SYSTEM CALIBRATION OF SCANNING ELECTRON MICROSCOPES

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ABSTRACT

Traditionally Laboratory Calibration of instruments or its components offered satisfactory tools to a wide range of users. Most of this satisfaction was due to the ease and convenience of testing instrument components in the environment of a laboratory. While this mode of calibration can work perfectly well for assessing properties of individual components, it is certainly not convenient for more complicated systems, In fact, sometimes, its results could be even misleading since it does not take into account the compensation/deterioration effect between the different system components. Hence, the notion of System Calibration is regarded by many as the proper approach for realistic conclusions about the performance of the system in question.

In this paper the mathematical models are presented together with results of system calibration for the SEM systems. The mathematical model used includes a number of twenty parameters (6 parameters pertinent to the first micrograph, 6 parameters pertinent to the second micrograph and 7 parameters common to both micrographs). Repeatability assessment of the calibration is performed and analyzed based on proper statistical testing.

I. INTRODUCTION

Eisenhart [2] was among the pioneers in the development of the concept of system calibration. He views calibration of instruments as a refined form of measurement and in order to have a measurement process we have to attain statistical stability known in industrial disciplines as the state of statistical control. His definition of a measurement process can be best understood by referring to figure 1.

Maune (1973) and Nagaraja (1974) applied this concept as a production process for the electron micrographic system. However, with the electron microscope no one tried to develop an entire measurement system calibration, as Eisenhart viewed it. This would require repeated application of the calibration procedure, until future behaviour of the system could be accurately predicted.

The scanning electron microscope used in this study is the Coates & Welter 100-4. Its magnification varies from 20X to 100,000X continuously with a working distance of approximately 25 mm. The specimen stage allows for an ω - rotation ranging between 0° and 90° with 0.2° increment, a κ - rotation from 0° to 360° and X & Y shifts of upto 0.8" with a minimum graduation of 0.001".

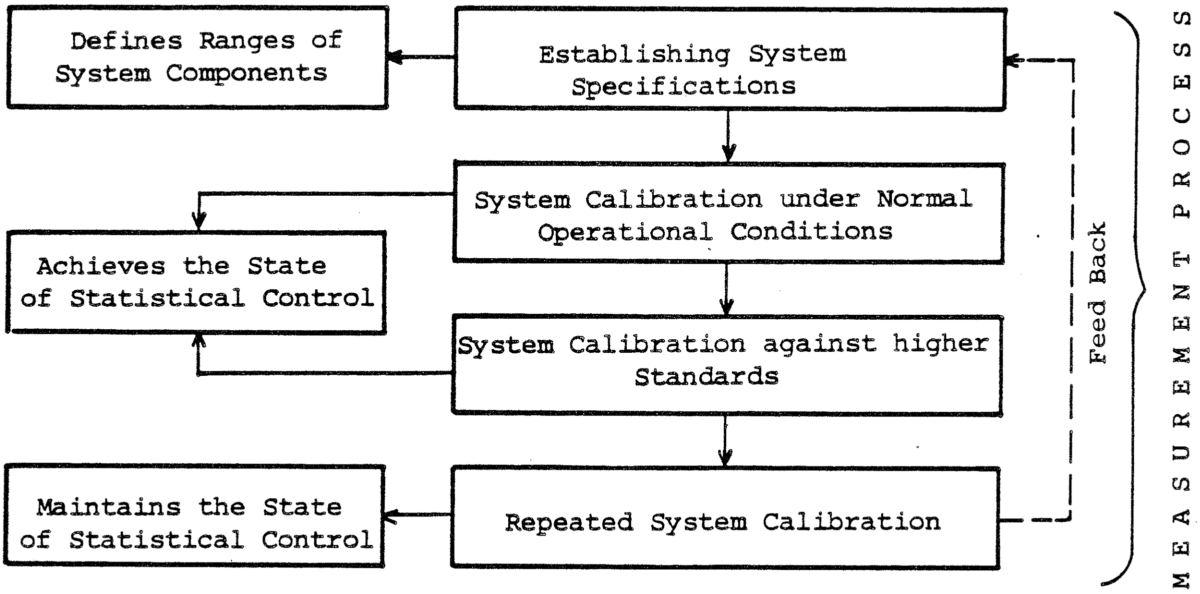

Fig. 1 Measurement Process According to Eisenhart Concept

II. MATHEMATICAL MODELING

II. 1. Coordinate Systems

The projection type used in developing the mathematical model here will be based on the principles of parallel projection. This seems to be appropriately justified, in view of the range of magnifications being used [4], [5].

