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Evaluating scanning electron microscopy for the measurement of small-scale topography

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Abstract

For predicting surface performance, multiscale topography analysis consistently outperforms standard roughness metrics; however, surface-characterization tools limit the range of sizes that can be measured. Therefore, we evaluate the use of scanning electron microscopy (SEM) to systematically measure small-scale topography. While others have employed SEM for similar purposes, the novelty of this investigation lies in the development and validation of a simple, flexible procedure that can be applied to a wide range of materials and geometries. First, we established four different options that can be used for sample preparation, and we measured quantitative topography of each using the SEM. Then the power spectral density (PSD) was used to compare topography among the four preparations, and against other techniques. A statistical comparison of PSDs demonstrated that SEM topography measurements outperformed AFM measurements at scales below 100 nm and were statistically indistinguishable from (highly labor-intensive) TEM measurements down to 16 nm. The limitations of SEM-based topography were quantified and discussed. Overall, the results show a simple generalizable method for revealing small-scale topography. When combined with traditional stylus profilometry, this technique characterizes surface topography across almost seven orders of magnitude, from 1 cm down to 16 nm, facilitating the use of physical models to predict performance.

1. Introduction

Surface properties depend on surface topography, but the relevant size scale varies widely across applications [1]. Because topography varies over length scales [2–4], then there is a need to combine topography measurements at many different scales. This multiscale analysis has been shown to predict performance more accurately as compared to conventional roughness metrics [5, 6]. Many real-world surfaces contain topography with features that span many orders of magnitude which cannot be captured using a single technique because of limitations on resolution and scan size. In these cases, capturing all relevant scales of roughness may require the combination of measurements at different sizes,

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often involving more than one measurement technique. Stylus and optical profilometry are widely used for large-scale characterization, but the resolution is limited to a few microns. Atomic force microscopy is commonly used to measure the smaller scales, but is limited by tip-radius artifacts [7, 8]; especially with tip wear, it can exhibit artifacts as large as 100 nm in lateral scale [9]. In prior work by the present authors, we demonstrated transmission electron microscopy (TEM) as a method for characterizing surfaces from 100 nm down to the atomic scale. However, TEMs are relatively uncommon, and the sample preparation can be time-consuming and costly. Therefore, the present investigation evaluates the use of SEM to evaluate small-scale topography.

Many prior investigations have used SEM as a method to measure surface topography. As far back as 1985, Suganuma [10] extracted surface topography by using two secondary-electron detectors placed symmetrically about the incident beam. By subtracting the two images, the topography could be computed with a height-resolution of approximately 2 nm. Podsiadlo & Stachowiak [11] advanced the topic of SEM topography further by using feature-detection to quantitatively characterize the surfaces of wear particles. However, Liu *et al.* [12] questioned the extraction of surface roughness with SEM. The authors quantitatively measured the topography of AlTiC substrates coated with diamond-like carbon using SEM and then compared it to measurements taken with an AFM. The authors determined that the extraction of surface properties from SEM proved inconclusive and advocated the use of AFM for the extraction of RMS parameters. Other sophisticated methods have been developed for quantitative SEM measurements of 3D topography, such as stereo microscopy [13] or a four-detector approach [14]. While these techniques were successful, planar distortions and detector asymmetry were present and the method was inappropriate for samples containing high curvatures and overhangs. Other authors used cross-sectioning to extract topography, which has the advantage of combining surface information with sub-surface information that is revealed deeper in the section [15]. Shi *et al.* [16] compared cross-section methods against geometry-based methods such as stereoscopic imaging and rotation-based serial imaging. A 3D FIB-EBSD method produced reliable 3D recreations of surface structures but was slow, destructive, and limited in resolution by the thickness of each FIB layer. In contrast, the stereoscopic tilt-imaging and sample rotation were high-throughput and non-destructive but suffered similar limitations in resolution and also the presence of distortions. Recently, Ding *et al.* [17] used standard metallographic sample preparation to create cross-sections for side-view SEM imaging in the context of flooring topography to understand friction performance. This work showed good correlation between stylus profilometry and SEM topography measurements, even despite the non-conductive nature of the samples. Yet charging artifacts were observed in the SEM, which limited the maximum magnification for analysis. Ramos *et al.* [18] used SEM to investigate the monofractal and multifractal properties of a Piper Leaf surface architecture. The SEM images were utilized to qualitatively and quantitatively characterize the surfaces of the leaves.

Two challenges become obvious from the above examples of SEM-topography measurement: (1) there are a wide array of methods for measuring topography, some of which require complex instrumentation; and (2) there is little work validating the accuracy of SEM measurements against other techniques. The motivation for this investigation is

to address these two challenges by accomplishing the following corresponding objectives: (1) establish a simple yet flexible SEM-based method for measuring topography, and (2) validate the SEM-based measurements against previously published results. In order to achieve this, the topography of a wear-resistant ultrananocrystalline-diamond coating was measured in the SEM using four different sample-preparation techniques. First, the SEM measurements were compared against previously published stylus-profilometer measurements, to evaluate their accuracy at the large scale. Second, the SEM measurements were compared against previously published AFM and TEM measurements to evaluate performance at the small scale. Finally, the four different sample preparation techniques were compared against each other, for validation that all four options give statistically indistinguishable results.

2. Methods

2.1. Experimental methods: Preparing samples for SEM topography

The present investigation employs and compares various methods for creating a sample for SEM investigation. Cross-sectioning is a commonly used technique in materials science for revealing the internal structure of a material, and the same general approach can be used to reveal the surface topography. As described in Khanal *et al.* [19], this is an effective technique as long as efforts are taken to preserve the original surface and prevent damage. The cross-section approach requires time and effort to perform but has the advantage that it can be performed on any part, component, or sample. By contrast, Khanal *et al.* [19] also described the “wedge-deposition method”, where a material is deposited on a wedge or thin blade, and then imaged in profile. As discussed at length in Ref. [19] the wedge-deposition approach is significantly simpler, requiring no sample preparation at all; but it has the disadvantage that it only applies for materials that can be deposited (for instance, coatings that are deposited by evaporation, physical vapor deposition, chemical vapor deposition, spray coating, etc.). In Khanal *et al.*, one method for cross-sectioning was compared to the wedge-deposition method using TEM. By contrast, the present work uses SEM instead of TEM (harnessing the advantages discussed above), and compares four separate methods of sample creation, as shown in Fig. 1.

In the present investigation, the same ultrananocrystalline diamond surface was prepared using each of the four techniques described in Fig. 1. While these techniques are extensively documented in other works, a brief description of their implementation will be included here. First, in Method 1, a sample was created using the wedge-deposition method (Fig. 1a), exactly as described in Khanal *et al.* [19]. A microfabricated wedge (Hysitron Picoindenter wedge substrates, Bruker, Billerica, MA) was coated with UNCD, deposited using a tungsten hot-filament chemical vapor deposition system with parameters as described in Ref [20]. The wedge-deposition method is simple to perform, requiring nothing more than the addition of a witness sample to the chamber where the deposition is occurring. The witness sample can be a wedge, similar to the one used here, but could also be any sharp blade-like sample, including a razor blade or a thin foil of metal. The only relevant consideration is that the coating on the witness sample should be representative of the coating on the

real-world use-case. This may require matching the material of the witness sample to that of the use-case, and/or matching the height and other conditions during the deposition.

