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FUTURE POSSIBILITIES OF PRECISION MAPPING WITH ELECTRON MICROSCOPY

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ABSTRACT

Mapping potentials with micrographs from Scanning Electron Microscopes (SEMs) and Transmission Electron Microscopes (TEMs) are considered. Fundamental projection geometry and associated distortions related mathematical models are presented. Available hardware and methods of mapping in view of the problems related to the provision of controls are discussed. Accuracy and reliability considerations as well as the associated ideas on stability and repeatability at such microscope systems are presented. All these indicate great possibilities in the increased use of electron microscopes in various mapping problems, representative cases of which from several fields of science and engineering are discussed.

INTRODUCTION

Electron micrography has opened new approaches to scientific-engineering tasks in numerous fields. Basically, two types of electron microscope (EM) systems, TEM (Transmission Electron Microscope) and SEM (Scanning Electron Microscope), are used in obtaining quantitative data.

Single, two-dimensional, micrographs are generally satisfactory for many purposes. However, there is a growing demand for three-dimensional information of objects under very high magnifications. Such information can be "direct" (i.e., positional information related to size and shape) or "derived" (e.g., change-parameters like velocity, volume change, etc.; statistical parameters like area distribution, standard pointing accuracy,etc.; or other associated parameters like stress, force, etc.). The standard photogrammetric procedures with certain modifications and innovations appear to offer the most for high accuracy quantification of such microscopic object measurements.

The history of the SEM goes back nearly as far as the conventional TEM. The early theoretical and experimental works relating to field emissions of electrons from surface were reported during the 1930's or before (e.g., Schottky, 1923; Millikan and Eyring, 1926; Fowler and Nordheim, 1928 and others later on). Müller (1937) built the first reported field ion microscope and by 1951 he successfully achieved images showing atomic resolution of the emitter surface. During the 1950's and 1960's, several commercial versions of the TEM and the SEM, respectively, were available. The instruments have been greatly improved during the 1970's. This has been associated with their increasing applications in numerous fields.

The use of TEM for stereoscopic measurements of object features was considered at the early stage of its development (Helmcke, 1954). What one can see in the TEM micrograph is a pattern of light and dark areas produced by the passage of electrons through a thin slice of the specimen. The image is magnified and is displayable on a fluorescent screen. To obtain a micrograph for mensural purposes, however, the electron beam is defocussed below the specimen and projected onto a photographic emulsion surface. The loss of surface features and comparatively high resolution (~0.3 nm) are two important characteristics of a TEM micrograph. If three-dimensional data of the object are needed, it must be no larger than the thickness of the specimen slice. This serious limitation does not apply to the SEM where considerable depth of field and relief contrast are two normal features.

Simply stated, in the SEM, the surface of the specimen (object) is impinged upon by the focussed electron beam. The radiation is used for two synchronous scanning beams, one sweeping over the surface of the specimen and a corresponding second beam sweeping over a fluorescent screen, which is recorded with a camera (often one capable of recording the scene almost instantaneously, like a Polaroid Land Camera). The micrograph thus obtained shows the surface features of the object. Comparatively low resolution (~6.0 nm) and absence of subsurface details are the two important characteristics of the SEM micrograph.

The uses of scanning methods on TEMs (STEM) and transmission methods in SEMs (TSEM) have been very successful in recent years (Kimoto, 1973). Such combination instruments retain the basic features of each instrument and are sometimes combined with X-ray microanalysis capability.

PROJECTION GEOMETRY

A micrograph is a projected record of the object. The projection geometry and associated distortions need to be understood and mathematically modeled for mapping applications. By using rigorous calibration procedures, it has been established (Ghosh and Nagaraja, 1976) that an EM system can often be represented by a mathematical model for an effective *central (perspective) projection* and the photo coordinates can be expressed by:

$$x = X_R \frac{C_x}{\underline{Z} - Z_R} \quad \text{and} \quad y = Y_R \frac{C_y}{\underline{Z} - Z_R} \qquad \text{Eq. 1}$$

where x,y are the photo (micrograph) coordinates;

X_R, Y_R, Z_R are the object-space coordinates after rotations (see below);

$\underline{Z}$ is the projection distance to the reference datum of object;

and C_x , C_y are the two different projection constants for the respective equations.

On the other hand, in view of very small object and extremely large magnification, an EM involves a very small field angle of projection. This can be viewed as *parallel projection,* which can be expressed by

$$\begin{bmatrix} x \\ y \\ 0 \end{bmatrix} = \begin{bmatrix} K_1 & 0 & 0 \\ 0 & K_2 & 0 \\ 0 & 0 & 0 \end{bmatrix} \begin{bmatrix} X_R \\ Y_R \\ Z_R \end{bmatrix} \qquad \text{Eq. 2}$$

where K_1, K_2 are the scale factors along X_R, Y_R directions, respectively.

