

## A Kossel Camera Designed for the Cameca Electron Probe Microanalyser

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The camera described is built as an attachment for the Cameca electron probe microanalyser, interchangeable with the standard specimen-stage drawer. It can be used for transmission-pattern recording, or by the incorporation of a special specimen holder, for back-reflexion patterns. In both modes, films are kept in air and are parallel to the specimen surface. The features incorporated in this camera, associated with computer calculations of an analytical treatment of the patterns, take full advantage of Kossel patterns, as a very easy to use, very fast and very accurate X-ray crystallographic technique.

### Introduction

Early in the use of electron probe microanalysis, Castaing & Guinier (1951) showed that an electron probe instrument, in which a very small X-ray source is obtained, is ideally suited for the production of Kossel or divergent beam X-ray patterns.

Although there has long been interest in this X-ray crystallographic technique and it has been illustrated by some remarkable applications, it can be said that development to full potential has been hampered by difficulties related to the calculations involved in making a proper use of these patterns, and by technological problems encountered in recording them.

It is the purpose of this paper to present a new design of camera which makes possible easy and fast recording of divergent-beam X-ray patterns, and to emphasize that such a design, in conjunction with related computer programs, makes the use of these patterns very simple, even for people not really trained in crystallography.

### Design considerations

#### General

The requirements considered for this instrument were of two different kinds. First, the patterns should be easily obtainable in either the transmission or the back-reflexion mode; second the need for an analytical and computerized analysis of the pattern should be fully met.

#### Electron source

An electron probe microanalyser, or a scanning electron microscope, is ideally suited to provide a microsource of X-rays, but, as it has to be used for many other purposes, the change over from the usual techniques to Kossel micro-diffraction has to be completed within a few minutes.

We have used an electron probe microanalyser built by Cameca. The camera has been built as an attachment, interchangeable with the standard specimen-stage drawer. The camera is substituted directly for the specimen chamber without disrupting any adjustments of the electron-optics column.

#### Specimen holder

We need to examine reasonably large specimens. The orthogonal *XY* motions enable more than 20 mm to be traversed in the specimen plane, with coordinates shown on external micrometers.

The analytical computerized technique we use (Hälbig, Ryder & Pitsch, 1969), is valid for any crystal orientation. It gives the most accurate results when the specimen surface is exactly parallel to the film plane. This can be machined usually to an accuracy of better than a few tenths of a degree.

The knowledge of the centre of the pattern and the specimen-film distance is not needed. A goniometer stage is not necessary. In these conditions specimen handling is very simple.

In both stages, transmission and back-reflexion, the specimen can be observed by using scanning images

obtained with secondary electrons or with the specimen current. One also needs to have an optical microscope in order to recognize metallographic features which might not give contrast in electron images.

#### Film chamber

The source-to-film distance should be as large as possible as it is the major variable by which the contrast of the patterns can be improved. On the other hand, it is desirable to make the X-ray divergent angle intercepted by the film as large as possible, in order to record enough conic sections for interpretation and analysis. If the film is large enough, prints and enlargements are not needed. This saves time and avoids problems related to uneven shrinkage of photographic prints.

It was decided, as a compromise, to work with semi-aperture angles between 35 and 50°. Furthermore in transmission the specimen-film distances may be variable between 50 and 170 mm and in reflexion are generally fixed at 90 mm. Films of standard format, either 13 × 18 or 9 × 12 cm are used.

A major design characteristic of the film chamber is the use of a window to separate the specimen and the film – this allows the specimen to be kept in a vacuum while the film is in the air. This has been done both for the transmission and for the back-reflexion modes. An air film chamber offers many advantages, as discussed by Vieth & Yakowitz (1966). It allows multi-exposure without disturbing either the vacuum, the specimen or the alignment and it permits immediate processing. Films are wrapped in black polyethylene foil and need not be shielded against back-scattered electrons. The vacuum is not disturbed by films out-gassing. With direct access to the film there is no need for any shutter. The filters can be easily inserted in the X-ray paths.

These conditions have been obtained in the back-reflexion mode by tilting the specimen against the beam axis, as the angle of incidence of the electrons on the specimen has no effect on the patterns. With such a geometry there is no need to punch a hole in the film near the centre of the pattern for the primary beam to pass through. Thus, our film is undamaged, and an important part of the pattern is not cut off.

With the X-ray wavelengths involved, an air path increases exposure times by less than 20 or 30%. On the other hand, soft bremsstrahlung radiations are absorbed before reaching the film, and this does, in fact, improve the contrast. Air scattering along the distances involved is negligible.

The window material is mylar 200 µm thick. Such

windows are very reliable; they have been used in this instrument for more than eighteen months without replacement.

