3302
doi:10.1017/S1431927621011375
Microsc. Microanal. 27 (Suppl 1), 2021

Toward Determination of the Surface Roughness of Particles from a SEM Image

Ardian Gojani[1], Charlie Tobias[2], Deniz Hülagü[3], Knut Rurack[2] and Vasile-Dan Hodoroaba[3]

[1]Federal Institute for Materials Testing and Research (BAM), Division 1.9 Chemical and Optical Sensing, Berlin, Germany, [2]Federal Institute for Materials Testing and Research (BAM), Division 1.9 Chemical and Optical Sensing, Berlin, Berlin, Germany, [3]Federal Institute for Materials Testing and Research (BAM), Division 6.1 Surface Analysis and Interfacial Chemistry, Berlin, Berlin, Germany

Determination of surface roughness of micro- and nanoparticles is a challenging problem that does not have a general solution. Instead, one adopts solutions specific to an application. Some solutions are the use of atomic force microscopy (AFM) [1], or the evaluation of the fluctuations from a circular circumference of the projected profile extracted from a transmission electron microscopy (TEM) image [2].

In this communication, we address the issue of roughness measurement by investigating if the grayscale values from SEM images can be used for surface roughness determination of spherical particles. Unlike the TEM micrograph, in a high-resolution SEM micrograph details of the surface morphology are visible on the nanometer scale. The spherical particles investigated in the present study are paramagnetic beads, which will be decorated with antibodies and used in a microfluidic-based immunoassay. Their functionalization depends on the surface roughness because this determines the amount of binders that can be attached to the surface and their orientation, i.e. the accessibility of the binding sites. The investigated particles, shown in Figure 1, have a polystyrene (PS) core mixed with polyvinylpyrrolidone (PVP) with different molecular weights (10 kDa for 'SEM 1' and 360 kDa for 'SEM 2'), coated with silica. The magnetic feature is introduced through iron oxide nanoparticles at the interface between polymer core and silica shell. The diameter of the particles is 1.0 µm +/- 1% (the scale bars in the image correspond to 100 nm).

The method proposed here relies on topographic contrast, which is generated by the secondary electrons (SE). The grayscale values in the recorded image correspond to the number of SE that have reached the detector, in our case an InLens SE1 detector. This number depends on several factors, including primary beam incidence angle (i.e. local surface morphology), SE trajectory to the detector, and sample composition, but also beam current, and detector sensitivity, to name a few. The relationship between SEM image grayscale values and height of the structures on the particle surface can be achieved only by applying specific methods, such as 3D reconstruction of stereo imaging [3]. Hence, the proposed method gives only relative measurements and relies on at least a single absolute measurement for setting the scale for grayscale value to height conversion. Since the particles are produced in a well-controlled and reproducible process, the height information can also be obtained from the size of the nanoparticles deposited on the surface, which can reliably obtained in a straightforward manner from SEM image analysis, thus serving as an internal height reference. In our study, the effect of variation in sample composition on the number of emitted SE is negligible, and the SEM image acquisition conditions were the same (accelerating voltage 20 kV, working distance 2.6 mm, magnification 200,000 times).

The foundation of the applied method here is the determination of grayscale value fluctuations along an inscribed circle centred on the particle centre, as shown for two SEM images in Figure 1. As a first step of analysis, the image grayscale values are normalized, such that the variations between different image acquisition conditions is removed. Next, a series of grayscale value profiles along circles of different radii (from 185 to 463 nm; SEM images in Figure 1 show only the cases for 278 nm) are collected. From this series of profiles, the variation of the grayscale values, shown as lateral profiles in Figure 1 bottom, is measured using the root mean squared value. This value, then, corresponds to the scale of surface roughness for one circle. The averaged value along all radii, i.e. of the whole series of profiles, determines the surface roughness of the entire particle.

This method is tested in two modes: (i) as a measurement of the fluctuations of grayscale values, and (ii) as a measurement of the fluctuations of the differences of grayscale values. The latter connects directly to the effect of higher SE emission from inclined surfaces, as compared to flat ones, due to the larger SE yield for larger incidence angles. Nevertheless, the results did not show large relative deviations and were within the standard deviation of the roughness measurement.

