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Roughness characterization in positive and negative resists

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Abstract

Different roughness parameters, such as the root mean square deviation (rms or σ), the correlation length L_{cor}, the fractal dimension D and the Fourier spectrum, are presented and compared. The scaling behavior of σ determining the L_{cor} as well as the dependence of σ and D on the exposure dose for two negative tone (wet- and plasma-developed) and one positive tone resist are investigated. The experimental analysis reveals an interesting interrelation (inverse behavior) between σ and D which is not predicted by theory, and elucidates the dependence of L_{cor} on the exposure dose.

Keywords: Metrology; Roughness; Fractal dimension; Correlation length; Resists

1. Introduction

Since the lithographic processes for this decade are facing the demands of sub-100 nm range characteristic dimension, the roughness of lithographic materials starts playing a significant role in the quality of the printed lines and the CD (critical dimension) control. CD metrology tools can now evaluate line edges and provide a 3σ line edge roughness (LER), which shows the importance of the subject. In the last few years, several works [1–11] have appeared in the literature studying different aspects of this problem. However, many relevant questions still remain open, motivating further work in the field. One such question refers to the determination of the best and most complete set of parameters characterizing the roughness of a curve or surface. A second open problem of constant interest has to do with the experimental study of the dependence of these roughness parameters on material properties and conditions of the lithography process. In this paper, we investigate some aspects of these problems giving emphasis to the possible interrelations between roughness parameters

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predicted either by theory or revealed by experimental analysis. The theoretical interrelations can help us understand deeper the first question and choose the best set of roughness parameters, while the experimental ones may shed light on the mechanisms responsible for the roughness formation and dependence on the particular process condition or material properties.

The paper consists of three more sections. Section 2 presents some roughness parameters, namely root mean square deviation σ, fractal dimension D, correlation length L_{cor}, and Fourier spectrum, and discusses their relations as predicted by theory. In Section 3, the behavior of σ and D vs. exposure dose is examined for experimental surfaces of both negative and positive tone resists and an interesting interrelation between σ and D is deduced. For the same surfaces, a scaling analysis of σ is carried out giving the dependence of L_{cor} on the exposure dose. Section 4 summarizes our conclusions.

2. Roughness parameters

A remaining crucial issue in roughness studies is the lack of a formalism that can describe a real, irregular curve or surface accurately and uniquely. This is why a lot of methods and parameters for the roughness characterization have been used in the past. However, in the field of lithography the most commonly used parameter is the root mean square deviation σ (usually 3σ values are reported).

Obviously, the great advantage of σ is that it can be calculated easily. Many scanning electron microscope (SEM) and atomic force microscope (AFM) instruments provide algorithms for calculating σ automatically. On the other hand, σ is limited to giving information on the important 'vertical' magnitude of the roughness without saying anything about the spatial complexity of surfaces or profiles. In addition, it is well known that σ depends on the size L of the sample of the surface we measure. Dynamical scaling theory predicts that this dependence may follow a power law for a specific range of L values. In particular,

$$\sigma(L) \sim L^{a} \quad \text{for } L < L_{cor} \tag{1a}$$

$$\sigma(L) \sim \sigma \quad \text{for } L > L_{cor} \tag{1b}$$

where a is sometimes called the roughness exponent ($0 < a < 1$) and L_{cor} denotes the correlation length after which no height–height correlations exist and $\sigma(L)$ converges to its final value σ [12,13].

Obviously, the estimation of σ is reliable if and only if the size of the sample we measure is larger than the L_{cor} since then the measured value σ becomes independent of the sample size L (Eq. (1b)). Furthermore, it has to be emphasized that statistics over many samples of size L is needed in order for the power law behavior of Eq. (1a) to appear. So, $\sigma(L)$ in Eq. (1a) is in fact an average over many samples with size L, randomly chosen on the total larger surface or line edge. The appearance of power law means that the surface can be described as self-similar or more generally self-affine and can be studied by fractal geometry [13]. Fractal geometry is an extension of Euclidian geometry, which studies self-similar or self-affine objects by using the notion of non-integer dimension. This dimension, referred to as fractal dimension D, is limited by an upper or lower bound; a rough fractal profile will have $1 \leq D \leq 2$ depending on how much area it fills, and a two-dimensional fractal surface will have $2 \leq D \leq 3$ depending on how much volume it occupies [14]. Thus, a very smooth curve or

line will have $D \cong 1$, while a smooth surface will have $D \cong 2$. Rough curves and surfaces will have, respectively, $D \cong 2$ and $D \cong 3$.

