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Thermionic vacuum Arc deposited Cu and Co nanostructured multilayers: synthesis and characterization

D. RĂSLEANU, D. ILIE, V. IONESCU, V. MOCANU, M. G. MUREŞAN, I. M. OANCEA – STĂNESCU*, V. CIUPINĂ, G. PRODAN, E. VASILE[a], I. MUSTAŢĂ[b], V. ZAROSCHI[b], C. P. LUNGU[b]
Department of Physics, Ovidius University, Constanta, 900527, Romania
[a]*Metav-CD S.A., Bucharest, 050025, Romania*
[b]*National Institute for Laser, Plasma and Radiation Physics, Magurele, 077125, Romania*

The paper presents an approach for obtaining and the characterization of several nanostructured materials with special resistive magnetic properties. The Cu and Co thin films are grown by the physical Thermionic Vacuum Arc method (TVA). Successive layers of Cu and Co were deposited on glass, silicon and ceramic substrates using tungsten crucibles containing Cu and Co metals, respectively. The morphological and structural investigations were achieved by means of electron microscopy techniques: TEM (Philips, CM120ST) and SEM. The compositional analysis of the films together with the atomic percentage determination of the constituent elements were performed by the Energy Dispersive X-ray Spectroscopy (EDS) analysis.

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Keywords: Multilayers, Thermionic vacuum arc, Cu, Co

1. Introduction

The thermionic vacuum arc method is using an electron beam emitted by an externally heated cathode. The cathode is a based heated tungsten filament cathode. The electron beam is accelerated to a high anodic voltage and can evaporate the anode materials as neutral pure particles. It facilitated the deposition of the anode materials on a substrate. The electron energy and current intensity on the substrate are not too high. When the anode potential is increase up to a certain value, the evaporation rate increases enough to allow an electrical discharge to ignite in the evaporated pure material. The discharge maintains even when the discharge current is as low as a few hundred mA. The discharge can burn only in the presence of external cathode heating source at these intermediate arc currents [1-4].

In the Fig. 1 is presented the experimental set-up [5].

In order to study the phenomena which influencing the magnetorezistive effect in the alternate layers, we consider one multilayer with two magnetorezistive layers: (Co) separated by one conductive nonmagnetic layer (Cu).

The magnetoresistive effect ΔR is described by the formula:

$$\Delta R = [R(0) - R(H)] \tag{1}$$

where $R(H)$ is the resistance in the presence of an external magnetic field H and $R(0)$ the resistance in null magnetic field.

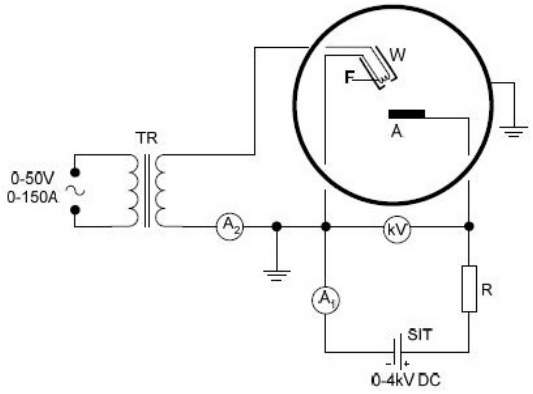


Fig. 1. The electrical arrangement experimental set-up.

The magnetoresistance, $\frac{\Delta R}{R(0)}$ [1,6-8], is defined as:

$$\eta = \frac{\Delta R}{R(0)} = \frac{[R(H) - R(0)]}{R(0)} \tag{2}.$$

2. Experimental

Successive layers of Cu and Co were deposited on glass, silicon and ceramic substrates using tungsten crucibles containing Cu and Co metals, respectively (see fig. 2). The minimum pressure in the vacuum chamber was $4 \cdot 10^{-5}$ Torr. The deposition thickness was controlled to be of approximately 4 nm for Cu and approximately 10 nm for Co, using a Cressington quartz balance monitor mtm 10.