#### Geometrical limits

All these requirements had to be met both in the transmission and in the back-reflexion modes of recording the patterns. In our design, we decided to build a camera with two separate specimen holders, one for each mode. This was found to be the most economical way of realization, compatible with mechanical accuracy, and the easiest configuration to fit in the room available in a specimen-holder drawer.

### Description

#### Transmission stage (Figs. 1, 2).

The specimen is held in metal frames which can be machined with various openings. The holder carries a polished thorium oxide standard for electron-beam alignment and is connected to the specimen current amplifier.

The specimen can traverse *X* and *Y* over 24 mm. It lies in an horizontal plane, adjustable with a *Z* movement to the focus plane of the standard optical microscope of the microanalyser (magnification 400 ×).

#### Back-reflexion stage (Figs. 3, 4).

The specimen is inserted in an holder similar to the massive specimen holder of the electron probe ana-

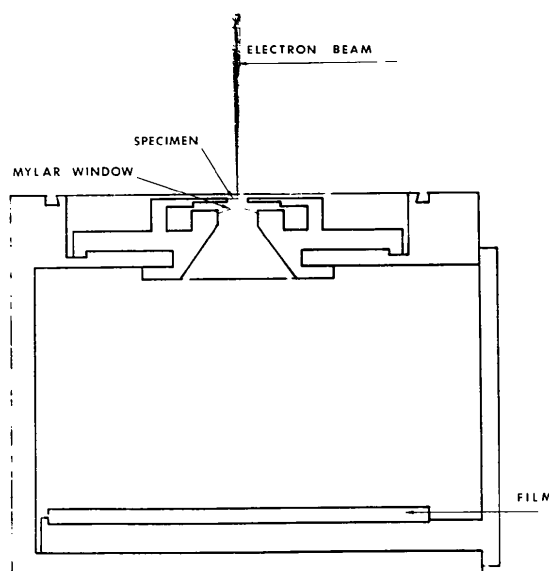


Fig. 1. Transmission stage, schematic.

Table 1. Examples of operating conditions using Kodirex film

	Target	Beam	Film distance	Exposure time
Transmission	Fe 30 µm	30 kV 60 nA	100 mm	10 min
Back reflexion	Fe massive	35 kV 8 nA	90 mm	5 min

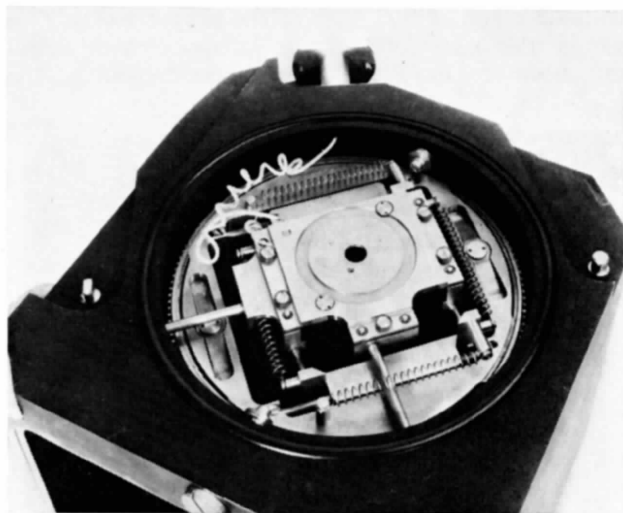


Fig. 2. Transmission stage, specimen holder.

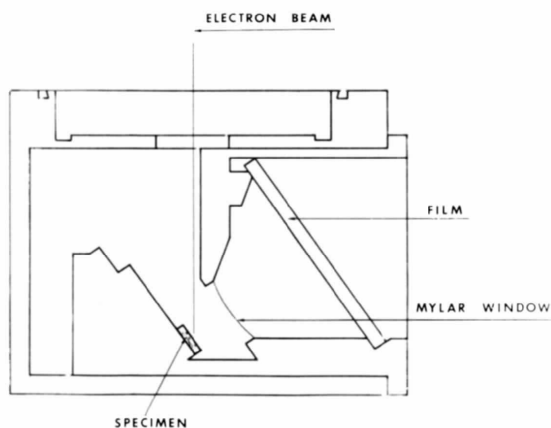


Fig. 3. Back-reflexion stage, schematic.

