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THE COMPARISON OF THE STRUCTURAL PROPERTIES OF MULTILAYERED SYSTEMS Cu/Ni OBTAINED BY THE ELEKTROCHEMICAL AND FACE-TO-FACE SPUTTERING METHODS

PORÓWNANIE WŁAŚCIWOŚCI STRUKTURALNYCH UKŁADÓW WIELOWARSTWOWYCH Cu/Ni UZYSKANYCH METODĄ ELEKTROCHEMICZNĄ I ROZPYLENIA JONOWEGO ŚCISKAJĄCEGO

The investigations of the structural properties of Cu/Ni multilayer systems for following samples: Si(100)/[Cu(20Å)/Ni(t)] × 100, Si(100)/[Ni(20 Å)/Cu(t)] × 100 were performed. The samples were produced using electrochemical and face-to-face sputtering methods. It was affirmed that multilayers obtained by face-to-face sputtering method, in comparison with samples obtained by electrochemical method, were characterized by better quality of the structure. The samples made by the face-to-face sputtering method have smaller value of the interface roughness, higher quantity and quality of satellite peaks. The half widths of peaks for both superlattices are similar. Additionally, in reflectivity measurements the presence of Bragg peaks was stated. They are used to determine the value of period of the multilayer system. These type of peaks was not present in samples made by electrochemical method.

Keywords:multilayer system, electrochemical and face-to-face sputtering methods, satellite peaks

W pracy przedstawiono wyniki badań właściwości strukturalnych układów wielowarstwowych Cu/Ni. Wielowarstwy te otrzymano dwoma metodami: elektrochemiczną oraz rozpylenia jonowego. Na podstawie przeprowadzonych badań udowodniono, że próbki uzyskane metodą rozpylenia jonowego charakteryzują się lepszą jakością struktury. Przejawia się to: w mniejszej wartości szorstkości, obecnością wierzchołków satelitarnych, zbliżoną wartością szerokości połówkowej wierzchołków supersieci.

1. Introduction

The X-ray diffraction ranks to the most important, non-invasive and often applied methods of the investigation of the structural properties of multilayers systems. The privity of parameters appointed from the analysis of X-ray diffraction spectra allows on extraction of conclusions regarding the interlayer exchange coupling in the magnetic multilayers systems.

On the basis of the position of the satellite peaks visible on the X-ray diffraction pattern we could qualify [1] value of the period superlattices (Λ)

$$\Lambda = \frac{(m-n)\cdot\lambda}{2\cdot(\sin\theta_m - \sin\theta_n)} \tag{1}$$

where: Θ_m, θ_n – angles for the positions of the peaks of the order m-th and n-th ,

λ – the length of the radiation of X-ray.

The average interlayer distance was definited through the position of zero peak, from the formula (2):

$$\overline{d} = \frac{\lambda}{2\sin\theta_0}. \tag{2}$$

The reflectivity mesaurments, beside information of relating values of thickness of period bilayers, deliver information about of the quality of the interlayer surface (roughness). Large value causes the disappearance of the antiferromagnetic coupling [2]. The value of the bilayer period λ (the sum of the thickness of the layers of the material A and B) can receive on the basis of angular Bragg peaks positions:

$$\lambda = \frac{\lambda_X}{2}\sqrt{\frac{n^2-m^2}{\sin^2\theta_m - \sin^2\theta_n}} \tag{3}$$

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where: θ_m, θ_n – the angular Bragg peaks positions of the order m-th and n-th.

The aim of this paper is to compare of the structural proprieties of Cu/Ni multilayers systems which were obtained by the electrochemical and face-to-face sputtering methods.

2. Experimental deatalis

The Cu/Ni multilayer systems were consisted of the ferromagnetic Ni layer and the nonmagnetic Cu layer. These layers were deposited on the crystalic substrate Si (111).

The following samples were studied:

- Si(100)/[Cu(20Å)/Ni(t)] × 100, where t = 10 Å; 12 Å; 14 Å; 15 Å; 16 Å; 20 Å; 25 Å; 30 Å; 40 Å; 60 Å;
- Si(100)/[Ni(20 Å)/Cu(t)] × 100 where t = 8 Å; 9 Å; 10 Å; 12 Å; 18 Å; 20 Å;

These samples were obtained by two methods: electrochemical [3, 4] and face-to-face sputtering [5].