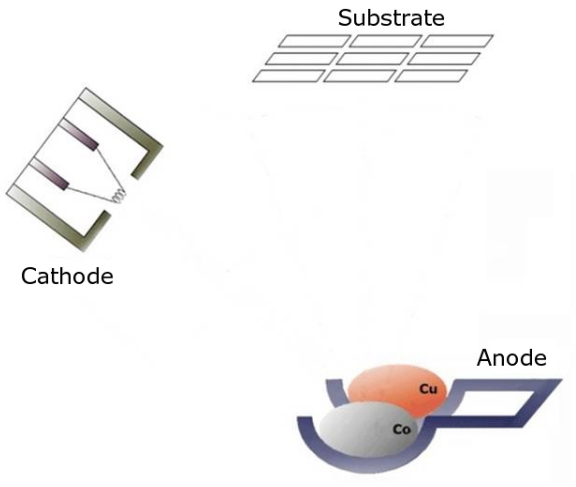


Fig. 2. The geometry of the deposition.

The experimental deposition parameters are presented in Table 1 ($I_{filament}$=24 A, p=4·10^{-5} Torr.)

Table 1. Deposition parameters.

Layer number	Metal	Anodic voltage (kV)	I_{anode} (mA)	Layer thickness (nm)
I	Co	1.2	100	11
	Cu	0.8	80	3.9
II	Co	1.6	132	10.4
	Cu	0.8	80	4
III	Co	1.6	121	10.1
	Cu	0.8	80	5
IV	Co	1.6	120	3

The morphological and structural investigations were achieved by means of the electron microscopy techniques: TEM (Philips, CM120ST), SEM and EDAX.

3. Results and discussion

TEM measurements were made using a higher resolution electron microscope, Philips CM 120, operating at an accelerating voltage of 120 kV and capable of a point-to-point resolution of 2 Å.

The well definite diffraction rings from SAED pattern (Fig. 3 and Fig. 4) indicate the polycrystalline state of the thin film investigated.

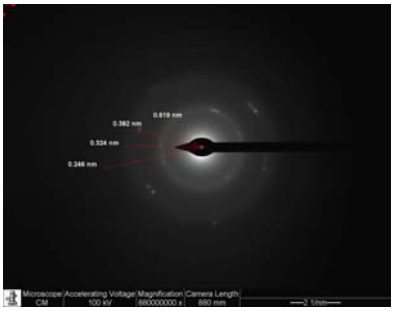


Fig. 3. The SAED image.

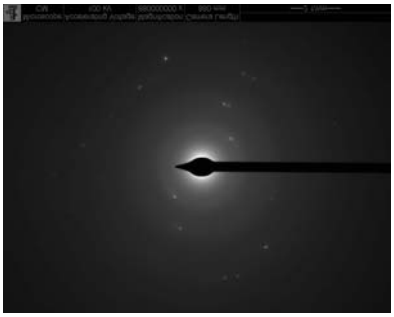


Fig. 4. The SAED image.

From the BF-TEM image of the film (Fig. 5 and Fig. 6), we can say that the sample investigated has a grain structure, which consist in many small grains of relatively uniform size forming a morphologically homogeneous film.

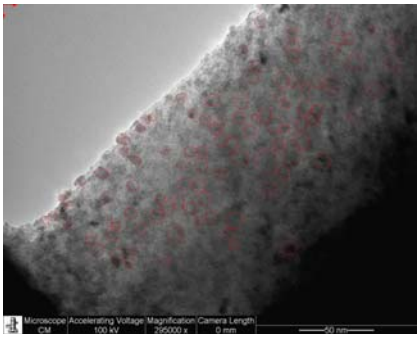


Fig. 5. The BF-TEM image

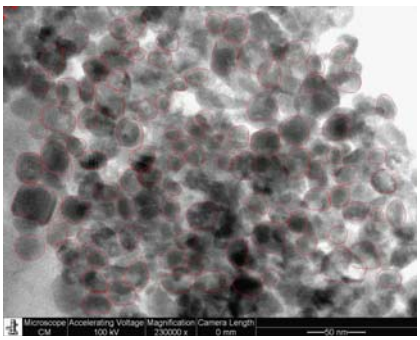
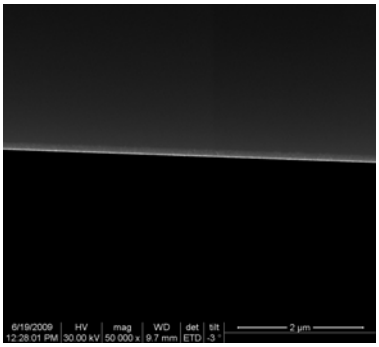
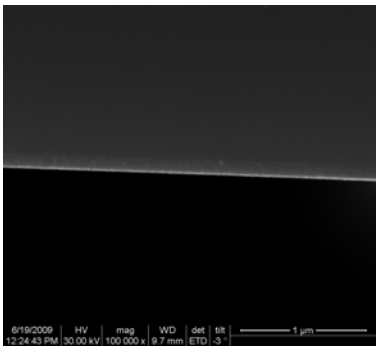


