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STEREOMETRY OF CRYSTALLINE PHASES OBSERVED IN SCANNING ELECTRON MICROSCOPY

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Abstract

A study of edges and angles of known samples has been performed by means of stereoscopic pairs with electron microscopy in two and three dimensional systems. The derived equations have been included in a computer program. Measurements on stereoscopic pairs with different tilt angles using several crystals of galena, calcite and other objects with known dimensions have been performed. The optimum tilt angles have been obtained by comparison with the actual values, and a logarithmic ratio between these angles and the stereoscopic pairs magnification has been found. The errors obtained using this system of calculation are lower than 6 % in the measure of angles and 15 % in the case of edges.

Key Words: Scanning electron microscopy, stereoscopic pairs, stereometry, optimum tilt angles, measurements of angles and lengths.

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Introduction

The photograph of an object is considered as a conic perspective.When photographs are available they can be used to obtain the real dimensions of the photographed object if the necessary means to solve tridimensionally the conversion of such a conic perspective into a dihedral system (Couderc 1974).

Scanning Electron Microscopy is particularly useful in this case, because its great depth of focus enables the surface topography to be seen in perspective; however, it is sometimes insufficient. An experienced operator could find the two-dimensional sections and the projections useful, but the sensation of reality, the accuracy, and the true parameters of the tridimensional images can be obtained only by using stereoscopic techniques.

There are five important advantages in the technique of stereometry:

a) Highly accurate measurement.
b) Data that cannot be obtained by any other technique.
c) Low cost, compared with other methods.
d) This method avoids interactions between the measuring system and the object to be measured.
e) Measurements can be obtained at any time after taking the images.

Studying the true dimensions of a crystal, starting from the artificial space created by a SEM stereograph, requires a pair of micrographs of the crystal taken from different points of view, that is to say, at different tilt angles to the horizontal plane, and with the same degree.of magnification (fig. 1).

It has been proven that using small tilt angles in aerial stereometry (Daneo, 1955 and Florence, 1972) causes erroneous results.

This report describes a method for determining the optimum tilt angle between the images forming a stereographic pair, and affords a suitable way to perform the stereopsis (estimation of homologous images and the subsequent calculation of the three dimensions).

The equations used were derived from the published works of Gotthardt (1942), Lane (1969), Wret and Robertson (1983) and Knoesen and Kritzinger (1983). Using these equations a computer program was developed to calculare the desired stereographic parameters, thus reducing computational time and errors.

In order to find the optimum tilt angles in stereometry, models with well-known dimensions have been studied. Comparing the results obtained with the known values the total error can be estimated.

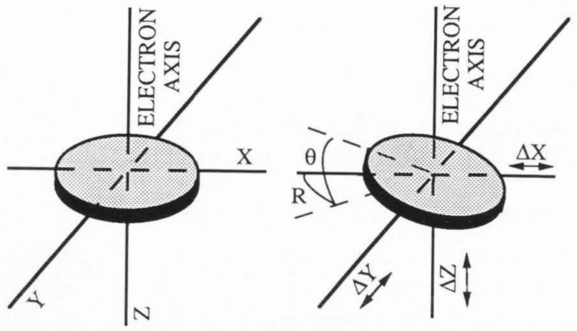

Figure 1. The specimen can be tilted about an axis perpendicular to the electron beam direction and rotated around an axis perpendicular to the specimen plane.

Theoretical Method

In micrographs the coordinate axes of the system (fig. 2) are arranged in this way: Z-axis must line up to the microscope electron beam, OY to the tilt axis of the specimen, and OX must be perpendicular to the other two, OY and OZ axes, and must go through point O (origin of the coordinate system).

Stereometry equations. a) The length l_o (is the hypotenuse of the right-angled triangle defined by the legs h_o and k_o), of a crystal edge in a two-dimensional system (plane XZ) can be determined considering fig. 2, where:

θ_D, θ_I = tilt angles in a micrograph.

$\Delta\theta$ = difference between the tilt angles.

M_D, M_I = magnification degree in a micrograph.

a_D, a_I = lengths of the edges measured on the micrographs.

