Surface micromorphology analysis of Cu/Ni nanocomposite thin films by power spectra density and fractal geometry

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Copper (Cu) and nickel (Ni) nanoparticles have been grown simultaneously on glass and silicon substrates by RF sputtering method to form three Cu/Ni nanocomposites at different deposition times. The existence of Cu and Ni peaks in the X-ray diffraction (XRD) profiles confirms the crystalline structure of samples with Cu and Ni atomic content which have also been characterized by Rutherford backscattering (RBS) method. Moreover, the structural and morphological properties of the prepared nanocomposites have been compared with respect to their morphologies by means of atomic force microscopy (AFM) analysis. In order to compare the surface roughness over different spatial frequency ranges and evaluate surface quality, power spectral density (PSD) of each sample has been extracted from AFM data and also, the experimental and theoretical results have been compared. The fractal nature of these nanocomposites has been finally discussed.

Keywords: *Cu/Ni nanocomposite; power spectral density; crystalline structure; surface micromorphology; atomic force microscopy*

1. Introduction

Spherical particles with 1 nm to 100 nm diameter [1–4] and high surface to volume ratio are known as nanoparticles (NPs) [5–8]. The efforts to create new nanoparticles and nanocomposites with novel physical properties for various applications, such as sensors, nanomedicine, and catalysts are still continued. The behavior of metallic nanocomposites deposited on substrates, such as silicon and glass, has been studied. Among metallic NPs, Cu and Ni are good candidates for economic reasons compared to other metals like silver and gold [9–13]. However, one of the most significant problems of catalysts is their short lifetime which has been solved by fabrication of nanocomposites in the way that each metal keeps their activity. Nanocomposites are a mixture of two or more materials with separate phases with at least one nanoparticle phase. Moreover, multiple metal composition shows different magnetic and chemical properties which make them more applicable

due to their unique role in sensors, water splitting, and electrical devices [14–17]. In the present study, Cu/Ni two-phase nanocomposites have been considered. The FCC structure of both Cu and Ni with only 2.5 % lattice mismatch, along with the solid solution form of Cu/Ni, is the main reason for making Cu/Ni nanocomposites [18–20].

In the wide range of applications, surface morphology affects the functionality of materials. Compared to RMS roughness, which is a well-known parameter for surface morphology analysis, PSD is the more accurate method for characterization of surface morphology and lateral distribution, which performs decomposition of the surface profile into its spatial wavelengths. Moreover, PSD provides useful information about surface quality and microstructure [8, 21–26].

Unique properties of Cu/Ni nanocomposites are governed by surface effects and surface topography. It is worth to mention that the surface of nanocomposites is not random, so fractal approaches are appropriate for morphological characterization. In order to complement the recent works, Cu/Ni nanocomposites were prepared

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Table 1. Deposition conditions of Cu/Ni samples

Sample No.	Target	Initial pressure [Pa]	Deposition time [s]	RF power [W]
#1	Cu/Ni	2.4	20	200
#2	Cu/Ni	2.4	25	200
#3	Cu/Ni	2.4	30	200

by radio-frequency (RF) sputtering method at different deposition times. Rutherford backscattering (RBS), X-ray diffraction (XRD), and atomic force microscopy (AFM) were used for studying their structural and morphological properties.

Fig. 1. Schematic of experimental set up.

2. Experimental

Copper and nickel were sputtered using RF sputtering system with 13.56 MHz power supply to form Cu/Ni nanocomposites. Two electrodes with different areas were fixed in a reactor in which the smaller one was a copper/nickel composite disc with 9 cm diameter as a powered electrode. Deposition process was performed at room temperature on glass and silicon substrates as the grounded electrode. The chamber was evacuated to a base pressure of about 2.4 Pa. The deposition process was performed at a constant power value of 200 W at different times from 20 min to 30 min. RBS analysis was carried out by protons with energy of 1.4 MeV to 1.8 MeV to investigate Cu and Ni atomic content. XRD patterns were investigated to

reveal the crystalline structure of the films. DMC DS 95 series AFM in non-contact mode was used to evaluate characteristics and topography of the films. The schematic of deposition process is presented in Fig. 1 and the deposition conditions of three specimens fabricated at various deposition times are summarized in Table 1.

