CORRELEATION BETWEEN SIMMULATION AND MICROANALYTICAL EXPERIMENT IN MULTILAYER NANOSTRUCTURES TYPE ANALYSIS

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Atunci când sunt evaluate sisteme multistrat nanodimensionale prin spectrometrie cu dispersie după energii (EDS), sunt necesare standarde privind precizia datelor analitice generate de interacţiunile electron-material al substratului şi grosimea efectivă a filmelor depuse ca şi filme ultrasubtiri. Acest studiu îşi propune să investigheze efectul energiei fasciculului de electroni acceleraţi asupra adâncimii de penetrare în nanostructuri multistrat de tip Cu-Ni-Cu-Fe-Ta-Ag de diverse grosimi depuse pe suporţi de Si (100) prin metoda cu arc în vid termo-ionic (TVA), folosind măsurători microanalitice EDS şi simulări matematice bazate pe modelul Monte Carlo. Sunt stabilite relaţii între energia fasciculului de electroni şi adâncimea de penetrare în probe.

When assessing nanodimensional multilayer systems by energy dispersive spectrometry (EDS), new standards of accuracy are required for the analytical data generated by the electron-matter interactions from substrate and the effective thickness of deposited ultrathin films. This study aims to investigate the effect of acceleration electron beam energy on the penetration depth on Cu-Ni-Cu-Fe-Ta-Ag multilayered nanostructures of various thicknesses deposited onto Si (100) wafers by thermionic vacuum arc (TVA), using extensive microanalytical EDS measurements and mathematical simulations based on Monte Carlo model. Relationships between the electron beam energy and penetration depth into samples are established.

Keywords: software simulation, nanolayers, microanalysis, depth of X-ray excitation

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1. Introduction

The possibilities offered by the new deposition methods of thin films have reached unimaginable limits till now, as currently is possible to deposit structures even with subnanometric dimensions, metallic and non-metallic, in various combinations. Reducing the thickness of deposited layers led to the need to improve the inherent resolution in terms of separation and quantification of elements in the microanalytical methods. The recent scientific literature indicates a variety of analysis methods oh the thin and ultrathin layers in terms of morphology, surface and internal structure, but also the composition of the nanostructure. These methods offer us very precisely qualitative and quantitative imagistic and compositional details in terms of any form nanostructures, but undoubtedly, they involve high cost starting with the high performance equipment acquisition till the prices of the supplies and maintenance.

Given these issues, by this paper, we propose the application of an innovative method, a tandem between the Monte Carlo mathematical modelling and simulation, using a CASINO® specialized software and the microanalytical experimental EDS type analysis. The purpose of applying this methodology is primarily to reduce costs and spending time dedicated to the deposits (due to the quantitative results provided by simulation programs) and secondly to the microanalysis, because the very precise mathematical estimates lead to the use of fixed parameters of some cheap emission sources, of wolfram. These one allow sufficient excitation energy of the K and L lines of heavy elements and offer compositional proportional results with the ratio of chemical elements deposited in nanodimensional layers, at different depths of multilayer structures like [1, 2].

The use of precise simulation programs for the electron beam-sample interactions enables the visualization of the interaction volumes between accelerated electron beams and samples, as well as an accurate calculation of the signal intensity resulting from this impact. Among the advantages of using these analyzing programs lies the possibility of the thoroughly planning and interpretation of the microanalytical (EDS) results [3, 4, 5].

In this paper, by microanalytical measurements and mathematical simulations based on Monte Carlo model were established relations between electron beam energy and penetration depth into the samples. A gradually electron trajectory simulation is produced, using random numbers to approximate the scattering angles based on theoretical probability distribution or empirical models. [6, 7]. The depth where X-rays signals is produced during the electron beam-solid matter interaction is strongly dependent on sample density and accelerating voltage. The penetration depth of electrons and the sample interaction volume depends on the incidence angle, current, acceleration voltage and atomic number (Z) of the sample [8, 9, 10].

More exactly, we analyzed by EDS microanalysis, the chemical composition of multilayer structures type Cu-Ni-Cu-Fe-Ta-Ag with different thicknesses deposited by Thermionic Vacuum Arc (TVA) onto Si (100) wafers, using a perpendicular incidence of the accelerated electron beam, at the variable voltages, so that the X ray signal come mainly from the desired layer to be investigated.

