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Roughness analysis for textured surfaces over several orders of magnitudes

Laura Vepsäläinen[a], Petri Stenberg[b], Pertti Pääkkönen[b], Markku Kuittinen[b], Mika Suvanto[a], Tapani A. Pakkanen[a,*]

[a] Department of Chemistry, University of Eastern Finland, Joensuu Campus, P.O. Box 111, FI-80101 Joensuu, Finland
[b] Department of Physics and Mathematics, University of Eastern Finland, Joensuu Campus, P.O. Box 111, FI-80101 Joensuu, Finland

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ABSTRACT

Multiscale structured surfaces have roughness distributions at various spatial frequencies that affect surface properties of materials. A recently developed filtered power spectral density (FPSD) method for surface roughness characterization was generalized to comprise structures from micro- to nanoscale. Furthermore, a uniform analysis method for micro- and nanoscale characterization over five orders of magnitudes was found by combining optical profilometry data, at the microscale level and atomic force microscopy data, at the nanoscale level. The FPSD method was also combined with structure simulation for multiscales, thus the roughness distributions can be designed and studied without the fabrication of structures. Furthermore, the FPSD simulation offers a design tool for structure–property correlations.

1. Introduction

Features on surfaces span many orders of magnitudes, which offer characteristic properties. At the micro- and nanoscales there are six orders of magnitudes that range from the visible surface textures down to the atomic lever structures. The horizontal features of the surface structures are either periodic or random, while the vertical features account for the surface roughness properties. In multiscale structures there is an interplay of surface features in different scales, which yields important physical and chemical surface properties.

Multiscale structures are characteristic of natural surfaces, where hierarchy also plays an important role [1–12]. For instance, randomly distributed micropapillae of lotus leafs covered with nanostructures cause the special wettability, cleaning, and adhesion properties of the leafs [3,4]. Similarly, water-strider can walk on water due to the durable and robust orientated needle-shaped microsetae and elaborate helical nanogrooves locating on its legs [6,7]. Overall, the multiscale structures affect the material properties, and the topography has to be noticed in the design of new materials [13]. A case in point is biomimetics, which imitates natural multiscale and hierarchical structures onto artificial surfaces in order to mimic unique features of nature [14–18]. A number of biomimetic materials imitating plant leaves [19–26,28], butterfly wings [27–29], and insect compound eyes [30–32] have been developed in order to achieve properties such as superhydrophobicity [19–23,25,27,28], self-cleaning [19–23,25,27,28], low-adhesion [20–22,26–28], anisotropic wetting [25,26], structural color [27,28], anti-reflection [30,31], and anti-fogging [32]. The surface properties, resulting from the multiscale structures, are due to the surface textures being at different length scales, whereas chemical properties depend less on material topography.

Inspired by natural structures, surfaces consisting of multiscale structures are fabricated by a variety of methods, for instance, by lithography [19,21–23,31,32], deposition [20–22,27–29,32], transfer [19,24–26,29,30,32], and etching techniques [31,33]. A common approach toward producing multiscale structured surfaces is to combine mechanical techniques in microscale to nanoscale structures formed by chemical self-ordering processes. An illustration of this is anodized aluminum oxide (AAO) that forms a self-ordered hexagonally packed pore structure inside domains [34–36], which has been combined, for instance, with lithography [37] and transfer [38] techniques.

In order to understand the material properties, multiscale surface structures have to be characterized in horizontal and vertical directions for all length scales. Microscopic techniques, such as a scanning electron microscope (SEM) and a transmission electron

* Corresponding author. Tel.: +35 8405028982; fax: +35 8132513390.
E-mail addresses: laura.vepsalainen@uef.fi (L. Vepsäläinen), petri.stenberg@uef.fi (P. Stenberg), pertti.paakkonen@uef.fi (P. Pääkkönen), markku.kuittinen@uef.fi (M. Kuittinen), mika.suvanto@uef.fi (M. Suvanto), tapani.pakkanen@uef.fi (T.A. Pakkanen).

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microscope (TEM), offer high quality visual images of horizontal and vertical directions, but provide limited topographical information and quantitative data concerning surface roughness and spatial orientation or periodicity of the structures. However, these properties are important when the roughness distributions and spatial frequencies that affect the function of the structures are to be considered. Microscopic methods are also less useful in characterizing large surface areas in detail. Profilometric methods yield structural information over large surface areas, which can be analyzed by various mathematical variables. Atomic force microscopy (AFM) offers precise surface data particularly at the nanoscale, while the profilometric measurements are most useful for the microscale [39,40].

