MICRO-RANGE APPLICATIONS USING SEM & TEM MICROGRAPHS

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ABSTRACT

The image forming and other operations in a scanning electron microscope are somewhat different from those in a transmission electron microscope. It is sufficient to note that the TEM image is obtained as a result of the scattering power of each point in the specimen as the beam passes through, while SEM image is obtained by collecting and displaying the secondary electrons which are generated at the surface of the specimen. This results in two different images for the same object. The SEM image resembles to a great extent a conventional photograph in that it reflects the surface features. The electron beam in case of TEM system penetrates the object before it forms the image, thus revealing a great deal about the internal structure. The TEM image thus resembles an X-ray photograph.

Due to the fact that the geometry of image formation in both systems is near parallel projection, conventional plotting instruments based on central projection principles do not offer the best solution. In this paper we will present a system composed of a Zeiss Stereocord G-2, an Electronic System DIREC 1, a Hewlett Packard Calculator model 9825A and a Hewlett Packard XY plotter model 9872A. With such an analytical system, it was possible to plot contours of microscopic objects of up to 5 nanometers contour interval from SEM stereo micrographs. It was also possible to plot internal compositions and structures from TEM stereo micrographs.

I. INTRODUCTION

There is a growing demand among electron microscope users to extract metric information from micrographic imagery. Many of those working or using electron microscopes are usually satisfied with the descriptive or qualitative information basically because of their unawareness of the potentials of photogrammetric techniques. Their common goal is to investigate the shape and size or associated characteristics of microscopic objects. Single, two dimensional micrographs have been frequently used. However, it seems that the scientific community is looking for more accurate three dimensional information about micrographic objects and therefore it is adequate to name such an area of application as Micro-Range Photogrammetry.

II. 3-D PLOTTING FROM SEM & TEM MICROGRAPHS

Three dimensional information could be with regard to discrete points or continuously drawn lines. While other attempts have been done in the area of discrete 3-D coordinate information [1], [2] & [4] very little work have been made for continuous 3-D mapping.

The object which has been mapped in this paper, the carbon black, a precisely manufactured form of polycrystalline graphite is an extremely finely divided material consisting of small spheroids (20 to 50 nanometers in diameter) fused together into aggregates of approximately 20 to 80 spheriods [5]. Figure 1 shows a (TEM) micrograph of one of the aggregates magnified 30,000 times.

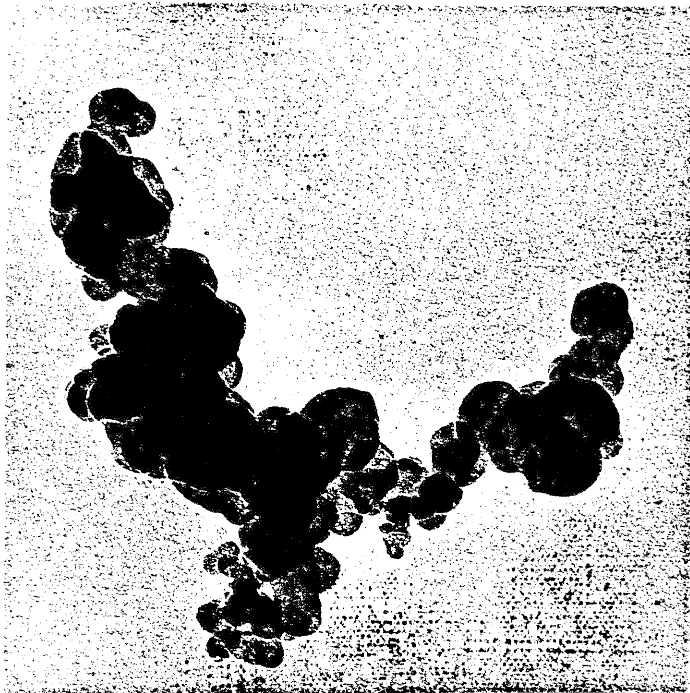

Figure 1. (TEM) micrograph of carbon black aggregate composed of several spheroids

II.1. System Hardware (PPS)

The components of the Photogrammetric Plotting System (PPS) from SEM and TEM micrographs is composed of:

A Zeiss Stereocord G-2, An Electronic System DIREC 1,
A Hewlett-Packard Calculator model 9825 A and A Hewlett-Packard XY Plotter model 9872A. Figure 2 shows the configuration of the system.

