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TEM AND COMPUTING PROCEDURES IN CRYSTAL STUDIES

I. CALIBRATION OF THE ELECTRON MICROSCOPE

by
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Abstract: *Tables and diagrames neeessary for the calibration of the microscope are given. Based on these data, accurate calculations, either on the screen during the observation or on the positive prints afterwards, can be executed.*

1. INTRODUCTION

The JEOL 120 CX electron microscope has a eucentric side entry goniometer stage with tilting facilities within the range of $\pm 60°$. With such a stage the tilt axis can be adjusted so that the image of the specimen can be always tilted by an axis passing through a diameter of the screen.

It is well known that for each one of the functions of the microscope (SA DIF, SA MAG etc.) there are different rotations of tbe image, due to the differeut imaging procedure by the magnetic lenses and to their different excitation. Therefore, it is necessary for the operator to know the direction of the tilt axis on the screen for each function. Once this is known, the rotation relationship between different cases will be also known. This calibration enables the use of the instrumental coordinate system[1] and, therefore, a lot of tilting experiments can be easily carried out.

The results following will be given for both systems, the one which the operator uses and tbe other one for positive prints, because, in electron microscopy, people usually refer their results to positive prints, i.e. to photographs printed with emulsion down.

2. *DEFINITIONS*

a) Directions on the screen (for the operator).

The calibration was made in terms of the instrumental system in order to have the coordinates of a direction compatible with computing procedures, which usually are constructed for positive prints. This means that, if the direction of the real position of the mechanical tilt axis (MTA) is from South to North ($\overrightarrow{SN}$), as in Fig. 1, then its image $\overrightarrow{PQ}$ is characterized by a negative rotation angle $-\varphi$ if the entrance point is lying in the eastern semicircle (as in Fig. 1) and by a positive one if the entrance point is in the weastern semicircle (zero angle at point N). The tilt angle δ, which is readed on the goniometer stage, is taken as positive for counter-clockwise rotations and vice versa. When observing the screen, positive tilt values correspond to directions towards the left side of the direction $\overrightarrow{PQ}$. For example, if for the case of Fig. 1 δ was positive, the correct direction would be $\overrightarrow{OA'}$ and not $\overrightarrow{OA}$. The angle POA′ is characterized as $+\alpha$ according to the system of coordinates proposed[1], because it is on the left side of $\overrightarrow{PQ}$.

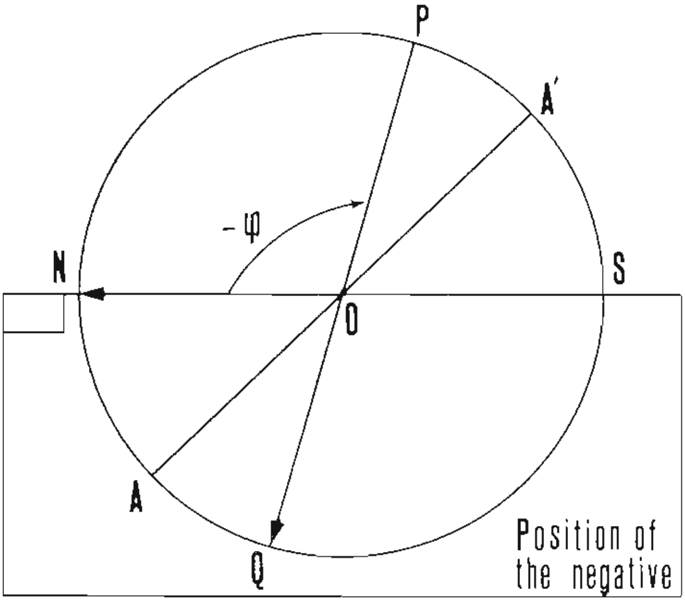

Fig. 1

b) Positive prints.

For the case of positive prints, the roration of the image is always refered to the direction of the image of the MTA, PQ in Fig. 2. The plane of the diagram corresponds now to a Wulff's net, with PQ along the zero-degrees meridian. The tilt angles readed on the goniometer stage, have the same sign as in (a). Positive values of α are now on the right side of $\overrightarrow{PQ}$. For example, $POA' = +\alpha$ for the situation in Fig. 2.