The vertical direction of a surface structure is frequently analyzed by conventional roughness parameters [39–41]. A commonly used parameter is the root-mean-square (RMS) roughness (R_q) which is shown in Eq. (1) [41],

$$R_q = \left[\left(\frac{1}{l} \right) \int_0^l Z^2(x)\, dx \right]^{1/2} \quad (1)$$

where l is the sampling length and $Z(x)$ is the height of the assessed profile at any x position. It can be seen from Eq. (1) that the R_q value offers height information as an average value over the analyzed structure but contains no description of the horizontal spatial distribution of the surface features. Therefore, roughness differences between spatial frequencies cannot be separated, and structures with different properties may have identical roughness values. The horizontal roughness distributions can be studied by filtering methods by dividing the surface into arbitrary ranges [42]. Another approach is to measure areas of various sizes, when roughness is obtained in different scales [42,43]. However, the disadvantage of these methods is that they offer little information about surface periodicity or orientation. Roughness differences at spatial frequencies have been analyzed by the filtered power spectral density (FPSD) method [44], which combines power spectral density curves (PSD) of horizontal features and filtered root-mean-square (RMS) roughness values. This method has been used for microscale structures and for profilometric measurements.

The complete characterization of the surface structures requires a multiscale analysis in order to examine the connection between structure and function. The aim of our work is to generalize the FPSD method in order to cover both micro- and nanoscales and to find a uniform measurement technique for their characterization. In addition, the FPSD simulation will be demonstrated for multiscale structures. We also propose a design tool that employs structure–property correlations.

2. Materials and methods

2.1. Fabrication of the structures

Silicon wafers were coated with negative tone hydrogen silsesquioxane (HSQ) electron beam (Ebeam) resist XR-1541 from Dow Corning. This resist was patterned by an Ebeam patterning tool, Vistec EBPG 5000+ ES HR, with an acceleration voltage of 100 kV and a dose of 4500 μC/cm^2 for the sample with the largest diameter and 5000 μC/Cm^2 for the other samples. Exposed resist was developed with a sodium hydroxide buffered solution that contained MP 351 developer (from Rohm and Haas Electronic Materials) and deionized water at a ratio of 1:3. The patterns for the sample with the largest diameter were etched into a silicon substrate by a hydrogen bromide based plasma etching process, in order to produce a structure which is directly suitable for an optical profilometer measurement. For the etching process, HSQ with a 200 nm thickness was used as a mask material. After etching, residues of the mask were removed in buffered hydrogen fluoride, which led to a plain silicon sample. For the other samples, no additional silicon etching is needed because the final pattern height is determined in the spin coating process and the resist patterns can be used as test structures as they are.

Aluminum foil (Alfa Aesar Puratronic®, 99.997%, thickness 0.25 mm) was cut into a 4.0 cm × 4.0 cm plate and preprocessed as described earlier [38,45]. The plate was nanostructured by an anodic aluminum oxide (AAO) process, as in previous studies [38,45].

2.2. Characterization of the fabricated structures

The heights of the silicon structures were determined by a stylus profilometer (Dektak 150+) from Veeco (currently Bruker AXS). The height of the etched structure was 530 nm, whereas the height of the other silicon structures was 110 nm.

The fabricated silicon structures and anodized aluminum oxide structure were imaged with a Field Emission Scanning Electron microscope (FE-SEM) (Hitachi S-4800). The structures were fixed onto a stub, with carbon and copper tapes. The accelerating voltage was 3 or 5 kV and the working distance was 9.7–9.9 mm.

The height profiles of the silicon and AAO structures were measured by an atomic force microscope (AFM) (Autoprobe M5) from Thermo Microscopes using standard aluminum coated silicon probes in noncontact mode. The measurement areas of the silicon structures were 100 μm × 100 μm–5 μm × 5 μm with pixel resolutions of 0.39–0.020 μm, respectively. For the AAO structure, the measurement area was 2 μm × 2 μm with the pixel resolution of 0.0078 μm.

Furthermore, one silicon structure with the largest diameter was measured by an optical profilometer (OP) (Wyko NT9300) from Veeco (currently Bruker AXS) in order to combine two different surface measuring techniques, the optical profilometry and AFM. The measurement area was 94 μm × 125 μm, and the pixel resolution of the measurement was 0.20 μm. All the measured height profiles were analyzed by an optical profilometer program, Vision (Vision 4.20), in order to determine PSD curves. The PSD curves were processed by Fourier filtering in order to obtain filtered roughness values at various spatial frequency ranges. The filtering ranges were 0.01–0.1, 0.1–1.0, 1.0–10, and 10–100 1/μm for the silicon structures and 0.1–1.0, 1.0–10, and 10–100 1/μm for the AAO structure. As micrometers, these ranges represent periodicities of 100–10, 10–1, 1–0.1, and 0.1–0.01 μm.

2.3. Simulation of the height profiles

The height profiles corresponding experimental silicon structures were simulated from the input data of an Ebeam device with a MATLAB® (R2011a)[1] code. The height parameter was set to 110 nm. The simulated structures were analyzed by the optical profilometer software, Vision, similarly as performed with the fabricated structures. The measurement areas and resolutions of the simulations matched the parameters of the experimental structures, thus allowing a direct comparison of the corresponding structures.

3. Results and discussion

Silicon pillar structures were fabricated to illustrate the frequency analysis at the transition regime of the micro- and nanoscales. In addition, the largest silicon structure demonstrates the combination of the profilometric and microscopic

[1] MATLAB is a registered trademark of The MathWorks, Inc.