With such analytical system, it is possible to apply the principles of parallel projection which is in accordance with the geometry of electron micrographs and also correct for some or all of image deformations.

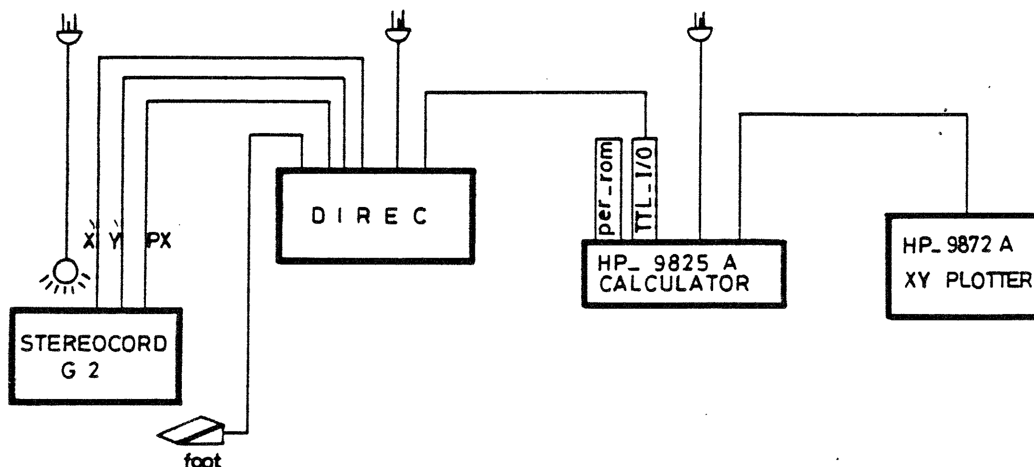

Figure 2 Configuration of the System Hardware

II.2. Parallel Projective Transformation

Due to the limited capacity of the system computer, it was decided to adopt a rather simplified approach in developing the parallel projective transformation.

It was assumed that the object (X,Y) coordinates can be computed from x', y' of the left photograph after accounting for the tilt. This is acceptable for most users due to the large magnifications used. (Z) coordinates were computed based on parallax differences between corresponding points on both micrographs. The calibrated tilt angle and magnification were used. It is further assumed that both ϕ and κ can be constrained to zero since statistical testing at a significance level $\alpha = 0.05$ proved their insignificance [3].

Accordingly, the formula for parallel projective transformation can be derived by referring to figure 3.

$$\tan\theta = \frac{1}{2}\left(\frac{\Delta P}{\cos\theta}\right)/Z_L$$

$$Z_L = \Delta P/2\,(\cos\theta)(\tan\theta) = \Delta P/2.\sin\theta$$

Taking the effect of magnification (M) into account, we get the expression for Z_L as follows:

