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Abstract
A surface engineering method based on the electrostatic deposition of microparticles and dry etching is described and shown to be able to independently tune both amplitude and spatial roughness parameters of the final surface. Statistical models were developed to connect process variables to the amplitude parameters (center line average and root-mean-square) and a spatial parameter (autocorrelation length) of the final surfaces. Process variables include particle coverage, which affects both amplitude and spatial roughness parameters, particle size, which affects only spatial parameters, and etch depth, which affects only amplitude parameters. Correlations between experimental data and model predictions are discussed.

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Comments
Method to Generate Surfaces with Desired Roughness Parameters

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A surface engineering method based on the electrostatic deposition of microparticles and dry etching is described and shown to be able to independently tune both amplitude and spatial roughness parameters of the final surface. Statistical models were developed to connect process variables to the amplitude parameters (center line average and residual stress) and a spatial parameter (autocorrelation length) of the final surfaces. Process variables include particle coverage, which affects both amplitude and spatial roughness parameters, particle size, which affects only spatial parameters, and etch depth, which affects only amplitude parameters. Correlations between experimental data and model predictions are discussed.

Introduction

Surface energy and surface forces are often closely related to surface topography. Physically, surface topography is decided by the atomic arrangement at the surface of a material, which in turn may be decided by the material properties, processing method, and environment. Surface topography may be changed by external forces (e.g., during wear) or internal forces (e.g., residual stress). Under stable conditions, surface topography can be described by surface roughness parameters, which include both spatial and amplitude parameters. Amplitude roughness parameters describe height information. For example, the central line average (CLA) is an average value measuring points departing from a center line, and the root mean square (rms) is the standard deviation of profile ordinates. Spatial parameters describe spatial surface information. For example, the autocorrelation length (ACL) is the distance over which points on a profile can be treated as independent. In engineering, the ACL is often defined as the distance over which the autocorrelation function decays to 1/e of its original value. Besides these commonly used parameters, other parameters are also used to describe surface topography, including statistical parameters for peaks as well as fractal dimensions and the Hurst exponent for self-affine surfaces.

Surface roughness parameters are widely used to connect surface topography to a variety of surface phenomena. For example, the amplitude parameters (CLA and rms) have been shown to affect adhesion, friction, and wear as well as optical loss in the waveguide. The spatial parameter (autocorrelation length (ACL)) has been used to model the optical properties of a waveguide, substrate stresses in coating, and adhesion of thin elastic films as well as the real area of contact and the friction behavior of rough surfaces.

To systematically study roughness effects and use them for specific applications, it is of interest to develop a processing method that can generate surfaces with the desired roughness parameters. Commonly used processing methods, such as grinding and polishing, either lead to a large range of roughness variation or the lack of ability to control both amplitude and spatial roughness parameters. Most micro/nanoscale fabrication methods, such as wet/dry etching, micromolding, and pulsed laser machining, are generally used to realize deterministic structures or may not be suitable for processing large areas because of the sequential nature of operation. Recently, we have shown that a microparticle-based surface processing method using electrostatic deposition and dry etching can generate random surfaces with the desired ACL. This method is able to generate random surfaces that are not deterministic (i.e., random) and has the advantage of being applicable to large areas, which can potentially translate to high throughput. In this article, this method is shown to be able to tune both amplitude and spatial parameters of the final surface by controlling the process variables of particle size, particle coverage, and etch depth.

Experimental Details

Surface-Processing Technique. The proposed process is shown schematically in Figure 1. We used a silicon substrate to illustrate the process. First, a clean silicon (100) surface with a negatively

Figure 1. Process sequence involving the electrostatic deposition of particles and subsequent dry etching to generate random rough surfaces.
charged native oxide layer was achieved using a Piranha etch (3:1 H$_2$O$_2$/H$_2$SO$_4$). Next, a uniformly ionic layer was realized. A polycationic native oxide layer was achieved using a Piranha etch (3:1 ratio structures with C$_3$F$_8$ and SF$_6$ feed gases. During this line-of-sight etching process, particles act as temporary masks that result in “hillock”-like features on the substrate. The remaining silica particles were then removed using 49% hydrofluoric acid. To remove fluorocarbon carryover generated during the dry etch, all samples were cleaned in Piranha etch for half an hour, followed by Milli-Q water rinsing.