The specialized literature indicates many possibilities of using the EDS method into the multilayer analysis, but it does not offer the accuracy's certainty for investigating the nanostructures, due to the very high depth of penetration of the accelerated energy beams, so that it could be obtained X-rays signals characteristic to the desired depths [1, 8, 9]. To improve the quality of the microanalysis results in these cases, our team proposes to coat the nanodimensional multilayer with thicker layers, of chemical elements that are not in the analyzed structure, of a very different atomic number and without lines of analysis which may overlap slightly with the elements of interest.

The study aims to obtain a correlation between mathematical simulation based on Monte Carlo model and the EDS microanalytical experiment regarding the effect of electron beam energy on the penetration depth using EDS analysis of Cu-Ni-Cu-Fe-Ta-Ag multilayer structures with different thicknesses deposited by Thermionic Vacuum Arc (TVA) onto Si (100) wafers.

2. Experimental

X-ray spectroscopy methods determine the presence and quantity of chemical elements by detecting the characteristic X-ray emitted from atoms irradiated with high energy beams. The characteristic X-ray energy is given by the difference between the energy of two electrons located on different layers and well defined, being also dependent on the element's atomic number. The EDS spectra lines are representations of X-ray intensity depending on the energy range between 0.1 and 20keV and may include both heavy and light elements, as the K lines of the light elements and M and L of the heavy elements are located on the same range. The lateral size of the interaction's volume may be significantly greater than the diameter of the electron beam, which is very important to know for the EDS analysis of some micro zones of massive samples [10, 11].

In the microanalysis methods case, the ZAF methods bases on the supposition that the analyzed sample is massive and has a microscopic flat. This is an important thing for the analysis of the thin films, because the matrix's effect is reduced. The Standardless method (used in this study) is recommended when standards are not available as models and it is based on the intensity prediction of X-ray which should be obtained from a standard of a pure element, in the same instrumental conditions. The method is useful when the sample is composed of

transition elements with the K lines in 5-10 keV area. In the case of 3 keV smaller lines, significant errors can occur due to the detection of several families of lines, K, L and M [1, 8].

The multilayer Cu-Ni-Cu-Fe-Ta-Ag type nanostructures used for analysis in this paper were obtained using TVA method (detailed described in other papers) at the National Institute of Laser, Plasma and Radiation Physics (INFLPR), Bucharest-Magurele [12, 13, 14]. In the experiments presented in this paper, the voltage applied to the anode, had values of kV order (Table 1). To achieve nanodimensional multilayer films, we used a special anode system, consisting from a cylindrical graphite disc, in which there were positioned four crucibles with the specific deposition materials (Cu, Ni, Fe, Ta, Ag), each material being deposited in the necessary order, without external interference during the deposition session. The substrates used consisted of Si wafers (100) 10×12mm, positioned at a distance of 250mm from the discharger. The deposition rates of each material were determined during the deposition process with a quartz microbalance. The deposition conditions and the thin layers thicknesses are presented in Table 1.

Table 1.

Operating parameters and thickness of deposited layers.

Elem.	U (V)	I (mA)	Rate (Å/s)	Thickness structure 1 (515nm)	Thickness structure 2 (523nm)	Thickness structure 3 (543nm)	Thickness structure 4 (703nm)
Cu	600	350	6	3	5	10	50
Ni	1300	50	0.1	3	5	10	50
Cu	1800	90	0.2	3	5	10	50
Fe	2100	120	0.2	3	5	10	50
Ta	1500	110	0.1	3	3	3	3
Ag	1700	100	0.12	500	500	500	500
Total				**515**	**523**	**543**	**703**

Samples with mentioned structures have been used for the EDS elemental analysis of the perpendicular incidence. After deposition, the samples were placed in the working chamber of the SEM microscope for analysis by standardless method. Imaging results were obtained with a Philips XL 30 ESEM TMP microscope and X-ray spectra generated were determined and analyzed by an energy dispersive spectrometer EDAX Sapphire, with a resolution of 128eV, at University "Politehnica" from Bucharest. Depending on electron beam accelerating voltage and the analyzed elements K, L and M emission lines were used.