$$Z_L = \Delta P/2.M.\sin\theta \qquad \dots\dots\dots\dots\dots\dots(1)$$

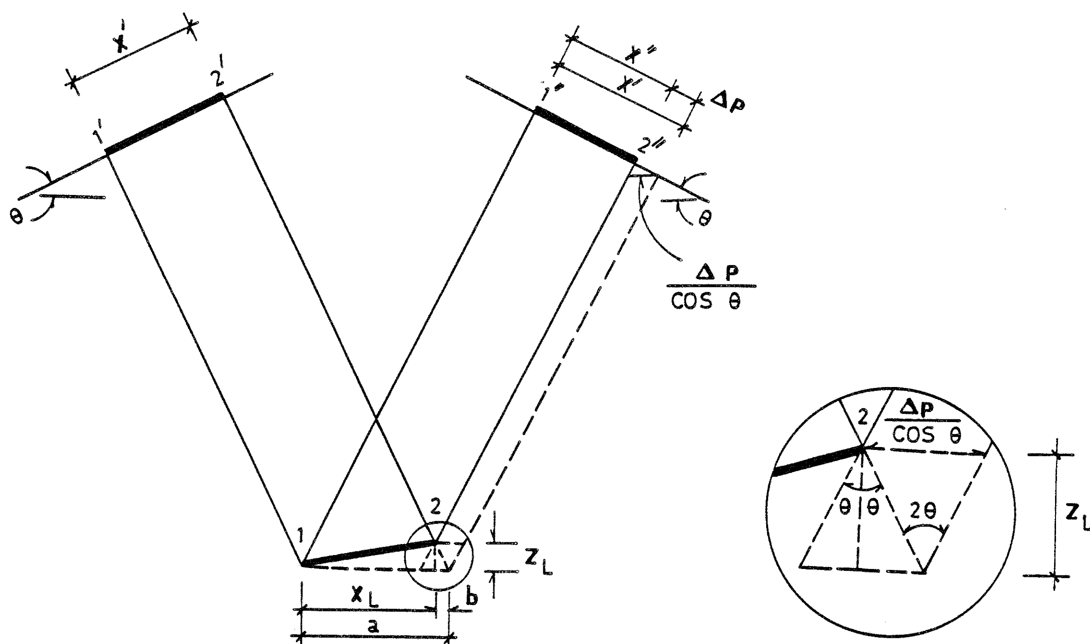

Figure 3. Height difference between two points based on parallel projection

Also,

$$X_L = a-b$$

$$X_L = \frac{x'}{\cos\theta} - Z_L \cdot \tan\theta \qquad \text{..................(2)}$$

In case of carbon black aggregates, the maximum height difference between any two points does not exceed 0.5 µm. Then with the value of θ equals 7.5°-value used for producing micrographs - the second term in expression (2) can not exceed a value of {(0.5)(tan 7° 30')}, which equals 0.0658 µm. With the accuracy of any plotting system, this value is absolutely negligible. This is true for most microscopic objects. Accordingly, expression (2) can safely, after considering the magnification, be reduced to:

$$X_L = x'/M.\cos\theta \qquad \text{..................(3)}$$

The value of Y_L will be directly proportional to the value of y' on the micrograph due to the parallelity condition. Accordingly, we have:

$$Y_L = y'/M \qquad \text{..................(4)}$$

Using equations 1, 3 and 4, the simplified parallel projective transformation for the transmission electron microscope expressed in matrix form is given as follows:

$$\begin{bmatrix} X_L \\ Y_L \\ Z_L \end{bmatrix} = \frac{1}{M} \begin{bmatrix} \sec\theta & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & (\frac{1}{2})\operatorname{cosec}\theta \end{bmatrix} \begin{bmatrix} x' \\ y' \\ \Delta P \end{bmatrix} \qquad \text{..................(5)}$$

where,

X_L, Y_L, Z_L	are the model coordinates
x' , y'	are the x and y left micrographic coordinates
M	is the calibrated magnification of micrograph
θ	is the calibrated angle of tilt
ΔP	is the parallax difference

Equation (5) has been used for plotting from TEM micrographs. Using one magnification for both x and y is justified for TEM since earlier calibration proved that difference between magnifications in x and y directions is insignificant [3].

Unlike TEM, the scanning electron microscope has different magnifications in x and y directions. Accordingly, equation (5) is modified to give

$$\begin{bmatrix} X_L \\ Y_L \\ Z_L \end{bmatrix} = \begin{bmatrix} \sec\theta/M_x & 0 & 0 \\ 0 & 1/M_y & 0 \\ 0 & 0 & \text{cosec}\,\theta/2.M_x \end{bmatrix} \begin{bmatrix} x' \\ y' \\ \Delta P \end{bmatrix} \quad \ldots\ldots\ldots\ldots(6)$$

where,

M_x is the calibrated magnification in x-direction

M_y is the calibrated magnification in y-direction

III. SYSTEM OUTPUT

Based on equations (5) and (6) soft-ware were developed to compute model coordinates as well as to plot planimetry and contours. The program also allows for the calculation of other parameters of interest such as volumes, areas, perimeters, spatial and horizontal distances. The language used by the HP 9825A calculator is called HPL. Figure 4 shows a block diagram of the program main operations.

Besides the main parallel projective equations, the program uses three simple formulae to compute different distances between points. For two points (X_1, Y_1, Z_1) and (X_2, Y_2, Z_2) the formulae are as follows:

Difference in Height (Δ Height) $= Z_2 - Z_1$

Horizontal distance (Hordist) $= \sqrt{(X_1 - X_2)^2 + (Y_1 - Y_2)^2}$

Spatial distance (Spadist) $= \sqrt{(X_1 - X_2)^2 + (Y_1 - Y_2)^2 + (Z_1 - Z_2)^2}$

The program accumulates the area and perimeter of a contour. The area is computed based on dividing the whole contour into trapizoids of known dimensions, then using simple numerical integration.