The same material was also used to create cross-section samples. In particular, a silicon wafer was inserted in the same deposition run that was used for the wedge-deposition sample (described above), ensuring that the material is as similar as possible. The coated silicon wafer was then used to create cross-sections in three different ways. In Method 2, the simple-fracture method was used (Fig. 1b), whereby the wafer was scribed using a diamond scribe and cleaved into pieces, exposing the fracture surfaces. Because of the brittle behavior of the silicon and the diamond materials, the fracture process is expected to induce very little damage into the surface topography or sub-surface structure. This technique therefore presents a nearly equipment-free method to produce SEM cross-sections of brittle materials with little time or effort. In Method 3, this same UNCD-coated silicon wafer was subjected to the mechanical-polishing technique (Fig. 1c); this process is also called metallographic sample preparation, and is exceptionally common in materials science and engineering [21]. While not required, the present investigation used the common practice of including an additional (sacrificial) sample of the same material mounted within the same epoxy resin. This practice prevents the possibility that differences in polishing rate between the mounting resin and the sample could cause rounding of the surface of interest. In this case, an auto-polishing system (MultiPrep 8, Allied, Compton, California, USA) was used to mechanically polish the sample to a sub-micron surface polish. After leveling the sample using grinding, the sample was polished using successively finer grits, progressing along 9, 6, 3, 1, 0.5, and 0.01 μm slurries. Each step was used to remove a material thickness approximately corresponding to three times the grit size of the previous polishing step. After polishing, the sample was sonicated in ethanol for 15 min to remove any residue present from the polishing process prior to imaging. Finally, in Method 4, a sample was created from this same wafer using the process of ion-polishing (Fig. 1d). Here, the material is milled using a beam of Ar ions that approach the cross-section of interest at a grazing-incidence angle. In this work a Fischione Instruments model 1060 ion mill using an argon plasma at 4keV a focus of 50%, a grazing incidence of 7° from the surface, and a lateral rocking angle of 70° . Prior to milling a UNCD-coated silicon coupon was released from the bulk using the simple-fracture method described above, but any cutting or breaking method could have been used to expose the surface. Once exposed, the glancing-angle ions were used to remove material, exposing the fresh surface.

2.2. Data processing methods: Extracting quantitative topography from side-view SEM images

Once the samples were created, they were imaged in profile using SEM, as shown in Fig. 2a. Images were collected on a field-emission scanning electron microscope (Sigma VP, Zeiss, Oberkochen, Germany). Samples were mounted using double-sided carbon tape to mitigate sample-charging effects. The sample and chamber were then plasma cleaned using an EM Kleen with an RF of 50 W, pressure of 45 mTorr and a run time of 8 mins (stable plasma exposure was approximately 6 mins). This was done to mitigate any carbon deposition on the sample under high acceleration voltages. The samples were then imaged using an

aperture setting of 7.5 mm, an acceleration voltage of 15kV and an in-lens secondary electron detector.

Once imaged, the profile of the surface was quantified (Fig. 2b). Generally, there is flexibility in how this step is performed. For some samples, edge-finding routines such as those that come standard in programs like ImageJ or MATLAB can be used to find the boundary between the light-colored sample and the dark-colored background. In the present work, a manual point-picking method was used, where a trained user visually selects a sequential line of points that define the boundary. Here, this was implemented through a custom algorithm (MATLAB, MathWorks, Natick, MA) that has been used extensively in prior investigations (for example, Ref. [22]). Similar algorithms exist in ImageJ and other freely-available software. This profile extraction must be performed in calibrated units, using the scale bar of the SEM image. The final result is a series of x,y coordinates (Fig. 2c), not dissimilar from the output of a stylus profiler.

The SEM-measured topography will be compared against stylus-profilometry measurements collected in a prior investigation [9]. In that work, the data were collected on a stylus profilometer (Alpha-Step IQ, KLA Tencor, Milpitas, CA) with a 5- μm diamond tip. One-dimensional line scans were taken at a scanning speed of 10 $\mu\text{m s}^{-1}$. 20 measurements were taken for each surface at 7 different scan lengths, ranging from 0.3 to 10 mm. These measurements were collected at random orientation and did not show any significant variation with direction. To remove the tilt of the sample and the bowing artifact from the tool, a parabolic correction was applied to all the measurements.

2.3. Topography-analysis methods: Computing metrics for (1) a multiscale analysis using the power spectral density (PSD); and (2) scalar analysis using root-mean-square (RMS) parameters

First, a multiscale analysis of topography was performed using the one-dimensional power spectral density, a widely used (e.g., [23]) multiscale topography metric. The PSD separates the surface topography into contributions from different wavevectors. From a mathematical standpoint, the PSD is the Fourier transform of the autocorrelation function, or equivalently the square of the Fourier transforms of the height itself. Here the PSD $C(q)$ is computed according to the practices described in Ref. [8]:

$$C(q) = L^{-1} |\tilde{h}(q)|^2 = L^{-1} \left| \int_0^L h(x) e^{-iqx} dx \right|^2 \quad \#(1)$$

The details of the calculation of the PSD can vary across different software implementations. Here, we use the open-source analysis code from the freely-available web application <https://contact.engineering>, which is fully documented in Ref. [24]. Furthermore, a power spectral density can be calculated from 2D (line-scan) or 3D (area-scan) measurements of topography; at all points in this paper, PSDs were computed using the line-scan approach to ensure an apples-to-apples comparison across all techniques.

Second, the surface topography was analyzed using scalar roughness parameters: the RMS height h_{rms} , RMS slope h'_{rms} , and the RMS curvature h''_{rms} . From a topography measurement, these quantities can be computed in real-space from the line profiles as: [9]

$$h_{rms}^2 = \frac{1}{L} \int_0^L h^2(x) dx, \quad h'_{rms}{}^2 = \frac{1}{L} \int_0^L \left(\frac{dh}{dx} \right)^2 dx, \quad h''_{rms}{}^2 = \frac{1}{L} \int_0^L \left(\frac{d^2h}{dx^2} \right)^2 dx \quad \#(2)$$

However, the value computed from a single measurement will vary strongly with the size scale of that particular measurement (as discussed in Ref. [9]). To avoid this problem, these same parameters can be calculated equivalently, according to Parseval's law, in frequency space. Here, we combine the computed power spectral densities from each measurement (different techniques and different length scales) and then take the averaged PSD as a complete representation of the surface. Then, we integrate over the positive-frequency domain, according to Ref. [9].

$$h_{rms}^2 = \frac{1}{\pi} \int_0^\infty C(q) dq, \quad h'_{rms}{}^2 = \frac{1}{\pi} \int_0^\infty q^2 C(q) dq, \quad h''_{rms}{}^2 = \frac{1}{\pi} \int_0^\infty q^4 C(q) dq \quad \#(3)$$

The result yields RMS parameters that describe the surface as a whole, rather than any individual measurement.