For the stuctural characterization of the Cu/Ni multilayers the X-ray diffraction method have been used. The diffractions spectra were obtained using Cu lamp giving the wave length of the X-ray λ_{Cu} = 1,54056 Å. The reflectivity and Θ-2Θ measuremts have been performed using *Wingix* and *ProfitFile* software, respectively.

3. Reasults and discussion

3.1. Structural properties sputtering Cu/Ni multilayers

The X-ray diffraction pattern for 100×[Cu/Ni] multilayers, where the thickness of Cu layer was constant and the thickness of Ni layer were changing (10 Å⩽ t_{Ni} ⩽ 60 Å) are presented in Fig. 1. The satellite peaks from (111) and (200) planes, which characterizated multilayer system, are also visiable in this figure. However, the vertical lines on this plot show the position of peaks corresponding to the (111) and (200) planes of the fcc structure cubic Cu and Ni, respectively. The zero peaks occur between two theoretical values for fcc (111) and (200) planes. On diffractograms (Fig. 1) the increase in the thickness of the Ni layer caused the displacement the maximum of peaks from (111) and (200) planes in the direction of the tabulate value for Ni.

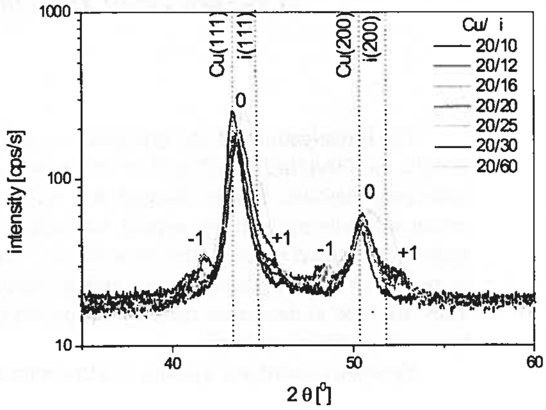

Fig. 1. Diffractograms of Cu/Ni multilayers with constant Cu layer thickness (20 Å) and varied Ni layer thickness (10 Å⩽ d_{Ni} ⩽ 60 Å). Dashed lines showed the tabulate positions of peaks for materials Cu and Ni

From the fitting of the measurment (Fig. 2), the satellite peaks are clearly visible for multilayers about the Ni layers thickness even:16 Å; 20 Å; 25Å; 30 Å.

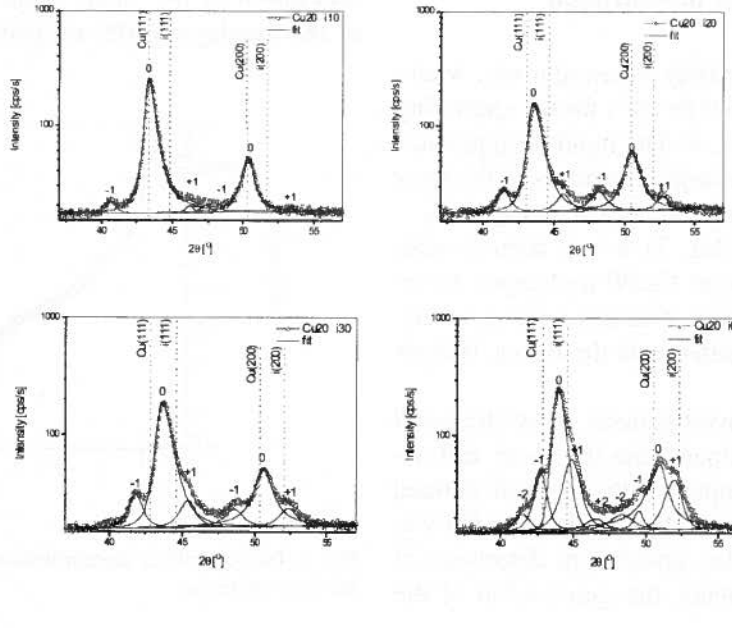

Fig. 2. Results of the fitting measurment Θ-2Θ of 100×[Cu(20 Å)Ni(t)] multilayers

In this case Cu and Ni are chemical elements neighbouring in the periodical system, both their constant lattices and the interplanar distances are very similar to themselves (Table 1). The intensity of the satellite peaks is low in the comparison with zero peak.