Accumulating the perimeter is much simpler than accumulating the area since it is the sum of the horizontal distances between successive points.

After properly inserting and centering the stereo micrographs on the stages of the stereocord G-2, the x and y parallaxes should be eliminated near the left fiducial center using P_x and P_y knobs. Then moving near the other fiducial center there should no y parallax left after removing the x parallax. Sometimes a small correction for the P_y setting is needed to eliminate or average residual parallaxes.

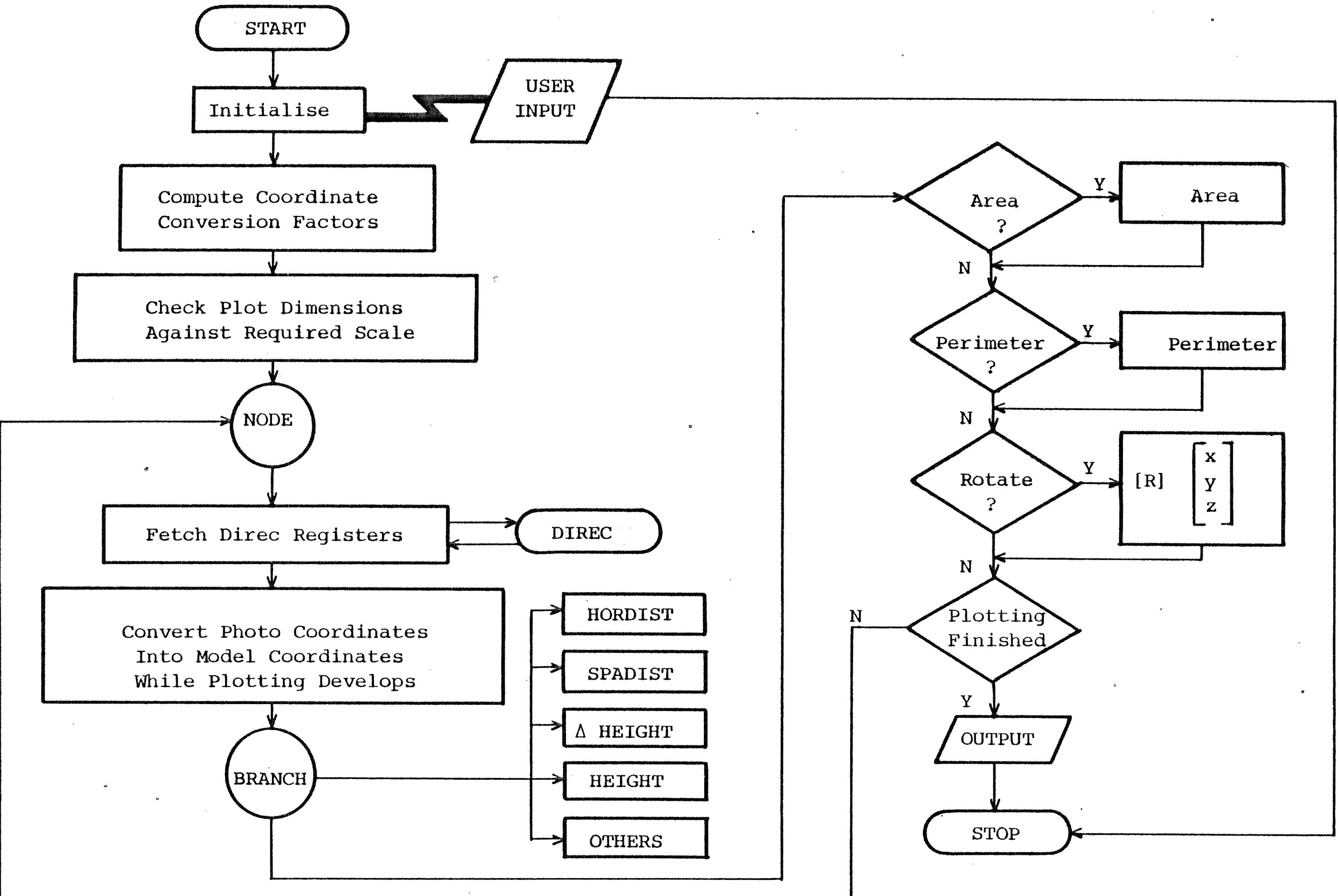

Figure 4. Block diagram of computer program (HP 9825 A)