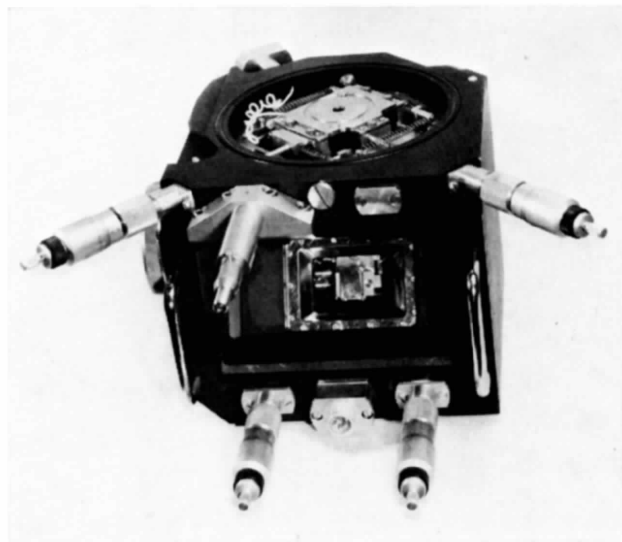


Fig. 4. Back-reflexion stage, and overall view of the camera.

lyser. It can be 20 mm square and 8 mm thick. The holder carries a fluorescent screen for electron-beam alignment; and it is connected to the specimen current amplifier.

The specimen lies in the lower part of the camera, and its plane is tilted by  $55^\circ$  to the beam axis. The mylar window which separate the air film chamber from the vacuum is transparent. Thus, an optical microscope can be inserted in this air chamber, in front of the specimen, and adjusted so that the point bombarded by the electrons can be looked at. Because of the long focal distance, magnification is only 200 times. Thus, any point on the specimen can be selected by either scanning or optical microscopy. The microscope includes a metal shield to protect the operator. But it is important to point out that, because of the low probe currents, X-ray emission is very low as compared to standard X-ray tubes.

The specimen lies at a relatively long distance from the focusing lens of the microanalyser, but with the low beam currents needed the probe diameter is only one micron in these conditions.

#### *Operating conditions*

Table 1 gives examples of operating conditions.

### **Applications**

#### *Efficiency*

The camera was designed in 1971 and has been operating since January 1973. This attachment is shared between the IRSID and the CEA/SRMP electron probe microanalysers. In fact, it is only used a small part of the time because the instruments are used for other kinds of work. In this limited period of eighteen months as many as a thousand patterns have been recorded as a result of the very easy and efficient way the attachment works. Such efficiency has been fully exploited with the help of computer programs which were written in order to treat the patterns.

#### *Computation*

Indexing of the lines is carried out by comparison with standard stereographic projections. These can be drawn by a computer, from a program valid for any crystal structure in any orientation (Philibert, Tixier & Waché, 1971). Such a comparison is generally easy to perform and requires no special crystallographic knowledge.

Only those conics needed to define the four points,  $P_i$ , of the Hålbjg, Ryder & Pitsch analytical method (1969), are indexed. The distances between those four points and point  $T$ , the intersection of  $P_1P_2$  and  $P_3P_4$  are then measured on the film. The whole operation generally takes about five minutes. The following calculations are done by a computer in a few seconds; Fig. 5 is an example of a transmission pattern. Fig. 6 is a back-reflexion pattern used for orientation.

The orientation is calculated, even for non-cubic

crystals, with an accuracy generally better than one degree. The computer gives the position of the normal to the specimen plane relative to a given stereographic triangle or it can plot a stereographic projection of poles for this direction. The crystal is fully oriented as not only the normal, but also two known directions ( $P_1P_2$  and  $P_3P_4$ ) perpendicular to it, are given.

For lattice constant determination a method is used (Tixier & Waché, 1970), which can be applied to almost any pattern as it does not require a defined conics configuration. It makes it possible to compute the lattice constant for a cubic crystal or one of the parameters of a non-cubic one. The program includes a refraction correction.

As there are redundancies in the input parameters, the programs contain internal checks in order to give an estimation of the accuracy and to detect any errors in the input data.

### Studies

This camera has been used for systematic studies of orientation relationships in low-carbon steel sheets. For instance we have obtained maps on which the orientations of the grains observed on the micrographs could be distinguished for a series of heat treatments.

It was possible to obtain accurately the orientation of grains with a diameter down to five microns.

Other applications include studies on polygonization, measurements of lattice constant and phase identifications.

