A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 55 2010 Issue 1

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X-RAY MEASUREMENT OF THE Cu/Ni MULTILAYER PERIOD

RENTGENOWSKI POMIAR PERIODU WIELOWARSTWY Cu/Ni

Results of the X-ray examination of a Cu/Ni multilayer obtained by the magnetron deposition technique are presented in the article. The multilayer composed of one hundred bilayers was deposited on a Si (100) silicon substrate. The thickness of the Cu sub-layer was 2nm, while the thickness of the Ni sub-layer was greater, being 3nm. The grazing-incidence X-ray diffraction (GXRD) method with X-ray incidence angles ranging from 0.5 to 22.5° was employed for the investigation. The X-ray beam of a wavelength of λ_{Co}=0.17902nm in the diffraction angle range of 2Θ=43÷63° was used. The obtained diffraction patterns were analyzed for the greatest intensity of the main peaks (111) and (200) and their associated satellite peaks S-1 and S+1. As a result, the total intensity of all peaks as a function of X-ray incidence angle was determined. It was found that the greatest intensity was exhibited by diffraction reflections recorded at X-ray incidence angles in the range of 5÷10°.

Keywords: Cu/Ni multilayers, magnetron sputtering, grazing-incidence X-ray diffraction

W artykule prezentowano wyniki badań rentgenowskich wielowarstwy Cu/Ni otrzymanej techniką osadzania magnetronowego. Wielowarstwa złożona ze stu biwarstw została osadzona na podłożu krzemowym Si (100). Grubość podwarstwy Cu wynosiła 2nm, natomiast podwarstwa Ni była grubsza i wynosiła 3nm. Do badań zastosowano metodę dyfrakcji promieniowania rentgenowskiego przy stałym kącie padania (GXRD), w której kąty padania promieniowania wynosiły od 0,5 do 22,5°. Użyto promieniowania o długości λ_{Co}=0,17902nm w zakresie kątów dyfrakcji 2Θ=43÷63°. Otrzymane dyfraktogramy analizowano pod względem największej intensywności pików głównych (111) i (200) oraz zintegrowanych z nimi pików satelickich S-1 i S+1. W wyniku, określono całkowitą intensywność wszystkich refleksów w funkcji kąta padania promieniowania. Stwierdzono, że największą intensywność posiadały refleksy dyfrakcyjne zarejestrowane przy kątach padania promieniowania w zakresie 5÷10°.

1. Introduction

Multilayers of differential constituent composition have become recently the interest of materials engineering. This interest has resulted from the possibility of obtaining materials of unique physical properties, which could find their application in various branches of industry. Multilayers composed of Cu and Ni are interesting mainly owing to the gigantic magnetoresistance (GMR) effect being characteristic for thin-layered ferromagnetic/diamagnetic material systems [1-3].

These particularly interesting properties of Cu/Ni multilayers result, *inter alia*, from the identical crystallographic structure (fcc) of the both elements and a small crystalline lattice misfit, which amounts merely to 2.5%. Cu/Ni multilayers are technologically easy to produce by many techniques, including the PVD [4-14].

From the point of view of multilayer production technology and multilayer properties, it is essential to determine precisely the parameters of a multilayer, such as the thickness of the period (bilayer) or the perfection of the crystalline structure [7,10-15]. The X-ray diffraction method is a basic non-destructive technique that enables the determination of these parameters. In the present work, two methods of X-ray examination were used for the determination the multilayer period based on the satellite peaks associated with the basic (111) and (200) and the GXRD reflections to identify the possibility of the best representation of the main and satellite peaks.

2. Methodology and the material

A Cu/Ni multilayer deposited on a monocrystalline Si(100) silicon substrate was examined. The multilayer, which was composed of 100 bilayers, was produced by

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the magnetron deposition technique. The thickness of the Ni sub-layer is 3nm, while the thickness of the Cu sub-layer is equal to 2nm. The multilayered structure was examined by X-ray measurements using a radiation wavelength of λ_{Co}=0.17902nm. Two methods of measurement were carried out: Θ–Θ and GXRD, both in the diffraction angle range of 2Θ=43÷63°.