Figure 2 shows the different coordinate systems and transformations involved in developing the projective equations.

II. 2. Transformation

Let points "P" in the object and "O" the origin have their coordinates in the object based coordinate system as (X_p, Y_p, Z_p) and (X_o, Y_o, Z_o) respectively.

Accordingly the coordinates of point P in the X_1, Y_1, Z_1 system will be:

$$\begin{bmatrix} X_1 \\ Y_1 \\ Z_1 \end{bmatrix} = \begin{bmatrix} X_p - X_o \\ Y_p - Y_o \\ Z_p - Z_o \end{bmatrix} \qquad \text{...........(1)}$$

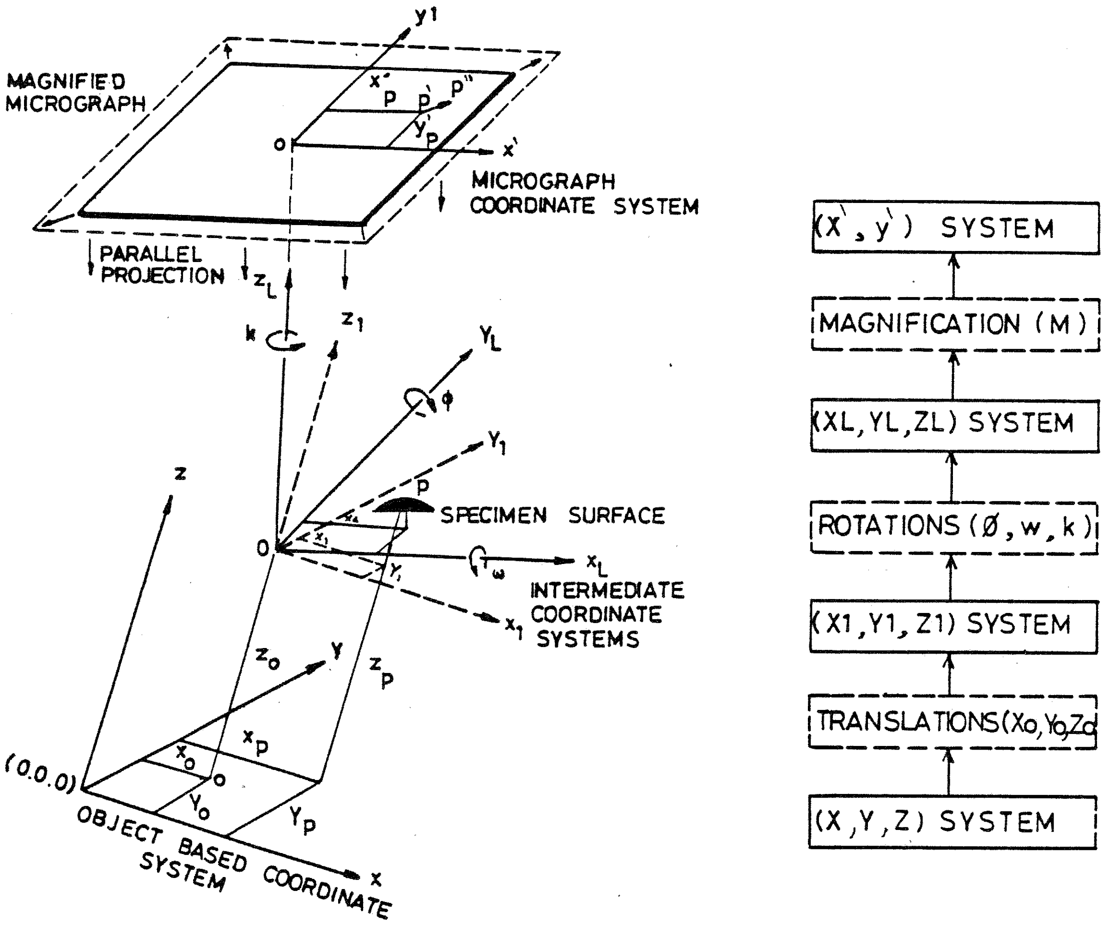

Fig. 2. Coordinate Systems and Transformations

Then considering the rotations (ω, ϕ, κ), we have

$$\begin{bmatrix} X_L \\ Y_L \\ Z_L \end{bmatrix} = \begin{bmatrix} R \end{bmatrix} \begin{bmatrix} X_1 \\ Y_1 \\ Z_1 \end{bmatrix} \qquad \ldots\ldots\ldots\ldots (2)$$

where R = Rotation matrix = $R_\kappa \cdot R_\omega \cdot R_\phi$ ....(3)

Finally, the transformation from the (X_L, Y_L, Z_L) coordinate system into the (x', y') micrographic system will involve a scale change as well as a parallel projection from a 3-D space into a 2-D space as follows:

$$\begin{bmatrix} X' \\ Y' \\ 0 \end{bmatrix} = \begin{bmatrix} M & 0 & 0 \\ 0 & M & 0 \\ 0 & 0 & M \end{bmatrix} \begin{bmatrix} X_L \\ Y_L \\ Z_L \end{bmatrix} \qquad \ldots\ldots\ldots\ldots (4)$$

where M is the magnification

II. 3. Distortions

Three groups of distortions are considered, scale, radial and spiral distortions.

a) Scale distortion

Four different parameters were carried to represent the magnifications for both micrographs. For each micrograph(J), two separate magnifications in x and y (M_{xj} , M_{yj}) were used in the mathematical model.

b) Radial distortion