In view of rotations and translations to a selected origin, one gets:

$$\begin{bmatrix} X_R \\ Y_R \\ Z_R \end{bmatrix} = M \begin{bmatrix} X - X_o \\ Y - Y_o \\ Z - Z_o \end{bmatrix} \qquad \text{Eq. 3}$$

where X_o, Y_o, Z_o are the coordinates of the selected origin in the object system;

M is the orientation matrix;

and X,Y,Z are the object-space coordinates before rotations and translations.

Distortions

It has been found practical and convenient to consider the parallel projection as normal in EM systems and the deviations from parallel projection as distortions. The various distortions as mathematically modelled are listed below in the order of their relative magnitudes as have been established from research studies reported by Nordberg (1972), Maune (1973), Nagaraja (1974) and Ghosh and Elghazali (1977). It may also be noted that the scale affinity (i.e., difference in scale factors K_1 and K_2) may be considered as one form of distortion.

Perspective Distortion, defining the difference between the perspective and parallel projections, can be expressed by

$$\left.\begin{aligned} x_{parallel} &= x_{pers} - x_{pers}\{\nabla + \nabla^2 + \cdot\cdot\} \\ y_{parallel} &= y_{pers} - y_{pers}\{\nabla + \nabla^2 + \cdot\cdot\} \end{aligned}\right\} \qquad \text{Eq. 4}$$

where $\nabla = \frac{1}{\underline{Z}}\{(X - X_o)c\omega s\phi - (Y - Y_o)s\omega + (Z - Z_o)c\omega c\phi\}$;

and c,s prefixes indicate cosine and sine functions, respectively, of the rotation angles as indicated under Exterior Geometry.

Radial Distortion, considered with regard to the fiducial center/principal point/principal beam of the micrograph, either positive (in outward direction) or negative (in inward direction) and expressed by

$$\Delta r = k_0 r + k_1 r^3 + k_2 r^5 + \cdots \qquad \text{Eq. 5}$$

where r is the radial image point distance from the photo center;

and k's are certain constants.

Equation 5 can be simplified for use in practice. Considering that k_0 is equivalent to a scale factor (and thus contained in K_1 or K_2 of Eq.2 and that terms of 5 and higher order in r can be neglected in practice, for the x and y components, one gets

$$\left.\begin{aligned} \Delta x &= \Delta r\frac{x}{r} = k_1 r^3\frac{x}{r} = D_1 x^3 + D_2 x y^2 \\ \Delta y &= \Delta r\frac{y}{r} = k_1 r^3\frac{y}{r} = D_3 y^3 + D_4 x^2 y \end{aligned}\right\} \qquad \text{Eq. 6}$$

where the D's are certain constants.

Spiral Distortion, indicating spiral or rotational twist of the electron beam can be expressed by

$$\left.\begin{aligned} \Delta x &= S_1\frac{y}{r}r^3 = S_1(x^2 y + y^3) \\ \Delta y &= S_2\frac{x}{r}r^3 = S_2(x^3 + x y^2) \end{aligned}\right\} \qquad \text{Eq. 7}$$

where S_1 and S_2 are certain constants.

The degree of stability of the EM system influences the distortions. The reliability of the equations, therefore, would depend on calibration and evaluation of the EM system done with regard to specific working conditions. The obtainable refinement of coordinate data may be illustrated in the following example from the calibration of one SEM with micrographs of magnification 5000× and based on 50 known grid points randomly distributed over the entire format. The standard errors(deviation) of point location are:

When uncorrected for any distortion ..	.. ±300 nm
When corrected only for scale affinity	.. ±110 nm
When corrected for all the distortions	.. ± 23 nm.

It may be noted with interest that Maune (1973) indicated and modelled another type of distortion, viz., *Tangential Distortion,* which, according to Nagaraja (1974), can be contained within the mathematical model for the spiral distortion for all mapping applications of micrographs.

EXTERIOR GEOMETRY

Although there is really no "exterior" space in the EM systems, certain parameters defining separate configurations are inherent and, analogous to conventional photogrammetry, must be identified in precision mapping and are categorized as "exterior". This is especially so with regard to multiple micrography as is necessary in three-dimensional mapping.

In a rectangular three-dimensional system of reference, the "elements of orientation" are the three translations (along X,Y,Z axes) and the three rotations (ω, ϕ and κ around X,Y and Z axes, respectively).

