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Investigation of the topography of magnetron-deposited Cu/Ni multilayers by X-ray reflectometry and atomic force microscopy

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This paper presents the results of research of Cu/Ni multilayers magnetron-deposited on an Si (100) substrate. The thickness of Cu sublayers was identical in all multilayers and equalled 2 nm. The thickness of Ni sublayers varied between 1.2 and 3.0 nm. The surface topography of the multilayers was examined using the AFM (research of different areas). The interface roughness and period thickness were characterized by X-ray reflectometry (XRR). The roughness of the multilayers achieved by the XRR method was very similar for all 0.6 nm samples examined. However, the values of the roughness parameter R_a obtained by AFM examination were in the range of 0.09–0.32 nm, and depended on the size of the area examined. Based on the function obtained from the AFM measurements, taken on surface areas varying in size, an area of 1.5–4.0 cm^2 was determined, for which an agreement between the roughness parameter, as determined by the AFM method, and the XRR method results would be expected.

Keywords: multilayers Cu/Ni, X-ray reflectometry (XRR), atomic force microscopy (AFM), roughness.

1. Introduction

Multilayers built of two different types of magnetron-deposited layers have become the focus of scientific research owing to their potential usefulness for modern nano-equipment [1]. Moreover, it has been demonstrated that multilayers consisting of nanometre-order layers exhibit better mechanical properties, which makes them candidates for wear-resistant applications [2].

Cu/Ni multilayers are technologically interesting because of the relative ease of making layers characterized by strong adhesion to the substrate. This is due to the fact that both Cu and Ni have an *fcc* structure, and their lattice misfit is a small as 2.5%.

In addition, the Cu-Ni solution is a solid solution within the entire concentration range [3].

A considerable development of non-destructive techniques used for testing nanomaterials has been observed in recent years. For multilayers, the most important non-destructive testing methods are: atomic force microscopy (AFM) and X-ray reflectometry (XRR). The low-angle X-ray diffraction is the most advantageous for specimens whose layer thickness is less than approx. 100 nm. The image of low-angle examination depends strongly on the surface topography and the interfacial area [4–6]. This method serves also for determining the thickness of layers (the measuring uncertainty of this method is relatively small, being approx. 1%). The non-destructive determination of layers on the nanoscale is becoming increasingly important, *e.g.*, for quality control in the semiconductor industry [7, 8].

2. Material and methodology

The material for the investigation was 6 multilayers, each consisting of 100 Cu/Ni bilayers. The multilayers were made by the method of magnetron sputtering onto an Si (100) substrate at the Institute of Molecular Physics of the Polish Academy of Sciences (PAN) in Poznań. The thickness of Cu sublayers was identical in all multilayers and equalled 2 nm. The thickness of Ni sublayers varied between 1.2 and 3.0 nm (Fig. 1).

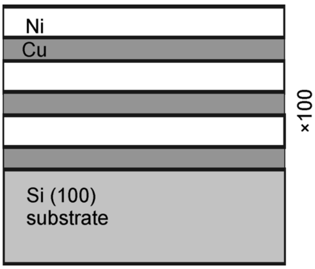

Fig. 1. A schematic diagram of the arrangement of sublayers in the multilayers investigated.

The surface topography of the multilayers was examined by AFM, with the tapping method, using a Veeco device. The coat surfaces were scanned within the area of 25 μm^2. The estimation of the surface roughness parameters, R_a (mean arithmetical profile deviation (1)) and R_z (mean roughness height (2)), was done using the Nanoscope software, and

$$R_a = \frac{1}{l}\int_0^l |y|\,\mathrm{d}x \approx \frac{1}{n}\sum_{i=1}^{n} y_i \tag{1}$$

$$R_z = \frac{1}{5}\left(\sum_{i=1}^{5} h_{\max i} - \sum_{i=1}^{5} h_{\min i}\right) \tag{2}$$

where: l is the elementary segment, y is the profile height: h_{max} – maximum height, h_{min} – minimum height; n is the number of measurement points.

The roughness of the coats was also estimated by XRR. In this method, the roughness σ_{XRR} can be determined from the equation for the reflectivity:

$$R = R_0 \exp\left(-\sigma_{XRR}^2 q^2\right) \tag{3}$$

where: R_0 is the ideal surface reflectivity, q is the scattering vector component normal to the surface.