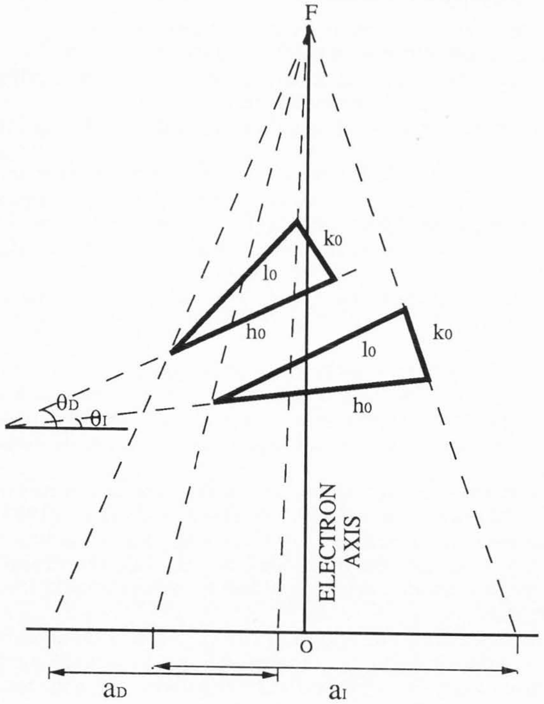

Figure 2. Geometry of the edge in two dimensions.

Gotthardt (1942) and Lane (1969) obtained the parameters h_o and k_o using the equations:

$$h_o = \frac{\frac{a_I}{M_I}\sin\theta_D - \frac{a_D}{M_D}\sin\theta_I}{2\sin\Delta\theta}$$

$$k_o = \frac{\frac{a_I}{M_I}\cos\theta_D - \frac{a_D}{M_D}\cos\theta_I}{2\sin\Delta\theta} \quad [1]$$

therefore, the distance l_o is:

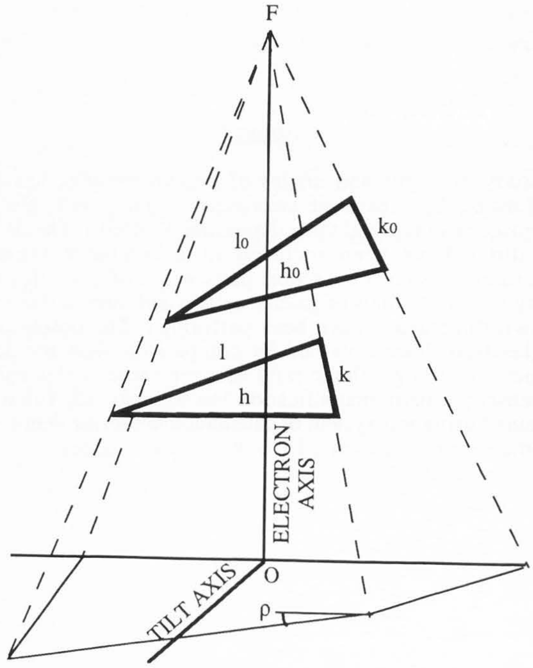

Figure 3. Geometry of the edge in three dimensions.

$$l_o = (h_o^2 + k_o^2)^{1/2} \quad [2]$$

This expression depends on parameters that can be measured on the stereopair micrographs.

When obtaining the micrographs one has to consider:

$M_I = M_D = M$

θ_I = 1.5708 rd., as it is completely horizontal

$\theta_D = \Delta\theta = \theta$

the above equations are simplified as follows:

$$h_o = \frac{a_I \sin\theta - a_D}{2 M \sin\theta}$$

$$k_o = \frac{a_I \cos\theta - a_D}{2 M \sin\theta} \quad [3]$$

and they can be introduced into equation [2].

These results are similar to the ones used by Heidenreich and Matheson (1944) and Martin et al. (1976).

Lane (1969), developed calculations for a three-dimensional system in which according to figures 3 and 4:

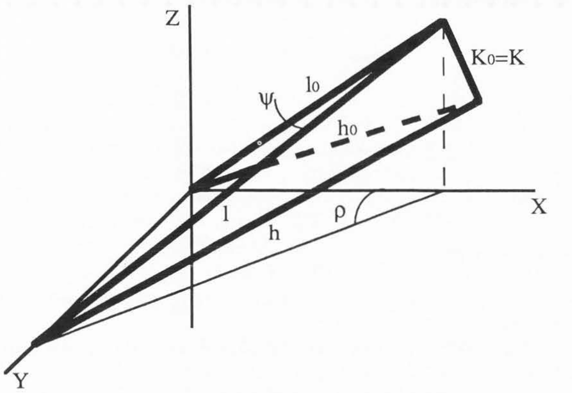

Figure 4. 2-D and 3-D dimension angle relationships

$$\tan \psi = \tan r \cos\left[\theta + \arctan\left(\frac{k_o}{h_o}\right)\right] \qquad [4]$$

where:

ρ_D, ρ_I = angles of deviation from plane XZ of the edge projections on plane XY.