Expressions (5) are used after Klemperer and Barnett (1971)

$$\begin{aligned} \Delta x &= D_1 x^3 + D_2 xy^2 \\ \Delta y &= D_3 y^3 + D_4 x^2 y \end{aligned} \qquad \ldots\ldots\ldots\ldots\ldots(5)$$

where D's are the coefficients of the radial distortion

c) Spiral distortion

This is caused by the spirally trajectory of electrons in the electron column as well as in the CRT and is expressed by

$$\begin{aligned} \Delta x &= S_x (x^2 y + y^3) \\ \Delta y &= S_y (x^3 + xy^2) \end{aligned} \qquad \ldots\ldots\ldots\ldots\ldots(6)$$

where S_x and S_y are the coefficients of spiral distortion.

Then combining eqn. (4) with the distortion coefficients and more over considering the following two assumptions,

* $\phi_1 = \phi_2 = \Phi$; since the SEM stage does not allow for a rotation around the y axis and accordingly a common Φ angle is introduced as a wobble about the y-axis.

* Defining an arbitrary datum for measuring elevations by setting $Z_{oi} = 0$,

would result in the final set of the projective equations (7)

$$\begin{aligned} x_i' = Mx_1 \; \{ & (X_i - X_{o_1})(\cos\kappa_1 \cos\Phi + \sin\kappa_1 \sin\omega_1 \sin\Phi) + (Y_i - Y_{o1}) \\ & (\sin\kappa_1 \cos\omega_1) + Z_1(-\cos\kappa_1 \sin\Phi + \sin\kappa_1 \sin\omega_1 \cos\Phi)\} \\ & + D_1 x_i'^3 + D_2 x_i' y_i'^2 + S_X\,(x_i'^2 y_i' + y_i'^3) \qquad \ldots\ldots(7a) \end{aligned}$$

$$\begin{aligned} y_i' = My_1 \; \{ & (X_i - X_{o1})(-\sin\kappa_1 \cos\Phi + \cos\kappa_1 \sin\omega_1 \sin\Phi) + (y_i - y_{o1}) \\ & (\cos\kappa_1 \cos\omega_1) + Z_i(\sin\kappa_1 \sin\Phi + \cos\kappa_1 \sin\omega_1 \cos\Phi)\} \\ & + D_3 y_i'^3 + D_4 x_i'^2 y_i' + S_Y\,(x_i'^3 + x_i' y_i^2) \qquad \ldots\ldots(7b) \end{aligned}$$