This method is still under development and the next step is its characterization and validation using a 3D reconstruction of two SEM images taken at different tilt angles. The initial results provide a good basis for quantitative surface roughness characterization from a single SEM image. This approach can constitute a valuable alternative compared to stereo microscopy requesting two micrographs, or projective measurements as required by TEM.

https://doi.org/10.1017/S1431927621011375 Published online by Cambridge University Press

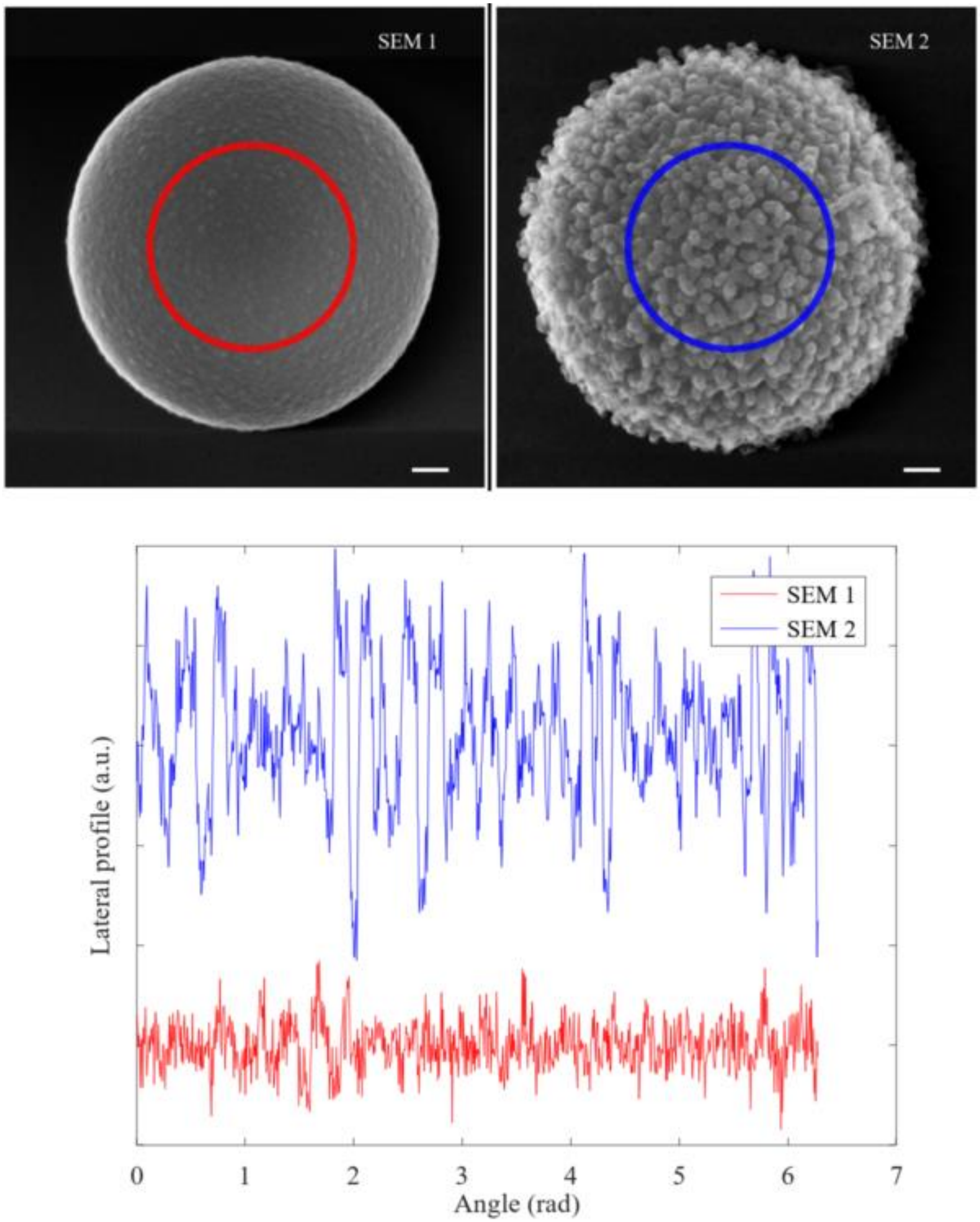

Figure 1. Figure 1. SEM micrographs of two particles with different surface morphologies and their lateral profiles as extracted along the circles depicted in the SEM images.

References
[1] B. Ilhan, et al., Colloids and Surfaces A 560 (2019) 50-58

https://doi.org/10.1017/S1431927621011375 Published online by Cambridge University Press

[2] D. Sarma, et al., Applied Surface Science 426 (2017) 446-455
[3] S. Roy, et al., IEEE EMBS 34 (2012) 4361-4364

https://doi.org/10.1017/S1431927621011375 Published online by Cambridge University Press