Fractal dimension D can be estimated through the roughness exponent a, as $D = 3 - a$ for a fractal surface [13]. However, because of the statistical character of Eq. (1a), the determination of a and hence D from Eq. (1a), requires a lot of samples to be measured and so it is quite time consuming. For this reason, we choose to estimate D by the variation method proposed by Dubuc et al. [15], which is based on the analysis of a single sample. Dubuc et al. have shown the superior performance of this method compared to other methods when applied to digitized profiles or surfaces. In fact, the variation method provides a new way of counting the total number of boxes (cubes) N required to entirely cover the profile (surface) as a function of the size of the box (cube) ϵ. The D will be the slope of any linear region of the plot of ln (N (ϵ)) vs. ln ($1/\epsilon$) (see Fig. 1a).

Another traditional method, providing information on the structure of a profile or a surface at different frequency regimes, is the Fourier transform. In Fig. 1b, the amplitudes $F(\omega)$ of the Fourier transform of a typical AFM line scan of a negative tone resist are shown. We observe that it roughly consists of two parts; a horizontal of constant amplitude at low frequencies and a power law behavior at higher frequencies. According to Parceval's theorem:

$$\sigma^2 = \frac{1}{2\pi} \int_{-\infty}^{+\infty} |F(\omega)|^2 \, \mathrm{d}\omega \tag{2}$$

the rms value σ is related to the power spectrum of the Fourier transform.

Moreover, according to the fractal theory [14], the exponent d of the power law in the spectrum of the amplitudes is related to D according to the relation $d = 2.5 - D$. However, it has been shown [16] that the accurate estimation of D from the exponent of the power law in the Fourier transform requires a large number of data points (more than 1000), which is not the case for the profiles we get from lithography metrology.

Therefore, we may infer that although theoretically σ and D are linked to features of the Fourier transform, for experimental surfaces and profiles, inspecting the Fourier spectrum is not a reliable and safe way to estimate them. For this reason, in the following, we will restrict ourselves to the investigation of σ and D characterizing the 'vertical' and 'horizontal' dimension of the roughness correspondingly, and the scaling analysis of σ leading to the estimation of L_{cor}.

3. Roughness of experimental surfaces

In this section, we will present the results from the calculation of the roughness parameters σ and D and the scaling behavior of σ for various resist surfaces. Our aim is to investigate the effect of different materials and process parameters as well as the possible interrelations between these roughness parameters. The scan size of the AFM samples of the surfaces we analyzed was 2.5×2.5 μm^2 and the grid lines 300 except from the samples of the epoxy resist (EPR) surfaces (see below) which were 1×1 μm^2 and the grid lines 512. The measurements were taken with a Digital Instruments Nanoscope III AFM in the non-contact mode and with a Topometrix TMX 2000. Comparison between instruments showed little difference in the measurements.

First, we examine the dependence of σ and D on exposure dose for various resist surfaces. Let us