We studied the effect of electron beam energy (ranging between 5 and 30kV in steps of 5kV for 150 life seconds) on the penetration depth into the nanostructurated multilayers. The dead time during the spectrum collection was kept into the range 20 - 40%. Samples were positioned at a takeoff angle (TOA)

of 35º from X-radiation detector. During tests, the working conditions were kept in order to minimize any effect on the statistical nature of the production of radiation. Using the program EDAX analysis, the results were normalized using the ratio of the intensity spectrum. The magnitude of errors should not be significant in this study, as operating conditions were optimized [15].

As the deposited layers were made from chemical elements with close Z number, the structures being similar, but with very different thicknesses, it is supposed that a correct analysis would highlight approximately double concentrations of Cu, the cumulative signal from Ni and Fe, towards the combined signal of Ni and Fe. We shall try to determine the approximate acceleration voltages recommended for analysis of nano-dimensional multilayer structures depending on the thickness of the analyzed layers. Since it is known that the penetration depths of the energy needed to obtain the characteristic X-ray touch for the micrometers order for light elements and near the size of a micrometer for heavy elements, there is the possibility that the initial assumption would not outlined in any of the studied cases. From this reason, in order to obtain at higher accelerating voltages required for K lines' excitation of the heavier elements, results closer to the assumption that in terms of concentration wt.% Cu = Ni + Fe, we performed EDS microanalysis on samples with structures according to data from Table 1. The silver has been chosen as the difference of atomic number is large enough and there are no suppositions for any spectral lines of interest.

3. Results and Discussion

X-rays are separated in the series K, L and M in accordance with layers that their electrons fill. The atom's ability to generate characteristic X-ray photons varies when it is irradiated with characteristic X-rays photons or electrons. First of all, there is a competition between the emission of X-ray photons and Auger electrons when the electron refills a vacancy in an inner layer, and secondly there is a competition between generating series K, L and M of X ray. Another difference between K, L and M lines consists in their energy levels. The K series' energy is largest than the L and M series. Generally, there is more likely to be detected L lines of heavy elements than K lines, since the latter have energies greater than 20keV [1, 8, 15].

The CASINO® software (used for this research) is based on a single scattering algorithm, and models the interaction of low energy beams and thin solid samples. The model considers that the electric charge density is uniform throughout the system and a large scanning area and a defocused beam is assumed in order to have a one-dimensional problem, where the electric field is only a function of the z axis [3, 5, 6, 7, 16].

As the electrons traverse the sample, the program had to correct the trajectories at the interface between two regions crossed. In this case, no angle deviation is calculated and a new random number is generated to calculate the distance L, from the new region. This method produces a more accurate distribution of the maximum penetration depth of electrons in homogeneous and multilayer samples having the same chemical composition as compared with the use of the same random number used to calculate the length *L* in each new region [5, 16].

The data recording were performed in three-dimensional matrices of cubic elements representing the energy loss of all simulated electrons' trajectories. One of the simulation results was the energy contour lines representation calculated from the center of the interaction and revealed the percentage of energy that is not included in the line [5].

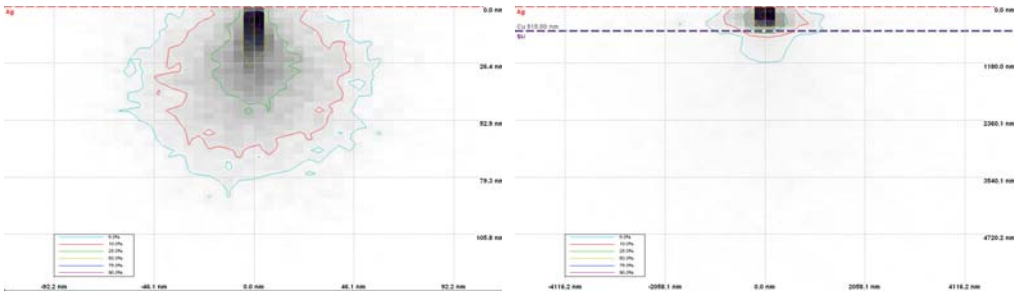

Fig. 1. Energy by position distribution in the interaction volume of the accelerated electron beam in the nanostructures Si_Cu3_Ni3_Cu3_Fe3_Ta3_Ag500 type at 5kV (left), and 30kV (right).