3. Results and discussion

Fig. 2. RBS spectra of typical sample #2, experimental (red line) and simulation (green line).

The content of 49 % Cu and 40 % Ni in the typical sample #2 was extracted from the RBS spectra shown in Fig. 2. The experimental and simulated results have been marked with thin red and thick green lines. Si and O peaks at 100 keV and 600 keV are related to the substrate, while the peaks between 1400 keV and 1500 keV come from Cu and Ni. SIMNRA software was applied for RBS simulation. Cu content was established as 42 %, 49 % and 55 % and the thickness was about 130 nm, 145 nm

and 153 nm, for the samples #1, #2, and #3, respectively. The thickness of Ni layers was 80 nm, 92 nm and 98 nm for the same samples, respectively.

Fig. 3. X-ray diffraction profile of Cu/Ni nanocomposite of sample #2.

Crystallized Cu and Ni NPs in the typical sample #2 can be seen in the XRD profiles in Fig. 3. The sharp peaks at 43.1° and 74.6° are related to Cu (1 1 1) and Cu (2 0 0), while the peaks at 44.2° and 50.4° are related to Ni (1 1 1) and Ni (2 0 0), respectively. All mentioned peaks show good crystallinity of prepared nanocomposites. The Si peak at 58.9° is related to the substrate.

In Fig. 4, fine-grained structures of all samples are presented in $1.0 \mu\text{m} \times 1.0 \mu\text{m}$ AFM images which were used for surface analysis.

PSD plots of the samples in Fig. 5 were extracted from AFM images by FFT algorithm. The plateau in low-spatial frequency region has an inverse slope compared to high spatial frequency and can be characterized by k-correlation model [27] as follows:

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

in which PSD_{ABC} is the auto-covariance function, and f is the spatial frequency. Here, A is the height of the rough surface at low spatial frequency [28], B is the slope of a line which connects two points

Sample #1

Sample #2

Sample #3

Fig. 4. $3.0 \mu\text{m} \times 3.0 \mu\text{m}$ 3D AFM images of all samples.

of the surface, and C gives information about nature of the roughness at high spatial frequency. In Fig. 5, the assigned region (a) is the high frequency region in which the behaviors of the curves are not the same. A direct relation exists between PSD and

Fig. 5. PSD plots of all samples extracted from AFM images by FFT algorithm.

Fig. 6. Typical power spectral density versus frequency (K) showing different slope values in different parts of sample #2.

deposition time for all samples which is inverse at -1.4 to -1.2 . These changes in region (a) confirm the complexity of the film surface with fine features [29] which make it a suitable region for surface investigation.

Fractal dimension (D_f) is another important parameter which gives valuable information about surface irregularities through the following equation:

$$D_f = 4 + \frac{\beta}{2} \quad (2)$$

which increases with the slope decrease in region (a) [30]. The results concerning the fractal dimension for each sample are summarized in Table 2.

The slope variations in Fig. 6, denoted by lines 1, 2, and 3, indicate fractal dimension of sample #2 which is best fitted in line 3. The relationship between the slopes and fractal dimensions for different frequency regions are also summarized in Table 3.

In addition, the direct relationship between increase in deposition time and the fractal dimension is shown in Fig. 7.

Fig. 7. Dependence of fractal dimension on deposition time for all specimens.

4. Conclusion

The main purpose of this work was comparing the analyzed PSD spectra obtained from AFM data. This procedure was applied for studying the morphology of three Cu/Ni nanocomposites which were synthesized by RF sputtering method at different deposition times. The results demonstrated that PSD provides the most comprehensive

Table 2. The slope and fractal dimension (D_f).

Sample No.	Slope	Fractal dimensions (D_f)
#1	-3.90	2.05
#2	-3.65	2.17
#3	-3.23	2.39

Table 3. The relation between slopes and fractal dimensions for different frequency regions.

Frequency region	Slope	Fractal dimension
high	-5.90	1.05
mid	-5.05	1.48
low	-3.32	2.34

understanding of the influence of deposition time on morphological features and surface morphology of the films. Moreover, the fractal approach showed the complexity and irregularity of Cu/Ni thin films deposited at different deposition times from 20 min to 30 min, revealing that fractal dimension increased with increasing deposition time.

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