Height Measurement of Interplanetary Dust Particles by Scanning Transmission Electron Microscopy (STEM)

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Interplanetary dust particles (IDPs) comprise an important source of extraterrestrial materials available for study of our solar system and originate from either the asteroid belt or from short period comets. IDPs from cometary sources are particularly important as they constitute the only physical samples of comets available; all known meteorites are derived from the asteroids, the Moon or Mars. By measuring the densities of IDPs and using an appropriate atmospheric entry heating model, it is possible to determine whether an individual IDP has been derived from an asteroidal or cometary source region. Calculating the density of an IDP requires knowledge of both its mass and volume, which can be determined by using a combination of secondary and transmission electron microscopy techniques. We have developed methods to measure both of these parameters and thus routinely measure densities for individual IDPS in the size range of 5 -15 μm. Mass determination is done using EDX spectra collected on whole particles by comparison of the FeKα line of the IDP with the FeKα line of a stainless steel sphere of known size arid composition. Calculating particle volume requires measurement of both the cross-sectional area of an IDP and its height. A simple method to measure approximate heights of individual IDPs utilizes the narrow depth of focus in a JEOL 1200EX STEM equipped with a secondary electron detector. It is the purpose of this paper to describe this technique with the hope that similar methods can be adopted by others who may have a need for height measurements.

Sample Preparation

The IDPs are captured in the Earth's stratosphere on collector flags which have been mounted to the wings of U2 and other aircraft flown by NASA. The particles on the collector flags are then individually removed and placed on a carbon-coated Nucleopore filter (polycarbonate substrate) which has been attached with epoxy resin to a rectangular-shaped carbon planchet over a large centrally-located hole. The carbon planchets have dimensions of 1" x 0.25" and replace the quick-change specimen retainer used in the JEOL 1200EX sample exchange rod which is normally used to clamp two TEM grids. Prior to mounting particles, a mask of Pd from a 200-mesh square TEM grid is placed on top of the carbon-coated Nucleopore filter allowing placement and number/letter cataloguing of up to 100 individual particles; these particles are then analyzed by EDX to determine which are extraterrestrial. Since this configuration will block the electron beam beneath the sample, a small hole should be drilled through the carbon planchet near the nucleopore filter to allow the electron beam in the TEM to penetrate to the viewing screen allowing the operator to properly saturate the beam and perform important microscope alignments.

IDP Height Measurement

Particle height measurement is performed with secondary electron images in scanning mode and is possible because of the narrow depth of focus due to a relatively large beam convergence angle. This is different than conventional SEM where the convergence angle is much smaller due to the sample residing beneath the objective lens. The incident beam convergence angle, which can be measured for each STEM, is largely a function of the second condenser lens aperture size. Smaller diameter CL2 apertures decrease the convergence angle and increase the depth of focus whereas a large CL2 aperture increases convergence angle and thus decreases focal depth. This can be seen in Figures 1 and 2, which were taken with 20 µm and 100 µm condenser lens apertures, respectively. In Figure 1, an approximately 7 µm high IDP is in focus from the top of the particle to the substrate on which it rests. This compares to the much narrower depth of focus seen in Figures 2a and 2b which were obtained with a 100 µm condenser lens aperture and thus larger convergence angle. In general, to minimize the depth of focus, selection of the largest CL aperture is preferred. Of course the selection of CL aperture size will be dependent on other important parameters such as the sample height, the required signal-to-noise ratio, susceptibility of the sample to beam damage and charging effects. For our studies on IDPs in the size range of 5 - 15 µm, the scanning mode of the JEOL 1200EX STEM with a 100 µm CL aperture provides an approximate depth or focus of about 0.5 µm.

The height of each IDP is measured, after all suitable microscope alignments have been done, using differential focusing between the top of the particle and the substrate on which it rests with a calibrated objective focus knob. Calibration of the objective focus knob step-size is done on carbon-coated 9.870 µm ± 0.057 µm polystyrene spheres, available from a commercial manufacturer, which are placed on the Nucleopore filter near the IDPs. The spheres are exceptionally smooth and thus it can be difficult to find features to focus on their top surfaces; focusing at the 'equators' is much easier due to the perceived edge of the sphere although somewhat less precise. Calibration on gold wires of known size where small features are likely to be visible near the top surface may provide better results. We measured an objective focus step size of 13.7 nm which compares to a JEOL calibration value of 13.0 nm given on the display panel CRT, a difference of about 5%. On microscopes without this parameter listed, simply counting the number of clicks of the objective focus knob should give similar results

In practice, particle height measurement is done by first finely focusing on a representative feature near the top of the IDP (Figure 2a) in reduced raster mode at a magnification as high as practically possible, typically greater than l00,000X. Higher magnifications allow a perceived narrower depth of focus for a particular resolution and beam convergence angle and thus allow finer focusing on small features. The step position on the CRT screen is then noted and the sample translated to a hole in the Nucleopore filter near the IDP without changing magnification as this will reset the step position number. The objective focus knob is turned and refocused on the edge of the hole (Figure 2b) and either the number of clicks of the objective focus knob or the number of steps observed on the CRT display is noted. This procedure is repeated three times and an average of the three measurements is used as the final value. Errors between the three measured heights should typically be less than 10%. Height measurements are dependent on precisely focusing on detailed features such as holes or rough surfaces; improved focusing can be achieved by using the y-modulation mode of a linescan until high frequency ripples are observed in the waveform. At focus, intensity steps in the waveform show the steepest slope and reproduce the finest details.