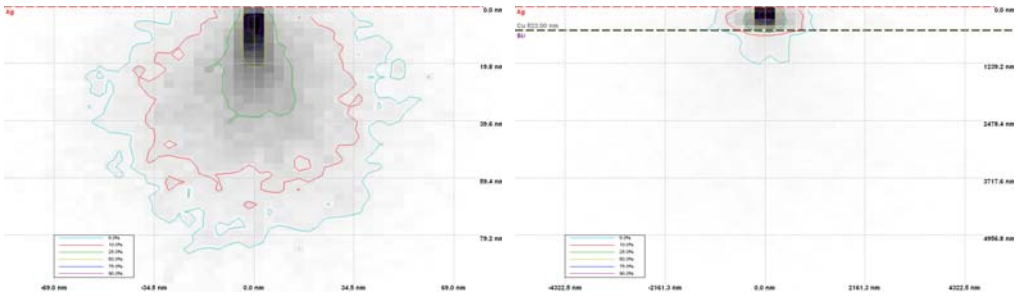

Fig. 2. Energy by position distribution in the interaction volume of the accelerated electron beam in the nanostructures Si_Cu5_Ni5_Cu5_Fe5_Ta3_Ag500 type at 5kV (left), and 30kV (right).

The lines marked by 10% represent the limit between the area that contains 90% of the absorbed energy and the rest of the sample. Absorbed energy densities are represented by different shades of grey, darker as the density is increasing [5, 16]. From the electron energy loss in the sample we were able to determine the characteristic X-ray radiation generated. X-ray intensities were

normalized as a function of depth. The function was calculated from X-ray intensity generated in a ΔZ film thickness with the same chemical composition. This information is useful for a better selection of SEM microscope parameters used in the analytical qualitative and quantitative investigation of nanofilms. The calculations used did not take into account relativistic effects, since these effects become important at energies above 50keV.

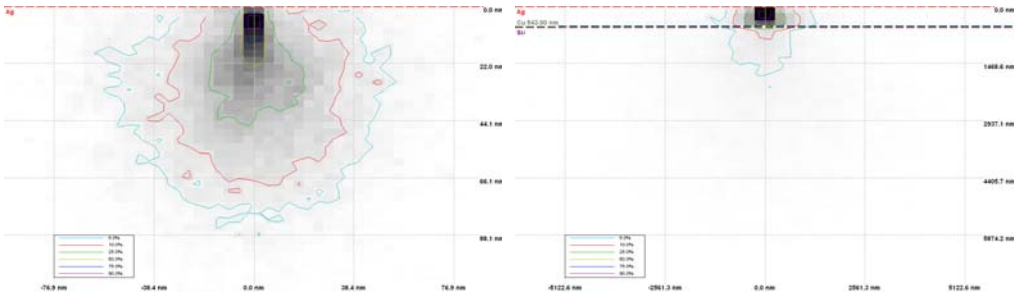

Fig. 3. Energy by position distribution in the interaction volume of the accelerated electron beam in the nanostructures Si_Cu10_Ni10_Cu10_Fe10_Ta3_Ag500 type at 5kV (left), and 30kV (right).

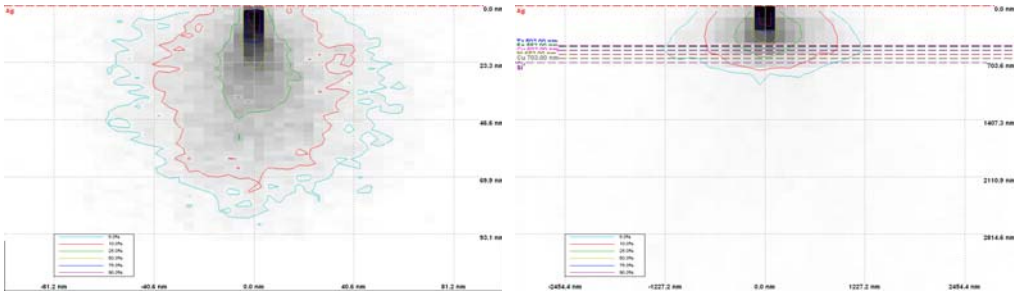

Fig. 4. Energy by position distribution in the interaction volume of the accelerated electron beam in the nanostructures Si_Cu50_Ni50_Cu50_Fe50_Ta3_Ag500 type at 5kV (left), and 30kV (right).

