STRUCTURE OF Fe-Cu COATINGS PREPARED BY THE MAGNETRON SPUTTERING METHOD

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This work presents the results of our research concerning the synthesis of metallic Fe-Cu coatings by use of the magnetron sputtering method. The structure of the coatings synthesized during two modes of pulsed magnetron sputtering was compared . In our experiment the pulsed magnetron power supply generated a series of pulses gated at 1kHz - standard pulsed mode (SPM) and 2 Hz - low frequency pulsed mode (LFPM). The analysis of the microstructure by means of SEM and TEM show that obtained coatings are characterized by nanocrystalline structure. Additionally the optical emission spectra (OES) during the copper and iron sputtering were measured.
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1. INTRODUCTION

The conventional magnetron sputtering is a plasma deposition technique which is often used for various kinds of films deposition; it permits to control density and composition of the films even for low thicknesses (from nm to μm scale). Pulsed magnetron sputtering has recently become a very popular method of thin film deposition. In such process the plasma is switched on and off cyclically. The usage of pulsed power supplies allows the performance of very stable processes independently of whether dielectric or conductive layers are deposited. [1]. The non-reactive magnetron sputtering process of metallic coatings using a pulsed power supply is presented in this paper. It is well known that the columnar structure is the typical morphology form of coating materials produced by the magnetron sputtering even for pulse techniques when the magnetrons often work with frequency values of the order of 10^2 kHz.

Taking into account the experience concerning the IPD method [2,3,4] which use the impulse plasma for layers preparation and as a consequence of that it allows to obtain amorpho-nanocrystalline or nanocrystalline structure of coating materials, we expected more favorable structural properties of the obtained coatings. The aim of our research described below, concerning the synthesis of metallic coatings by use of the magnetron sputtering method was to compare the structure of the coatings synthesized during two modes of pulsed magnetron sputtering: standard pulsed mode and low frequency pulsed mode.

2. EXPERIMENT

The coatings were deposited by magnetron sputtering technique using a dual-gun system. The two WMK 50 magnetron guns [5] were used. Magnetrons were powered by 10 kW pulse power supply DORA PS, operating in DC mode with a frequency of 170 kHz and modulation in range of 2 Hz to 2 kHz [6].The power transmitted into the plasma was controlled by number of current pulses per time unit. In arrangement called pulsed magnetron sputtering maximizing the effective power transmitted directly to the target was used, what has influence on the efficiency of the sputtering process.

In our experiment the pulsed magnetron power supply generated a series of pulses gated at 1kHz (standard pulsed mode (SPM) of magnetron sputtering in the case of our setup) and 2 Hz (low frequency pulsed mode (LFPM) of magnetron sputtering).

The magnetron targets of 50 mm in diameter sputtered in this experiment were made of iron and copper. Fe, Cu and layered Fe/Cu coatings were fabricated in the Ar atmosphere. The chamber was pumped by standard vacuum system (diffusion pump + rotary pump). The gas pressure during sputtering process ranged from p= 0.5-0.8 Pa. The coatings were deposited on silicon and sapphire (001) substrates located in parallel to targets, keeping substrate-target distance 13 cm.

The Fe, Cu and Fe/Cu coating were deposited as a function of deposition time and number of the elemental layers. The morphology and microstructure of the deposited coatings were characterized by using scanning (SEM) and transmission (TEM) electron microscopes. The crystalline phases of the Fe, Cu and the layered Fe/Cu coatings were identified by an X-ray diffraction measurements. Optical emission spectra (OES) were measured during the copper and iron sputtering using the WMK-50 magnetrons. Optical signals emitted from the discharge area were collected through the view port window (transparent in 200-1100 nm range) by a fiber optic cable. An averaged signal over the whole space between the target and substrate was collected. This was sufficient to estimate what population of the particles dominates within the plasma region.

3. RESULTS

The results of our investigation show that the Fe-Cu coatings are characterized by nanocrystalline structure. X-ray diffraction measurements revealed polycrystalline structure of the layer's materials (Fig. 1). Fig. 2 shows structure of the Fe-Cu coating, together with corresponding electron diffraction pattern. Electron diffraction pattern shows series of rings that can be attributed to the nanocrystalline structure of the coating materials which was composed of fcc-Cu and bcc-Fe phases.

