STRUCTURAL PROPERTIES AND COMPOSITION ANALYSIS OF Cu–Ni–Co ALLOY THIN FILM*

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The main purpose of this paper is to investigate the composition of $Co_{20}Ni_xCu_{80-x}$ alloy, which has some interesting magnetic properties. The Cu–Ni–Co alloy film with a few nanometers thickness (10-50nm) was deposited onto glass substrates by physical vapor deposition technique.

Transmission Electron Microscopy (TEM) analysis carried out by Philips, CM120ST operating at 100 kV with Cs = 1,2 mm and ≈ 2 Å resolution. The polycrystalline state was confirmed by using SAED technique, that exibits a typically pattern for this type of material. BF-TEM was useful to examine the morphology of thin film by mean of the grain size measurements that show a film with nanocrystalline grain with a narrow distribution around mean diameter of grain equal to 4 nm. EDX analysis was performed to complete compositional analysis; concentrations of the film components was determined, and a map with the distribution of these concentrations was also obtained.

Key words: thin film, EDX analysis, Transmission Electron Microscopy.

1. INTRODUCTION

Numerous studies of magnetic thin films which includes in their compositions Fe, Ni, Co, Cr, Al have been carried out because of potential applications in magnetic sensor technology and in computer read/write heads, in microelectromechanical systems MEMS, and ultralarge scale integration ULSI devices.

Giant magnetoresistive materials, as $Co_{20}Ni_xCu_{80-x}$ granular thin films, are used in the fabrication of spin-valve sensors, which is the prime device for current drive heads in the magnetic recording industry.

A magnetic field sensor based on GMR effect can directly detect a magnetic field and any changes of this field. It means that it can be used in

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variety of magnetic sensors to detect such parameters as displacement, torque, position, current and many others.

Co-Ni and Co-Cu based alloys like CoFeNi, CoCuNi, FeCoCu, CoNiAg thin films form promising alternatives for magnetic media and sensor industry [1–9].

F. Wang *et al.* [1] reported a microstructure of the granular CoNiCu film showing a matrix of Cu in which nanoparticles of Co-Ni are well distributed; this structure is very different from that of the granular Co-Cu thin films, in which full grown Co microparticles are embedded in a Cu matrix.

2. EXPERIMENTAL

This thin film were deposited by thermal evaporation technique at 10^{-5} Torr on glass substrates, with TS = 300 K – temperature of substrate, and the rate of deposition was 10 nm/s. The thickness of the film d was measured using a Cressington Thickness Monitor mtm10, and we found the value d = 50 nm.

The microstructure of the CoNiCu thin films was analysed by transmission electron microscopy (TEM). TEM measurements were made using a higher resolution electron microscope, Philips CM 120, operating at an accelerating voltage of 100 kV and capable of a point-to point resolution of 2 Å.

With the Bright–Field Transmission Electron Microscopy (BF–TEM) image, we studied the granular structure of the film, and we plotted a grain size distribution for this alloy.

Using the spectral EDX analysis, we investigated the metallic composition of the thin film, and we found the approximate concentration of each element; a map with the distribution of density concentration for those elements was also obtained.

3. RESULTS AND DISCUSSIONS

The well defined diffraction rings from SAED pattern (in Fig. 1) indicate the polycrystalline state of the thin film investigated. The diffraction rings shown in this pattern are corresponding to the interplanar distances of 0.2 nm, 0.174 nm, 0.124 nm, and 0.104 nm, which fits well with the distances of the (111), (113) diffraction planes for the fcc phase of Cu, and with those of the (200), (110) planes for the fcc phase of Ni and for the hcp phase of Co, respectively.

So, the layer contain a mixture of fcc and hcp nanocrystalline graines.

TEM analysis results of NiCo alloy thin films deposited by vapour deposition indicated that the films were face–centered cubic (fcc) in structure [3].