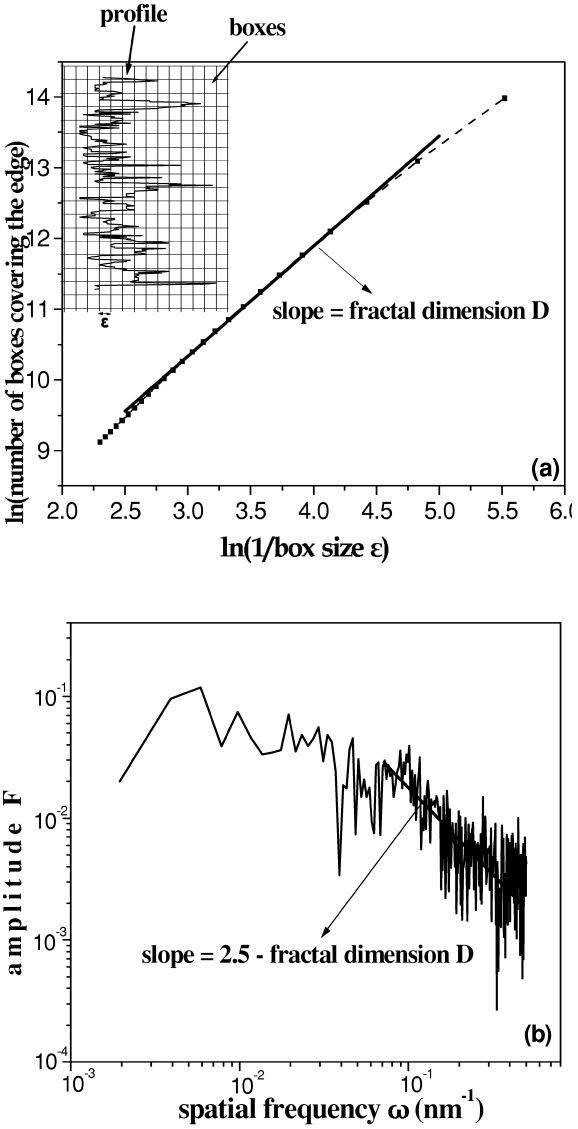

Fig. 1. (a) Estimation of the fractal dimension *D* of the profile shown in the inset. (b) Fourier amplitude spectrum for a typical AFM line scan of a negative tone resist. Notice the different behavior at low and high frequencies as well as the relation to *D*.

start with a wet-developed negative tone epoxy resist (EPR). This resist is extremely sensitive to e-beam and to deep ultraviolet (DUV) radiation and is developed with organic solvents [17]. A modified version of EPR can be developed with standard aqueous developer and is promising for micromachining applications [18]. EPR with 1% photoacid generator (PAG) concentration was used prebaked at 110 °C, e-beam exposed at 50 KeV to form 10×10 μm^2 pads, post-exposure baked at 100 °C, developed in propylene glycol methyl ester acetate (PGMEA), and measured in the AFM. Analysis of the measurements was performed with the methods described in Section 2. Fig. 2 shows

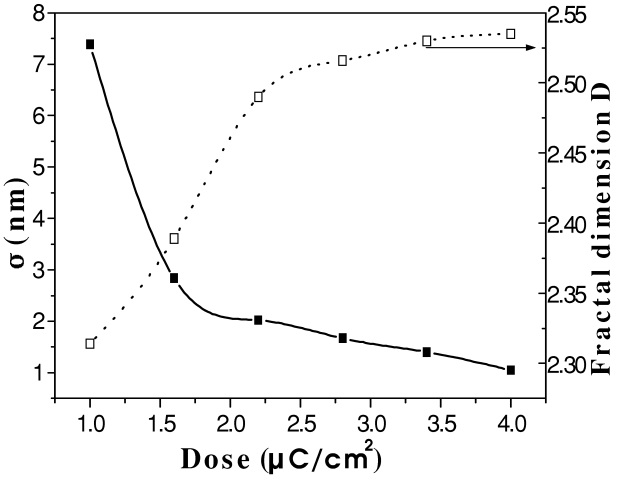

Fig. 2. Experimental values of σ and D vs. exposure dose for negative tone wet-developed epoxy EPR with 1% PAG. Note the opposite behavior of σ and D vs. dose.

the dependence of σ and D on exposure dose in the region 1.0–4.0 $\mu C/cm^2$ (experimental values). Two observations could be made. First, we observe that the σ goes through a maximum at low doses, i.e. at the onset of the contrast curve. As the dose increases, the σ drops quickly up to 2 $\mu C/cm^2$ and then continues decreasing but with a lower rate. On the contrary, the behavior of D seems to be the opposite. It increases following the increase in exposure dose. Here, we can also distinguish two regions: at doses lower than 2 $\mu C/cm^2$, D increases quickly, whereas at higher doses the increase is slower.

The same behavior has been observed in the second type of surfaces we analyzed. These come from a negative tone siloxane resist with oxygen plasma development in a high-density plasma (HDP) reactor (Alcatel MET inductively coupled plasma reactor). In particular, the resist is a commercial polydimethylsiloxane (PDMS) by Aldrich with a broad MW distribution ($M_n = 60{,}000$, $M_w/M_n = 2$) [8]. The resist was coated on hard-baked novolac at approximately 100 nm thickness. Although the resist without any sensitizer is very sensitive to 157 nm radiation, here it was exposed with 50 KeV electrons, and developed in methyl isobutyl ketone (MIBK). After wet development of the siloxane top layer, oxygen plasma development of the bottom layer followed in the HDP reactor. A break-through-step (BTS), without bias voltage and containing fluorinated gases and oxygen, preceded the pure oxygen plasma development. Fig. 3 shows the results of these dependencies for σ and D_F. The σ curve exhibits similar behavior with that of the EPR resist, with the important difference that at useful doses σ of the plasma-developed siloxane resist takes significantly lower values than the σ of the wet-developed EPR surfaces. The fractal dimension D at low doses, increases quickly and then displays a broad maximum before starting slightly to decrease at high doses. Generally, we observe for a second time that σ and D_F behave in an opposite way; when the first decreases the second increases.