The stage plate containing the object in EM systems has, invariably, four possible movements: (1) Uniaxial tilt corresponding to ω or ϕ in conventional photogrammetry; (2) Rotation around the direction of the principal electron axis, corresponding to κ in conventional photogrammetry; (3) X-translation, contributing to the "base" of the stereo model; and (4) Y-translation, analogous to y-parallax for the entire photo. There is a projection distance in reality. However, in effect, this is nullified by the enormous electronic magnification. Therefore, the focal length (or, projection constants, C_x and C_y in Eq.1) and the projection distance ($\underline{Z}$ in Eq.1) may be combined in ratios to define magnifications (or, scale factors K_1 and K_2 as in Eq.2) as separate parameters.

Stereo model and Orientation

The spatial intersection of conjugate rays is performed by a procedure of *relative orientation* which ensures the *condition of coplanarity* of the two conjugate rays with the base (i.e., three vectors, see Ghosh, 1979). Relative orientation becomes extremely simple if, for generating the second micrograph, one uses only the essential elements (i.e., the tilt and the associated translation). This can be performed analogically at a restitution instrument (may be an analytical plotter) or computationally at a calculator.

The stereo model replica of the object obtained after relative orientation requires *absolute orientation* before any meaningful mapping data may be extracted. In computational approaches, this is performed by using the three-dimensional transformation equations. In instrumental approaches, it is done in two steps: (a) *Scale correction (or Scaling)* by having measurements against dimensions of known values, e.g., replica grid or other usable "standard"; and (b) *Tilting, Rotating and Translating* the model to fit the coordinate system in which the final mensural data are acquired.

Accuracy considerations

With regard to the accuracy of the mensural data, one must consider several points:

1. The "standard" used for scale determination: Diffraction gratings are known to be about the best for this purpose. It is a matter of ingenuity as to what extent these can be used for scale-control/calibration.

2. The measuring instrument: Note that the mensuration capability also depends on the type of point observed and the observational capability of the observer.

3. The intersection geometry: The optimum parallactic angle with EM stereo micrographs is usually between 10° and 15°.

4. The magnification at the micrograph against the limiting resolution provides a compromise situation which the mapper can not ignore. The model/mapping scale depends very much on this compromise.

Stability and Repeatability: Stability tests on parameters (or, elements) in EM systems indicate that with the usual consideration that 90% of the checked items should pass a test, even at the significance level of 0.02, the parameters are statistically stable (Maune, 1973;Nagaraja, 1974 and Elghazali, 1978). Repeatability tests on X,Y,Z coordinates by Elghazali (1978) indicate results consistent with those of testing the parameters. These appear to be even better, being statistically stable at a significance level of 0.01 generally for all the three coordinates. One study by Ghosh et al (1978) on scale repeatability error of the micrographs indicated that the errors stayed within ±1.17% for one SEM and within ±2.07% for one TEM.

HARDWARE AND METHODS

Instrumental Mapping

The EM imaging systems having parallel or very nearly parallel projections and the conventional photogrammetric plotting instruments having perspective projections (optical or mechanical), they are mutually incompatible in principle. Stereo models produced at these instruments with EM micrographs show significant affinity of scale and model deformation.

One way of alleviating this effect will be to consider an appropriate scale factor for the heights (Z) of points and utilize developed (ad hoc) nomograms for continuous mapping of features as has been demonstrated by Oshima et al (1970) in using a Wild A7 stereoplotter. A second possibility exists in modifying such a stereoplotter for EM applications. One such successful modification of a Wild B9 Aviograph was reported by Wood (1972). The third possibility of using "camera lucida" type instruments in obtaining 3-D data was found to be unsuited to production of large volumes of data (Boyde, 1968). The fourth possibility of using instruments with the capability of correcting the final deformed model (such as Zeiss Stereotope) has been successfully demonstrated by Ghosh (1971). Such a system interfaced with a computer and a plotter gives a simple analytical plotting system and has been found to be extremely suited to EM applications (see Ghosh et al, 1978).

The other possibility is the use of specialized instruments developed specifically for EM applications, albeit with their instrumental limitations. One such example is the EMPD (Electron Micrograph Plotting Device) Model 2 (developed by Cartographic Engineering Ltd., U.K.), first discussed by Boyde and Ross (1975). The design philosophy of the EMPD includes two basic assumptions, viz., (a) that a simple optical-mechanical solution is adequately efficient; and (b) that distortion-free photographs are available. Assuming parallel projection systems and the same magnification in each photo of the stereo pair, such an instrument is capable of reducing the data in the simplest form known to photogrammetrists.

