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Characterization of Microroughness Parameters in Cu and Cu_2O Nanoparticles Embedded in Carbon Film

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***Abstract*—**The morphological parameter of a thin film surface can be characterized by power spectral density (PSD) functions which provides a better description to the topography than the RMS roughness and imparts several useful information of the surface including fractal and superstructure contributions. Through the present study Nanoparticle copper/carbon composite films were prepared by co-deposition of RF-Sputtering and RF-PECVD method from acetylene gas and copper target. Surface morphology of thin films is characterized by using atomic force microscopy (AFM). The Carbon content of our films was obtained by Rutherford Back Scattering (RBS) and it varied from .4% to 78%. The power values of power spectral density (PSD) for the AFM data were determined by the fast Fourier transform (FFT) algorithms. We investigate the effect of carbon on the roughness of thin films surface. Using such information, roughness contributions of the surface have been successfully extracted.

***Keywords*—**Atomic force microscopy, Fast Fourier transform, Power spectral density, RBS.

I. Introduction

THE properties of the tin films are usually substantially influenced by their microstructure and surface morphology[1]. It has also been recognized that the nature and structure of the substrate has a strong influence on the structure and morphology of the thin films [2]-[4]. In general, the micro roughness characterization of a thin film surface can be carried out using optical or mechanical profiler or an atomic force microscope (AFM). However, AFM can be advantageously used with high vertical and spatial resolution to extract micro roughness information of the surfaces [5]. Such roughness information obtained from AFM can be advantageously employed to the successful development of low scatter optical multilayer coatings. In addition, such information about the surface roughness is extremely useful for the alternate optical characterization of thin films using spectrophotometer. Three types of roughness estimation are often used - root mean square (rms) roughness, average roughness and peak to valley roughness. But the direct measurements of all of these yield only the height information of the surface morphology and not the total characterization of surface. Another way of describing the surface morphology is its spectroscopic nature, i.e., frequency distribution (power spectral density or PSD technique). However, the underlying assumptions of Fourier transform (FT) which is the background of PSD method make its use rather limited. In this work analysis of the atomic force microscopy (AFM) images yields thin film roughness values and power spectral density (PSD) plots which provides valuable information on spatial frequencies of height distribution. From these data, correlations between film morphology, thickness, and specific deposition conditions are established. Nanoparticle copper/carbon composite films were prepared by co-deposition of RF-Sputtering and RF-PECVD method from acetylene gas and copper target. The Copper content of our films was obtained by Rutherford Back Scattering (RBS) and it varied from .4% to 78%.The power spectral density (PSD) functions of all the surface profiles of each film have been computed and combined in to a single PSD profile covering large spatial frequency bandwidth. These experimentally derived PSDs have been fitted with appropriate analytical models. From this fitting, the surface characteristic parameters relating to fractal properties (substrate dominated), roughness information of the effect of carbon during the sputtering copper Nanoparticles have been obtained for all the films. Such information has helped to understand the influence of deposition conditions on micro roughness introduced by the PSD components and pure film. In addition, one can advantageously use such information in tailoring a surface morphology according to the requirements by choosing appropriate deposition conditions. The details of the surface topographic study are presented in the subsequent sections.

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II. Experimental Details

Nanoparticle copper/carbon composite films were prepared by a capacitance coupled RF system with 13.56 MHz power supply. The reactor consists of two electrodes with different area size. The smaller electrode was a copper disc with 7.5 cm diameter as a powered electrode. The other electrode with 13 cm diameter was grounded via the body of the stainless steel chamber. Deposition was performed on glass and silicon substrates on the grounded electrode at room temperature. The distance between electrodes was 5 cm. The chamber was evacuated to a base pressure of about 10^{-3} Pa prior to the deposition and then the pressure rises to ambient pressure using only acetylene gas. The pressure measurement during deposition was done by a pirani gauge that its head was outside plasma environment. Deposition was done in constant RF power regime and different initial pressure. The RF power

was 200 W and the initial gas pressure was set at different pressure from 1.3 Pa to 6.6 Pa. We selected this zone because below 1.3 Pa mainly copper was sputtered and above 6.6 Pa only deposition of amorphous carbon occurs. The films that were grown with initial pressure from 1.3 to 6.6 Pa contain both carbon and copper. The copper and carbon atomic content in the bulk and the surface of the films were obtained from RBS analysis. We obtained RBS spectra of the samples by protons with energy 1.4–1.8 MeV. The resonance scattering by carbon nuclei is in this range. Considering atomic sensitivities for C and Cu, the percentage of copper and carbon of our films was obtained. AFM on non-contact mode was used to obtain the surface topography of the films and copper grains [12].