TABLE 1

The tabulate values of lattice constant and the interplanar distances for Ni and Cu

Chemical element	a [Å]	d (111) [Å]	d (200) [Å]
Ni	3.5238	2.0345	1.7619
Cu	3.6150	2.0871	1.808

The dependence of interplanar distances versus the thickness of the ferromagnetic layer in Cu / Ni multilayer is presented in Fig 3a. The growth of the Ni layer thickness is caused the change the average value of the interplanar distance of the Cu / Ni multilayer for both the peaks coming from the planes (111) and (200). These interplanar distances decrease and approach to the tabulate value for Ni. Such tendency is the result of the increasing of the Ni layer thickness, for which the value lattice constant is smaller than for Cu.

Together with the growth of the Ni layer thickness in Cu/Ni systems the growth the period value is observed. These values are obtained on the basis of Eq. (1) and are depicted in Fig . 3b. The deviation from dashed line, appointed period on the established process, is the measure of the planar perfection of the growth of the multilayers systems.

(a) (b)

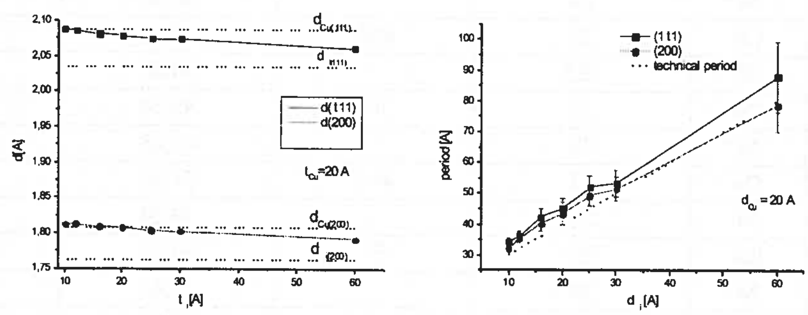

Fig. 3. The interplanar distances versus the Ni layer thickness in Cu/Ni systems (a). The period value of Cu/Ni multilayer versus the Ni layer thickness (b)

3.2. Reflectivity measurment

The results of the reflectivity measurements, where angle 2 Θ was changed from 0 to 10 °, for the sputtering samples are presented in Fig . 4. The rapid disapperance these curves is caused by the large roughness of the layer surface or the interlayers areas in multilayers systems.

On the basis formula (Eq. 3) it has been posible to calculate the period value of Cu/Ni multilayer. In order to obtain the best precision of structural parameters, similarly as for Θ-2 Θ measurements the fitting Wingix software (Fig. 5) is used.

The results of these investigations show the good agreement with results obtained from the X-ray diffraction measurement. The samples with the well defined structure of the superlattice are characterized by the visible Bragg peaks and it makes possible to determine of the periods values. Additionally, the gradual fall of the oscillation of the small value of the surface roughness or the interlayer RMS are persented (Table 2).

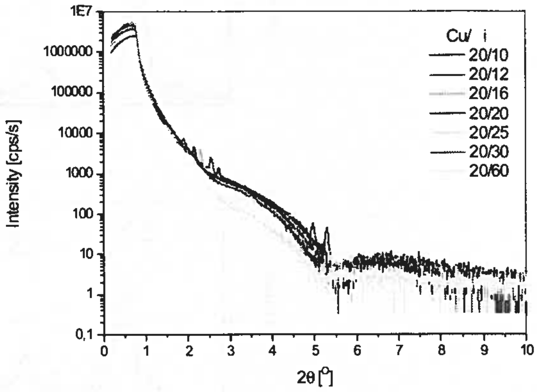

Fig. 4. The reflectivity measurment of Cu/Ni multilayers with varied Ni layer thickness

TABLE 2

The Cu and Ni layers thicness, the period value and the roughness value for Cu/Ni multilayersig

Samples	Llayer	Thickness [Å]	Period refl. [Å]	Period Θ-2Θ [Å]	Roughness RMS [Å]
Cu20/Ni10	Cu	22	32,6	34,28	7
	Ni	11,6		31,89	7
Cu20/Ni12	Cu	22	36	35,85	7
	Ni	14		35,38	7
Cu20/Ni16	Cu	22	40,5	42,19	7
	Ni	18,5		40,00	7
Cu20/Ni20	Cu	22	44	45,24	7
	Ni	22		43,25	7
Cu20/Ni25	Cu	22	49,5	51,93	8
	Ni	27,5		49,20	8
Cu20/Ni30	Cu	22	51	53,21	7
	Ni	29		51,45	7
Cu20/Ni60	Cu	22	82	88,03	7
	Ni	60		75,52	7
				78,72	
				84,52	