2.4. Statistical Methods: Evaluating the statistical significance of differences between surfaces

One of the main goals of this paper is to quantitatively compare measured surface topography across techniques, which requires a method to determine whether two power spectral densities have statistically significant differences or are (statistically) indistinguishable. To achieve this, we followed and slightly adapted the method established by Chrostowski *et al.* [25, 26]. In particular, a statistical comparison was performed pairwise between all PSDs, using the four-step approach shown in Fig. 3 and described below.

First, segmentation.—If needed, the two PSDs under comparison are divided into regions of interest, such as the ‘roll-off region’ or the ‘self-affine region’. The reason for this step is that many PSDs contain regions with distinct slopes; therefore, it may not make sense to perform statistical testing on the curve as a whole. In the present analysis, this segmentation was only used to remove the artifacted (q^{-4}) portions of the SEM-measured PSDs; the remainder of the curve was not subdivided further.

Second, regression analysis.—In this step, we performed linear regression to the log-log data. While any arbitrarily complex equation can be fitted to the measured data (with or without weighting) [25], linear regression was chosen here for simplicity and ease of use. Assuming a portion of the one-dimensional PSD can be described by a power-law equation $C^1D(q) = C_0 q^{-1-2H}$, then the log-log plot can be fit to a line according to: $\ln(C^1D) = \ln(C_0)$

+ $(-1 - 2H)\ln(q)$. For each pair of measured power spectral densities, three separate line fits are generated using ordinary least squares regression (which include log transformation for both $C(q)$ and q): one fit using the first PSD, one fit using the second PSD, and one fit to both of the PSDs as a combined data set. Each of the three fits yields best-fit values for C_0 and H , and each one yields a maximum likelihood of those fit values, designated $\max L(C_0, H | C_{measured}^{1D})$. The maximum likelihood function is a standard statistical output; its form will vary with the underlying equation that was fit, but it is straightforward to calculate and is automatically output from most statistical software. For simplicity of notation, we designate all fit parameters by θ_i and all measurements of PSDs as Y_i , following the notation of Ref. [25]. For the linear-regression analysis, we further designate the two separate measurements using the subscripts 1 and 2, and the combined data using the subscript 1 U 2.

Third, likelihood-ratio test—Finally, we determine the statistical similarity of the two PSDs using hypothesis testing. While the fit will *always* be better with more fitting parameters, a likelihood-ratio test enables the evaluation of whether the improvement in fit is better than it would be from random chance. This statistical test uses the test statistic the log-likelihood ratio:

$$W_{test} = -2 \ln \left[\frac{\max L(\theta_{1 \cup 2} | Y_{1 \cup 2})}{\max L(\theta_1 | Y_1) * \max L(\theta_2 | Y_2)} \right] \quad \#(4)$$

If the null hypothesis is correct (i.e., the two PSDs are indistinguishable), then the test statistic should be equal to a chi-squared distribution with the number of degrees of freedom equal to the difference in model parameters. Since the numerator has two fit parameters (as mentioned, θ contains both fit parameters C_0 and H), and the denominator has four fit parameters, then the critical value of the test statistic, for 95% confidence and 2 degrees-of-freedom, is $W_{crit} = 5.991$.

Fourth, statistical determination.—Finally, if $W_{test} > W_{crit}$ then the null hypothesis can be rejected, and the two PSDs are considered statistically significantly different. Otherwise, the null hypothesis cannot be rejected, and two PSDs must be considered indistinguishable. In the former case, the linear regression can be used to determine whether the primary differences are in the slope (i.e. Hurst exponent, fractal dimension) or the intercept (i.e., the vertical offset of the curves). Of course, the individual values for H and C_0 can still be reported if the data sets fail the likelihood-ratio test, but their differences cannot be considered meaningful. See Ref. [25] for an excellent discussion of the number of measurements that should be required to resolve differences of varying sizes.

3. Results and Discussion

3.1 Extracting topography, and topography metrics, from SEM images

Representative images from the side-view SEM measurements are shown in Fig. 4. Examples are shown at two different magnifications for all four of the samples.

Qualitatively, the samples appear approximately similar, though some differences are apparent. First, for the wedge sample (Fig. 4a), the appearance of the material in the lower half of the image is significantly different from the material in the lower half of the cross-section samples (Fig. 4b–c). This reflects the fact that the cross-section samples reveal the inside of the sample, i.e., the silicon sub-surface material, while the wedge sample looks only at the surface itself, i.e., only the UNCD material. However, this sub-surface material is not being characterized in this investigation. Rather, only the boundary between the light-colored material and black background is being found; therefore, this sub-surface difference will have no effect on results. Furthermore, while there is a difference in thickness of the sample between the various preparations, this effect was extensively studied in Khanal *et al.* [19] and it was determined not to have an effect on the statistical characterization of the surface. For each sample preparation, one image was collected in three locations at six different magnifications.

First, the multiscale metric—the power spectral density—was computed (see Methods) from the extracted topography profiles. Figure 5 shows the complete results from the wedge-deposition sample. The PSD was computed from each individual profile and then all PSDs are averaged together for a single multiscale statistical representation of the surface as a whole. There are no adjustable fitting parameters in the PSD analysis, it is shown that the measurements across scales from nm to mm all align reasonably well. For clarity, the typical slope of common topography artifacts (q^{-4}) is shown in red. Second, the scalar metrics—the RMS height h_{rms} , and RMS slope h'_{rms} —were computed (see Methods) in frequency space from the averaged PSD. The results for the wedge-deposition sample resulted in $h_{\text{rms}} = 14.8$ nm and $h'_{\text{rms}} = 0.46$. The values were calculated only from the reliable region. All quantitative topography measurements used in the present paper are uploaded to a repository and are freely available (see Data Availability statement for details). The following section will evaluate the accuracy of SEM-measured topography across size scales.

3.2. Evaluating the accuracy of the SEM-measured topography: Comparing against other techniques

The topography measured here using SEM is quantitatively compared against previous topography measurements on the exact same samples: ultrananocrystalline diamond from the wedge-deposition method. In the previous measurements, Gujrati et al. [9] used stylus profilometry, atomic force microscopy, and transmission electron microscopy. First, the SEM-measured topography is evaluated at the small scales, through comparison to the AFM and TEM data. Figure 6 shows the power spectral densities of topography as measured previously using TEM and AFM, as compared to those of the present SEM technique. Note that the prior work includes the removal of the tip-size artifacts for the contact-based techniques [9].

