USING THE SCANNING ELECTRON MICROSCOPE TO DETERMINE THE UNEVENNESS OF GRAIN SURFACES AND TO DRAW CONTOUR LINES

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ABSTRACT

This paper describes that the unevenness on a surface of a minute grain of quartz (SiO_2) can be determined with scanning electron microphotographs and illustrated with some contour graphs. Formulas for solving parallax and unevenness are derived and their precision is estimated. With information from a scanning electron microscope, the shape in three dimensions is determined and the contour map drawn on the stereometer of differential type with satisfactory results.

In addition, this paper also presents some features of the analytical process with scanning electron microphotographs.

1. INTRODUCTION

The Scanning Electron microscope (SEM) is a new type of electronic optical instrument, which has made a rapid development during the last ten years. Its principle of image-formation is different from that of the optical microscope. Instead of using a lens to enlarge the image, the image-formation is modulated, by signals stimulated by fine electron-beams scanning on the surface of the specimen in a way somewhat similar to television photography.

The SEM possesses a number of features: a wide range of magnification which can be regulated successively, a great depth of field, high resolution and so on. In order to take stereoimage pair, the stage plane of the specimen has to rotate a known angle to match the stereoimage pairs. The stereomodel of the object under study can then be made up. SEM has been used in many fields, such as biology, medicine, material analysis etc.. Up to now, only topographic observations and analyses of two dimensions on specimens have been made in our country, but their unevenness and contour lines have not been measured. This paper presents some of the results obtained in our experiment.

2. STEREOPHOTOGRAPH TAKEN WITH SEM AND ITS ANALYTICAL PRINCIPLES

By using the microstereopair taken with the optical microscope, the finely undulated surface of the minute object (specimen) can be measured. As the optical microscope has less depth of field, lower resolution, and magnification, it is difficult to use it to examine with precision the varying degrees of unevenness on the surface of the grain. Therefore, SEM was used to take the image pairs of a minute quartz grain under study and satisfactory results were obtained.

2.1 Photographic method

The surface of the specimen can be scanned by electron beam emitted from the electron gun (Fig. 1,b). The electron strength reflected by the surface of the specimen depends on the angle between an incident probe and the surface of a lash spot. The picture shown on the CRT screen is the picture of the specimen surface, which then can be taken with a 135 mm camera. To obtain the relief effect, photos should be taken from two angles. It is necessary to tilt the specimen stage at an angle of θ to the