In an effort to examine the general character of the opposite behavior of σ and D vs. exposure dose, we extend our calculations to the case of positive-tone resist surfaces. In particular, we studied a chemically amplified positive-tone acrylate-based resist (CAP) with 10% PAG concentration, e-beam

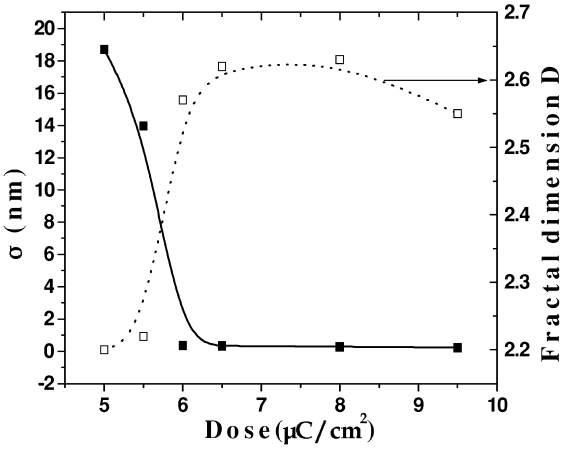

Fig. 3. Dependence of σ and D of a negative tone plasma-developed siloxane resist on exposure dose. Note: (i) at useful doses the values of σ are lower than those of wet-developed resists and (ii) the trend towards the opposite behavior of σ and D.

exposed and developed with diluted (20:1) standard tetramethyl ammonium hydroxide (TMAH) developer (the resist is an experimental one for e-beam and 193 nm lithography synthesized at the Institute of Microelectronics). Fig. 4 shows the results of our calculations. In contrast to the negative resists, here at low doses we observe an increase in σ and then a decrease as dose increases. Once again, the opposite behavior between σ and D is present. Therefore, it seems that this finding has a general character for surfaces fabricated by lithography process and examined vs. exposure dose. Let us notice that surfaces fabricated by other processes (for example vapor deposition or ion bombardment) do not show such opposite behavior of σ and D (see for example [19]). Thus, we conclude that this particular behavior may provide a piece of useful information about the mechanisms

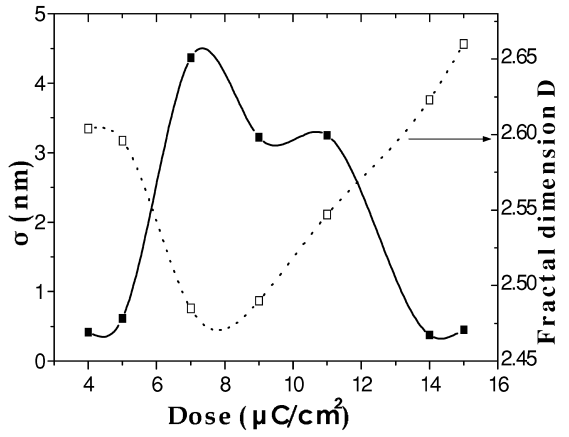

Fig. 4. Dependence of σ and D on exposure dose for a positive tone wet-developed resist with 10% PAG. Note: (i) the increasing behavior of σ at low doses not observed at negative resists and (ii) once again the trend towards the opposite behavior of σ and D.