The SEM-measured topography shows good agreement with the AFM and TEM measurements over the intermediate range of wavevectors (size scales) but appears to deviate at the small-scales. Well-known artifacts [8] cause the power spectral density to follow q^{-4} behavior, but typical explanations like tip-based artifacts [7] are not applicable. Instead, it is possible that the blurring artifact due to charging, which is visible in the

higher-resolution images in Fig. 4, is causing the same effect. The wedge topography (orange curve) appears to show a sudden deviation to q^{-4} -behavior (Fig. 5) at a wavevector of approximately $3 \times 10^8 \text{ m}^{-1}$, which corresponds to a length scale of 16 nm.

At the large size scale, the SEM-measured topography showed deviations from the prior stylus-based measurements, below a wavevector of approximately $1 \times 10^7 \text{ m}^{-1}$, or larger than 500 nm. To quantitatively determine the statistical significance of the difference between them, the likelihood-ratio test was performed (see Methods) between 1 μm and 1 mm (wavevectors of 103 to 10^6 m^{-1}). The likelihood ratio test showed that the two PSDs in that range were statistically significantly different (95% confidence).

Therefore, the SEM topography is only accurate up to a maximum size of approximately 500 nm. This result is somewhat surprising as the SEM has no maximum size limit of measurement. One possible explanation for the deviations of SEM-measured topography from true topography at the largest size scales is planarity distortions, as discussed in Ref. [29]; another possible explanation is the 1-to-1 nature of the measurement technique, such that there is a coupling of height resolution to lateral resolution in a way that does not occur in AFM or stylus measurements. In other words, for the SEM, a large lateral measurement size can only be accomplished by also increasing the size of an individual pixel. Overall, this large-scale limitation of the SEM technique must be accounted for; however, many other techniques (e.g. stylus profilometry) exist for measuring topography at size scales larger than 1 μm .

In summary, the SEM-measured topography has been compared against prior results for the same material based on multiple techniques. The power spectral density was used to determine a large-scale and small-scale cutoff where SEM-measured results began deviating from true topography. Using these two empirical cut-offs, the SEM measurement of topography is considered validated over a range of length scales from approximately 500 nm down to approximately 16 nm.

3.3. Evaluating the consistency of SEM-topography for the four proposed options for sample-preparation method

Here we compare the results from the four different approaches described in the Methods section to understand whether the sample-preparation technique significantly affects the measured results. To investigate this effect, the power spectral density was computed from all four of the different methods: wedge deposition, simple fracture, mechanical polished, and ion polished, as shown in Fig. 7. Qualitatively, the four power spectral densities appear similar, but with random fluctuations, and with an apparent upward deviation of the wedge sample from the remaining samples in the mid-range of size scales. The RMS height and slope for the four different sample preparation techniques are shown in Table 1.

Here, the results of the likelihood-ratio test are shown in Table 2. In the segmentation step, the artifacted regions were removed such that comparisons have only been performed over the determined range of SEM accuracy: 16 nm to 500 nm. Various assumptions of OLS were confirmed for the log-log data, including the assumptions that the independent variable and the residuals of the regression were normally distributed, as well as the

assumptions of linearity and homoscedasticity. The results showed first that the SEM and TEM measurements for the wedge-deposition sample yielded indistinguishable over the non-artifact region (Table 2, top row). This confirms quantitatively the qualitative conclusion that shown in Fig. 6. In addition, the subsequent rows show a pairwise comparison between the results from the four separate sample-preparation methods. The results show that the sample-preparation technique has no discernible effect on the resulting measurement of surface topography. In summary, the SEM-measured topography is as accurate as TEM down to a size of 16 nm, regardless of which sample preparation technique is used.

3.4. Achieving multiscale topography characterization by combining topography from SEM and stylus

The conclusion from this analysis is that the different options for sample-preparation method that are shown in figure 1 yield indistinguishable results, and therefore the recommendation is to use whichever method is (a) appropriate for the material of interest and then (b) simplest. For example, the wedge-deposition is the simplest for a material that is deposited onto a substrate, such as measuring the topography of a wear-resistant coating like diamond-like carbon or chromium nitride. The topography in a real-world use-case can be assessed by including some witness samples in the deposition chamber. However, this will not be possible for a bulk sample, or a material that has been modified after deposition, such as a semiconductor substrate that has undergone chemical-mechanical planarization. In that case, since the substrate is brittle, a simple-fracture approach could be used. Finally, in cases of a ductile material, such as measuring the topography of a metal component made with additive-manufacturing, the mechanical-polishing technique will be required. Finally, for the case of a very easily damaged substrate, such soft metals or certain ceramics that do not polish well, the ion-polishing method will be needed. In summary, for a wide variety of materials, SEM-measured topography can be accurately used to capture topography down to approximately 16 nm, with very little additional effort required.

Fig. 8 compares the multiscale topography as collected using two different approaches: first, the previously published multiscale PSD of UNCD [9], which was taken with stylus, AFM, and TEM; and second, a new multiscale PSD that is computed only using the present SEM analysis (only the reliable region of 16 nm to 500 nm) in combination with the stylus measurements. The aforementioned statistical test was applied to the two curves with a segmentation at 1 micron, delineating the self-affine and roll-off regions. The result of the test was that the two PSDs were found to be indistinguishable from one another across the whole reliable region.

There are two critical factors that must be considered during the use of the above method (stylus + SEM). First, the SEM-based technique results in a loss of information on the smallest-scale topography as compared to TEM, between the range of approximately 4 Å to 16 nm. There are some situations where this loss of measurement resolution could have a significant effect on predicted results (such as the adhesion of soft materials described in Ref. [30]), and other situations where there is a minimum size scale below which the roughness no longer contributes [31]). Thus, care must be taken to understand these effects. Second, as described in the prior investigation [9] the stylus profilometer must be set to

report the raw topography, absent any filters that are commonly used in ISO standards (e.g. ISO-21920 [32] or ASME B46.1[33]). This can be achieved by setting the stylus tool to report the “primary profile” as defined in ISO-21920 [32], (which is also called the “P-profile”) rather than the “roughness profile” (R-profile) or the “waviness profile” (W-profile). The above-described method will not apply if some scales of topography are being automatically removed in pre-processing of the data. With these two critical considerations, the above technique appears promising.

There are significant advantages of the stylus + SEM technique. The practical advantages of SEM over TEM are availability of SEMs, ease of use of SEM, and ease of sample preparation. SEM also has some advantages over AFM, including a lack of tip-radius artifacts, as well as the ability to measure overhangs, which become important in metals additive manufacturing (3D printing) and other complex geometries. However, there are distinct disadvantages of this cross-section SEM compared to AFM, including the difficulty of measuring non-conductive samples and the acquisition of line-scan topography instead of area-scan topography.

Overall, this study compared detailed measurements of UNCD using different combinations of techniques. A limitation of this study includes the fact that only a single material was evaluated, such that the findings could potentially fail to generalize to other materials. And with known limitations of SEM, such as with non-conductive materials, it is likely that the findings will not apply universally. Therefore, further investigation is required to fully validate and establish this method. Nevertheless, the findings of this study are encouraging for establishing the use of cross-sectional SEM in multiscale characterization of multiscale topography.