ψ = angle of deviation of the true edge from plane XZ.

We obtain:

$$h = \left[\frac{h_o^2 + k_o^2}{\cos^2 \psi} - k_0^2\right]^{1/2} \qquad [5]$$

since k = ko, the edge length will be:

$$l = (h^2 + k^2)^{1/2} \qquad [6]$$

If we refer to an edge lying on the plane which is parallel to the specimen holder (plane XY), we are in the special case where

$$k = k_o = 0 \qquad [7]$$

and hence the equations [4] and [5] are modified as follows:

$$\tan \psi = \mathrm{tg}\, \varphi \cos \theta$$

$$h = \left(\frac{h_o^2}{\cos^2 \psi}\right)^{1/2} \qquad [8]$$

b) In order to evaluate the angle γ_o between two edges of a crystal facet, which is on plane XZ (figs. 5 and 6), we can use equation [5] for edge A, and divide k_{oA} by a_I:

$$\delta_A = \arctan\left[\frac{\cos \theta - \left(\frac{a_D}{a_I}\right)}{2 \sin \theta}\right] \qquad [9]$$

In a similar way, for edge B:

$$\delta_B = \arctan\left[\frac{\cos \theta - \left(\frac{b_D}{b_I}\right)}{2 \sin \theta}\right] \qquad [10]$$

The angle γ_o between two edges in a two-dimensional system is thus:

$$\gamma_o = \pi - (\delta_A + \delta_B) \qquad [11]$$

These results are similar to those obtained by Wert and Robertson (1982).

The equations above are the clearest evidence of the necessity of using wide tilt angles in stereometry, since otherwise the smaller the angle, the higher the values (a_D/a_I), (b_D/b_I) and $\cos\theta$ approximates one, whereas $\sin\theta$ approximates zero, and therefore the error in the calculations increases considerably.

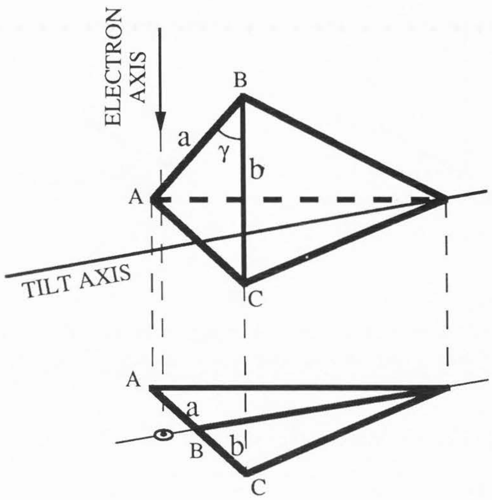

Figure 5. Geometry of the angle in two dimensions Side view and view along beam axis.

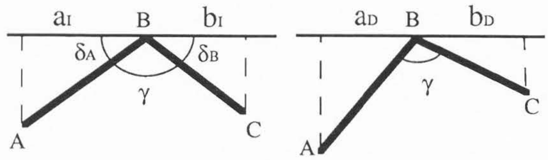

Figure 6. Geometry of the angle in two dimensions. Side view along tilt axis.

In a three-dimensional system, the angle γ between two edges A and B, both in the same plane, can be estimated according to Knoesen and Kritzinger (1983) (fig. 7) using the equations:

$$\cos \gamma = \frac{a_I\, b_I \cos(\alpha_{AI} - \alpha_{BI}) + V\, T}{\sqrt{a_I^2 + V^2}\,\sqrt{b_I^2 + T^2}} \qquad [12]$$

where

$$V = a_I\left(\frac{\cos\alpha_{AI}\tan R}{\sin\theta} + \frac{\sin\alpha_{AI}}{\tan\theta}\right) - a_D\left(\frac{\sin\alpha_{AD}}{\cos R \sin\theta}\right)$$

$$T = b_I\left(\frac{\cos\alpha_{BI}\tan R}{\sin\theta} + \frac{\sin\alpha_{BI}}{\tan\theta}\right) - b_D\left(\frac{\sin\alpha_{BD}}{\cos R \sin\theta}\right) \qquad [13]$$

α = deviation angle of the vector with respect to Y-axis.

