1 mm. The film diameter is 16 mm, the distance between sample and film is 3·6 mm. To change a film the camera must be taken out of the camera-holder and the filmholder out of the camera casing. The collimator, a lead glass capillary with an outer diameter of 0·35 mm, fitting in the filmholder with a small tolerance is held fast with soft solder. To eliminate the effects of light and background radiation, black plastic foil and aluminium foil are placed in front of the film.

In the centre, light is stopped by a rubber O-ring. This ring does not absorb much of the radiation reflected with wavelengths shorter than 2·5 Å.

4 Collimator

If a lead glass capillary is used, total reflection of x-rays on the inner walls of the capillary is possible. This results in a bigger total amount of radiation compared with the amount that is obtained from two platinum diaphragms with holes of diameters the same as that of the capillary. This is caused by the fact that in the case of a lead glass capillary the x-rays come from a larger area of the focus, see figure 4, A and B.

The maximum angle ϕ_{max} for which total reflection can occur is determined by the index of refraction n of the lead glass

$$\phi_{max} = \cos^{-1} n. \tag{1}$$

The index of refraction n is a function of the wavelength λ of the x-rays (Taylor 1961).

$$n = 1 - \frac{Ne^2\lambda^2}{2\pi mc^2} \tag{2}$$

where c is the velocity of light, e the electronic charge, m the electronic mass, and N the number of electrons per unit of volume.

For lead glass n becomes

$$n = 1 - \frac{4{\cdot}8 \times 10^{14}}{\text{metre}^2}\lambda^2 \tag{3}$$

(λ in Å).

To show that the exposure times obtained are much shorter with the lead glass capillary, a capillary with an inner diameter of 10 μm and length 11 mm is compared with a collimator composed of two platinum diaphragms separated by 11 mm. The diameter of the focus is 0·4 mm. The voltage of the tungsten tube is set at 30 kv.

For the lead glass capillary, larger diameters of the focus are effective (D2 and D3 in figure 4, A) than for the platinum collimator (D1 in figure 4, B). However, the effective diameter depends on the wavelength λ. The relative intensities of the

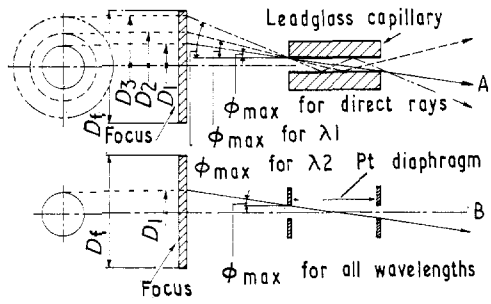

Figure 4 When a lead glass capillary A is used the total reflection against the inner walls results in a bigger total amount of radiation. Radiation of a larger region of the focus comes through the capillary, than in the case of the platinum diaphragms of the same diameter. $\lambda_1 < \lambda_2$

x-rays from the two collimators were calculated using expressions (1) and (3) (figure 5). For wavelengths larger than 1·3 Å, the entire focus is effective and the relative intensities of the two collimators have the constant ratio r_c, found to be 40·5.

It is interesting to compare this value with the ratio r_m measured using a flowcounter which has a measured range from 1·5 to 2·3 Å. Thus the measurements will not be influenced by the change of the spectrum below 1·3 Å (cf. figure 5). Putting

$$r_m = \frac{\text{total amount of radiation measured with the glass capillary}}{\text{total amount of radiation measured with the platinum collimator}}$$

different measurements gave values for r_m between 35 and 45, with an average value of 40, which agrees very well with the calculated ratio of 40·5. Thus it can be concluded that the total amount of radiation of these wavelengths which enters the lead glass capillary is transferred through it without appreciable losses. (This means that such a capillary can be used as a device in fibre optics for x-rays.)

A striking difference in performance between the diaphragm and the capillary collimators is that the latter results in a more divergent beam (cf. figure 4), as was also stated by Hirsch (1960). To determine the divergence in the two cases, the beam diameter at a distance of 1 mm from the collimator was measured (by means of a platinum foil and a flowcounter); for the lead glass capillary the measured diameter was 20 ± 2 μm. This means that

$$\phi = 0{\cdot}005 \pm 0{\cdot}001 \text{ rad.}$$

The angle between a beam which enters the collimator from the edge of the focus and the axis is about 0·004 rad. For the platinum collimator the measured diameter was 11 μm, this means a divergence of 0·0005 rad.

Instead of a glass capillary, it is possible to use two diaphragms of unequal diameter, one with a small hole very close to the specimen, and another with a larger hole further from it. However, a diaphragm which is very close to the specimen will interfere with the diffracted x-rays of the specimen, whereas in the case of a glass capillary the wall thickness near the specimen can be very small because only total reflecting x-rays come through; therefore, in principle a very tiny tip of such a capillary can be brought very close to the specimen without disturbing the diffracted beams.

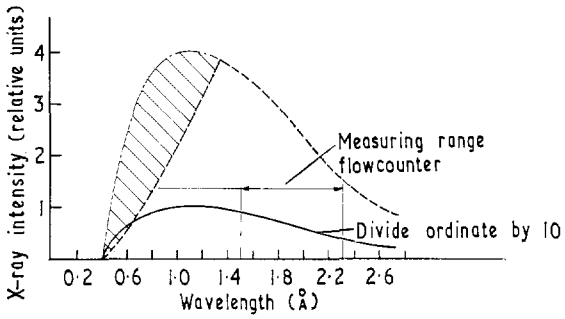

Figure 5 The spectra behind a lead glass capillary and a platinum collimator, both with a diameter of 10 μm at a voltage of 30 kv. The difference in the spectra is caused by the possibility of total reflection at the inner wall of a lead glass capillary. For wavelengths of more than 1·3 Å, the entire focus will send out radiation coming through the lead glass capillary by total reflection. The broken line indicates spectrum for lead glass capillary, the full line spectrum for platinum collimator

5 Results

One application of the camera was to study grain boundary migration during creep of highly pure aluminium. The positioning accuracy of better than 2 μm has proved satisfactory for the purpose. Laue back reflection patterns were made at 30 kv, 20 mA, using D10 Gevaert film and an exposure of 24 h.

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