On the basis of the θ-θ measurement, the calculation of the multilayer period thickness was made based on formula (1) derived from the geometry of X-ray diffraction on the interfaces in a layered arrangement, as shown in figure 1 [10]. The period size is equal to the thickness of two layers included in the multilayered arrangement.

$$\Lambda = \frac{\lambda}{2(\sin\Theta_i - \sin\Theta_{i-1})}, \qquad (1)$$

where:

i – main peak from the lattice plane,
$i\pm1$ – satellite peaks,
Λ – multilayer period thickness [nm],
λ – X-ray wavelength [nm].

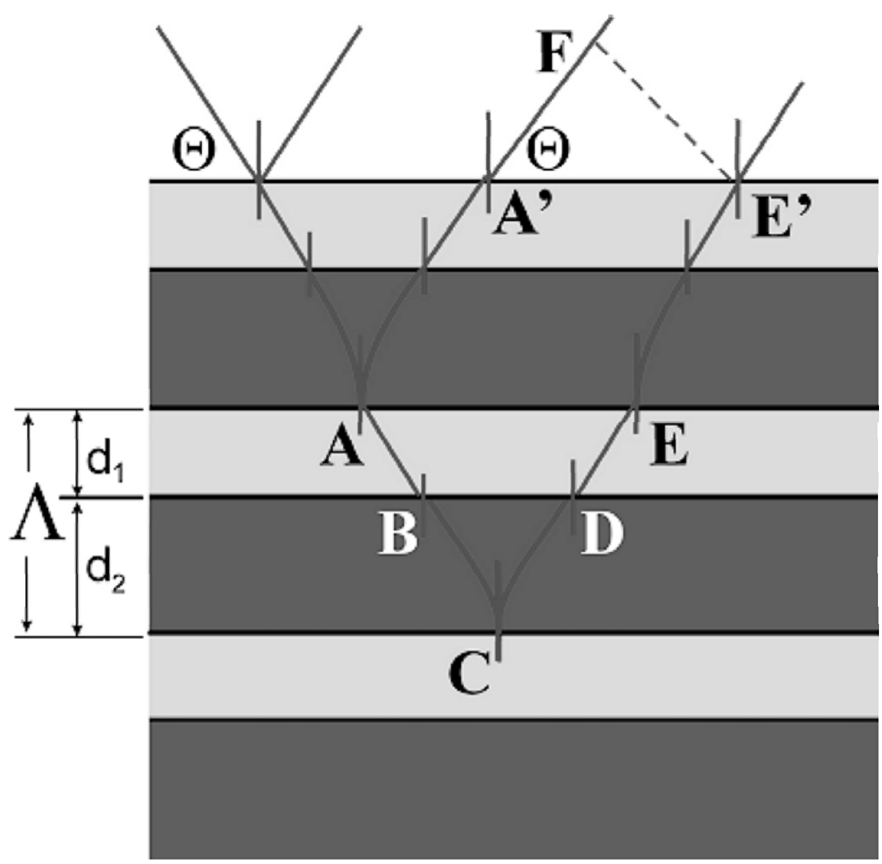

Fig. 1. Schematic diagram of X-ray diffraction on the interfaces of a multilayer, based on work [10]

3. Results and discussion

The examination results are shown in figure 2. Two main reflections, (111) and (200), were obtained in the diffraction pattern. These reflections are common to both multilayer constituents due to the similar values of interplanar distances. Additionally, their associated satellite peaks of the first order were recorded on either side of each of the main reflections. The reflections in figure 2a are described with the pseudo-Voigt curve using the Analyze software, as shown in figure 2a, and the angular positions of reflection maxima were determined. For the calculation of the multilayer period thickness, the positions of the main reflections and the satellite peaks – were used, because of their better representation on the diffraction pattern.