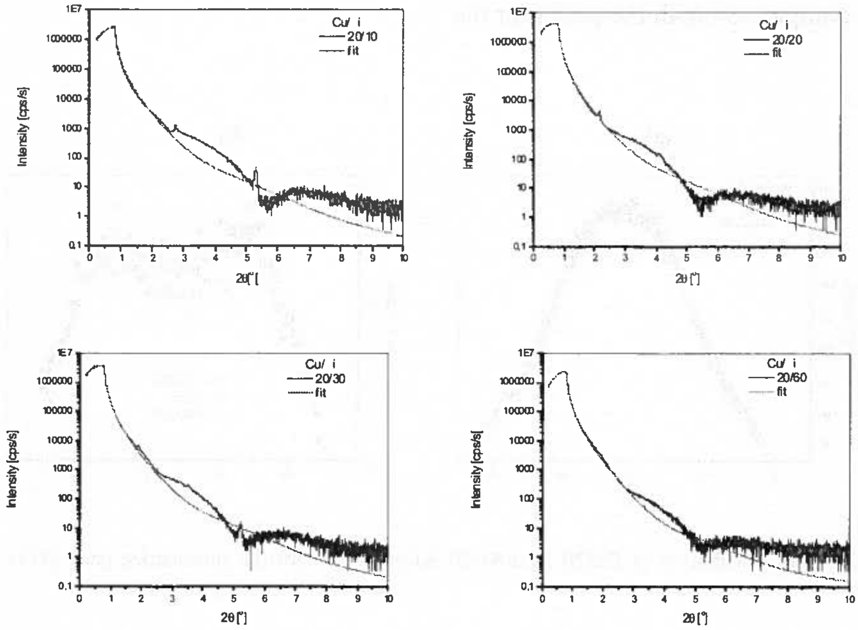

Fig. 5. The fitting results of the reflectivity measurments using the Wingix software

From results listed in the Table 2 one can see that the RMS roughness value, independently of the Ni layer thickness is similar. The periods values for all investigated systems are about 2 ÅA approx. larger than the values technologically established.

Comparing the period values of the Cu/Ni multilayers obtained from the Θ - 2Θ and the reflectivity measurments one can state that for the samples with the good structure quality, the period values are similar (Table 2).

3.3. The measurement of the distribution of the crystallite orientation directions (angle ω)

The results of the measurement of the distribution of the crystallite orientation directions, received for the 2 Θ angle agreed with the peak poitions coming from the (111) and (200) planes are presented in Fig . 6. These peaks are obtained by the Gaussian distribution of crystallografic directions. The results of analysis for the sample with the Cu and Ni layers thickness equal to 20 Åare shown in Fig. 7.

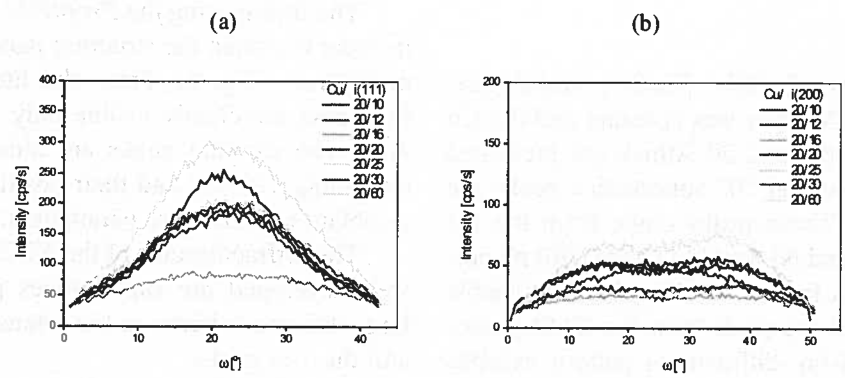

Fig. 6. The ω mesaurment on the superlattice peak from the (111) (a) and (200) (b) planes

From obtained earlier fitting and the ω measurements it is seen that the peaks coming from the (111) planes are characterized by smaller width and considerably larger intensity in comparison with the peaks for the (200) plane. This conclusion shows that the predominant orientation in all investigated of Cu/Ni mulitlayer is the (111) orientation.