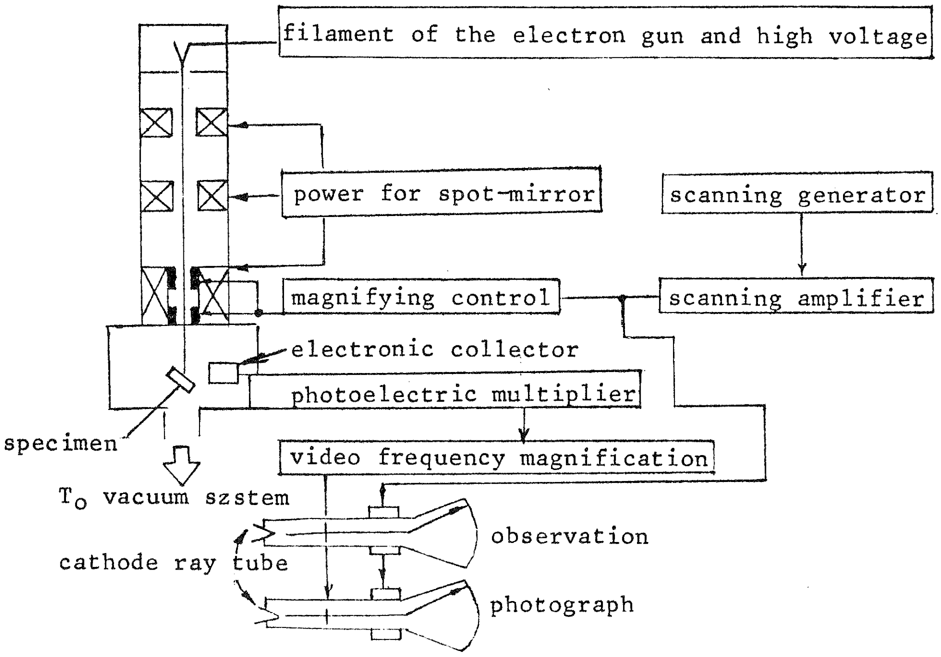


Fig. 1 Systematic block-diagram for scanning electronmicroscope

Y-axis or at an angle of Ω to the X-axis.

The stereopair can be obtained by photographing it twice in a rotation. The different heights on the surface of the specimen shows the different parallaxes. Compared with the magnitude of the specimen, the projection distance (about 60 centimetres high) in this sort of photopair is extremely great because of the extremely high magnification of the SEM. The beam forming the image can be regarded as a parallel beam. The best angle for the rotation of the specimen stage is $\theta = 10^0 \sim 15^0$ when the second picture is taken. If the angle of rotation is small, the relief effect will be poor. If the angle of rotation is big, it may lead to the distortion of the image, etc. . When the photo is being taken, a tiny grain of known size or a line of known scale is needed on the specimen stage in order to check the photographic scale.

2.2 Determination of Parallax P_M

In the photogrammetric work, the stereo-observation method is usually used to observe the photograph. This method is based on the properties of binocular sight. By means of the stereo-observation instrument, the coordinates and the parallaxes of the matching object point (on the point to be located) can be determined with high accuracy.

Microstereophotography belongs to the microclose range photography (the distance being very short). It is characterized by short photogrammetric bases and very few control points for checking the element of exterior orientation. The stereoparallax formed by the microphotograph is shown in Fig. 2.

When photos are taken, the specimen stage is titled at an angle θ, and thus the stereoparallax can be formed from Fig.2(b), we obtain the parallax P of object point M on the specimen.

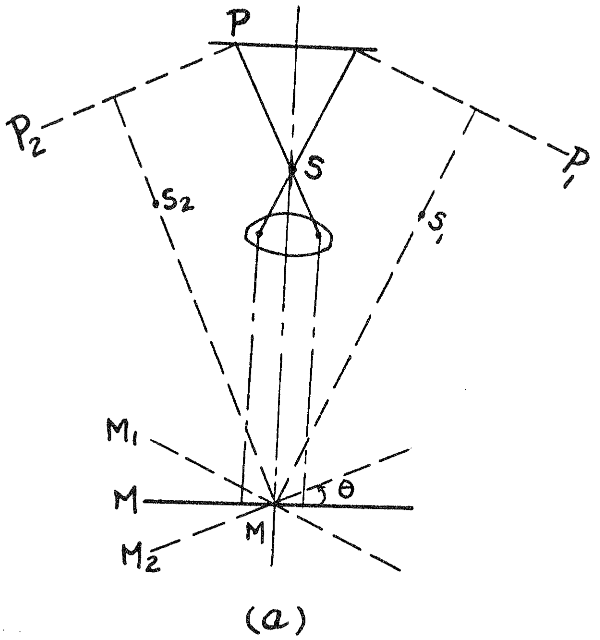


Fig. 2 (a)