CONTOURS

HEIGHT	AREA	PERIMETER
0.00	12	
5.00	9	
5.00	17	
10.00	8	
10.00	31	
10.00	315	
15.00	407	
15.00	353	
20.00	4511	
20.00	342	
25.00	2918	
30.00	2432	
35.00	1990	
40.00	1338	
45.00	1074	
50.00	258	
50.00	119	

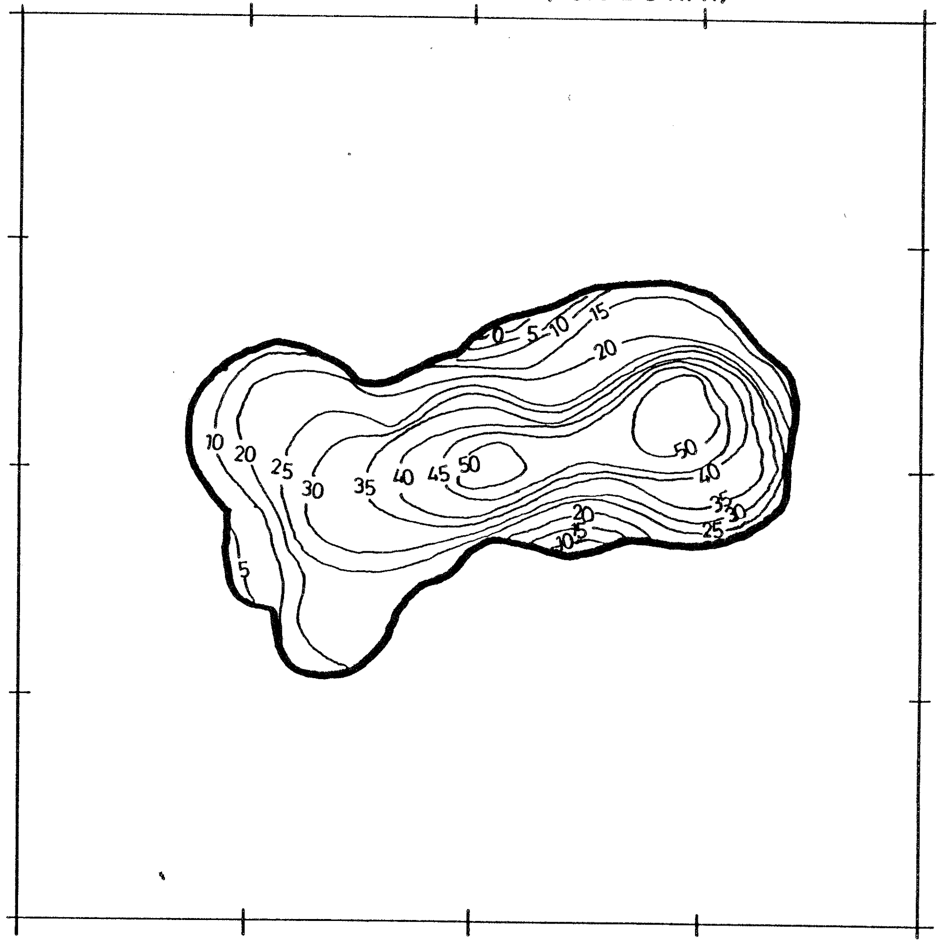

Figure 5. Contour plot from SEM micrographs using (PPS) contour interval = 5 nanometers

PARTICLES

HEIGHT	AREA	PERIMETER	#
21.70	8144	377	1
0.00	1923		2
5.01	868		3
10.01	871		4
15.02	1042		5
20.03	1011		6
18.38	222		7
18.38	300		8
23.37	326		9
23.37	423		10
30.04	1054		11
33.38	894		12
45.07	265		13
48.74	314		14