III. Results and Discussions

A. AFM analysis

Treatment of the AFM data allows quantitative information to be extracted on surface roughness. A systematic description of various analytical methods used for roughness characterization can be found in Ref [6]. Root-mean square roughness (R_{RMS}) which is defined as the standard deviation of the surface height profile from the mean height, is the most commonly reported measurement of surface roughness, and is given by:

$$R_{RMS} = \left[\frac{1}{N}\sum_{i=1}^{n}(h_i - < h >)^2\right]^{\frac{1}{2}} \quad (1)$$

Where N is the number of pixels in the image (or data points), h_i is the height of the i^{th} pixel, and h_i is the mean height of the image. Although the R_{RMS} evaluations are useful, they provide information only on the height variation, i.e., in the direction perpendicular to the substrate. Possible correlation between length scale of surface features and the roughness cannot be characterized using these values. For example, two images with exactly the same R_{RMS} values may have different surface morphologies. One way to examine more closely how the roughness varies with length scale is through power spectral density (PSD) analysis. Fast Fourier transform(FFT) can be used to obtain frequency distribution of the profile over the entire frequency range. The resulting function is the PSD function and is defined as the square modulus of FT. This is accomplished by calculating the square magnitude of the coefficients of the fast Fourier transform $|F(x,y)|^2$ of the digitized surface profile z(x,y). For a two dimensional (2D) isotropic surface profile, the PSD is obtained by performing 2D angular averaging of the $|F(x,y)|^2$ using the radius in the reciprocal space as the spatial frequency. The calculation used in this work normalizes the PSD with the number of pixels of every frequency resulting in units of length^4. The power spectral density is then plotted as a function of spatial frequency or wavelength and therefore provides valuable information. Hence, PSD explains a surface much better than the RMS roughness and provides very useful information on fractals and superstructures that may coexist in the microstructures [1]. It has been realized recently that the fractal geometry and scaling concepts can concisely describe the rough surface morphology.

B. Thin films and power spectral density functions

There have been large numbers of publications dealing with PSD calculations from the surface profile data. The computation of PSD function adopted in this paper is given by [7].

$$S_2(f_x, f_y) = \frac{1}{L^2}\left[\sum_{m=1}^{N}\sum_{n=1}^{N} Z_{mn} e^{-e\pi i\Delta L(f_x m + f_y n)}(\Delta L)^2\right]^2 \quad (2)$$

Where S_2 denotes the two-dimensional PSD, L^2 is the scanned surface area, N is the number of data points per line and row, Zmn is the profile height at position (m, n), f_x, f_y are the spatial frequency in the x- and y directions and $\Delta L = N/L$ is the sampling distance. PSD dependant on the spatial frequency f yields:

$$PSD(f) = \frac{K}{f^{-V}} \quad (3)$$

Where K has spatial length to the power and γ is the power of PSD. The γ is calculated as the inverse slope in the log-log plot of the high spatial frequency and the PSD [8].In our study, the length scale (L) considered is 3.0 μm, which gives a sampling rate of 3 μm/181 ~16.6nm. This corresponds to the spatial minimum frequency fmin = 1/3 μm = 0.33 μm^{-1}. The maximum frequency is limited by the sampling theorem of Nyquist frequency, fmax = 1/16.6 nm = 62.24 μm^{-1}/2 = 30.12 μm^{-1}. These form the upper and lower-bandwidth limitation of the PSD plots [8].

The fractal dimension of a set of two dimensional (2D) images can be calculated from the slope of a log S(k)–log k plot. The relationship between the power spectral density S (k) and the frequency k is given as [15]:

$S(k) \propto k^{-\beta}$

The fractal dimension D is related to the slope β of log– log plot

$D = 4 + \beta/2$

C. Surface morphology

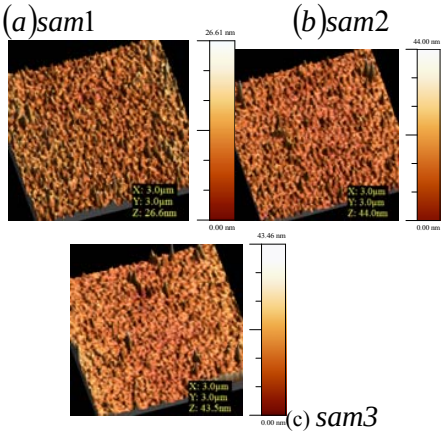

Fig. 1 AFM images of thin films with different percentage of carbon,

(a) 0.73 (b)0.54, (c) 0.555 and (d)0.05.Each images size is 3.0 μm x 3.0 μm

The AFM images showing the surface morphologies of the films are shown in Fig. 1. All of the AFM image sizes are of 3.0 μm x 3.0 μm areas

TABLE I
RMS ROUGHNESS FOR EVERY SAMPLE

Sample	RMS ROUGHNESS	Figure
Cu1	2.9775	1
Cu2	2.4183	2
Cu3	2.3556	3

We used AFM images to find the PSD of the films. Fig.1.