$$\begin{aligned} x_i'' = Mx_2 \; \{ & (X_i - X_{o2})(\cos\kappa_2 \cos\Phi + \sin\kappa_2 \sin\omega_2 \sin\Phi) + (Y_i - Y_{o2}) \\ & (\sin\kappa_2 \cos\omega_2) + (Z_i - Z_{o2})(-\cos\kappa_2 \sin\Phi + \sin\kappa_2 \sin\omega_2 \sin\Phi)\} \\ & + D_1 x_i''^3 + D_2 x_i'' y_i''^2 + S_X\,(x_i''^2 y_i'' + y_i''^3) \qquad \ldots\ldots(7c) \end{aligned}$$

$$\begin{aligned} y_i'' = My_2 \; \{ & (X_i - X_{o2})(-\sin\kappa_2 \cos\Phi + \cos\kappa_2 \sin\omega_2 \sin\Phi) + (Y_i - Y_{o2}) \\ & (\cos\kappa_2 \cos\omega_2) + (Z_i - Z_{o2})(\sin\kappa_2 \sin\Phi + \cos\kappa_2 \sin\omega_2 \cos\Phi)\} \\ & + D_3 y_i''^3 + D_4 x_i'' y_i''^2 + S_Y(x_i''^2 + x_i'' y_i''^3) . \qquad \ldots\ldots\ldots(7d) \end{aligned}$$

III. EXPERIMENT SET-UP

In this research a carbon replica grid, mounted on a 200 mesh copper grid, made from a master diffraction grating with 2160 lines per mm in crossed directions was used as the standard for SEM calibration.

Five sets of micrographic stereopairs were produced by the SEM using the nominal values for the magnification and the rotations (Magnification of 1200 and tilt angle of 10°). They were taken under the same operational conditions and accordingly they serve very well the purpose of assessing the stability and repeatability by way of comparing the adjusted parameters describing the systematic errors of the measurement process resulting from each of the five stereopair calibrations.

IV. RESULTS AND ANALYSIS

The solution of the twenty parameters mathematical model (7), was done using the minimum variance least squares technique. One unbaiased estimate of a population is preferable to another if the first estimate has a smaller variance than the second. The method of least squares is an application of this concept to a multivariate problem.

IV. 1. Validity of the adjustment procedure

After the adjustment, the photo residuals from the observations should follow a normal distortion. It has been shown (Hamilton 1964) that the statistic

$$\chi^2 = \sum_{i=1}^{n} \frac{(fi - Fi)^2}{Fi}$$

is distributed approximately as chi-square with degrees of freedom of (n-1). The test showed that the (x & y) photo residuals are normally distributed. Figure 3 shows the histogram constructed from the f (computed values) as well as the curve of the probability density function using the F (theoretical values) for the x & y photo coordinates.