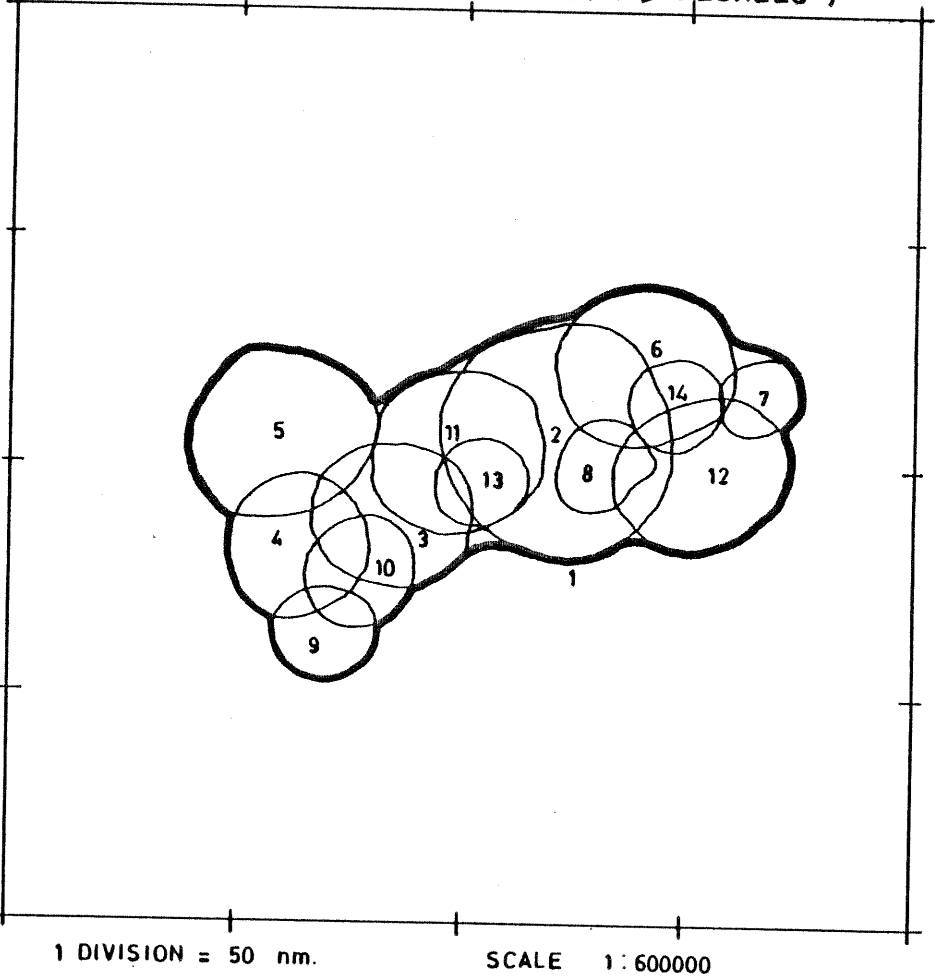

Figure 6. Plot from TEM micrographs using (PPS) revealing internal particle structure

Figures 5 and 6 show sample outputs from the photogrammetric plotting system (PPS) for SEM and TEM plottings respectively. In both plottings one has first to plot the outline of the whole aggregate after which its area and perimeter will be automatically recorded on the left side of the plotting sheet. In case of TEM plot each particle is plotted separately and its height above an arbitrary datum, its area and perimeter may be also recorded. In case of SEM plotting one was able to plot contour lines for the same aggregates which could not be done using TEM micrographs. By exercising enough care a contour interval of 5 nanometers was attained. (Fig.5).

IV. CONCLUSIONS

Plotting from SEM micrographs gives the surface feature of the object while TEM plotting reveals the internal structure of the object and thus, together they offer a unique tool of complete analysis of micro objects. The photogrammetric Plotting System (PPS) used in this research, has great potentials in plotting from electron micrographs or non-conventional systems since they can accomodate any type of mathematical projection. The repeatability of the PPS has been tested by plotting the same object ten independent times and recording the height of 14 points each time. A maximum σ_z = 8.61 nm and a minimum σ_z = 2.61 nm with an average σ_z = 4.37 nm was attained.

REFERENCES

[1] Boyde, A., "Practical Problems and Methods in the Three-Dimensional Analysis of Scanning Electron Microscope Images," Proceedings of the Third annual SEM Symposium, Chicago, Illinois, April, 1970.

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