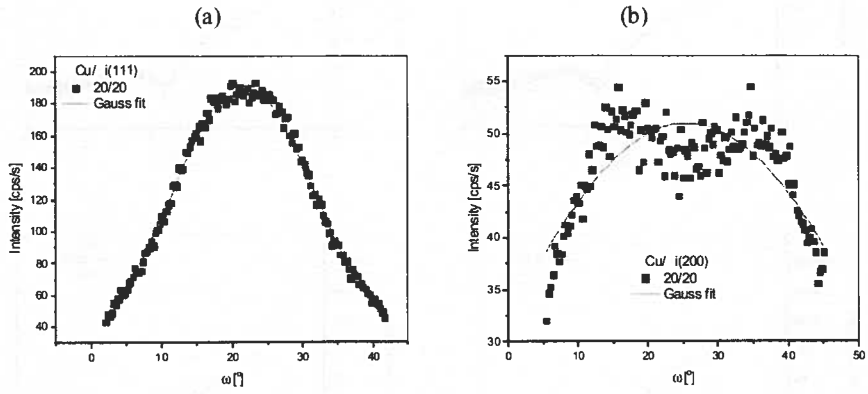

Fig. 7. The fitting results Gaussian distribution of Cu(20 ÅA)/Ni(20 Å) multilayer for the superlattice peak (111) (a) and (200) (b)

From the fitting results (Fig. 7 – the sample with the Ni and Cu thickness equal to 20 Å) it is observed that the texture in the (200) direction is worse than in the (111). The peak in the (200) direction is splitted, this proved that the planes in this direction are not parallel to the substrate (the sample surface).

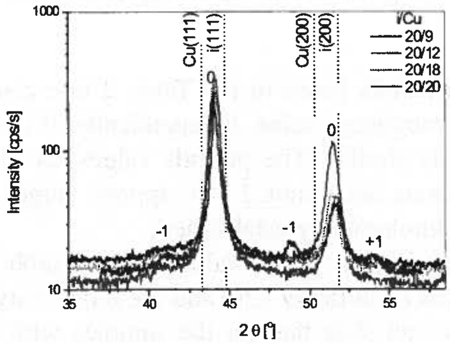

Fig. 8. Diffractograms of [Cu(t)/Ni(20 Å)]×100 multilayers, where tCu= 9 Å; 12 Å; 18 Å; 20 Å. Dashed line show the peaks positions for the Cu and Ni material

3.4. Structural properties electrochemical Ni/Cu multilayers

The diffractograms of 100× [Cu/Ni] multilayers, where the thickness of Ni layer was constant and the Cu layer is 9 Å; 12 ÅA; 18 Åand 20 Åthick are presented in Fig. 8. The order 1-st and "0" superlattice peaks are visiable in this figure. These peaks come from the fcc cubic structure for Cu and Ni the (111) and (200) planes. The 1 – st satellite peak from the (111) plane is visiable less in comparison with the peak from the (200) plane.

The analysis of X-ray diffractions pattern exhibits that the increase the Cu layer thickness is caused the displacement the peak maximum intensity at the axis 2θ, for the diffraction from the (111) plane for Cu. For the Cu 9 Åand Ni 20 Åmultilayer, these maximum is the nearest tabulated value of d111 for Ni.

The fitting using the *ProfitFile* software is performed in order to obtain the structure parameters of investgated multilayers (Fig. 9). From the fitting results the satellite peaks are clearly visible only for the sample Cu(20 ÅÅ). The satellite peaks are almost invisiable for the remaining samples and their positions are approximated to obtain the structure parameters.

The diffractograms of the Ni/Cu samples do not have well developed the superlattices peaks because of the large difference between the intensity of the 'zero' peak and the 1-st peaks.

Both the interplanar distances and the lattice constant of the componet materials of the multilayer are similar (Table 2). It does not allow the good fitting the simulated spectra to the experimental spectra and qualify of the superlattices parameters.