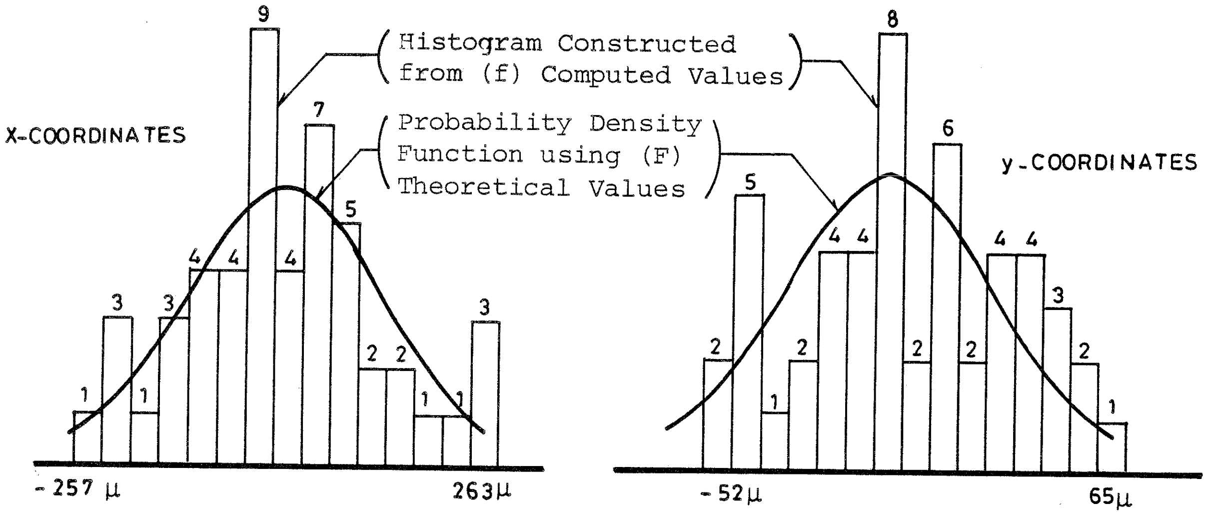

Fig.3. Goodness of Fit for x & y photo coordinates

Another important test is that of testing the apriori against the posteriori variance of unit weight. It has been proven that $\left(\frac{DF.\ \hat{\sigma}_o^2}{\sigma_o^2}\right)$ follows a chi-square distribution, Hamilton (1964). Accordingly, at a significance level (α) of 0.05, we have

$$P\left(\chi^2_{DF.,\ 0.25} > \frac{DF.\ \hat{\sigma}_o^2}{\sigma_o^2} > \chi^2_{DF.,\ 0.975}\right) = 0.95$$

Testing the null hypothesis $H_o:\ \hat{\sigma}_o^2 = \sigma_o^2$

against the alternative hypothesis $H_1:\ \hat{\sigma}_o^2 \neq \sigma_o^2$

at $\alpha=0.05$ resulted in accepting the null hypothesis for stereopairs #1, 2, 3 and 4 while the computed χ^2 of the fifth stereopair exceeded the upper value of the tabulated $\chi^2_{148,0.025}$.

IV.2. Stability and Repeatability

Table 1 shows the values of the adjusted parameters determined independently from the five stereopairs. The magnification in y-direction has been consistently larger than in x-direction. A maximum difference of 6.3% occured in the untilted micrograph of stereopair #5 while the average difference from the five cases was 4.8%. The tilted micrographs showed less difference between x and y magnifications with a maximum difference of 2.6% occuring in the tilted micrograph of stereopair #4 with an average of 1.8% from the five cases. The difference in magnification between the x and y directions is due to the scanning effect of the electron beam while the difference in magnifications between the tilted and untilted micrographs is attributed to the intervention of the operator during the imaging process.