4. Conclusions

Because of the well-established need for multiscale surface characterization, the purpose of this paper is to establish a simpler method for characterizing multiscale topography. This investigation has three primary conclusions. *First*, cross-section (or side-view) scanning electron microscopy was shown to accurately *measure* topography at lateral size scales from 500 nm down to 16 nm. Large-scale artifacts were attributed to planarity distortions and the 1-to-1 scale of SEM images; small-scale artifacts were attributed to charging that occurs at sharp corners. *Second*, four different options for sample preparation were evaluated and determined to be equivalently accurate within the limits of statistical testing (likelihood-ratio test). These techniques are (1) wedge deposition (for deposited coatings), and three cross-section methods: samples created with (2) simple fracture, (3) mechanical-polishing, or (4) ion-polishing. In general, the best technique to use will vary with the type of material. *Third*, it is suggested that a nearly complete multiscale topography can be achieved using *only* stylus profilometry and SEM; two techniques that are commonly available and reasonably quick and straightforward to perform. Care must be taken to (A) turn off all filters in the stylus profilometer and (B) limit the range of reliability of the SEM-measured topography. But with these caveats, this may present a straightforward method for characterizing all scales of topography over seven orders of magnitude, from approximately 15 nm to 15 mm.

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Data Availability

The data that support the findings of this study are openly available using the following DOIs:

- SEM-measured topography data for UNCD, created using Method 1: Wedge Deposition: <https://doi.org/10.57703/ce-zwcjk>.
- SEM-measured topography data for UNCD, created using Method 2: Simple Fracture: <https://doi.org/10.57703/ce-mqta3>.
- SEM-measured topography data for UNCD, created using Method 3: Mechanically Polished: <https://doi.org/10.57703/ce-b6ptc>.
- SEM-measured topography data for UNCD, created using Method 4: Ion Polished: <https://doi.org/10.57703/ce-c57vn>.

The previously published data for Ref. [9] can be accessed using the following DOI: <https://doi.org/10.57703/ce-jdyep>.

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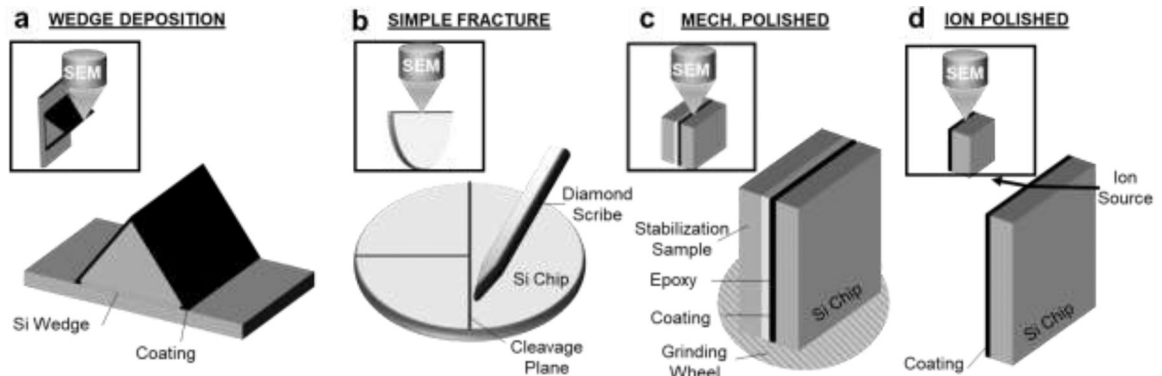


Figure 1: Four different options for sample-preparation were evaluated and compared.

(a) Method 1: The “wedge-deposition” method involves coating a wedge or thin blade with the material of interest and then imaging the apex of the wedge in profile in the SEM. It requires no further sample preparation and is applicable to any material that is deposited as a coating, for example those fabricated using physical vapor deposition, chemical vapor deposition, or spray coating. (b) Method 2: The “simple-fracture” method involves scribing the material of interest and then controllably breaking the material into pieces, exposing the fractured surface. This method involves little sample preparation, and is applicable to any brittle material, for example, silicon wafers and other functional materials used in electronics. (c) Method 3: In the “mechanical-polished” method, the sample is mounted in resin and subjected to grinding and polishing, as is common in materials science. This is often known as “metallographic sample preparation,” and is quite a general technique which can be used on metal samples, but also ceramics, polymers, composites, and many other materials. (d) Method 4: Finally, the “ion-polished” method uses grazing-incidence ion milling to gently remove material without significant damage. It is the most time- and equipment-intensive technique but is useful for samples that suffer damage during polishing or otherwise are incompatible with the other sectioning techniques.

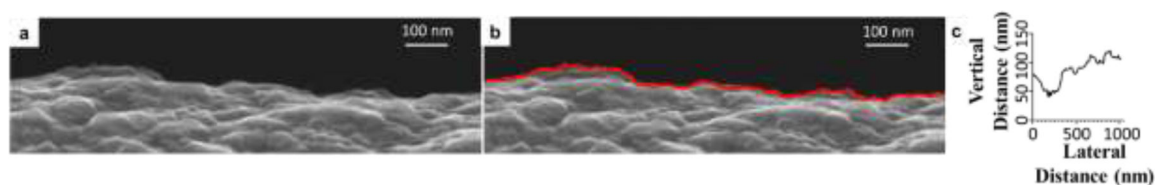


Figure 2: Extraction of a quantitative profile.

(a) A side-view SEM image reveals the boundary between the light-colored region (material) and the dark-colored region (vacuum). (b) This boundary is digitized using automatic edge-detection algorithms or manual point-picking, resulting in a quantitative 2D line profile (red line). (c) This line profile can be used and analyzed similar to the output of a stylus profilometer.

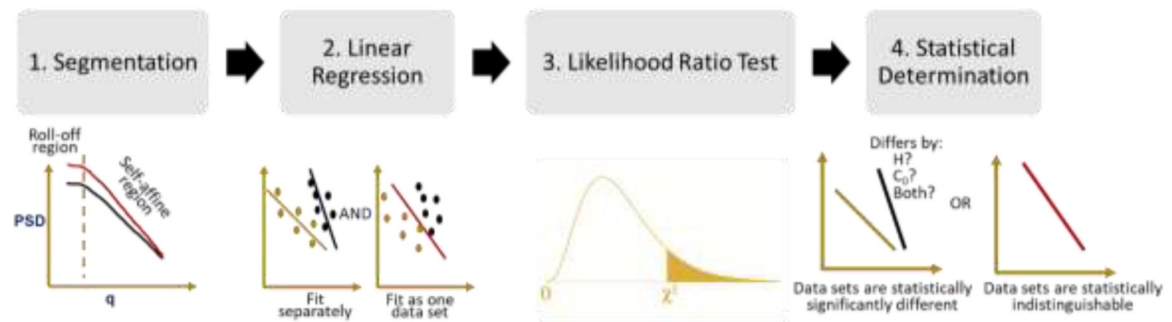


Figure 3: Statistical comparisons of PSDs.