### Future developments

Some accessories are planned in order to make possible other applications. For the divergent-beam applications, we need to build a retractable anti-

cathode made of thin foils of the proper metals. We would also like, for accurate lattice-constant determinations, to include a temperature control of the

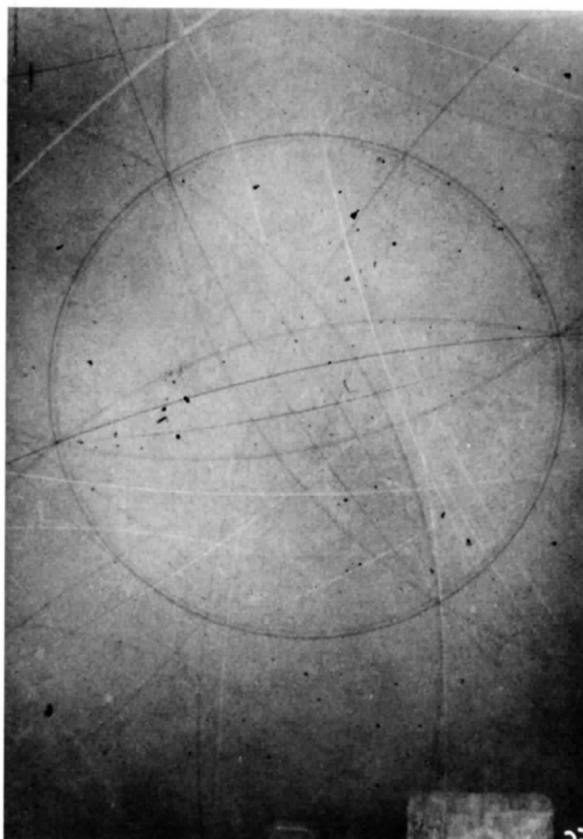


Fig. 5. Transmission pattern [Fe, Fe  $K\alpha$ , (110)]. Positive.

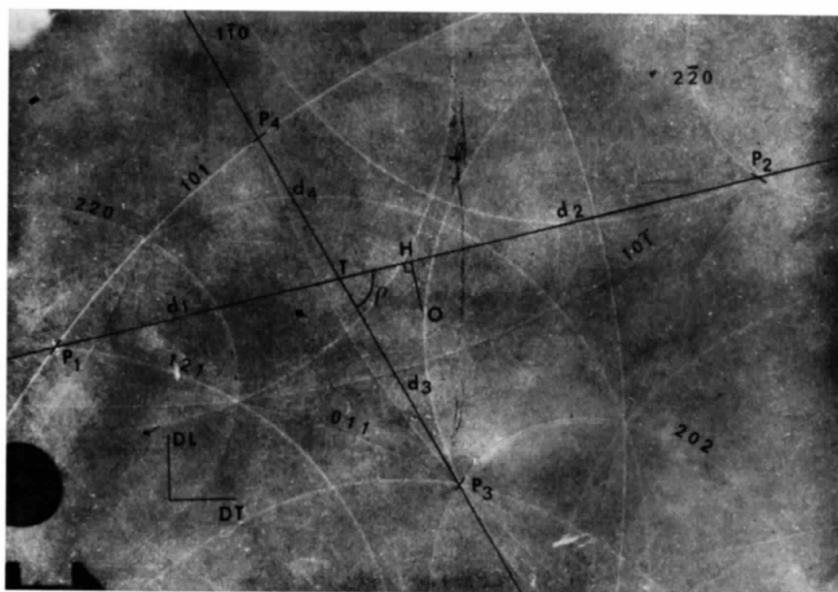


Fig. 6. Back-reflexion pattern [Fe, Fe  $K\alpha$ , near (100)] used for orientation. Positive.

specimen, as in the system described by two of us (Seguin & Maurice, 1975).

It has also been proposed that a device for *in situ* loading and heating of the specimen is installed. These modifications are possible because the specimen chamber, especially in the back-reflexion mode, is large with an easy access to the specimen and with ready-to-use electrical and mechanical connexions.

### Conclusions

This camera has proved to be efficient and easy to use. Other designs have been proposed, for example, by Bevis & Swindells (1973), Perkareev, Kotyokov & Christiakov (1971), Ullrich (1966), Brümmer, Brauer & Suvalski (1963), but this one is quite simple. It has many advantages over the others, in particular that the films are in the air and that patterns are recorded easily and quickly, in conditions which fit computer exploitation.

The results of such a technique are faster to obtain, more accurate and adapted to smaller crystals than other X-ray micro-crystallographic methods. Considering the crystal size, this technique fills the gap between X-ray diffraction and the electron microscopy of thin crystals. It is far easier to use and far more accurate than the pseudo-Kikuchi technique in scanning electron microscopy.

With the very low probe currents needed there are no radiation hazards for the operators.

As electron probe microanalysers or scanning elec-

tron microscopes are frequently available in laboratories where X-ray crystallographic techniques are also used, there is a real opportunity for sometimes using these instruments as very high performance microdiffractometers.

Assistance in the conception, design and realization of this instrument from M. Gervais, A. Giraud, G. Graton and A. Quennevat is gratefully acknowledged. (Patent Pending 72-18104.)

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