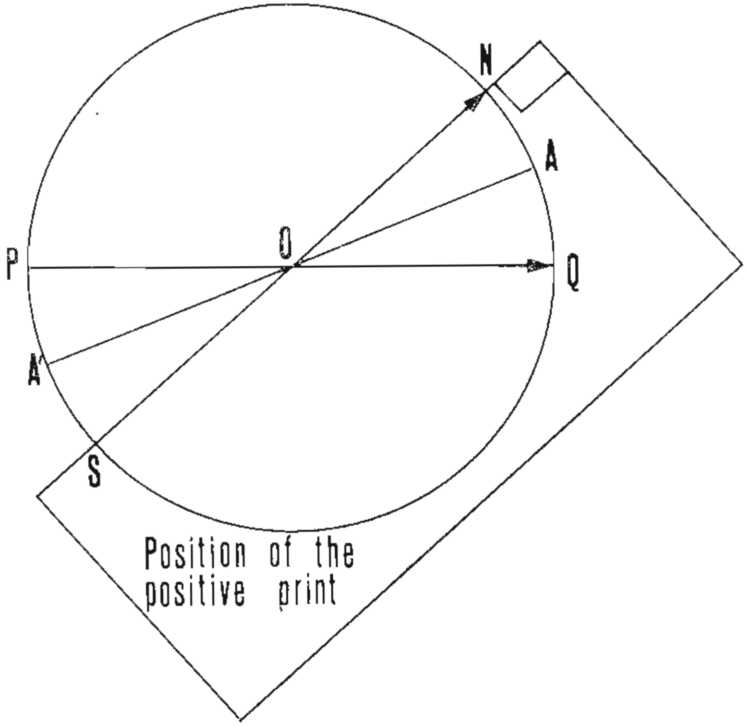

Fig. 2

3. RESULTS

The calibration was based on the above definitions and was made using a copper single crystal and it was checked using the twins of a GeS specimen. Table 1 gives the rotation angle φ of the image of the MTA for the different functions of the microscope. These values are for the operator and they are graphically presented in Fig. 3a while the corresponding graphs for positive prints are presented in Fig. 3b.

TABLE 1

Rotation angles calculated for the different functions of the JEM 120 CX electron microscope
(Accelerating Voltage 100 kV)

SA DIF		SA MAG		MAG		MAG (contin.)	
Camera length	Angle φ^0	Magnif. $(x10^{-3})$	Angle φ^0	Magnif. $(x10^{-3})$	Angle φ^0	Magnif. $(x10^{-3})$	Angle φ^0
32	-134,8	5.9	69	2,4	0	28	100
74	-111,4	8.9	86	2,9	-170	34	105
120	147,5	12	91	3,6	-157	46	115
190	152,3	17	-12,5	4,7	-135	56	130
390	170	23	-15	5,9	-120	83	9
580	-170	28	-13,5	8,9	-125	110	13
LOW	MAG	34	-12,5	12	-130	160	24
Magnif. $(x10^{-3})$	Angle φ^0	46	-7	14	84,5	210	32
0.8	- 87.8	56	1	17	87	260	40
1.2	-63	83	8	23	92,5	400	63
		110	30	SCAN :	Angle	φ = -170,5	

4. COMPUTING PROCEDURE

A basic problem in electron microscopy is to orient the crystal within the instrumental coordinate system in order to be easy for the operator to find the crystal coordinates (Miller's indices) of any observed direction and also to find the instrumental coordinates of a desired crystallographic direction.