R = rotation angle to obtain the stereopair.

Every parameter that appears in the equation is easily measured on the corresponding micrographs.

Computer Program

A computer makes it possible to automate the information; the numerical data are processed in a quick and accurate way, provided the appropriate programme is available.

The programme devised for the calculations is 3D-SEM-1. Its flowchart is shown in fig. 8.

The parameters required for this programme are:

- Magnification of the photographic plates on which the measurements have been taken.
- Tilt angle used to obtain the stereopair.

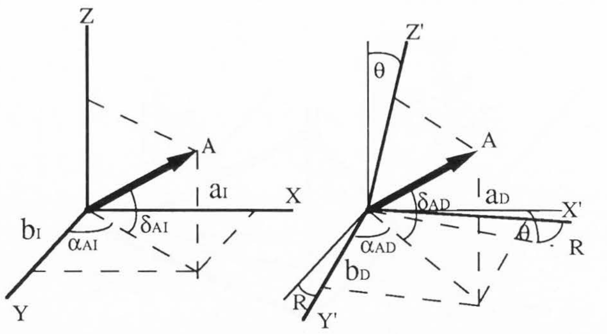

Figure 7. Geometry of the edge in three dimensions. Stereoscopic pairs of the vector A: in base XYZ and base X'Y'Z'

- Dimensions (in mm) of the edges projected on the planes of the two stereopair micrographs.
- Angle between those projections and the tilt axis.

When measuring this parameter, we have to specify that to obtain the true size of the edges, the smallest angle is to be used; and moreover, in order to obtain the angles between the edges on a plane, all the angles are to be measured anticlockwise.

Experimental Procedure

A JEOL JSM-35C scanning microscope (SEM) was used to obtain the stereopairs. The copies of the images were made on photographic paper, being careful to keep the magnification constant during the processing of each set of micrographs. The measurements on the paper copies were taken with a profile projector NIKON 6CT2 which is able to work at magnifications of x 10 or 20; the error in linear measurements was 0.001 mm, and 2.9 10^{-4} rd. in the angular ones. The calculations were made with a personal microcomputer.

The same process can be developed using an image-digitizer system by connecting a video camera to the microscope and using an interphase GEMLOCK and a digitizer DIGIVIEW.

To estimate the optimum tilt angles between the images of a stereopair, we used geometric models of known dimensions (Table 1). The first six tests were made at a maximum magnification of x 65, in view of the restrictions found when