AFM image analysis was performed to obtain the root mean- square roughness. TABLE I is summary of the rms roughness for every sample. The rms roughness of the films was not significantly different (2-3nm). Sample 1 has the greatest rms roughness.

The AFM images of the thin films grown to different thickness (deposition time) are shown in Fig. 1. The evolution of surface features with deposition time can be clearly seen in these photographs. Table 2 shows the thickness of the layers (1E 15Atom/Cm^2) and the percentage of each element in first layer and for every sample.

TABLE I I
THICKNESS OF THE LAYERS (1E 15ATOM/CM^2) AND THE PERCENTAGE OF EACH ELEMENT IN FIRST LAYER AND FOR EVERY SAMPLE

sample	THICKNESS OF THE FIRST LAYER (1E15ATOM/CM^2)	Cu	C	O
Cu1	1300	0.046	0 .73	0 .21
Cu2	2350	0.41	0.54	0.05
Cu3	1000	0.78	0 .05	0.17

C. PDS of the films

The PSD plots of the samples are shown in Fig. 2. Each PSD plots was calculated using the FFT algorithm for 3.0 μm x 3.0 μm AFM image data. All of the PSD plots exhibited typical features consisting of a plateau with the low spatial frequency and an inverse slope with high spatial frequency. The PSD plots feature of samples was characterized using the k-correlation model [10]. The k-correlation model for the auto-covariance function PSD_{ABC} for spatial frequency f is given by:

$$PSD_{ABC} = \frac{A}{\left(1 + B^2 f^2\right)^{\frac{(C+2)}{2}}} \quad (4)$$

With A, B, C being the function parameters, Here A is the magnitude at low spatial frequency, which is related to the height of the rough surface [11]. B determines the position of the 'knee', which is defined as the slope of a line connecting two points on the surface. C is the slope at high special frequency, which gives the nature of the roughness: it is a constant greater than 2. The PSD curves by the k-correlation are presented corresponding to the PSD plots as shown in Fig. 2.

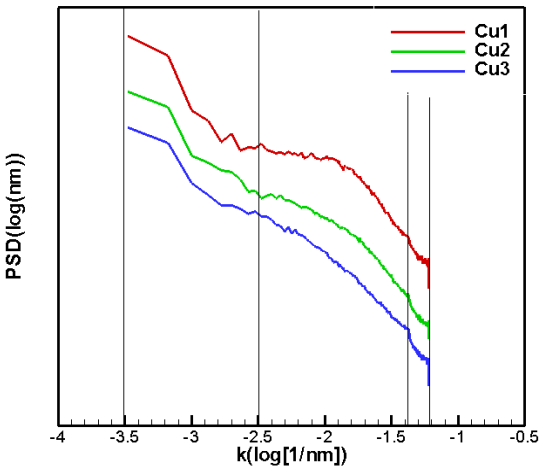

Fig. 2 The PSD plots of the samples

In comparison between sample 1 and sample 3, as it can be seen in table 2, the amount of C in sample 1 is more and as it is shown in figure 2 the PSD of sample 3 is more than sample 1 in lower frequencies and the PSD is more in sample 1 for upper frequencies. The psd in sample 2 is more than sam 1.and as it is shown in table 2, has the most amount of carbon. Sample 2 has a lower PSD in compare with sample 2.

D.Fractal dimensions

The fractal dimension (D_f) of the thin films was evaluated to characterize the surface morphology. The fractal dimensions of thin films were shown in Table 3. The dimension value determines the relative amounts of the surface irregularities at different distance scales. The fractal dimension of the surface can be obtained from the parameter C of the k-correlation [9].

TABLE I I I
FRACTALS FOR EVERY SAMPLE

Sample	Fractal dimensions(D_f)
Cu1	1.488
Cu2	1.443
Cu3	1.405

$$D_f = \frac{(7-c)}{2} \quad (5)$$

Since the fractal dimension of the thin film was around 1.5, the data of the fractals are the same as PSD analysis and they increase by the percentage of carbon.

IV. Conclusion

We have used the RMS roughness, fractal and PSD spectra derived from AFM measurements in order to obtain quantitative and qualitative morphology of tree samples. RBS analysis shows the percentage of elements in our samples. The sample 6 has shown a higher value of roughness (2.97) as compared to sample 8 and 7 (~2.3). The PSD study have yielded several interesting information about fractal nature and intrinsic roughness. The effect of carbon and copper on the roughness of the films was characterized by the PSD of fast Fourier transform algorithm. As it was shown the PSD and fractal of the surface are increasing by carbon as it is shown in figure 2 and table 3and the same result is obtained from rms roughness analysis in table 1 We found distinctly three inverse power values, which are divided by two 'knee' spatial frequencies of -2.5 and -1.5 psd is matched well with fractal and rms roughness results in -2.5 to-1.5 Region.

These data can satisfy the variations which can describe magnetic properties.they show useful information about morphology of the sureface which ended to so many other properties such as electrical and optical properties.

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