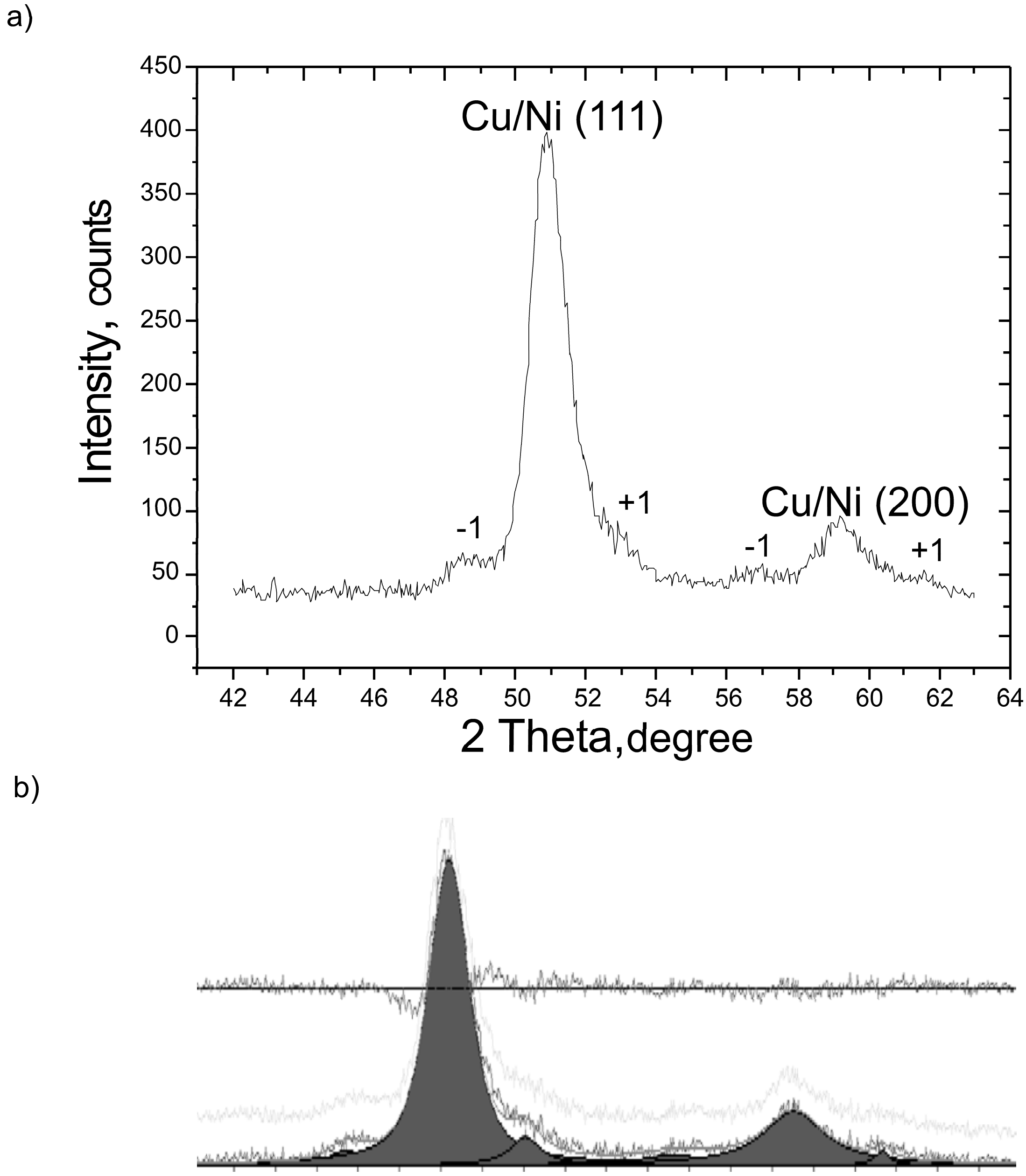

Fig. 2. XRD diffraction pattern of the Cu/Ni multilayer (a), fits of reflections obtained using the Analyze software (b)

The multilayer period thickness Λ (Eq. 1), is equal to 4.99nm as determined for the reflection (111), and 4.89nm as determined for the reflection (200). Thus, the results of period measurement for both reflections are very similar to each other and to the theoretical bilayer thickness (5.0nm) assumed for the deposition process.