The process variables that affect the final surface topography are particle size, particle coverage, and etch depth. Figure 2 shows the final topography as a function of particle size and etch depth measured using atomic force microscopy. Figure 3a−c shows the final topography of surfaces as a function of particle coverage measured using atomic force microscopy. Figure 3d shows the topography and cross section of a single hillock. The hillock has a shape and dimensions decided by the diameter of the particles used and the etch time. These results indicate that the topography of the final surface can be tuned by varying the process variables.

### Surface Roughness Measurement

The topography of the final surfaces with an etch depth below 1 μm were obtained using an atomic force microscope, AFM (Dimension 3100, Vecco Instruments, Santa Babara, CA), in contact mode with a commercial Si$_3$N$_4$ probe (radius ~50 nm) at a scan size of 60 μm × 60 μm with 256 × 256 data points. All surface roughness parameters reported were obtained from the AFM images. The topography of surfaces with an etch depth in excess of 1 μm were obtained using scanning electron microscopy, SEM (JEOL JSM-606LV), without any conductive coating.

### Results and Discussion

Figure 4 shows the effect of particle coverage and etch depth on the amplitude parameters, center line average (CLA) and the root mean square (rms) of the final surfaces. Both parameters increase with an increase in etch depth. Also, both parameters increase with increasing coverage up to a coverage of 50%, beyond which the parameters appear to decrease with an increase in coverage. We note that the upper limit of coverage for spherical particles on flat substrates is 74%, which corresponds to coverage for a hexagonal closed-packed (HCP) or cubic close-packed (CCP) structure. The amplitude parameters showed no dependence on particle size.

We present a statistical model to relate the amplitude parameters to the process variables. An inspection of the final surfaces shows...
that their roughness (Figure 5a) includes two independent components—one caused by the particles, which result in the hillock structures, and the other caused by dry etching. For a 1D case, details of a typical profile are shown in Figure 5b obtained using atomic force microscopy, which includes the two components of roughness described above. Figure 5c shows a schematic that simplifies the roughness as a superposition of two random processes. The roughness caused by the particles is approximated as a random pulse signal, where the pulse width is decided by the particle size, $a$. This representation assumes that the profile goes through the center of all particles. This assumption is reasonable when the particle size is much smaller than the profile length. Generally, dry etched surfaces result in random surfaces that are very smooth with small height variations of several nanometers or less.18 If this height variation caused by dry etching is much smaller than the dry etching depth, which is generally true for all of our experiments, then the roughness caused by the particles will dominate the amplitude roughness parameters of the final surface. We will therefore consider only the roughness caused by particles in the following model.

Referring to Figure 5b, if we denote the profile length as $L$ and the sampling interval as $s$, then the total number of heights measured as $N$ can be written as $N = \frac{L}{s}$. If we denote the coverage of particles as $p$, the etch depth as $a$, and the center of the pulse height as the zero position, then $pN$ points will have ordinates of $y_i = a$ and $(1 - p)N$ points will have ordinates of $-a/2$.

The ordinate of the center line $m$ can be estimated as follows:

$$m = \frac{1}{N} \sum_{i=1}^{N} y_i$$

$$= \frac{1}{N} \left( \frac{apL}{2} - a \frac{(1 - p)L}{2} \right)$$

$$= ap - \frac{a}{2}$$

With the center line defined, amplitude parameters CLA ($R_a$) and rms ($\sigma$) can be written as follows:

$$R_a = \frac{1}{N} \sum_{i=1}^{N} |y_i - m|$$

$$= p \left( \frac{a}{2} - m \right) + (1 - p) \left( m + \frac{a}{2} \right)$$

$$= 2ap(1 - p)$$

$$\sigma = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (y_i - m)^2}$$

$$= \sqrt{p \left( \frac{a}{2} - m \right)^2 + (1 - p) \left( m + \frac{a}{2} \right)^2}$$

$$= a\sqrt{p - p^2}$$

Both CLA and rms are linearly proportional to etch depth $a$ and are nonlinearly dependent on particle coverage $p$, which match the trends seen experimentally in Figure 4. Figure 6 compares the experimental results of CLA and rms as a function of both particle coverage and etch depth with the model predictions. The model matches the experimental data quite well. We note that coverage $p$ for the experiments is over an area, whereas the model describes a profile. However, the profile is a limiting case of an area, and the coverage remains the same. Amplitude parameters generally depend only on the distance of ordinates from the mean line as well as the number of ordinates a given distance from the mean line. Both of these measures are adequately accounted for by the height of the hillocks $a$ and the coverage $p$. Hence, particle size does not figure into the relations above. This means that these estimations can be used for different particle size combinations. The expressions derived for this approach can also be applied in cases for which the dry etching may contribute to the roughness in a non-negligible manner (such as for materials with inhomogeneities and grain boundaries). In these cases, if the background roughness due to etching can be described mathematically using process and material parameters, then our approach can still be used, and the final expression for amplitude roughness will include the superposition of two contributing terms—that of the hillock structures and that of the background. Certainly this superposition implies that the hillock structures and the background are independent.