The measurements were made on an X'PertMPD X-ray unit using a radiation wavelength of λ_{Cu} = 0.1542 nm in parallel beam optics. The divergence of the primary beam was 1/32°, and the distance of the divergence slit from the goniometer axis was 230 mm. The reflectometric curves were plotted with the WinGixa program.

3. Results

Figure 3 shows the topography of multilayers, as imaged by the AFM technique. In spite of all specimen surfaces having been washed with acetone, the surfaces still featured single convex artefacts markedly protruding above the average surface level. Experience gained from studies on other materials made by the magnetron sputtering method show that artefacts protruding above the coating surface are often met. They are made up of particles that have stuck together as a result of mutual collisions in the magnetron chamber, or are the result of non-uniform sputtering of the target. The largest artefacts occurred on the surface of the multilayer Cu/Ni = 2/2.5 nm. On this multilayer, as well as on the multilayer Cu/Ni = 2/1.2 nm, no region of surface area of 25 μm^2 was found which would have been free from artefacts, though on the latter of the multilayers they were much finer (Fig. 3).

The least roughness was shown by Cu/Ni = 2/1.4 nm (R_a = 0.09 nm) and Cu/Ni = = 2/1.5 nm (R_a = 0.11 nm) multilayers. The roughness values of multilayers with numerous artefacts were higher by 2–3 times. The parameter R_z for the smooth surfaces was greater than the parameter R_a by approx. 6 times, while for multilayers with the highest roughness it was greater by approx. 100 times. A comparison of the multilayer surface AFM images in Fig. 3 indicates the lack of any definite correlation between the Ni sublayer surface thickness and surface topography. There

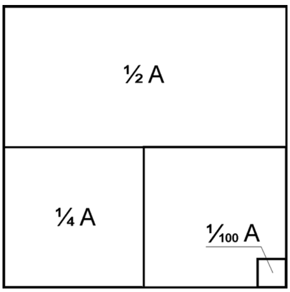

Fig. 2. The proportions of multilayer surfaces examined by the AFM technique. A – 25 μm^2 (5 μm×5 μm), $\frac{1}{2}A$ – 12.5 μm^2 (5 μm×2.5 μm), $\frac{1}{4}A$ – 6.25 μm^2 (2.5 μm×2.5 μm), $\frac{1}{100}A$ – 0.25 μm^2 (0.5 μm×0.5 μm).