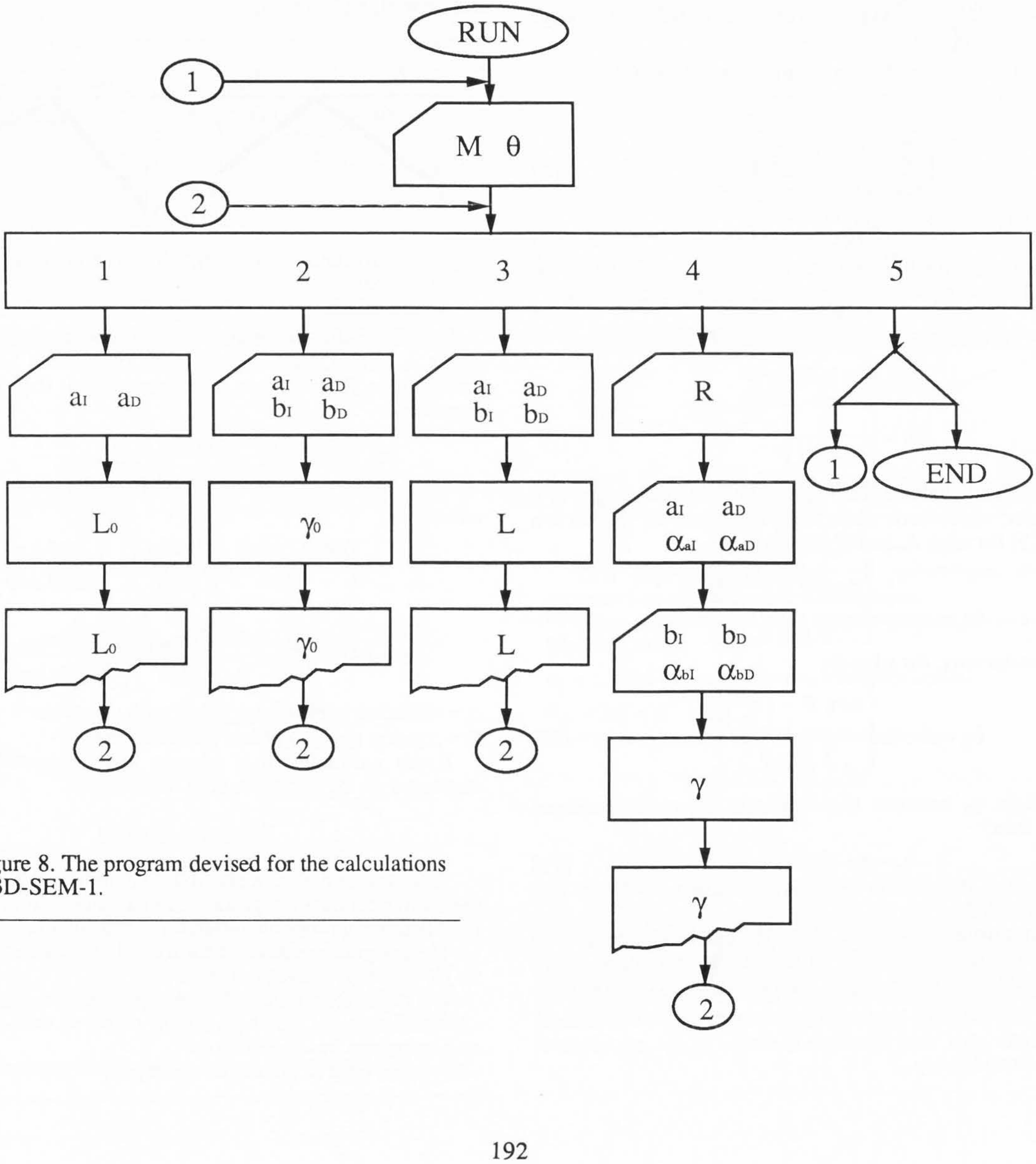

Figure 8. The program devised for the calculations is 3D-SEM-1.

Specimen	M	Model	Dimensions
1	14.8	Diamond penetrator	angle (γ)=1.48 rd
2	23.5	"	"
3	43.6	Big hardness indentations	edge (l)=1.59 mm and angle (γ)=1.48 rd
4	23.6	"	"
5	65.5	Small hardness indentations	edge (l)=0.67 mm and angle (γ)=1.48 rd
6	23.6	"	"
7	200	Calcite crystal	angles (γ_1)=1.99 rd and (γ_2)=1.15 rd
8	100	"	"
9	150	"	"
10	120	"	"
11	500	Galena crystal	angle (γ)=1.57 rd
12	1000	Twin in bronze	angle (γ)=1,57 rd
S-1	190	Simulated cube	edge (l)=0.10 mm and angle (γ)=1.57 rd
S-2	19	Simulated pyramid	edge (l)=6.40 mm and angle (γ)=0.91 rd

Table 1 Geometric models of known dimensions

working with large models, if compared with the usual specimen seen in the SEM. The models used were:
- Diamond penetrator used in Vickers hardness test; this consists of a regular quadrangular pyramid, whose opposite faces form an angle of 2.374 rd. at the vertex. For observation by means of SEM, it is covered with gold (fig. 9).
- Using this penetrator, two hardness indentations of different depths are made on a parallel-sided zinc specimen (fig. 10).

Only when measuring lengths can we work at higher magnifications using crystals of a well-known structure. The size of the crystal edges that are measured depends on parameters, such as the solidification speed or the indent depth; the angles forming the edges depend only on the crystalline structure of the specimen. This parameter is obtained in tests 7 to 12. The models used were:
- Calcite rhomb crystals.
- Galena crystals obtained by brittle fracture after plunging the original piece into liquid nitrogen.
- Bronze with 4 wt % Sn, annealed for 60 minutes at 700 ºC in order to eliminate any tensions, and then was air cooled. Subsequently, its surface was polished and it was compressed in the direction of the longitudinal axis; afterwards it was treated with an acid alcoholic solution of ferric trichloride in order to develop the microstructure (twins and grains boundaries).