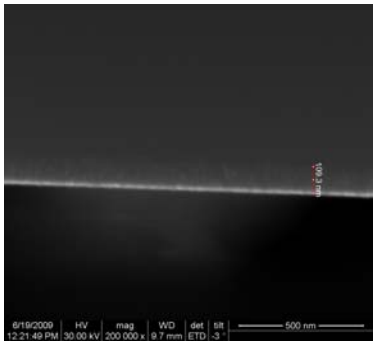
Fig. 6. The BF-TEM image



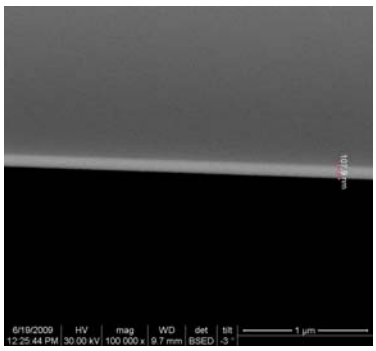
a) 50.000×



b) 100.000×



c) 200.000×



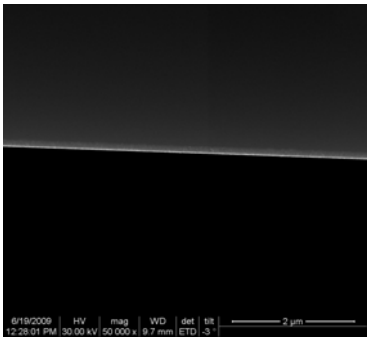
d) 100.000×

Fig. 7. The microstructural aspect of the thin film

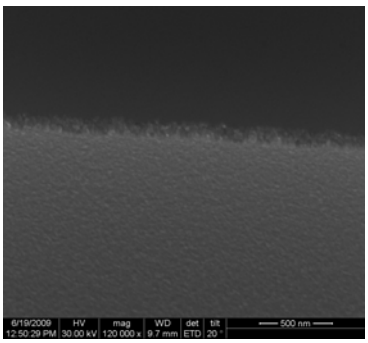
SEM measurements were made using QUANTA INSPECT with a field emission gun of 1.2 nm resolution, a spectroscope with energy dispersive X-ray (EDS) analysis with the resolution at the MnK of 133 eV. Using the spectral EDX analysis, we investigated the metallic composition of the thin film. We found a map with the distribution of density concentration for the elements.

The sample was studied in the cross-section. The cross-section was obtained by cutting with a glasses cutter (diamond) on the uncovered area. After the sample was put in the liquid nitrogen the fracture was produced .

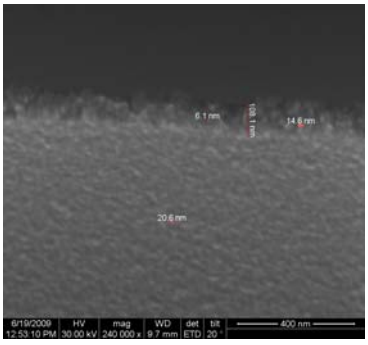
CoCu was deposited on a silicon substrate. The microstructural aspect in the section is presented in the Fig. 7 a, b, c (secondary electrons images - SEI) and the Fig. 7 d (backscattering electrons images - BSED).



a) 600.000×



b) 120.000×



c) 240.000×

Fig. 8. The surface appearance of the film at oblique incidence of the electron beam (inclination of 20 degrees)

In this image, at a 50,000x increase, details are observed in the cross – section the thin film deposition on the substrate (horizontally band light on the image). At a

200.000 times increase (Fig. 7 c) it is observed the uniform aspect of the layer with a 109.3 nm thickness. The predominant character of the film growth on the layer is columnar. The Fig. 7 d indicates a pronounced uniformity of deposited film.

In Fig. 8 a, for a 600,000 times increase and an oblique incidence of the electron beam on the specimen, it is shown a cross–section in the layer and the aspect of the deposited film surface. Fig. 8b (120,000x) presents a pronounced uniformity of deposited film and in this surface a homogeneous granular appearance for this deposit type.