In the granular thin films of CoAlAg, crystalline particles of fcc Ag and hcp Co were detected, after TEM analysis [10].
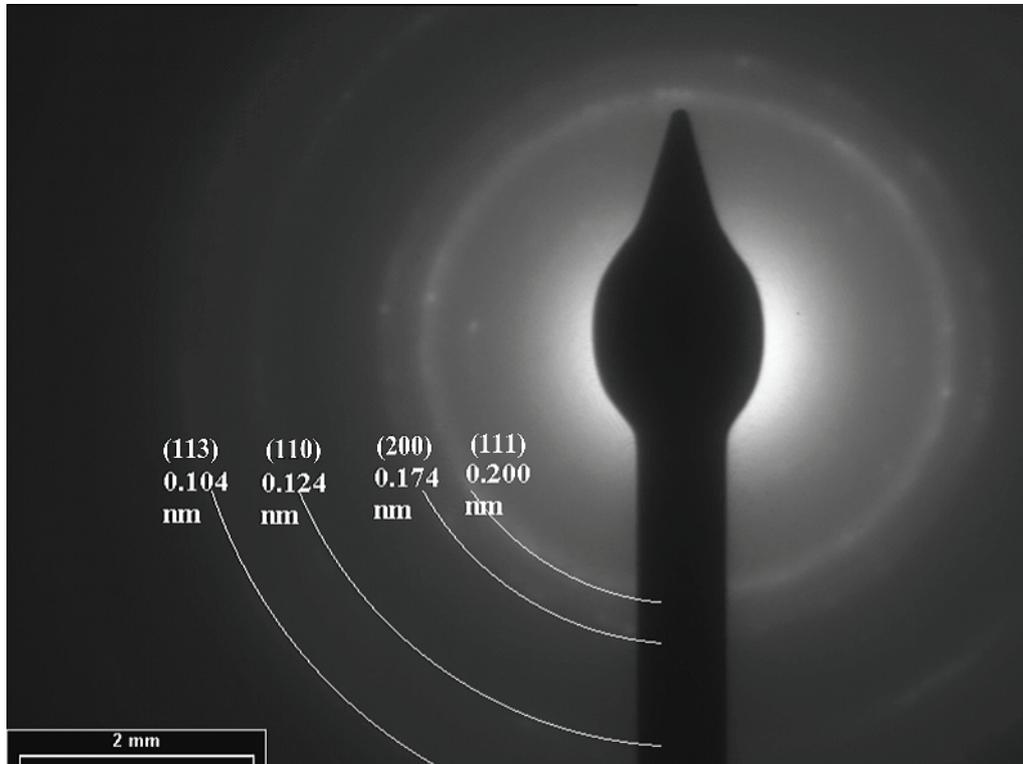
Fig. 1 – Typical SAED pattern of CoNiCu thin film.

From XRD and TEM analysis of CoFeNi thin films, N. G. Chechenin *et al.* [4] found that the layers consisted in mixtures of bcc and fcc crystalline grains.

From the BF–TEM image of the film, we can say that the probe investigated has a grainy structure which consists of many small grains of relatively uniform size forming a morphologically homogeneous film.

The size distributions of the crystallites, obtained from the diameter measurements of a few dozen grains, are plotted in Fig. 3. The distribution of grain sizes, as measured from BF-TEM image, was fitted to the lognormal curve. The mean grain size, D_m, is approximately 4 nm.

From the spectral EDX image presented in Fig. 4, we see three basic peaks, formed at the value of 6.8 keV, 7.6 keV, and 8.1 keV for the X-Ray Energy, witch are corresponding to the spectral lines of Co, Ni and Cu, respectively.

With some approximation, it can be determined the concentration of each metallic element from the thin film, using RONTEC EDWIN WinTools computer program.

We found the follow concentrations for the metals: 16.81% (± 2.23%) for Co, 25.77% (± 3.45%) for Ni and 57.43% (± 7.44%) for Cu.
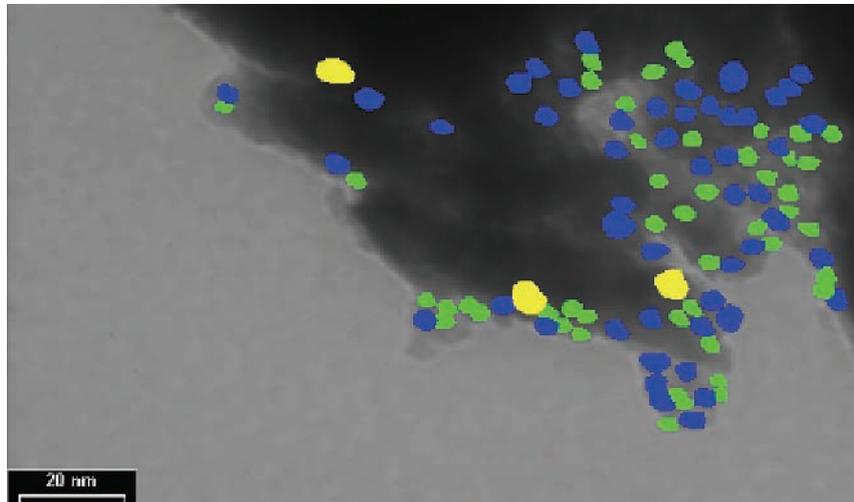
Fig. 2 – BF–TEM image of CoNiCu thin film.