To solve this problem, we are thinking of an algorithme, which

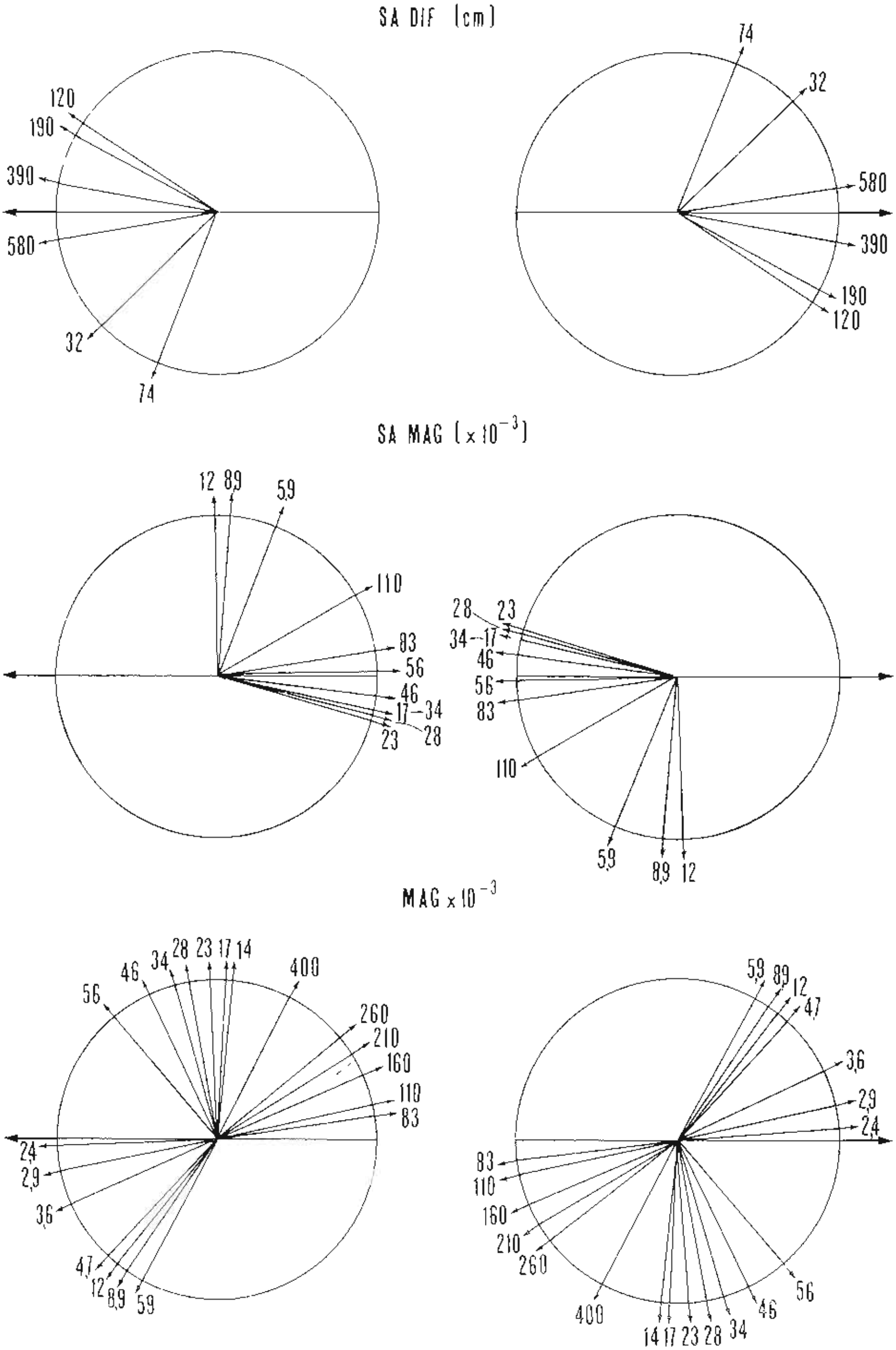
SA DIF (cm)
74
32
580
390
190
120
74
32
580
390
190
120
SA MAG (x 10^{-3})
12 8,9
5,9
110
83
56
46
17 — 34
28
23
28
23
34 — 17
46
56
83
110
5,9
8,9
12
MAG x 10^{-3}
56
46
34
28
23
17
14
400
260
210
160
110
83
2,4
2,9
3,6
4,7
12
8,9
5,9
5,9
8,9
12
4,7
3,6
2,9
2,4
83
110
160
210
260
400
14
17
23
28
34
46
56

Fig. 3

would be translated into a program working with an on-line computer, which would make, roughly speaking, indexing of diffraction spots and also would give to the operator the tilt angle neaded to reach a desired spot. The block diagram of such a logic is given in Fig. 4.