Fig. 8 c shows that the growth process was done by germination of nanoparticles with size 6–20 nm, distributed as columns on the surface.

In Fig. 9 one can see the distribution of elements Si, Co, Cu (in a cross-section through the sample) by the distribution characteristic X radiation SiK_{α}, Co K_{α}, CuK_{α}, in the micro area whose image is present in this image (top left corner). In the bottom of Fig. 9 is presented the spectrum of the X-ray energy dispersive analyzed micro area. It is shown the presence of elements Si, Co and Cu. The Si element comes from the substrate on which the deposition was made. Co and Cu are the elements present in the deposited thin film.

The distribution images of characteristic X radiation are present also in the fig. 10. The elements concentration is observed better by the relative density of colored points.

The spectrum of X-ray energy dispersive (Fig. 11), obtained on the deposited thin film, shows the presence of the elements Co and Cu in the cross-section of the film. The presence of the Zn element can be explained by the secondary reflections on the sample setting in the microscope.

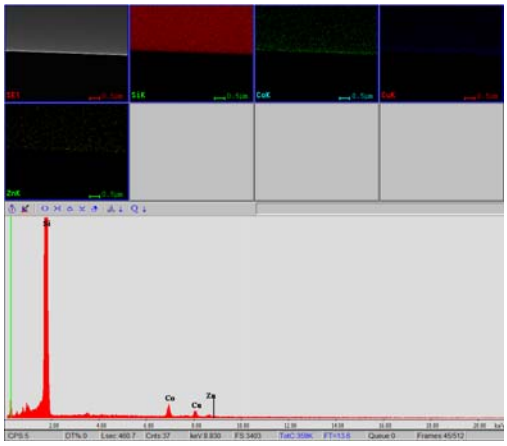


Fig. 9. The image of the secondary electrons and associate images of distribution in surface relative intensity of characteristic X radiation SiK_{α}, Co K_{α}, Cu K_{α} in the cross-section of sample.

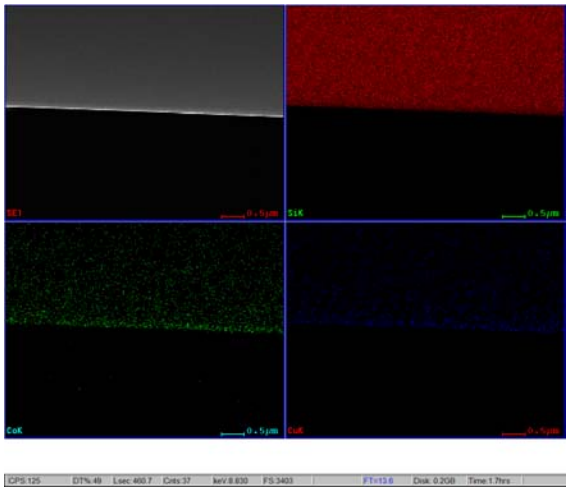


Fig. 10. The map distribution of the characteristic X radiation SiK_{α}, Co K_{α}, CuK_{α}

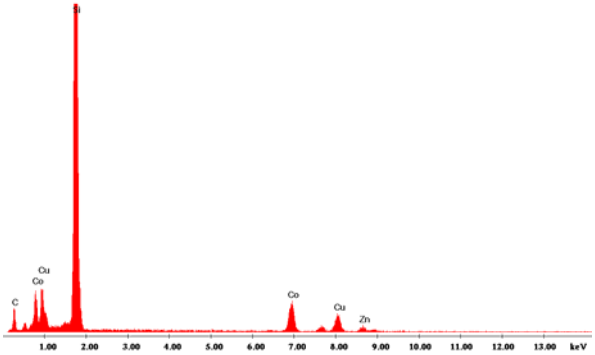


Fig. 11. Spectrum X-ray energy dispersive obtained on the deposited thin film

5. Conclusions

From the BF-TEM image of the multiplayer CoCu we were able to observe a morphologically homogeneous film, with small nanocrystalline grains, having he mean grain size approximately 8.36 nm and 20.33 nm. This multilayer thin film is nanostructured.

Based on the spectral EDS analysis we determined the map of the Co and Cu concentration. The concentration of Co is higher than Cu. The sample presented a pronounced uniformity of deposited film and in this surface a homogeneous granular appearance for this type of deposits. It is observed the uniform aspect of the layer with a 109.3 nm thickness.

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*Corresponding author: istanescu@univ-ovidius.ro