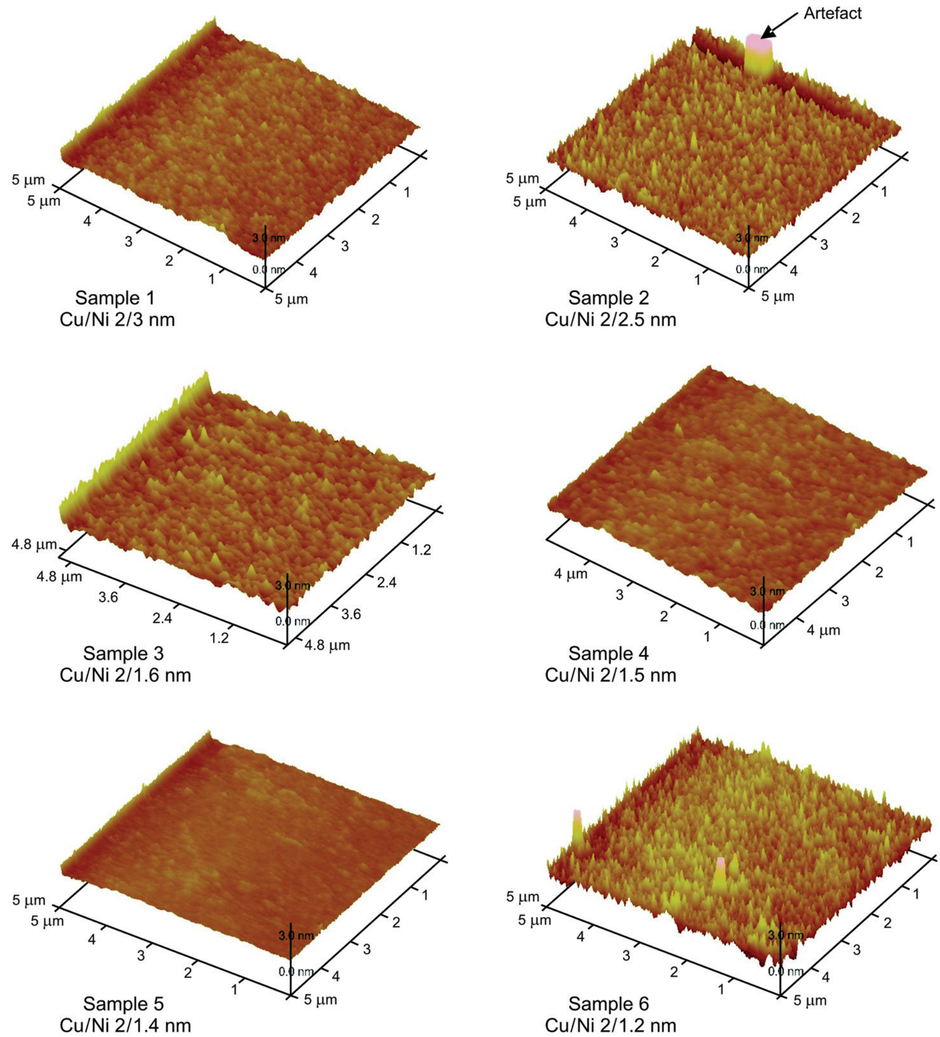

Fig. 3. The surface topography of Cu/Ni multilayers in AFM examinations. The highest lines at the top-left side of images of samples 1, 3 and 5, being an effect of the measuring method, were skipped in roughness calculation.

is a correlation between surface roughness and the Ni sublayer thickness in the range of 1.4–2.5 nm – with increasing sublayer thickness the multilayer surface roughness increases. However, the multilayers Cu/Ni = 2/3.0 nm and Cu/Ni = 2/1.2 nm deviate from this rule, exhibiting the opposite relationship.

Bearing in mind the statistic character of roughness measurement using the AFM technique, surfaces varying in area from 0.25 do 25 µm^2 were put through an analyis (Fig. 2). The calculation results are given in the Table.

T a b l e. The period thickness and surface roughness calculated by XRR and AFM technique.

Multilayer	X-ray reflectometry				Atomic force microscopy	
	Thickness [nm]			Roughness [nm]	R_a [nm]	R_z [nm]
	Cu	Ni	Period		A (25 μm²)	
Sample 1 Cu/Ni = 2/3 nm	2.0	2.60	4.60	**0.6**	**0.14**	5.97
Sample 2 Cu/Ni = 2/2.5 nm	2.0	2.30	4.30	**0.6**	**0.32**	44.90
Sample 3 Cu/Ni = 2/1.6 nm	2.0	1.85	3.85	**0.5**	**0.18**	9.71
Sample 4 Cu/Ni = 2/1.5 nm	2.0	1.95	3.95	**0.6**	**0.11**	6.57
Sample 5 Cu/Ni = 2/1.4 nm	2.0	1.80	3.80	**0.6**	**0.09**	23.7
Sample 6 Cu/Ni = 2/1.2 nm	2.0	1.55	3.55	**0.6**	**0.28**	23.40

A comparison of the roughness values calculated from surfaces of different size shows that the larger the region analyzed, the higher the estimated R_a values (Fig. 4). The increasing trend in the roughness magnitude with increasing surface area for two samples (with the thickest and thinnest Cu layer, respectively) is described with the logarithmic function (Fig. 5).

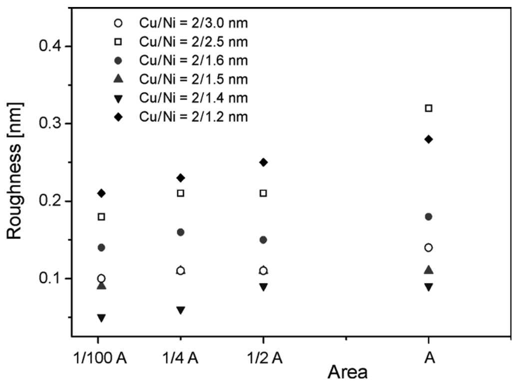

Fig. 4. The surface roughness of Cu/Ni multilayers, as determined by the AFM technique on surfaces of different area (A = 25 μm^2).

