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Mica Aspect Ratios Measured by Scanning White Light Interference Microscopy

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Platy minerals are utilized for their high aspect ratios (average plate diameter divided by plate thickness) in cosmetic, automotive, electronic, and construction applications. Conventional methods of measuring mineral aspect ratios include shadowing [1] and high tilt observation in the scanning electron microscope (SEM) [2]. For platy particles that are thin enough to be electron transparent, aspect ratio can be measured through automated measurement of particle diameter combined with measurement of scattering loss for measurement of particle thickness [3].

We previously reported the measurement of the aspect ratio of commercial talc particles (20 to 50 µm average diameter) using scanning white light interference microscopy (SWLIM) [4]. Comparison with aspect ratios measured using the high tilt SEM observation method showed good agreement. Here we report the results of an attempt to extend the SWILM method to smaller particles utilizing a commercial mica with average diameters < 10 µm.

Samples of mica were suspended in ethanol, placed onto clean coverslips, and coated with carbon. SWLIM analysis was performed on a WYKO NT-2000 surface profilometer in vertical scanning interferometry (VSI) mode. A field of view containing several particles was scanned at 100x magnification. In our previously reported results [4], we utilized the "Multiple Region Analysis" option supplied by the WYKO software [5] to measure the heights and diameters of individual particles, however, this software proved unable to recognize the present particles due to their small size.

Therefore, the X,Y and Z coordinates of a few points were manually recorded for each field of view and the WYKO image was saved for analysis with the imaging program ImageJ (http://rsb.info.nih.gov/ij/). In ImageJ, each field of view was calibrated in X, Y, and Z using the manually recorded data and the X feret, Y feret, and mean thickness for each particle was measured. The same samples were then placed in a JEOL 6500 field emission scanning electron microscope. Each field of view measured by the SWLIM/ImageJ method was located, and the X and Y feret diameters measured for the same particles. The sample was then tilted 60 degrees, and several measurements of thickness were made along each particle's edge. These measurements were corrected for the 60-degree tilt and an average thickness was calculated.

The mean aspect ratios of eighteen particles determined by the two techniques were found to be 13.5 +/- 5.1 (1 sigma) by SWILM and 10.1 +/- 3.1 by SEM. Application of student's t-test for paired data indicates the two values are different at the 95% confidence level [6]. Figure 1 compares the data obtained by the two techniques. Close agreement is seen between the two methods for average diameter, with R^2=0.98 and a slope of 0.95 [6]. Comparison of the particle thickness measurements from the two technique shows poorer agreement, with R^2=0.65, but with a slope of 1.02. The scatter in the thickness measurement is strongly reflected in the comparison of the aspect ratios measured by the two techniques, with R^2=0.31, with a resulting uncertainty in the slope of the best-fit line.

In the absence of individual particle aspect ratio standards it is hard to know which technique yields the most accurate results. The differences appear to arise from differences in thickness measurement, with a significant bias towards greater thickness in the SEM measurements compared to the SWLIM measurements. Some of this difference may arise from the fact that these two methods measure different manifestations of particle thickness, with SEM measurements derived from the particle thickness at the edges only, and SWLIM measurements from average thickness of the interior of the particle. Additional scatter (compared to our previous results from larger particles) may also be due to larger relative uncertainty in thickness measurement for thicknesses in the 100 nm range. We continue to investigate the cause(s) of these differences.

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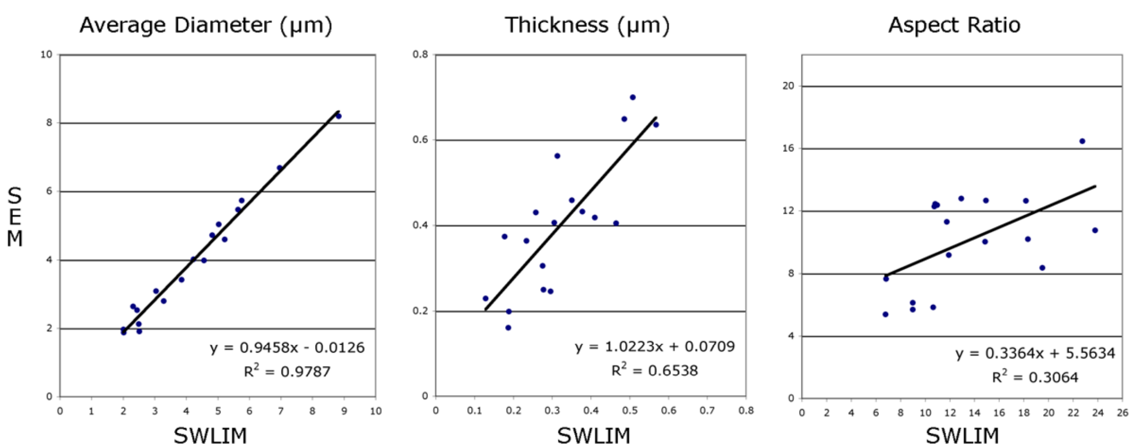

Figure 1. Comparison of diameter, thickness, and aspect ratios of mica particles by FESEM and SWLIM.

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