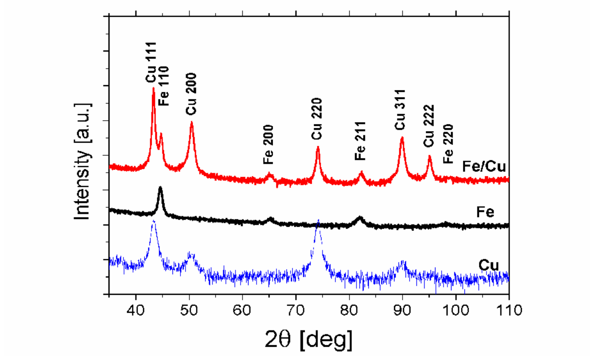

Fig. 1. X-ray grazing incidence diffraction patterns for Fe, Cu and Fe/Cu coatings deposited by (SPM) of magnetron sputtering

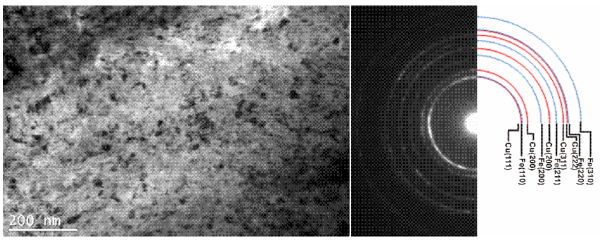

Fig. 2. Transmission Electron Microscopy (TEM) micrographs with corresponding electron patterns of the Fe/Cu coatings obtained (SPM) of magnetron sputtering

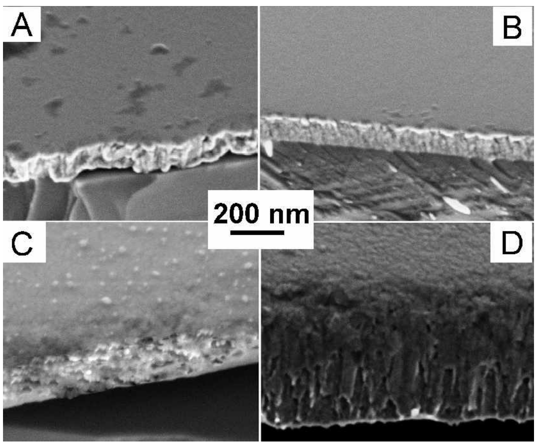

Fig. 3. The cross section view of the Fe (A,B) and Cu (C,D) coatings obtained by magnetron sputtering in the standard pulsed (A,C) and low frequency pulsed (B,D) mode

A comparison of the cross section view of the Cu, Fe and Cu/Fe coatings obtained during the low frequency pulsed mode of the sputtering source equals 2 Hz and standard pulsed mode of sputtering with 1kHz modulations are presented in Fig. 3 and Fig. 4. As one can see, the application of low frequency pulsed mode of magnetron sputtering resulted in production of more uniform and regular nanostructure. The coatings became flatter and more homogeneous than the coatings produced under standard pulsed mode. Additionally in the case of the copper, the obtained coating is two times thicker. It means that in the low frequency pulsed mode of the power supply the more efficient sputtering of the copper target was proceeded.

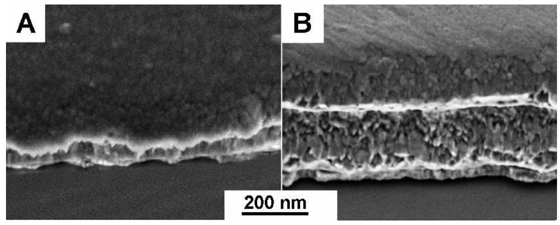

Fig. 4. The cross section view of the $(Fe/Cu)_{x2}$) coatings obtained by magnetron sputtering in the standard pulsed (A) and low frequency pulsed (B) mode

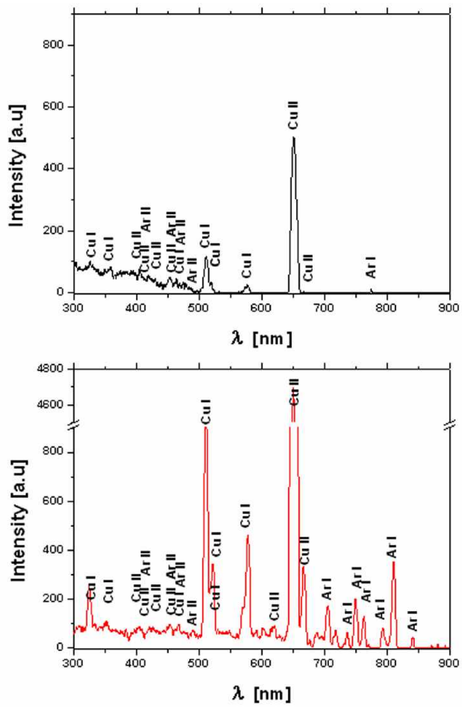

Fig. 5. Optical emission spectra from glow discharge during magnetron sputtering of Cu target: SPM (top), LFPM (bottom)