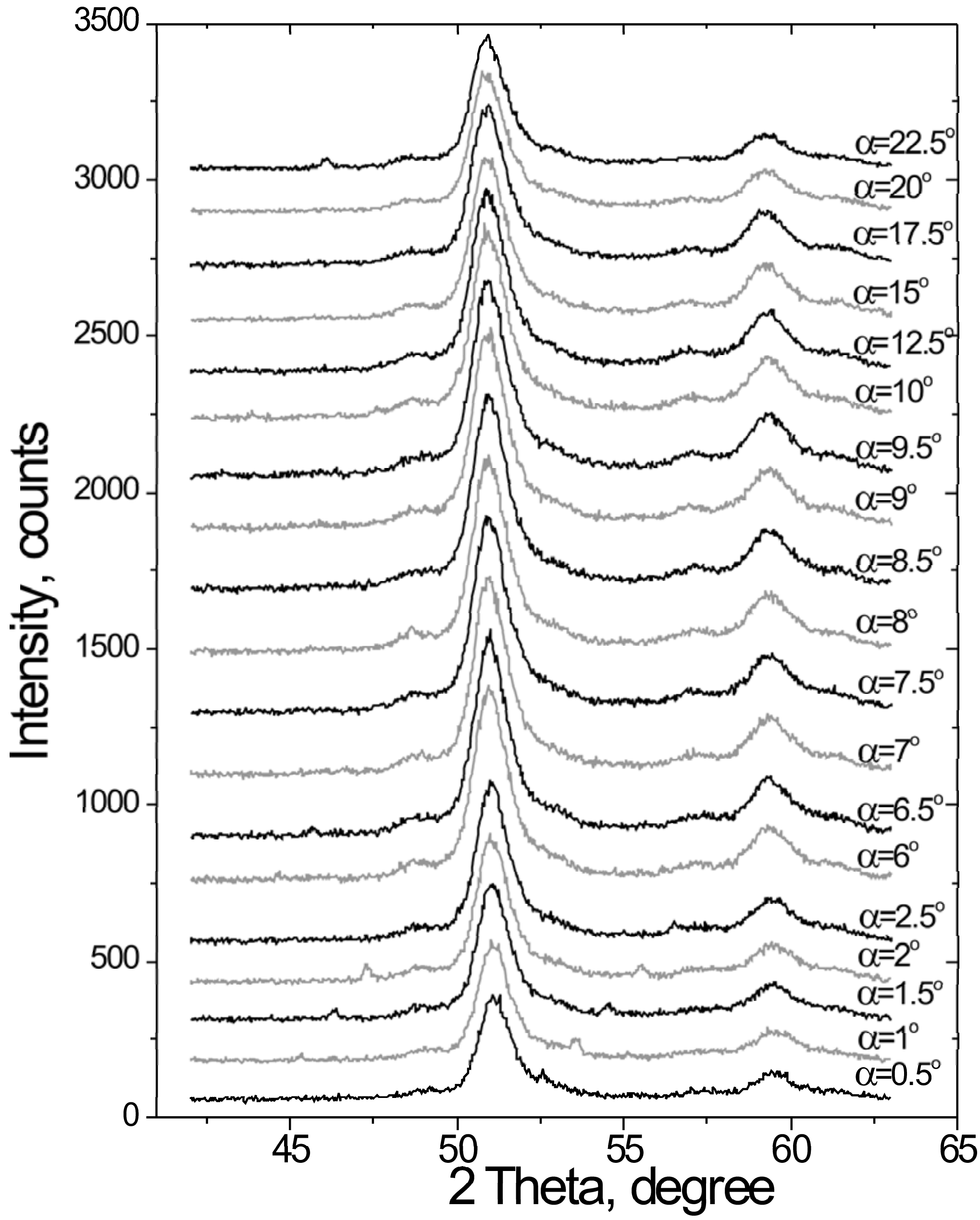

Fig. 3. Diffraction patterns of a multilayer sample using GXRD geometry

The GXRD measurement results are presented in figure 3. In order to establish the effect of the fixed-angle X-ray incidence on the quality of recorded satellite peaks, the total intensity of reflections was determined. The results of the total intensity measurement (I_{net}) are presented in figure 4 as a function of radiation incidence angle.