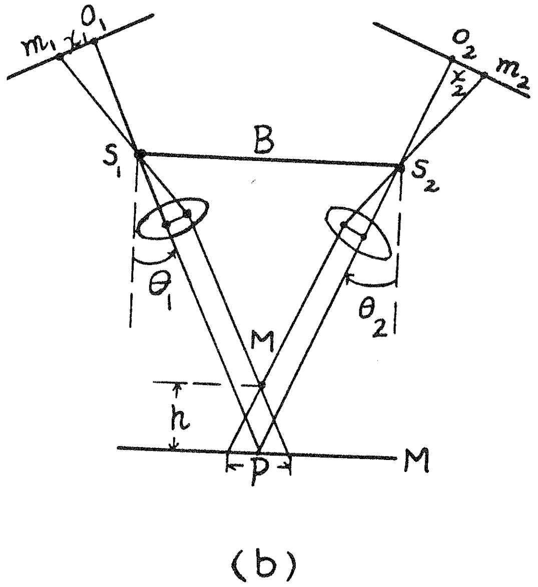


Fig. 2 (b) Illustrated microsteres image pair

$$P_M = h \cdot tg\,\theta_1 + h \cdot tg\,\theta_2 \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

when $\theta_1 = \theta_2 = \theta$,

$$P_M = 2h \cdot tg\,\theta \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (2)$$

The parallax expressed in terms of the photographic scale is as follows:

$$P_M = B_M \cdot \frac{1}{m} = \frac{2h}{m}\, tg\,\theta \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (3)$$

where: m = the denominator of the photographic scale.
θ = angle of rotation of the specimen about axis Y.
$\theta \approx 10^0 \sim 15^0$
h = the height of point M.

2.3 Analytical principles

The Stereo-photogrammetry is carried out on the microphotograph taken with the scanning electron-microscope. Its analytical principle is illustrated in Fig. 3. Point 0 and point M on the surface of the specimen and the scanning probe are shown in the figure. Point 0 is in the plane XY, while the scanning probe is perpendicular to it. The orthography of point 0 and point m can be obtained by the scanning probe and they are shown on the CRT screen. The distance L from point m to Q_xQ_x-axis is measured and then the specimen stage is rotated an angle θ about the Q_xQ_x-axis. The point M on the surface of the specimen moves to point M' and orthographies of point 0 and point m'(xy) are displayed. The distance L' from point m'(xy) to the Q_x axis is measured on the CRT screen. Both these two lengths of L and L' can be measured with the comparator on the photograph or with the squared plate graduated millimetre scale on the CRT screen. But the accuracy of the latter is not high.

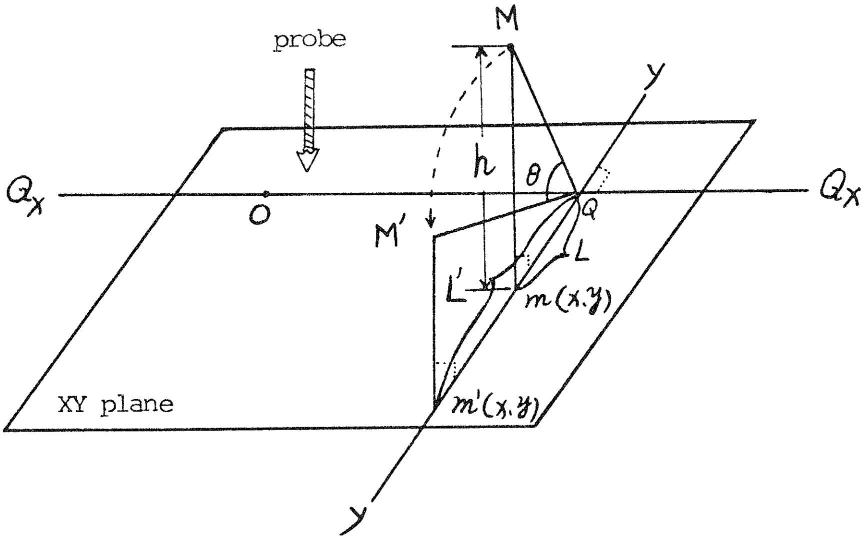


Fig. 3 (a)