The OES of magnetron plasmas for DC magnetron sputtering of copper and iron in argon atmosphere were studied. The emission spectra taken in the 300-900 nm radiation region obtained in the present experiment are shown in Fig.5 and Fig.6 respectively for copper and iron sputtering. The spectral lines were assigned to the corresponding wavelengths. The analysis of the emitted spectra shows the presence of different lines corresponding to excited state and ions of copper (Cu I, CuII [7]), iron (Fe I, Fe II? [7]) and argon (Ar I, Ar II [7]) originating from the target materials, as well as from the working gas. A very strong effect of the low frequency pulsed power supply on the emission lines was observed. Magnetron sputtering process carried out with the modulation equals 2Hz (low frequency pulsed mode – LFPM) resulted in a more intensive emission. We registered in case of iron sputtering four-fold increase and in case of copper sputtering seven-fold increase (Cu) in the intensity of the emission lines compared to the intensity of the emission lines obtained during standard pulsed mode (SPM, 1 kHz).

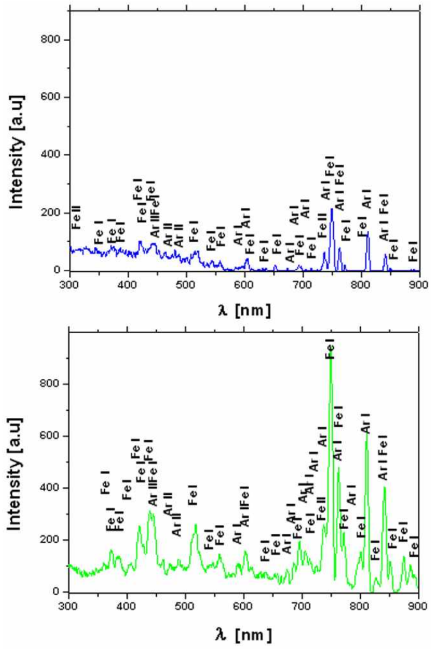

Fig.6. Optical emission spectra from glow discharge during magnetron sputtering of Fe target. SPM (top), LFPM (bottom)

4. CONCLUSIONS

In this paper we demonstrated the comparison of the structure of the metallic coatings synthesized during two modes of pulsed magnetron sputtering: standard pulsed mode (SPM) and low frequency pulsed mode (LFPM).

The results obtained during the experiments shown that the low frequency pulsed mode (2 Hz) of magnetron sputtering contributed to obtain flat smooth surface and more uniform structure than the layer produced under the standard pulsed mode (1 kHz). Additionally in the case of Cu coatings, the LFPM mode of magnetron sputtering had an effect on increase of the sputtering efficiency.

ACKNOWLEDGEMENT

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СТРУКТУРА Fe-Cu-ПОКРЫТИЙ, НАНЕСЕННЫХ МЕТОДОМ МАГНЕТРОННОГО РАСПЫЛЕНИЯ

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Представлены результаты исследований, связанных с синтезом металлических покрытий, нанесенных методом магнетронного распыления. Проведено сравнение структуры покрытий, синтезированных импульсным магнетронным напылением в двух режимах. В экспериментах использовались стандартный импульсный режим (SPM), генерируемый серией импульсов 1 кГц, и низкочастотный импульсный режим (LFPM) с частотой 2 Гц. Анализ микроструктуры с помощью SEM и TEM показал, что полученные покрытия характеризуются нанокристаллической структурой. Кроме того, были измерены оптические эмиссионные спектры (OES) в процессе ионного распыления меди и железа.

СТРУКТУРА Fe-Cu-ПОКРИТТІВ, НАНЕСЕНИХ МЕТОДОМ МАГНЕТРОННОГО РОЗПИЛЕННЯ

K. Nowakowska-Langier, K. Zdunek, R. Chodun, R. Nietubyc, R. Mirowski, J. Witkowski

Представлено результати досліджень, зв'язаних із синтезом металевих покриттів, нанесених методом магнетронного розпилення. Проведено порівняння структури покрить, синтезованих імпульсним магнетронним напилюванням у двох режимах. В експериментах використовувалися стандартний імпульсний режим (SPM), генеруємий серією імпульсів 1 кГц, і низькочастотний імпульсний режим (LFPM) з частотою 2 Гц. Аналіз мікроструктури за допомогою SEM і TEM показав, що отримані покриття характеризуються нанокристалічною структурою. Крім того, були виміряні оптичні емісійні спектри (OES) у процесі іонного розпилення міді і заліза.