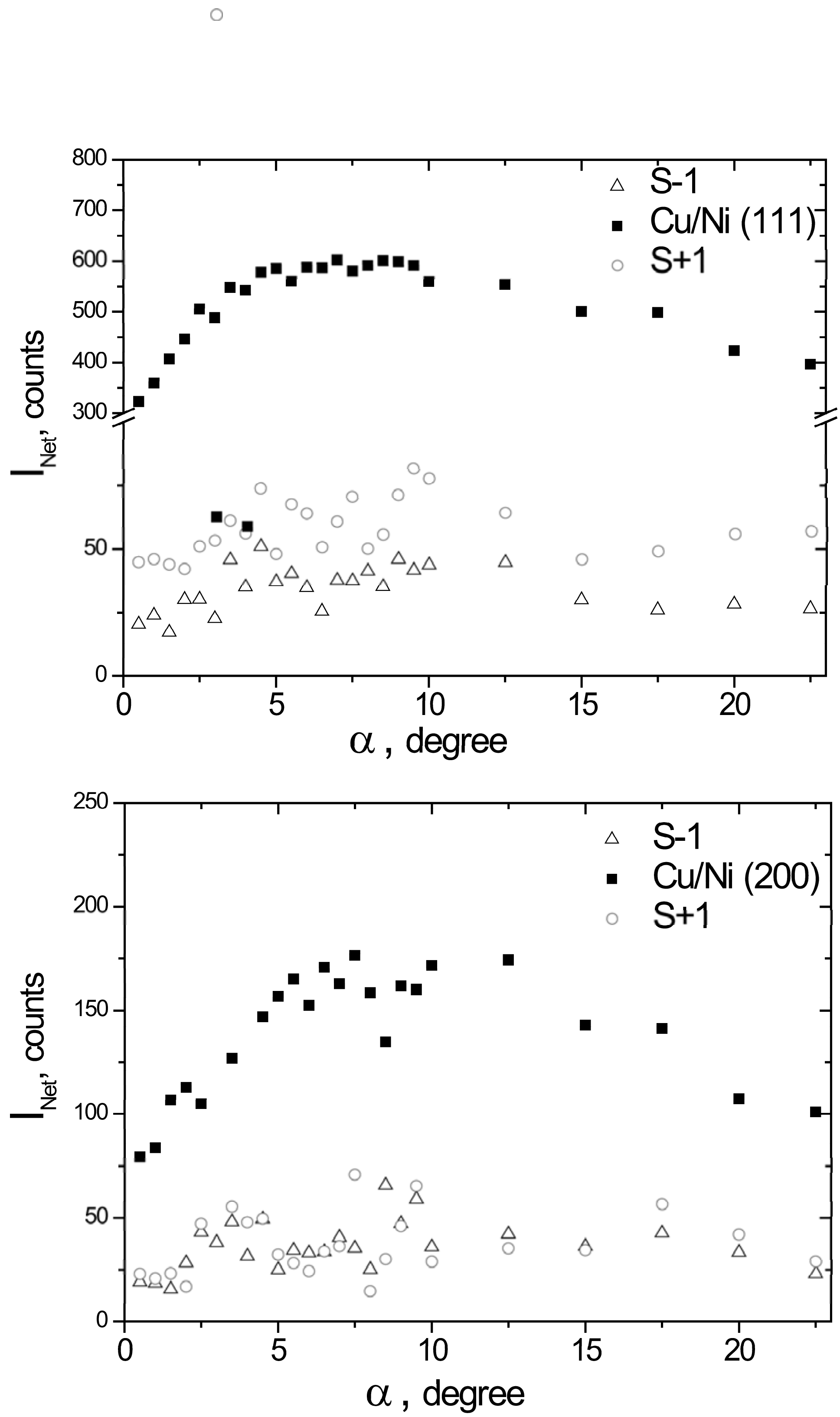

Fig. 4. Total intensity of reflections as a function of the X-ray incidence angle in GXRD measurements, Cu/Ni(111) (a), Cu/Ni(200) (b)

The total intensity of the main reflections and their associated satellite peaks increases with the increasing incidence angle to reach a maximum in the angle range of 5±10°, and then decreases its value with the further increase in the fixed angle of incidence (Figure 4).

For GXRD diffractions from figure 3, the period thickness was determined, and the obtained values are shown in figure 5. Regardless of whether the (111) or (200) reflection was used to determine the period thickness, the thickness values exhibited a similar scatter around the values of their arithmetical means. The average periods thickness of is equal to 4.96nm and 5.01nm, respectively.