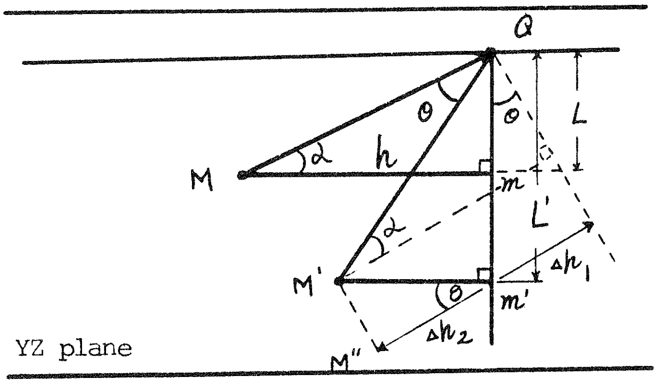


Fig. 3 (b)

From Fig. 3(a) and (b), we have

$$\Delta h_1 = L' \sin\theta , \qquad \Delta h_2 = \frac{L'\cos\theta - L}{tg\,\theta}$$

Substituting $h = \Delta h_1 + \Delta h_2$ into the above formula, and after rearranging the perpendicular distance between point M and point 0, we have the height h:

$$h = L'/\sin\theta - L/tg\,\theta \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (4)$$

where: L,L' can be measured respectively,
θ — the angle rotated about the specimen stage.

2.4 Estimation of accuracy

The image of the photographic specimen on the scanning electron microscope is orthographical, assuming that there is no distortion and that the angle θ is rather small. After differentiating the formula (4) and rearranging and simplifying it, we have the relative error of the height of the point to be located (object point)

$\frac{\Delta h}{h} = \frac{1}{\theta} \left(\frac{\Delta L'}{h} - \frac{\Delta L}{h} - \Delta \theta \right)$, the relative mean square error is:

$$m^2 \frac{\Delta h}{h} = \frac{1}{\theta^2} \left(2 m_L^2 + m_\theta^2 \right) \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \quad (5)$$

where: $\Delta \theta$ is the error of the angle θ
ΔL is the error of L or L'

The term ΔL means that the error in determining the position of the point on the photograph is contained. It depends on the resolution of CRT in a low magnification and on the diameter of the scanning probe on the specimen in a high magnification case.

Now the estimation of error in these two cases are discussed. Let the scanning region on the surface of the specimen be equal to $\ell \times \ell$. There are N lines of resolution on the CRT and the error in determining points on the CRT are proportional to $1/N$. From this, the error m_L in measuring L is given as follows:

At low magnification

$$m_L = C \left(\frac{\ell}{N} \right) \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \quad (6)$$

where C is a constant.

At high magnification, m_L is given by

$$m_L = C' d \quad \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \quad (7)$$

where C' is a constant and d = the diameter of a scanning probe. Substituting the formulas (6) and (7) into error equation (5), we have:

$$m^2 \frac{\Delta h}{h} = \frac{1}{\theta^2} \left(\frac{2}{h} \left(\frac{C\ell}{N} \right)^2 + m_\theta^2 \right) \quad \text{(the error at low magnification)} \quad \dots\dots \quad (8)$$

$$m^2 \frac{\Delta h}{h} = \frac{1}{\theta^2} \left(\frac{2}{h} (C'd)^2 + m_\theta^2 \right) \quad \text{(the error at high magnification)} \quad \dots\dots \quad (9)$$