This flow-chart demonstrates the statistical comparison of two PSDs. First, if needed, the two PSDs are divided into regions of interest; a typical division for many surfaces is between the ‘roll-off region’ and ‘self-affine region’. In the present data, this segmentation was only used to remove the artifacted portion of the curves. Second, the likelihood ratio test (main text) is performed for the two curves in the region of interest, determining whether they are distinguishable or indistinguishable. Third, if distinguishable, ordinary-least squares is used to determine whether they differ in slope (i.e. fractal dimension, or Hurst exponent[27]) or in offset (i.e. magnitude of the power spectral density).

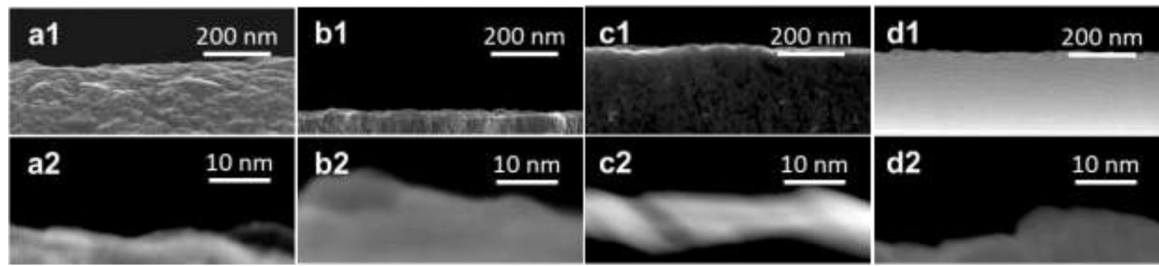


Figure 4: Side-view SEM images of the surface.

The various sample preparations (from Fig. 1) were imaged in profile in the SEM to reveal the surface topography. Representative images are shown here, at two different magnifications for each sample: (a) wedge deposition; (b) simple fracture; (c) mechanical polished; and (d) ion polished. In each image, the lighter-colored region represents the UNCD material and the black region on top represents vacuum. Therefore, the boundary between them represents one line profile of the surface of interest, extracted as shown in Fig. 2. We note that some blurring of the edge is visible in these images due to charging; this is a common corner effect in SEM, and thus its impact on results will be explicitly discussed.

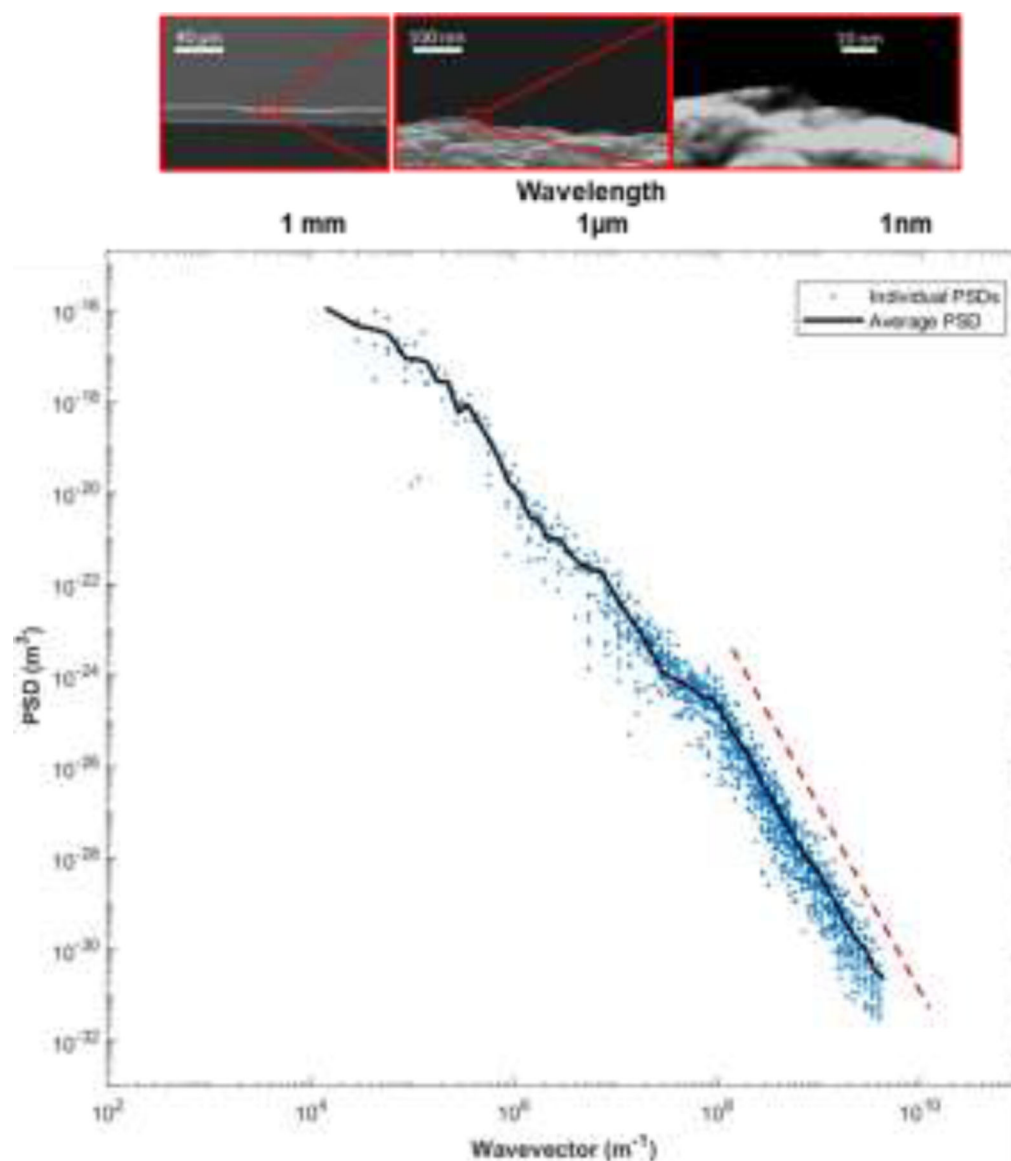


Figure 5: Multiscale analysis of SEM-measured topography using the power spectral density. After the surface was imaged (see Fig. 4), then the topography profiles were extracted (see Fig. 2), and each individual profile was used to compute its PSD (see Methods). Each individual PSD is plotted here (points) for the wedge-deposition sample. Then, all individual PSDs are averaged to create one single average PSD (line) that is a statistical representation of SEM-measured surface topography across all scales. The following section evaluates the reliability of different regions of this measured curve. For clarity, the typical slope of artifactual data (q^{-4}) is shown in red.