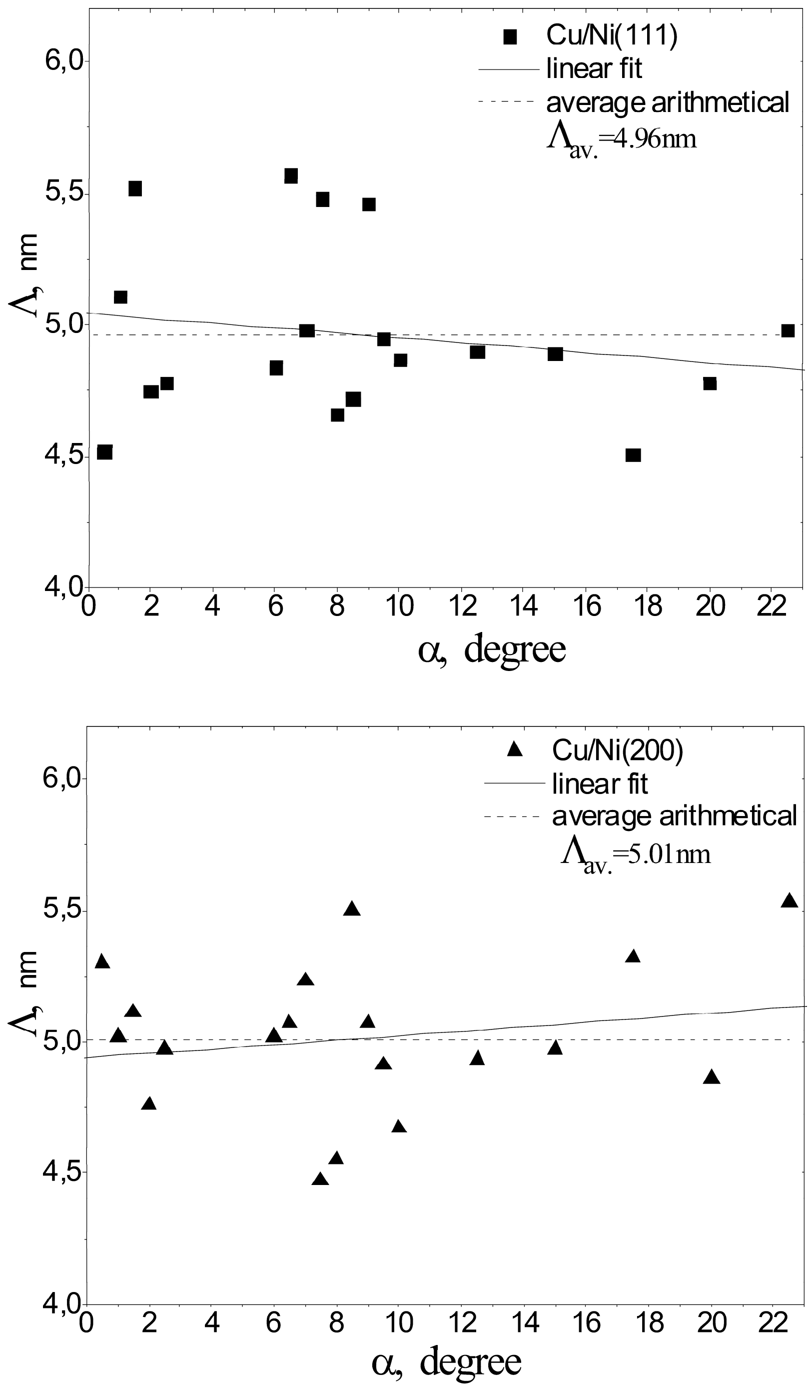

Fig. 5. Period thickness determined in GXRD measurements, Cu/Ni(111) (a), Cu/Ni(200) (b)

4. Conclusions

From fixed-angle measurements it was found that the intensity of main reflections and satellite reflections varied with X-ray incidence angle. Diffraction patterns recorded at incidence angles from the range of 5±10 are characterized by the greatest intensity. The use of diffractions obtained in this range for the determination of the period thickness does not affect the accuracy of its estimation.

From the analysis of the arithmetic mean of the thicknesses of all determined periods it was established that the determination of the multilayer period using the measurement of – geometry yielded values that are the closest to the assumed values, compared to the GXRD measurements used for this purpose.

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Received: 10 September 2009.