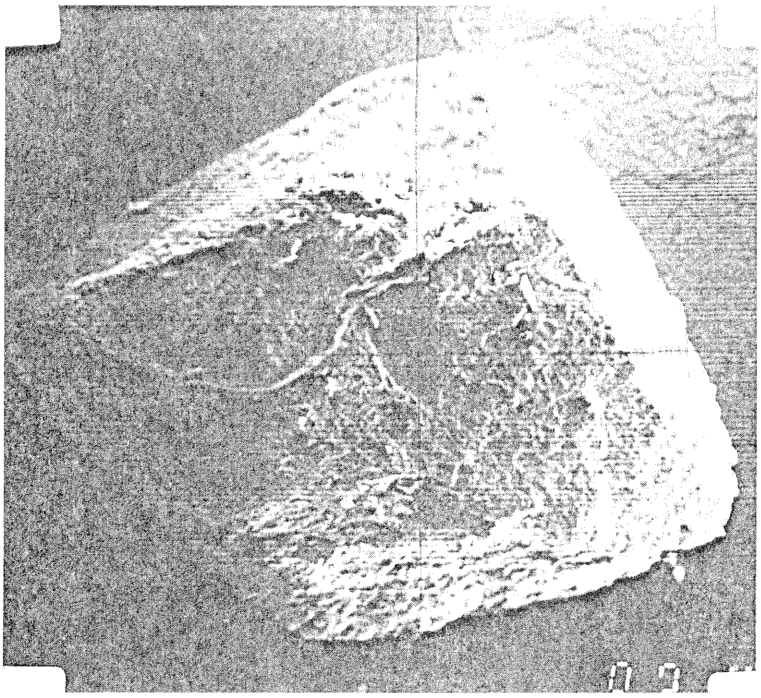
The diameter of the scanning probe d has influence on the error in measuring L, at 5000# magnification, $d \approx 10^{nm}$ ($1^{nm} = 10^{-9}$ m). It corresponds to the measuring accuracy 0.05 mm on the photograph. This is feasible, provided that the accuracy of the measuring instrument is less than 0.05 mm, such as the stereo comparator 1818.

3. CONTOUR MAP SURVEYING

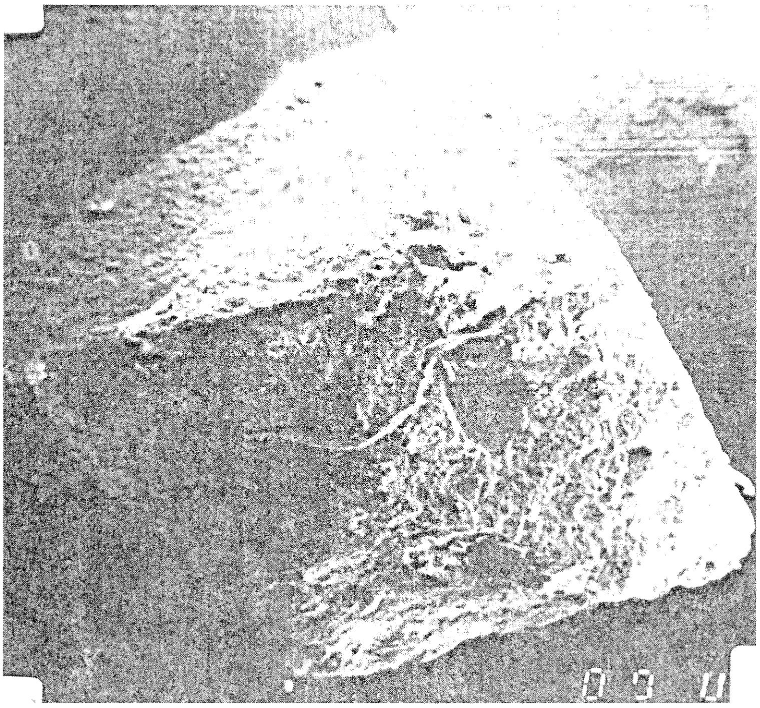
The photograph of the specimen which itself is taken by scanning electron microscope is in principle an image of the central projection, for its projection distance is rather long in comparison with the size of the specimen. The beam that forms the image can be taken as one that is of parallel projection instead of a central one. Therefore, the stereometer of the differential type or the analytical stereo-plotting instrument may be applied to contour map surveying. As there are no restrictions on the mechanical parts of the photo-principal distance, the tilt angle of a picture etc., these instruments are almost suitable for all kinds of photography. It should be pointed out, that when the microphotograph to center is used, lines joining the four corners of the photo or the centers of four sides should be used. Though the precision is not good enough, the height error from the errors of the photo-centering on the orthogonal projection is rather small. The relative orientation, modal-leveling, predetermined scale, isometric line surveying etc. of the microphoto-pair at the stereomapping are basically the same as those used in photography.