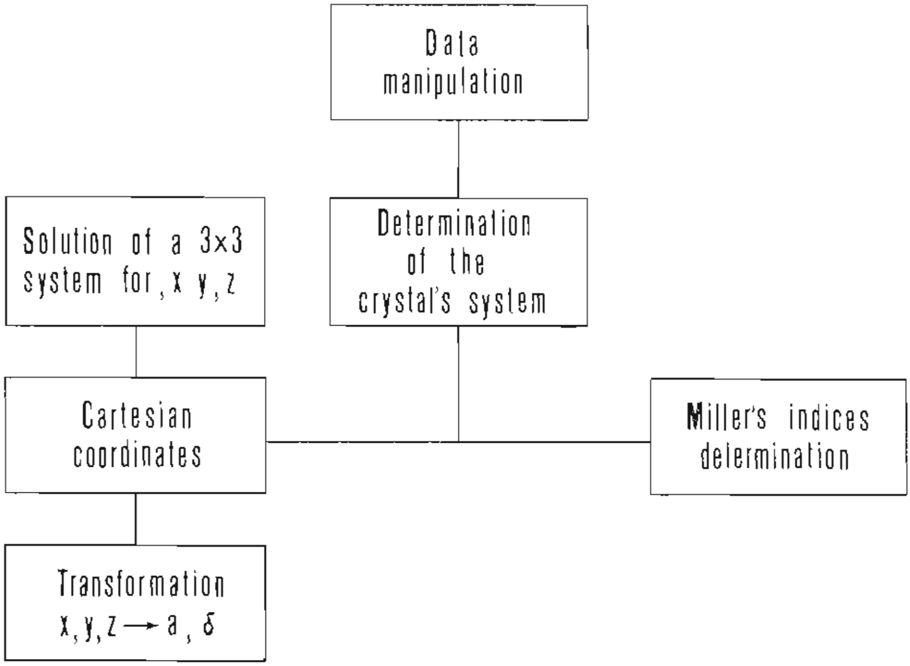

Fig. 4

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ΠΕΡΙΛΗΨΗ

ΤΕΜ ΚΑΙ ΥΠΟΛΟΓΙΣΤΙΚΕΣ ΜΕΘΟΔΟΙ ΣΕ ΜΕΛΕΤΕΣ ΚΡΥΣΤΑΛΛΩΝ

Ι. ΒΑΘΜΟΝΟΜΗΣΗ ΤΟΥ ΗΛΕΚΤΡΟΝΙΚΟΥ ΜΙΚΡΟΣΚΟΠΙΟΥ

Ὑπὸ

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(Ἐργαστήριο Β' Ἕδρας Φυσικῆς Πανεπιστημίου Θεσσαλονίκης)

Ἡ ἐργασία αὐτὴ ἀποτελεῖ τὴν πρώτη μιᾶς σειρᾶς ἐργασιῶν ποὺ ἔχουν ὡς σκοπὸ νὰ συνδυάσουν παρατηρήσεις στὸ ἠλεκτρονικὸ μικροσκόπιο μὲ ταυτόχρονους ὑπολογισμοὺς ποὺ θὰ γίνονται μὲ ἠλεκτρονικὸ ὑπολογιστή. Δίνει, γιὰ τὸ ἠλεκτρονικὸ μικροσκόπιο JEOL 120 CX τοῦ Φυσικοῦ τμήματος τοῦ Α.Π.Θ., τοὺς πίνακες καὶ τὰ διαγράμματα ποὺ εἶναι ἀπαραίτητα γιὰ νὰ καθοριστεῖ ἡ θέση τοῦ συστήματος συντεταγμένων τοῦ κρυστάλλου (δεῖκτες Miller) μέσα στὸ σύστημα συντεταγμένων τοῦ ὀργάνου. Ἔτσι ὁ χειριστὴς τοῦ μικροσκοπίου μπορεῖ νὰ κάνει ὁρισμένους σχετικὰ ἀκριβεῖς χειρισμοὺς τὴν ὥρα τῆς παρατήρησης ὅπως ἐπίσης ἀπόλυτα ἀκριβεῖς ὑπολογισμοὺς μετὰ τὸ τύπωμα τῆς φωτογραφίας. Τὰ δεδομένα αὐτὰ προβλέπεται νὰ συνδυαστοῦν μὲ ἕναν ὑπολογιστὴ on-line.