In our previous work, we developed a model relating process variables to the spatial parameter, the autocorrelation length (ACL).16 We provide a brief description of the model and focus on the results. Briefly, the final surface was treated as a superposition of the ACL due to the particle-based structures and that of the dry etching (as shown in Figure 5c). Dry etching is known to generate random roughness with an exponential autocorrelation function (ACF) on a silicon substrate.19 For other substrates, in addition to dry etching, grain boundaries or other inhomogeneous features may contribute to the representation of...
the ACF form. As long as the ACF for these features can be represented or quantified, the following approach to predicting the ACL of the final surface can be applied. The particles were modeled as pulses as described previously in the amplitude parameter model. The occurrence of particles (pulses) along a given profile length is treated as a random process, specifically, as a random telegraph signal (RTS), in which the pulse width is a variable following the Poisson distribution. Under conditions in which the particle size is small compared to the profile length, this Poisson approximation is reasonable. The RTS has an exponential autocorrelation function as well. Following our assumption of structure independence, the ACF of the final surface can be written as the superposition of the ACFs of the two random processes. The relation between the ACL of the final surface \( \beta^* \) and process variables is then given as the following equation:\(^{16}\)

\[
\frac{p}{d} = \ln\left(\frac{\sigma^2}{a^2/4}\right) - \ln\left[\frac{\sigma^2 + a^2/4}{e} - \sigma^2 e^{(\sigma^2/4a^2)}\right]^\frac{1}{2}\beta^* \tag{4}
\]

The final autocorrelation length thus depends on the coverage of particles \( p \), particle size \( d \), and etch depth \( a \) as well as the rms roughness and autocorrelation length of the surface resulting from the dry etching process, \( \sigma \) and \( \beta_1^* \), respectively. In the case in which \( \beta^* \gg \beta_1^* \) and \( \sigma \ll \lambda/2 \) (which is true for particle sizes in the micrometer range), eq 4 simplifies to a simple power law \( \beta^* \approx \lambda_{2p} \), which means that \( \beta^* \) is most sensitive to the hillock size \( d \) and particle coverage \( p \). This is reasonable because ACL is a spatial parameter and should not be significantly affected by amplitude changes resulting from \( a \) and \( \sigma \).

Figure 7a shows the effect of the process variables on the spatial parameter, the autocorrelation length (ACL) based on eq


4. Figure 7b shows the comparison between the experimental and model predictions. We did not have enough data points to compare against the prediction for particle size. We note that significant scatter in ACL is seen at lower values of coverage, which is attributed to particle clustering and implies that the proposed method may be applicable only for particle coverage larger than 20%. In the RTS model, the clustering effect is partially captured by treating the pulse length as a Poisson process, which allows particles to cluster together to form one pulse. This clustering phenomenon is not completely captured by our model, and we are currently investigating the use of an explicit structure function in our ACF description (rather than an RTS function) to include the effect of clustering. Furthermore, clustering can be minimized by employing techniques such as using functionalyzed particles or controlling drying conditions. We chose to retain the clustering phenomenon because of its ability to achieve larger values of ACL compared to surfaces without clustering (i.e., clustering allows a larger achievable range of ACL in our experiments).

From these models, it can be seen that particle coverage affects both amplitude and spatial parameters. The etch depth strongly affects the amplitude parameters, whereas the particle size affects only spatial parameters. This allows potential independent tailoring of amplitude and spatial parameters if desired. For example, for a given particle size, the particle coverage can be used to tailor the autocorrelation length by varying the immersion time in the colloidal solution. Then, the etch depth can be selected to obtain a target value of the center line average or the root mean square.

Conclusions

A surface engineering process that comprises the electrostatic deposition of microparticles and dry etching was shown to be able to tailor the surface structure and roughness parameters of an engineering material. This method has the potential to generate random surfaces with independent control of both amplitude and spatial roughness parameters. Models relating the key process variables—particle size, coverage, and etch depth—to amplitude and spatial roughness parameters were developed. The experimental results agreed with the model predictions fairly well for amplitude parameters whereas some discrepancies were observed in the case of the autocorrelation length as a result of the effects of clustering, which are not fully captured in our model.

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