4. TESTS

In these tests, we made use of the SEM in the Testing Center of Tongji University, to photo quartz crystals by the above-mentioned method. The stereopairs of the microphotograph were obtained (see Fig. (a) and (b)). On this photopair, the isometric line maps were surveyed with the stereometer of the differential type made in our country. As to the contour interval, the index contour is 40 μm and intermediate contour is 10 μm. The mapping method was carried out according to the general procedure of surveying. The result is that a better relief and a clear convex-concave are obtained. The model-leveling was done by the use of the level of the specimen stage. The photoscale is 100:1, Photofocal distance f = 600mm and isoline map cf. Fig. 5.

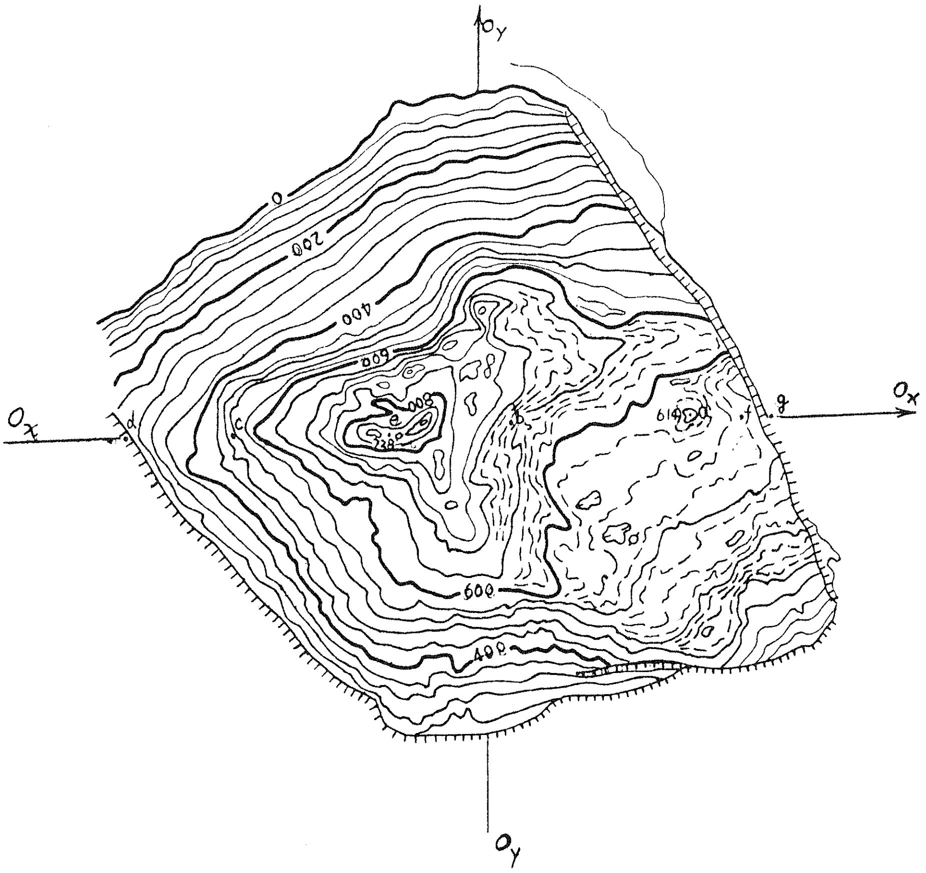


(a) Before rotation



(b) After rotating an angle θ

Fig. 4 Stereopairs for quartz grain taken by scanning electron microscope



Scale: 100 : 1

Fig. 5 Isoline map for quartz grain

Table 1

Point No.	L'_{mm}	L_{mm}	h_{mm}	Remarks
a	31.58	17.24	0.614	$h = L'/\sin\theta - L/tg\,\theta$ $\theta = 14^0.00$ Photoscale: 1:m = 100:1
b	8.01	-8.02	0.651	
c	-37.58	-51.68	0.519	
d	-54.51	-58.00	0.073	
e	-11.03	-29.50	0.728	
f	37.01	23.81	0.574	
g	40.05	41.01	0.008	

By formula (4), we calculated the heights (convex-concave) of seven particular points of the quartz grain on the sectional plane along the Q_xQ_x-axis. The results are given in Table 1. The seven high points on the sectional plane are shown in Fig. 6. The real line refers to the heights obtained by formula (4) and the dash line to the heights read from the isoline map.