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In the interplanetary dust particle shown in Figures 2a and 2b, 528 steps *were* measured between the top of the particle and the substrate on which it rests. Thus, at a calibrated objective focus step-size of 13-7 nm/step, the IDP is calculated to be 7.2 µm high. Close observation and comparison of these two figures shows the relatively narrow depth of focus in each image: in principle the heights of particles as small as 2 - 3 µm should be measurable and determination of perhaps even smaller particle heights is possible with larger condenser aperture sizes. One drawback with this technique is the susceptibility of the Nucleopore filters to cracking from apparent heating under high intensity electron beams. A prudent practice is to suitably coat the Nucleopore filters with sufficient carbon to allow excess charge removal and when possible to minimize the sample electron dose.

Conclusions

Using secondary electron images in STEM mode, we have measured the heights of over 200 IDPs in the size range of 5-15 µm. Height measurement errors are typically less than 10 % for the largest IDPs. The narrow depth of focus combined with surface-generated secondary electron signals makes this a useful technique for particle height measurement. Other methods, however, are also possible such as confocal microscopy or optical imaging in a compound microscope where small depths of focus are attainable. Each technique will have its

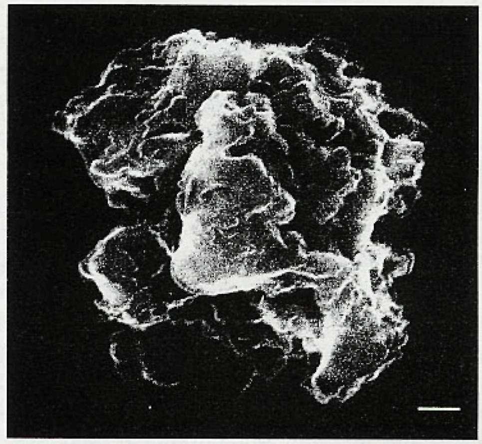

Figure 1: Secondary electron image taken in STEM mode of an interplanetary dust particle resting on a Nucleriore filter. The relatively large depth of focus shows the entire IDP from in focus from top to bottom and was obtained by using a 20 µm condenser lens aperture. Compare to Figures 2a and 2b. Scale bar = 1 µm.

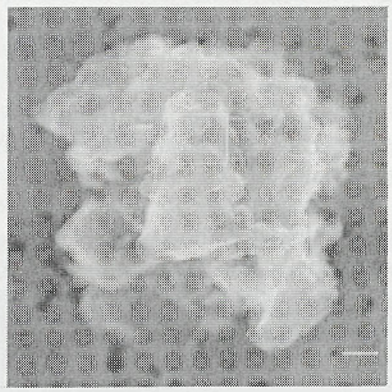

Figure 2a: Secondary electron image in STEM mode using a 100 µm condenser lens aperture and focusing on the feature visible on the top of the particle outlined in the white square. Note the out-of-focus substrate showing the relatively narrow depth of focus in this image and thus allowing particle height measurement. Compare to Figure 2b. Scale bar = 1 µm.

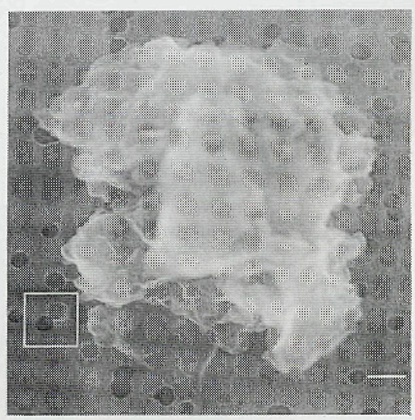

Figure 2b: Secondary electron image in STEM mode using a 100 µm condenser lens aperture. This figure is comparable to Figure 2a, but focused on the edge of a hole in the substrate shown in the white square. Note the out-of-focus upper surface of the IDP. A difference of 528 objective focus knob steps were measured between the top of the IDP and the substrate reflecting a height of 7.2 µm. Scale bar = 1 µm.

own unique advantages and the user must consider the requirements before choosing the most suitable method. For IDPs, where density measurement is the final goal, height is only one of several parameters measured with the electron microscope[4] thus scanning transmission electron microscopy is ideally suited. Height measurement with the STEM may find useftil applications in other fields such as the forensic sciences or failure analysis labs where measurement of feature depths in fractures may be required. Potentially this technique may also be useful for 3-dimensional reconstn~ction of complex particles by obtaining a series of thru-focus images from top to bottom at selected heights and using appropriate stereometry software. ■

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