Moreover, two geometrical models of stereopairs - cube and pyramid -, were simulated using a computer in tests S-1 and S-2.

From each of these models, and using different magnifications, sets of images with different tilt angles were made. The calculations were made for each of the possible stereopairs formed according to the following sequence (Stolz 1980):

a) The first image of the specimen was obtained in horizontal position, i.e. with a tilt angle of 0 rd. This made the subsequent calculations easier.

b) The structural detail to be observed was located on the microscope screen, as accurately as possible.

c) The specimen was tilted at an angle of θ rd.; this movement caused a distortion due to the perspective that is automatically corrected, and the change in position of the detail or structure, that is corrected after displacing the specimen-holder according to X - and Y - axes, or after rotating it R rd. If during this process the image is put out of focus, the restoration of the optimum working conditions must not be carried by changing the current in the microscope lenses, as this would change the magnification; to correct it displace the specimen-holder along the Z-axis.

d) After these adjustments we can obtain a second image, which together with the first one will form the stereoscopic pair. Repeating the process we obtain the necessary series of images.

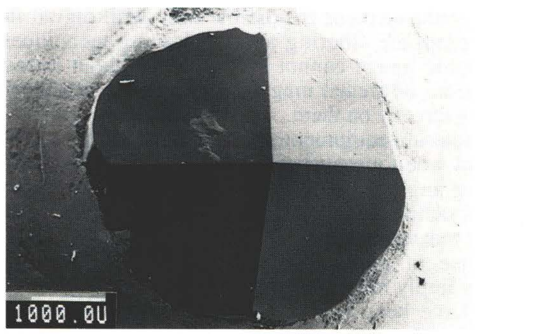

Figure 9. Diamond penetrator used in Vickers hardness test

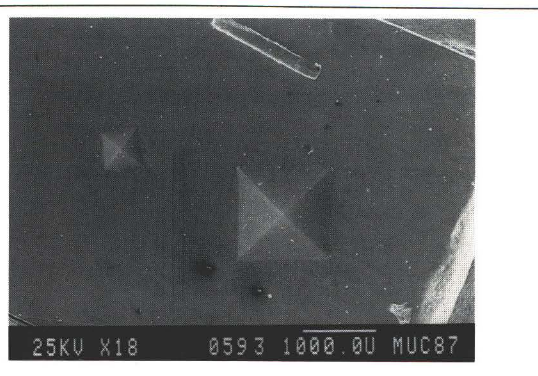

Figure 10. Two hardness indentations of different depths are made on a parallel-sided zinc specimen

e) Measurement of necessary parameters.
f) The three-dimensional parameters are obtained by using a computer program.
g) An appropriate statistical treatment of the results will allow us to predict the optimum tilt angle for each specimen.
h) To check the results, it was necessary to obtain the stereopairs with the optimum tilt angle.

By repeating the measurements and the calculations, and comparing the results with the true values, the total error is apparent.

Results and Discussion

Measuring involves an admission that we are making certain errors that will distort the final results. Apart from accidental errors due to an experimenter's imperfect work, and after correction of the systematic error of the equipment used to obtain and measure images, the resulting error is due to the limited accuracy of the instrument and the random errors made during measuring.

Image recording is performed using photochemical (photographic) or electronic (computer memory) methods. The former methods have the advantage of producing an image that is easily transported and reproduced, whereas the latter have the advantages of producing an internal analog to digital conversion, that allows the figures to be obtained directly.

Image enhancement is possible only with digital images, using appropriate filtering and reinforcing techniques. The photographic image cannot be restored without introducing imperfections of greater magnitude than those produced when measuring directly on them.

The metering equipment used has a sensibility threshold of 0.001 mm when measuring lengths, and of 0.0003 rd. when measuring angles. These data, together with an appropriate statistical sampling, allow us to state that the average error in the direct measurements is about 0.5 - 0.8 %.

The use of parameters more difficult to obtain will obviously imply higher errors; with the magnification interval that we have been using, this error could be estimated at between 2-5 %.

The data obtained in the series of images of the first tests are introduced in the program 3D-SEM-1. Its fourth section provides a certain set of values of the crystal angles, which vary linearly with the tilt angle used to obtain the stereopair. We obtain the optimum tilt angle by interpolation. Once the images corresponding to this angle are obtained, we repeat the calculation process, comparing the experimental values with the real ones, and the aggregate error can be estimated in each test.