Calibration / Parameters		#1	#2	#3	#4	#5
Pertinent to lst. Micrograph	Mx_1	12082	12073	12119	12144	11875
	My_1	12589	12669	12659	12715	12670
	Xo_1	0.52928(10^{-3})	0.56346(10^{-3})	0.56595(10^{-3})	0.59231(10^{-3})	0.62600(10^{-3})
	Yo_1	0.56855(10^{-3})	0.57253(10^{-3})	0.57186(10^{-3})	0.58805(10^{-3})	0.56005(10^{-3})
	ω_1	-0.27085(10^{-2})	-0.28770(10^{-2})	-0.26461(10^{-2})	-0.22654(10^{-2})	-0.25741(10^{-2})
	κ_1	0.24569(10^{-1})	0.14117(10^{-1})	0.11581(10^{-1})	0.88490(10^{-2})	0.11691(10^{-1})
Pertinent to 2nd. Micrograph	Mx_2	11813	11962	11893	11823	11735
	My_2	11993	11988	11992	11989	11992
	Xo_2	0.50075(10^{-3})	0.51377(10^{-3})	0.53413(10^{-3})	0.57872(10^{-3})	0.58944(10^{-3})
	Yo_2	-0.48088(10^{-5})	-0.55877(10^{-5})	-0.47088(10^{-5})	-0.35678(10^{-5})	-0.43719(10^{-5})
	Zo_2	0.84588(10^{-6})	0.98225(10^{-6})	0.82853(10^{-6})	0.62868(10^{-6})	0.76940(10^{-6})
	ω_2	-0.17412	-0.17400	-0.17417	-0.17442	-0.17420
	κ_2	-0.52886(10^{-3})	-0.30077(10^{-2})	-0.37094(10^{-4})	0.40419(10^{-2})	0.86994(10^{-3})
Common to both Micrographs	Φ	0.10758(10^{-7})	0.11997(10^{-7})	0.11790(10^{-7})	0.12329(10^{-7})	0.12520(10^{-7})
	D_1	0.10622(10^{-3})	0.98169(10^{-4})	0.96352(10^{-4})	0.10623(10^{-3})	0.69407(10^{-4})
	D_2	0.13629(10^{-3})	0.13796(10^{-3})	0.11833(10^{-3})	0.67879(10^{-4})	0.17153(10^{-3})
	D_3	0.28399(10^{-4})	0.36854(10^{-4})	0.35947(10^{-4})	0.37894(10^{-4})	0.40216(10^{-4})
	D_4	0.97800(10^{-5})	0.10363(10^{-4})	0.69366(10^{-5})	0.15384(10^{-6})	0.58537(10^{-5})
	S_X	0.18365(10^{-3})	0.17818(10^{-3})	0.17002(10^{-3})	0.13941(10^{-3})	0.17179(10^{-3})
	S_Y	-0.25553(10^{-3})	-0.26925(10^{-3})	-0.26550(10^{-3})	-0.26116(10^{-3})	-0.27630(10^{-3})

Table 1. Repeatability of Parameters from 5 calibrations

With the adjusted values of parameters, one can obtain their standard deviations from the corresponding variance covariance matrix. Accordingly the weighted mean for each parameter was computed together with its standard deviation. Since the true mean and true variance are unknown, we have to use the student statistic (t) to establish the confidence intervals. Accordingly,

$$P\ (\bar{x} - \frac{\sigma .\ t_{n-1,\alpha}}{x^{\frac{1}{2}}} < \mu < \bar{x} + \frac{\sigma .\ t_{n-1,\alpha}}{n^{\frac{1}{2}}}) = 1-\alpha$$

defines a 100 (1-α)% confidence interval for the mean. Now, if we test the null hypothesis H_o,

H_o: repeated parameter estimates lie within the confidence interval. Against the alternative hypothesis H_1,

H_1: repeated parameter estimates lie outside the confidence interval.

We can determine at what significance level (α) the parameters are considered to be statistically stable. Results of the test are given in table 2 for values of α(0.05, 0.025, 0.02 and 0.01).

Parameter	$\alpha = 0.050$		$\alpha = 0.025$		$\alpha = 0.020$		$\alpha = 0.010$	
	True	False	True	False	True	False	True	False
Mx_1		F5		F5		F5	T	
My_1		F1	T		T		T	
Xo_1		F5	T		T		T	
Yo_1		F4	T		T		T	
ω_1	T		T		T		T	
κ_1		F1		F1		F5	T	
Mx_2		F2,5	T		T		T	
My_2	T		T		T		T	
Xo_2	T		T		T		T	
Yo_2		F2	T		T		T	
Zo_2		F2,4	T		T		T	
ω_2	T		T		T		T	
κ_2		F2,4	T		T		T	
Φ	T		T		T		T	
D_1		F5		F5	T		T	
D_2		F4	T		T		T	
D_3		F1		F1	T		T	
D_4		F4		F4	T		T	
S_X		F4		F4	T		T	
S_Y	T		T		T		T	

Table 2. Results of Parameters testing for different values of (α)

In this table the letter (T) stands for "True" meaning accepting the null hypothesis, while letter (F) stands for "False" meaning rejecting the null hypothesis. Letter "F" is followed by the number of the stereopair which did not pass the statistical test.

V. CONCLUSIONS

From results presented in table 2, one can conclude that at the significance level of 0.01, all the parameters are statistically stable. If we accept the notion usually followed in practice, that 90% of the checked items should pass a test, then even at the significance level of 0.02, the parameters are statistically stable since only two parameters out of twenty did not pass the test.

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