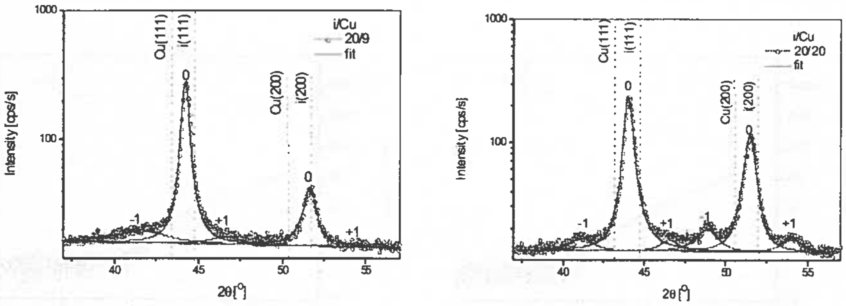

Fig. 9. Results of the Θ-2Θ fitting investigations of Ni/Cu multilayers obtained by electochemical method

The dependence of the interplanar distances versus the Cu layer thickness of Ni/Cu multilayer is presented in Fig. 10. Together with the growth of the Cu layer thickness insignificant growth of the value of the interplanar distance for (111) and (200) peaks are observed. This tendency is caused by the growth of the Cu thickness (larger than the lattice constant (Table 2).

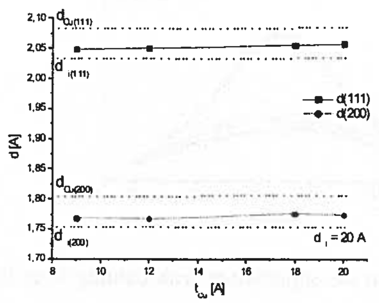

Fig. 10. The interplanar distances versus the Cu layer thickness of Ni/Cu multilayers

The value of the multilayer period and the half width of the peak are the next parameters which are derived from the peak position visible on the diffractogram. For the investigated samples the obtained period values is different from the technological one (e.g. for Ni(20 Å)/Cu(9 Å) the period is 38 Å). However, the value of the half width for the 1-st and 'zero' superlattice peaks coming from the planes e.g. (111) differ considerably from each other in every case of the investigated sample. It cause the worse quality of the structures in the comparison with the multilayers obtained the 'face-to-face' sputtering method.

3.5. Reflectivity mesaurement

The results of the reflectivity mesaurement of Ni/Cu multilayers are presented in Fig. 11. For the Ni/Cu multilayer with the changing Cu layer thickness, the reflectivity curves are characterized by very sharp fall of the reflection radiation and the abcence of Bragg peaks coming from the periodic of Ni/Cu superlattice. These samples are described by the large value of the surface roughness interlayer. The RMS roughness received from the fitting results (Fig. 12) increases with the increase of Cu layer thickness. The increase of RMS value from 25 ÅA to 37 ÅA is observed (Table 3). The absence of Bragg peaks proves that these samples do not create the periodic of Ni/Cu multilayer system.

TABLE 3
The RMS roughness value from the Wingix software fitting

Sample	RMS [Å]
Ni(20Å)/Cu(9 Å)	25
Ni(20Å)/Cu(12Å)	30
Ni(20Å)/Cu(18Å)	30
Ni(20Å)/Cu(20Å)	37

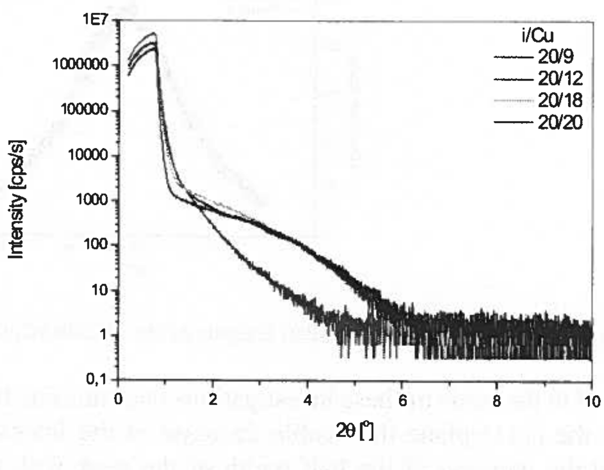

Fig. 11. The reflectivity measurement of Ni/Cu multilayers with varied Cu thickness layer