A precision analytical plotter, although somewhat beyond the reach of an average user,seems to be the ultimate in EM related mapping jobs. An interesting extension of this analytical concept will be found in the system discussed by Antos et al (1976) for generating high resolution, computer-enhanced and digitally reproduced images from a SEM. This system, developed at the Mead Technology Laboratories, Dayton, Ohio, consists of four components: (1) SEM with a digital scan generator; (2) Data acquisition system for digitizing and recording the image data on a computer-compatible magnetic tape; (3) Computer with image processing software; and (4) Digital image printer. Such a system simplifies the problem of applying the corrections due to various distortions by providing digital data directly accessible to photogrammetric corrections and/or calibration. In other words, the system can produce micrographs which include geometric corrections, image enhancement and image restoration.

Computer Mapping

The concept of DTM (Digital Terrain Model) has been widely used in many applications. There are two essential phases in works involving DTMs, viz., (i) *Data Sampling (acquisition)*; and (ii) *Data Interpolation (processing)*. Both require considerable amount of data storage and handling capabilities, which are efficiently possible with modern computer facilities. Furthermore, with currently available technology, computer mapping with DTM data and corresponding perspective diagramming as may be necessary in many applications are routinely possible.

The DTM approach also offers great possibilities of qualitative and quantitative studies of dynamic objects by way of "differential" mapping, i.e., the mapping of an object relative to itself (in view of changes in time and space or relative to another object with which it may bear some physical and dimensional relationship).

In these regards, it will be of interest to note that attempts have been made with non photogrammetric approaches for obtaining mensural data as well. These, however, have yielded results of rather limited scopes with uncertain and doubtful accuracies. For example, Lebiedzik and White (1975) described an interesting method which is based on simultaneous use of several electron detectors in SEM, from which instantaneous slope and orientation of each sampled point on the surface is computed.

CONCLUSIONS

The SEM has been well received in the materials sciences for surface examinations. Likewise, the TEM has been well established with biological sciences. Examination of deformed, corroded or crystalline surfaces previously performed with replicas (plastic imprints) in the TEM are now better done with SEM stereo-micrography. From the studies of insects to those of microfossils, with the SEM the scientists have seen and measured such details as have never been done before. Quantitative studies in blood cells and viruses were practically unknown before the advent of EMs. Studies on smoke particles (metallic or non-metallic), carbon-blacks (e.g., used in rubber technology) or bio-medical studies on cells and tissues would not be possible in the modern laboratories without the EMs.

The SEM and TEM micrographs can be visualized as analogous to ordinary photographs and X-ray photographs, respectively. By combining their features in three-dimensions for the same object, the scientist would obtain information equal of which is impossible with any other system. Recent research experience of developing procedures of such mapping (see Ghosh et al, 1978) indicates that the scope of the mighty EMs in combination with photogrammetry seems to be unlimited. Extended applications of photogrammetric techniques in these areas are expected to further human progress.

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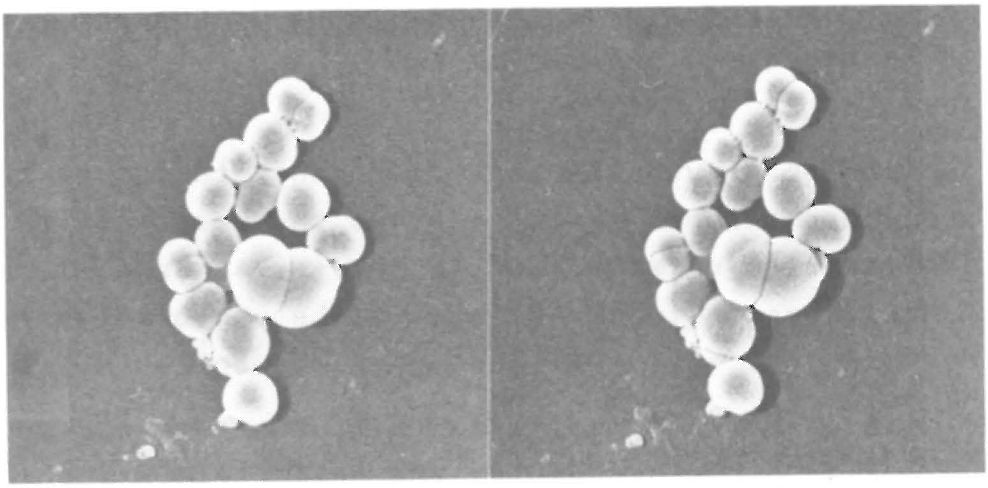

SEM Stereo-micrographs of a Carbon Black Aggregate
Magnification: 20k; Tilts: ±5°

[Courtesy, JEOL Ltd., Tokyo, Japan; Operator: Mr. Tomoyosi Watabe]