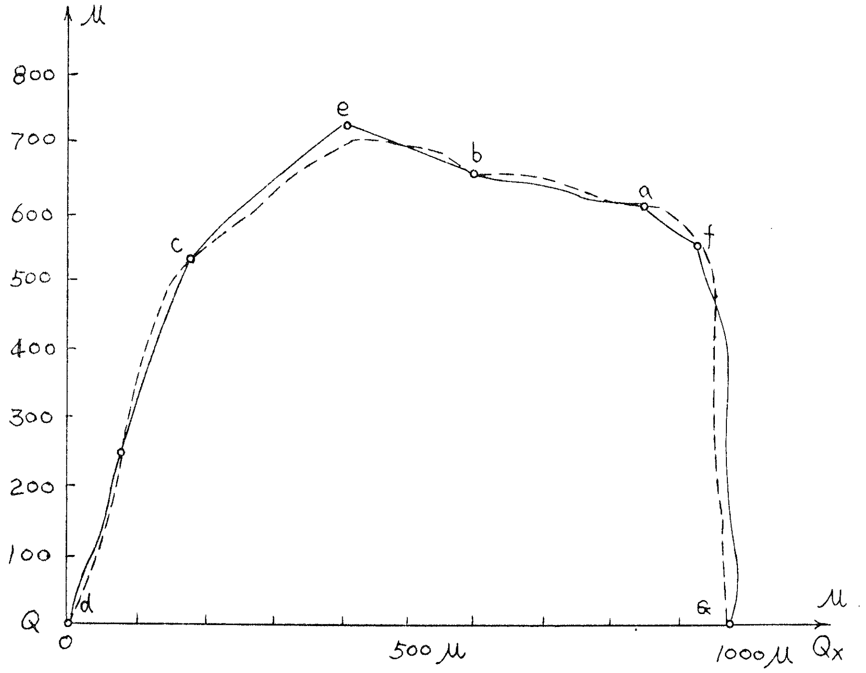


Fig. 6

—— real line is the height of the sectional plane $0 - 0_x$ obtained by analytic methods

....dash line is the height of the sectional plane $0 - 0_x$ read from the isoline map.

5. CONCLUSION

The main purpose of this test is to discuss the process and the basic analytical principle for the stereopair taken with the scanning electron microscope. The quartz grain used in this test is somewhat bigger, therefore we used low magnification, the scan width being about 720 μm, $\theta \approx 14^0.00$. From (8), the calculated error is about 2.5% ($C/N = 1/1200$, $m\theta \approx 15'$). This is identical with the precision of the isoline. Therefore, it is possible to use the scanning electron microscope to conduct stereophotography. The method is simpler and the analytical calculation is rather easy. It can quite satisfy the precision required for three-dimensional analysis in some scientific fields. If we use high magnification (more than $5000^{\#}$) and take all possible errors into consideration, then the accuracy can be highly improved.

REFERENCES

(1) Wang Zhizhuo: Photogrammetric Principle, Surveying Publishing House 1979.

(2) T. Oshima, S. Kimoto: Stereomicrography with a Scanning Electron Microscope, Photogrammetric Engineering, Volume 36, 08, August 1970.

(3) Li Guangwen, Li Weiqian, Yang Siyu: The Research and Experiments on Microstereo Photogrammetry, Surveying Journal, 1985. 3.

(4) Chen Sixi, Wang Chengrui: Metallic Electron Microanalysis , Mechanical Industry Publishing House 1982.