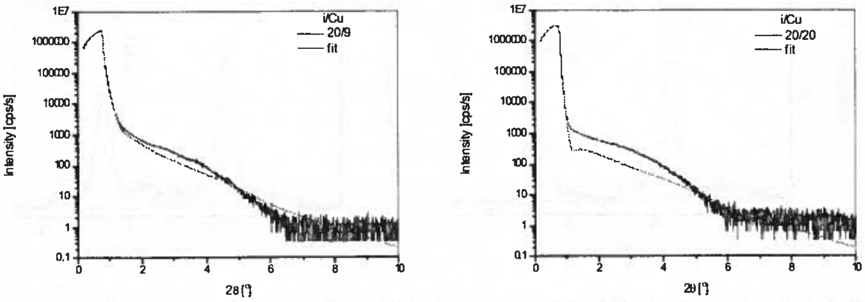

Fig. 12. The fitting results of reflectivity measurments of Ni/Cu multilayers

3.6. The measurement of the distribution of the crystallite orientation directions (angle ω)

The results of the measurement of the distribution of the crystallite orientation directions are depicted in Fig. 13. The obtained results are subjected to Gaussian analysis. The results of this analysis for the sample with the Cu and Ni layers 20 Åthick are shown in Fig. 14.

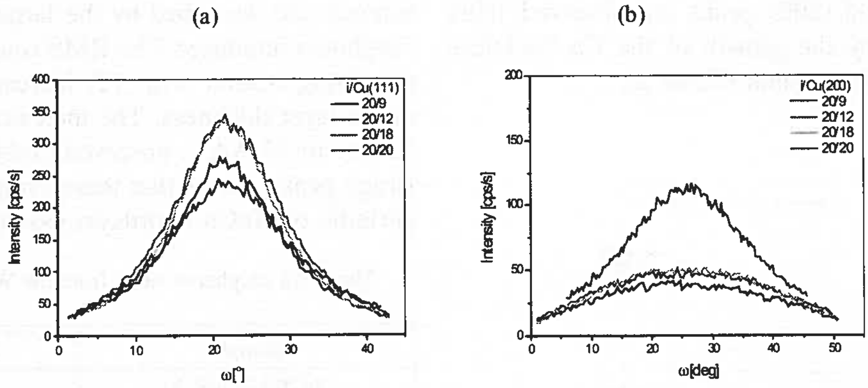

Fig. 13. The measurement of the distribution of the crystallite orientation directions on the superlattice peak coming from the (111)(a) and (200) planes (b)

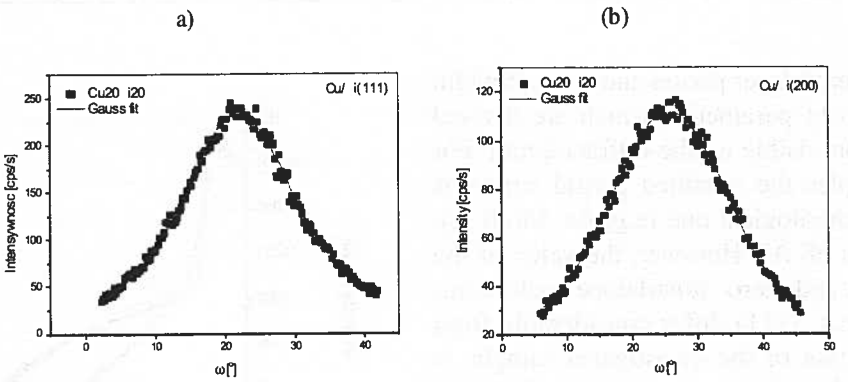

Fig. 14. The results of the Gaussian analysis of the Ni(20Å)/Cu(20Åmultilayer for the peak of (111) (a) and (200) (b) planes

On the basis of these investigations one can state that for the (111) plane the visible decrease of the intensity and the increase of the half width of the peak with the increase of the Cu layer thickness occur. From that we can conclude that the weak texture of the layer in [111] direction and the stronger texture visible in the [200] direction are present.

4. Conclusions

On the basis of conducted investigations one can state that multilayers obtained by the face-to-face sputtering method, in the comparison with samples obtained by electrochemical one are characterized by better quality of the structure:

- better defined superlattice structure (the larger intensity of satellite peaks, the half widths of peaks superlattice are similar),
- the smaller value of the interface roughness (about 4 times),
- the presence of the peaks in the reflectivity measurement, which gives the possibility to calculate the value of system periods and in the consequence of the thickness of Cu and Ni layers (these peaks are not visible in the case of electrochemical samples), [–] the increase of the total thickness of multilayered systems (face-to-face sputtering and electrochemical) leads to growth of peaks intensity coming from the (111) and (200) palnes, although predominant crystalographical orientation is (111).

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