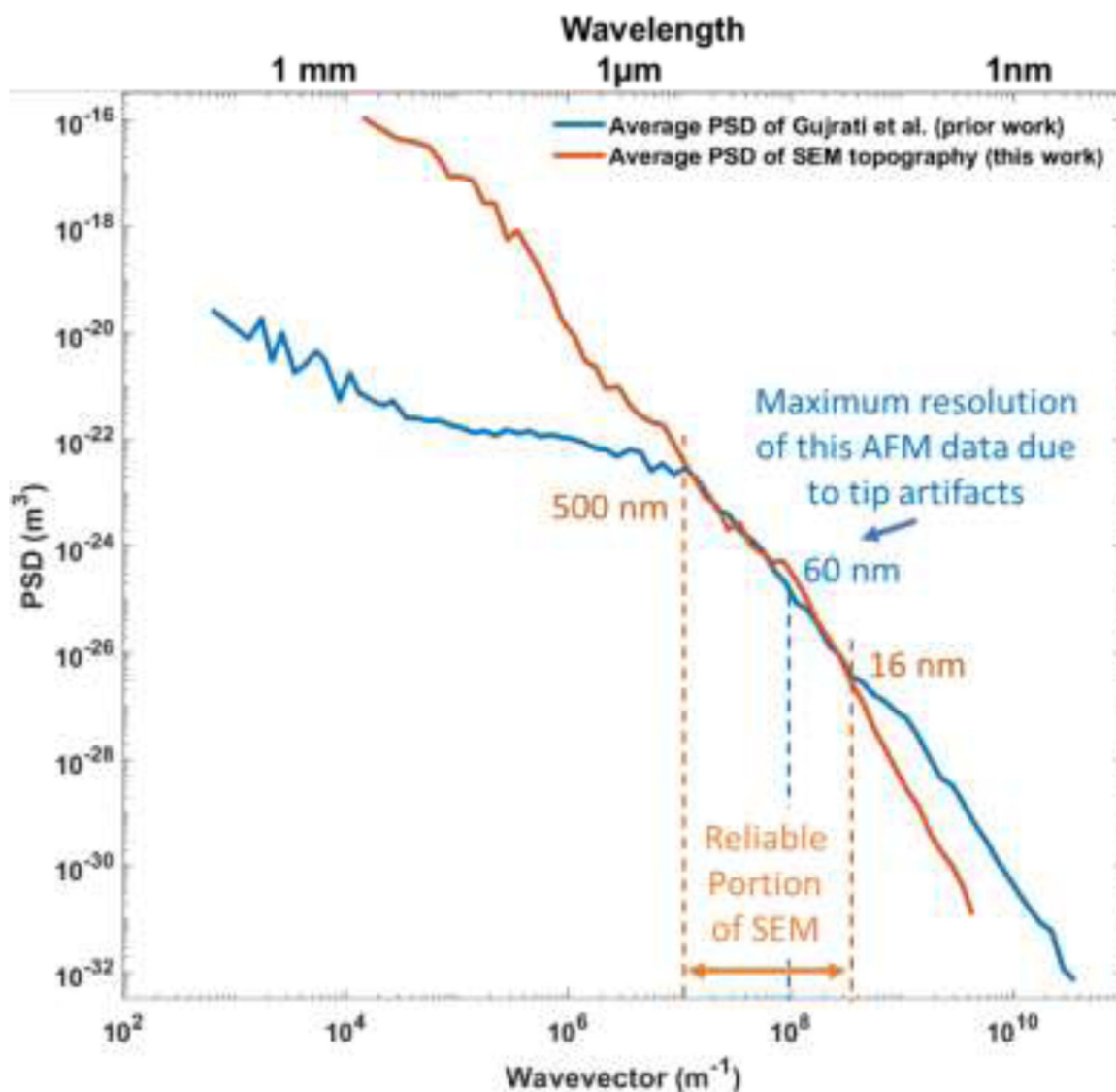


Figure 6: Validating the SEM-measured topography against prior results.

(a) The SEM-measured topography from the present work (orange curve) was evaluated by comparison against the previously measured topography [9] based on stylus, AFM, and TEM. The data agrees well over the intermediate wavevectors; however, it deviates from prior measurements at the large and small scales. The sources of deviation are discussed in the main text. The SEM-measured topography is determined to be accurate over a range of 16 nm to 500 nm. By comparison, the maximum resolution of AFM was limited to 60 nm in the prior work, and can be larger in cases where the tip radius is unknown or is actively blunted due to wear.

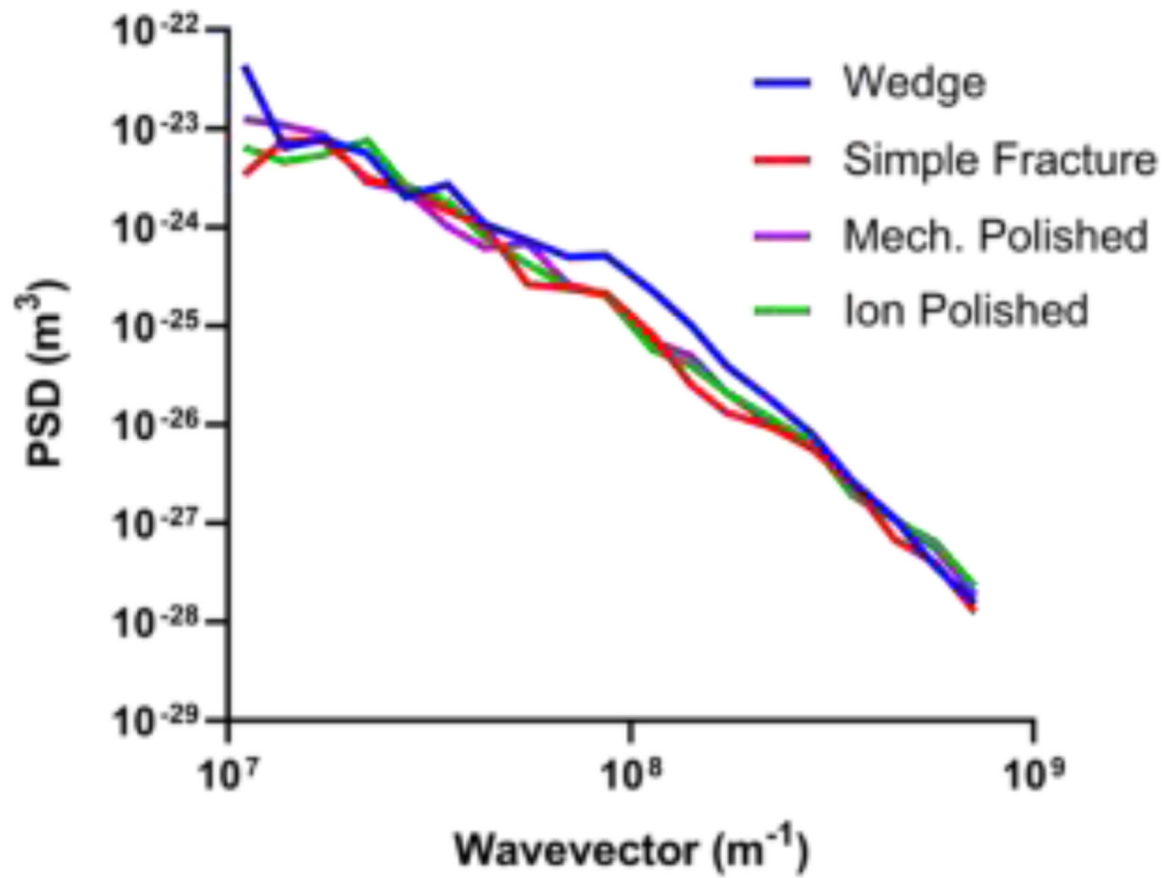


Figure 7: Comparing results across sample-preparation techniques.