The results of reflectometric measurement and their matching are shown in Fig. 6. Two fringes were recorded on the reflectometric curves at a distance of $\Delta(2\theta) \sim 1.4°$. With decreasing Ni sublayer thickness, the intensity of the peak pair increased, with the position of the peaks shifting towards larger angles. The period thickness and

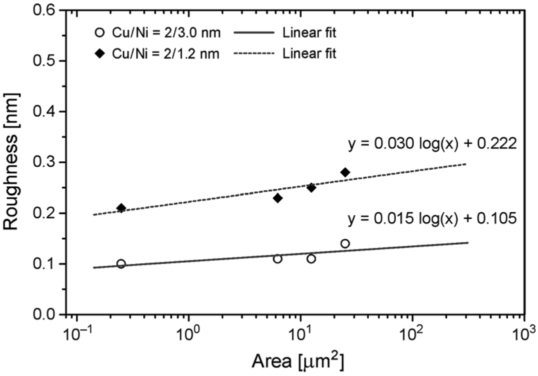

Fig. 5. Matching of the logarithmic function to the roughness measurement results obtained by the AFM method.

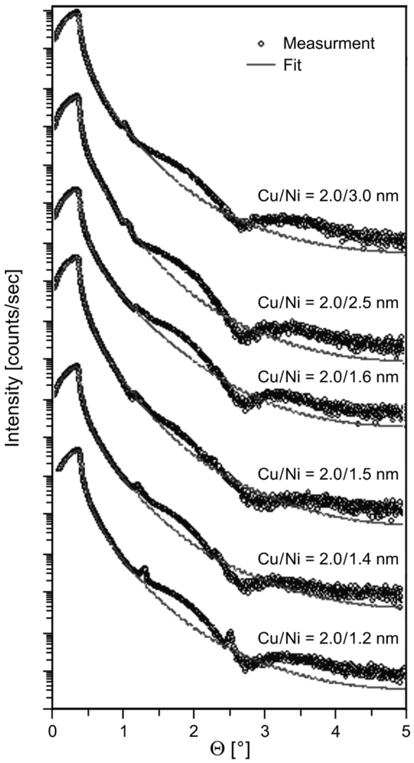

Fig. 6. Reflectivity from multilayers Cu/Ni.

surface roughness calculated on their basis are given in the Table. The roughness value obtained from X-ray measurements are similar for all the samples, amounting to 0.6 nm. Only for the Cu/Ni = 2/1.6 nm is the estimated roughness slightly lower, being 0.5 nm. The roughness values determined by reflectometry measurements are considerably greater than those obtained from AFM examinations. The difference results from the size of the area analyzed with both examination techniques. The maximum area analyzed with AFM was 25 μm^2, whereas the reflectometry measurement recorded reflections from surfaces of sizes ranging from several to several dozen square millimetres. Figures 4 and 5 show an increasing trend in the estimated roughness value with increasing area of the surface analyzed by the AFM method; thus, the reflectometry measurement results fit in with this relationship. Assuming the function formulae generated in Fig. 5 to be representative of the entire surface of both multilayers, it can be calculated that a roughness value identical to the one obtained from the XRR measurements would be reached by analyzing areas of $10^{12.6}$ μm^2 (~1.5 cm^2) and 10^{33} μm^2 (~4 cm^2) for the higher- and lower-roughness multilayer, respectively.

4. Conclusions

The AFM examination showed a correlation between surface roughness and the thickness of Cu/Ni multilayers, where the thickness of the Ni sublayer was contained in the range of 1.4–2.5 nm. More specifically, with increasing thickness of the Ni sublayer, the surface roughness of the Cu/Ni multilayer increased. For multilayers with extreme Ni sublayer thickness values of 1.2 and 3.0 nm, the opposite relationship was found.

No agreement between the roughness values obtained by the AFM and XRR techniques was obtained. The roughness values from AFM measurements were lower than those obtained from XRR measurements. The roughness values estimated by the XRR technique were similar for all multilayers, being in the range of 0.5–0.6 nm. Based on the function determined from the AFM measurements, taken on surface areas varying in size, an area of 1.5–4.0 cm^2 was determined, for which an agreement between the roughness parameter, as determined by the AFM method, and the XRR method results would be expected.

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