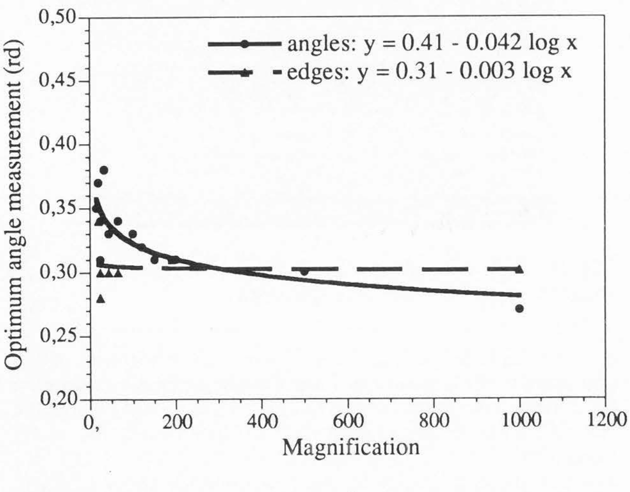

Figure 11. Optimum tilt angle measurement versus magnification.

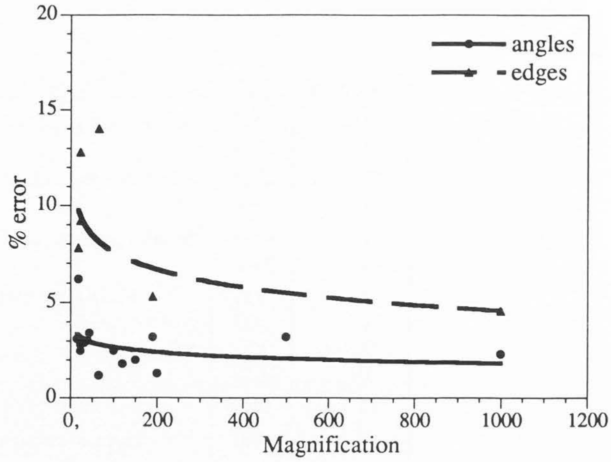

Figure 12. The errors obtained using this calculation process are lower than 6 % in the measure of angles and 15 % in the case of edges.

The graphic display of the optimum tilt angle in each test (y), versus the magnification (x), gives an experimental curve (fig. 11), according to this equation:

$$y = 0.41 - 0.042 \log_{10} x \quad \text{(for angles)} \qquad [16]$$

We need not assume that the process of the determination of the crystal edges is different from the determination of angles. When we introduce the data in the third section of 3D-SEM-1 program, we obtain a series of values of the crystal edges that we are studying. Using a calculation program similar to the one developed above, we can obtain the optimum tilt angles in each test. The variation of the tilt angles as the magnification changes provide us with an experimental curve (fig. 11) similar to the one above, and according to:

$$y = 0.31 - 0.003 \log_{10} x \quad \text{(for edges)} \qquad [17]$$

Comparing the experimental values with the real ones, we can estimate the aggregate error made in each test (fig. 12).

Conclusions

An overall view of the tests that were carried out confirmed that in using a stereoscopy system, we can obtain the value of the edges and angles of the crystals studied in SEM. It is even possible to reconstruct them using computer-assisted print systems. Both in two- and three-dimensional systems, by extrapolation or interpolation we can obtain the theoretical tilt angles that give optimum results in stereometry at any magnification.

The results obtained in tests at certain previously predicted tilt angles, had aggregate errors of between of 5 and 10 %, a value not at all unreasonable when dealing with measurements of this kind.

The advantages of this method are the following:

- It is a non-destructive method of measurement. Obtaining several micrographs does not disturb the material characteristics.
- It permits us to keep images and data of specimens that have already been destroyed, and thus make other tests; and also images of specimens that were damaged due to lack of stability or other external reasons. The documentation can be studied at a later time.
- The stereometric calculations allow us to estimate - though

with a slight error - the crystal edges and angles, always using a simple and economical method. Likewise, the calculation of the coordinates of the crystal edges offers the possibility of using a computer-assisted print systems, and allows a more thorough study of the crystal.

- The tests show the reliability of the methods of calculation, since the errors are quite small, if we consider the simplicity of such methods.

The main disadvantages of this method are:

- Slow image processing.
- Need for many measurements with the consequent errors.
- Difficulty in analysing very complicated surfaces, such as bent ones or those with overlapping objects.

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