The averaged power spectral density is computed for the SEM-measured topography of all four sample-preparation methods (legend). Results are only included for the region of wavevectors determined to be reliable (see previous section). The results from the different methods are qualitatively similar; a quantitative analysis is performed below.

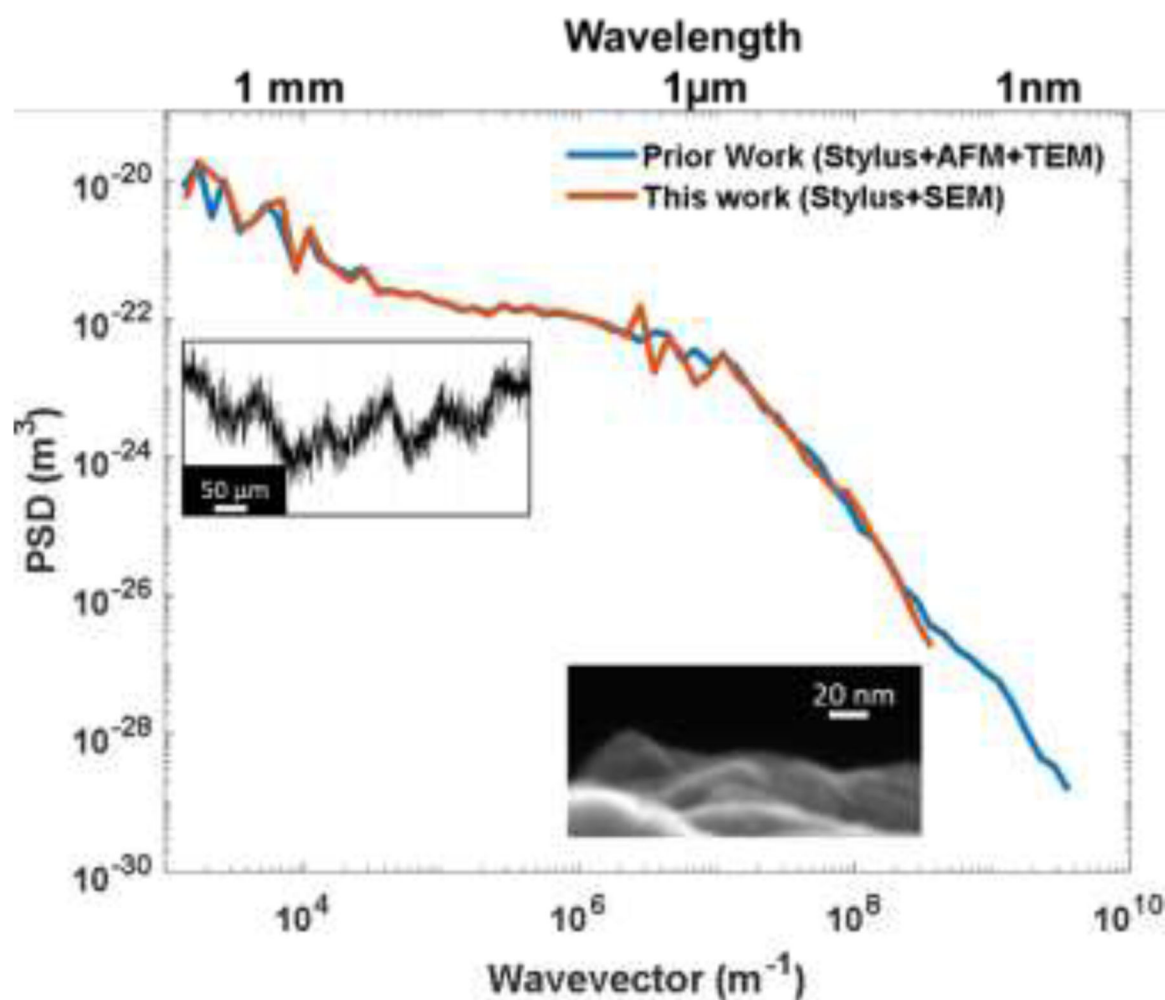


Figure 8: Measuring multiscale topography using only SEM and stylus.

Here we compare the previously reported comprehensive topography characterization of UNCD using TEM, AFM, and stylus (blue) against the PSD generated from a combination of only SEM and stylus (orange). The newer SEM-based analysis is far simpler and less time-consuming, while the results are statistically indistinguishable of the range evaluated. The only sacrifice is in high-resolution information below approximately 15 nm; in some conditions of materials system and performance criterion this may matter, but in other conditions this small scale of roughness will not contribute to performance.

Table 1:

RMS height and slope is extracted from the average 1D PSD, using equation 2, for each of the cross-sectional preparation methods for the SEM analysis of UNCD topography.

Treatment	h_{rms} (nm)	h'_{rms} (m/m)
Wedge deposition	14.8±0.4	0.46±0.14
Simple Fracture	14.4±0.2	0.33±0.12
Ion Milled	14.1±0.1	0.37±0.18
Mechanically Polished	14.2±0.3	0.34±0.12

Table 2:

The results from the likelihood ratio test comparing the averaged PSDs in the non-artifact region for each pairing of the measured PSDs for UNCD. The likelihood ratio W_{test} is compared against the critical value W_{crit} required to reject the null hypothesis ($\alpha = 0.05$). To save space, the wedge-deposition sample is abbreviated as just “wedge”.

Comparison (Sample 1: Sample 2)	Likelihood ratio W_{test}	Critical value W_{crit}	Statistical determination
Wedge SEM: Wedge TEM	0.618	5.991	Indistinguishable
Wedge SEM: Simple Fracture SEM	1.376	5.991	Indistinguishable
Wedge SEM: Mech. Polished SEM	1.652	5.991	Indistinguishable
Wedge SEM: Ion Polished SEM	1.527	5.991	Indistinguishable
Simple Fracture SEM: Mech. Polished SEM	0.921	5.991	Indistinguishable
Simple Fracture SEM: Ion Polished SEM	1.185	5.991	Indistinguishable
Ion Polished SEM: Mech. Polished SEM	1.247	5.991	Indistinguishable