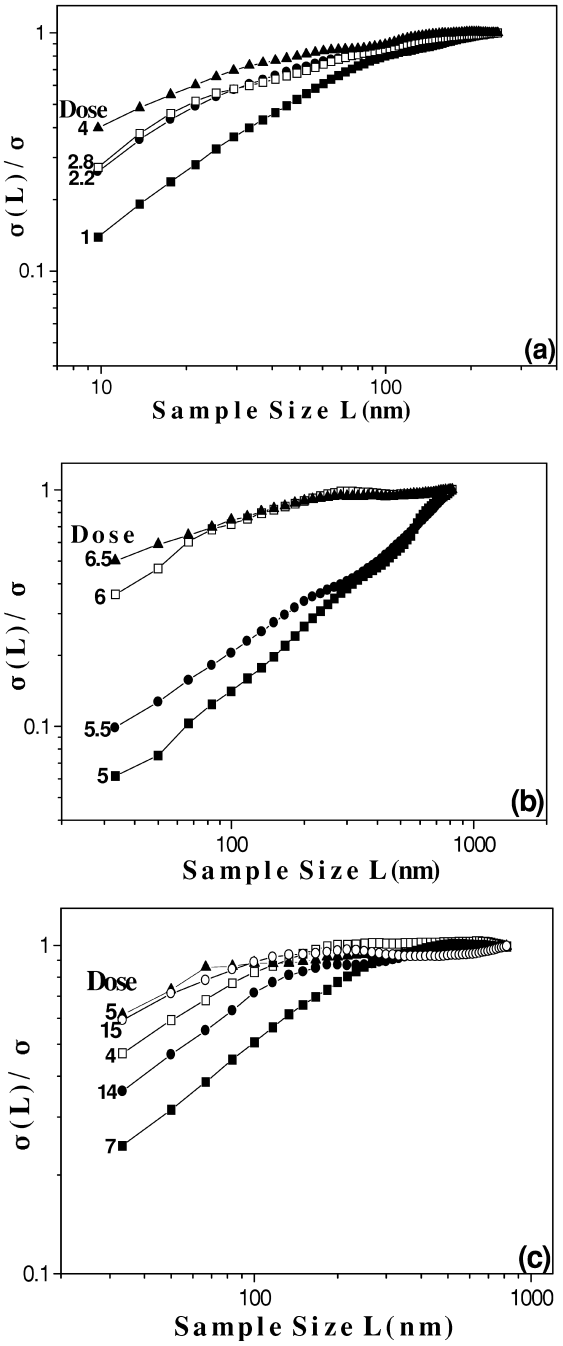

Fig. 5. Dependence of the average rms value $\sigma(L)$ on the sample size L (scaling behavior) for different exposure doses and for the negative EPR (a), the negative siloxane resist (b) and the positive CAP resist (c). The $\sigma(L)$ values of each curve are normalized to the saturated value σ of the corresponding dose. Note: (i) the dependence of the correlation length on the exposure dose and (ii) the power law behavior exhibited more clearly at doses that correspond to low values of D.

of the lithography processes which are largely responsible for the final morphology of surfaces and so the formation of their roughness.

Finally, the scaling behavior of σ (the dependence on sample size L) for the aforementioned resist surfaces is shown in Fig. 5a–c. Actually, the shown $\sigma(L)/\sigma$ curves come from an average of 16 samples (EPR surfaces) or nine samples (siloxane and CAP surfaces). A power law behavior can be observed in most cases. However, for surfaces with high D values, the power law is not very clear, indicating probably the need for better statistics. For the negative tone resists (Fig. 5a,b), as dose increases the convergence to the final value σ becomes faster and so the correlation length L_{cor} smaller. The value of L_{cor} for EPR lies between ~50 and 150 nm, whereas for siloxanes it is much larger (~200–800 nm). This difference may be due to the much smaller MW of EPR. For the positive tone resists, large L_{cor} are associated to high σ and low D values (Fig. 5c). The range of L_{cor} is ~60–300 nm. In all cases, an approximate inverse behavior of D and L_{cor} can be deduced, but the more detailed study of this behavior will be the subject of future work.

4. Summary and conclusions

In this paper, we presented and compared different roughness parameters (σ, D, L_{cor} and Fourier analysis). Then some of them were estimated for surfaces of positive and negative tone resists and experimental interrelations were deduced.

More specifically, according to theory the scaling behavior of σ is linked to D and saturates to its final value when $L \approx L_{cor}$ (Eq. (1b)). Furthermore, the Fourier spectrum is related to σ through Parceval's theorem (Eq. (2)), whereas its high frequency behavior is determined by the value of D. By analyzing the experimental surfaces, first we revealed a general trend towards an opposite behavior of σ and D when they are examined vs. exposure dose. Second, the existence of a power law behavior in the scaling analysis of σ was shown with some problems for surfaces with large D values. Finally, the dependence of L_{cor} on the exposure dose was clarified and an inverse relation between D and L_{cor} was hinted.

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