Data: Sample 109
Model: *LogNormal*

$\chi^2 = 1.3371$
$y_0 = 0.48105(\pm 0.57237)$
$x_c = 3.94195(\pm 0.01552)$
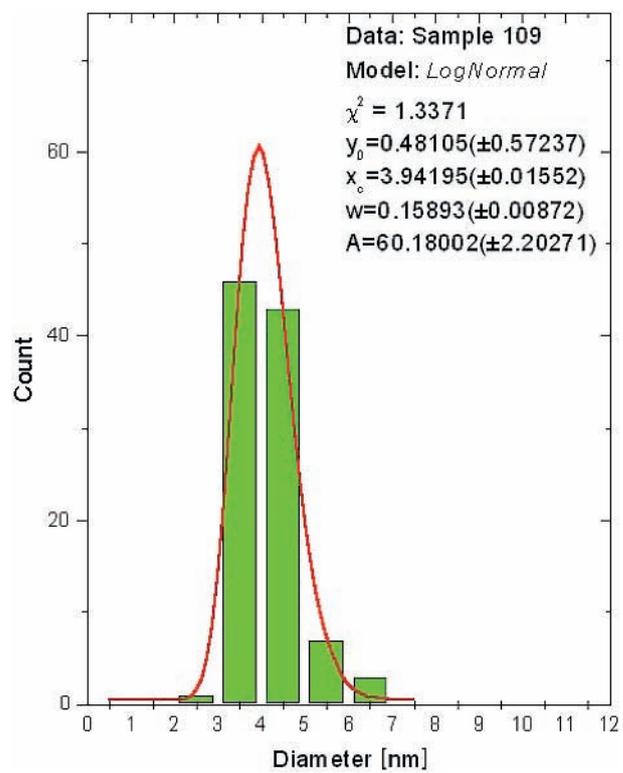$w = 0.15893(\pm 0.00872)$
$A = 60.18002(\pm 2.20271)$

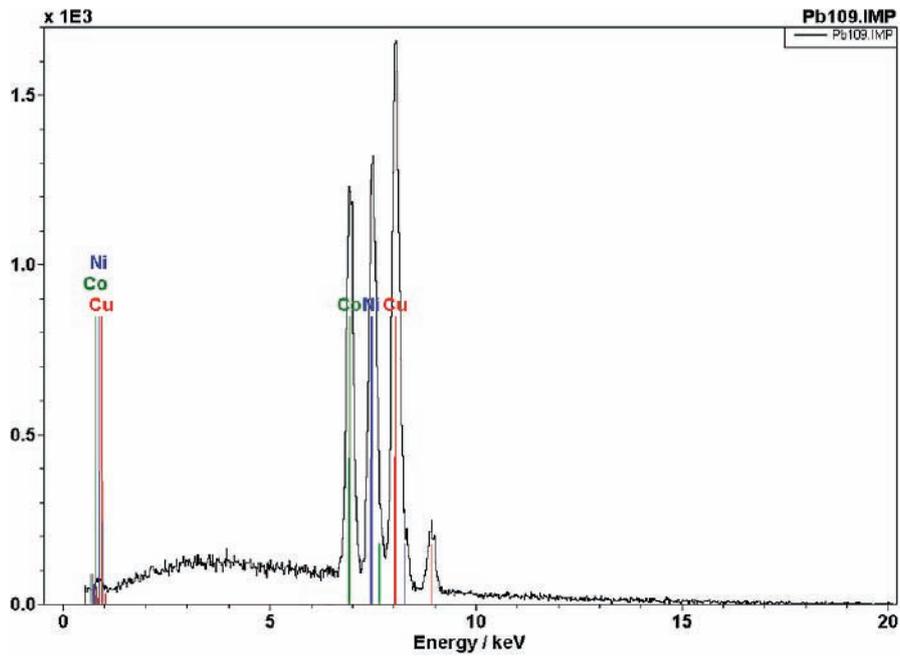Fig. 3 – Grain size distribution of CoNiCu thin film.

Fig. 4 – Spectral EDX analysis image of CoNiCu thin film.

Fig. 5 – Distribution of density concentration map for Co, Cu, Ni obtained from the EDX analysis of CoCuNi thin film.

It was reported that the MR ratio of Co-Ni-Cu alloys decreases monotonically with increasing Ni content, the ideal concentration for Ni being 5% [7].

From the map showed in Fig. 5 we can observe that exists an uniform distribution of the density concentration for the three metals.

CONCLUSIONS

From the BF-TEM image of the thin film we were able to observe a morphologically homogeneous film, with small nanocrystalline grains, having he mean grain size Dm = 4 nm.

With the Spectral EDS analysis we determined the concentration of these metals; we observed that exists a good correlation between the value of concentration for each metal, composing the alloy obtained by our group using the physical vapor deposition technique and the concentration wanted for this type of alloy thin film: $Co_{20}Ni_xCu_{80-x}$, which present some important magnetic properties, investigated into a future paper.

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