Quantitative provided data by the simulations of energy by position distribution have allowed drawing the depth variation curves of the characteristic X-ray emission and identification of layers that have been aroused in the application of voltages to accelerate electron beams between 5 and 30kV.

The differences between the curves drawn after the model simulation without Ag500nm additional coating layer and those of the cover layer are significant, to 30kV level the variations are between 2μm and 700nm, for individual layers of 50nm, 4.5μm and 600nm, for layers of 10nm, 620nm and 5.5μm, for layers of 5nm, between 5.7μm and 680nm or for 3nm layers.

For all accelerating voltages, the general appearance of the variation curves of the emission depth has a much smaller dispersion simulation for interaction with the Ag coated structures than with simple structures.

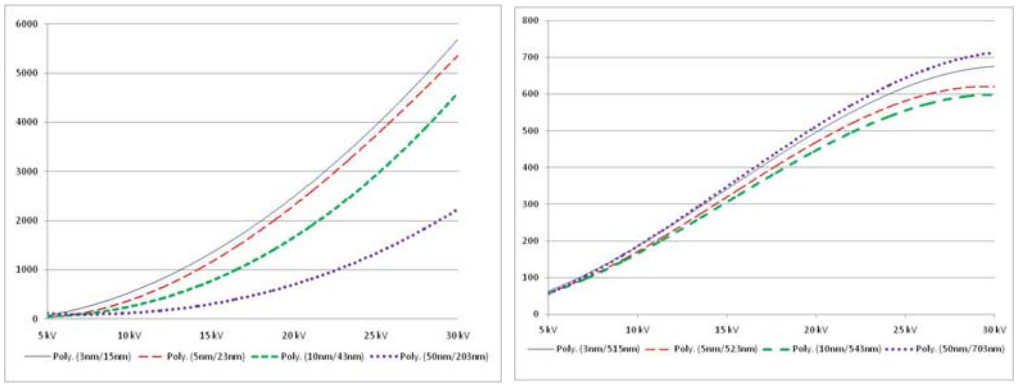

Fig. 5. Graphic representation of Monte Carlo simulations (energy by position distribution) regarding depth variation of X-ray signal source, versus deposited layers thickness and e-beam acceleration: Cu-Ni-Cu-Fe-Ta layers (left), with 500nm cover layer of Ag (right).

Overcoming the stage of simulation modeling, EDS analysis of samples with Ag, Ni, Fe and Ta resulted in six X-ray emission spectra for each set of samples, with acceleration voltage of electron beam of 5, 10, 15, 20, 25 and 30 kV, starting from perpendicular incidence to the direction of the electron beam, to an angle of 35° TOA to X-ray detector of the spectrometer. The results were used for plotting the variation curves of elemental concentrations (wt. %) versus e-beam acceleration voltage.

In terms of physical phenomena, when high energy electrons are interacting with a sample, they produce elastic or inelastic dispersals. The elastic dispersals produce retro-dispersed electrons (BSE), which are actually scattered electrons within the atoms in the sample. Inelastic dispersals produce secondary electrons (SE), which eject electrons from the sample's atoms. The electron scattering/dispersal leads to change in the direction of penetration of electrons in the sample surface. The interaction between the accelerated electrons of the beam and the atoms is produced in a given volume situated below the sample surface. This area is commonly described as having a pear-shaped, and its size is proportional to increasing electron energy within the sample surface [1, 8, 15].

In addition to BSE and SE electrons, in the interaction, X-rays are also produced and used in microanalysis. SE electrons have an energy level of a few keV order, and within the area of interaction they can escape only from a volume close to the surface sample from a depth of about 5–50nm, even if they are generated throughout the interaction volume [8].

In contrast, BSE electrons have an energy level close to that of incident electrons, allowing them to escape from a much deeper level of interaction volume, about 50–300nm. The spatial resolution of the SEM images is significantly affected by the large volume which shows the electron signal.

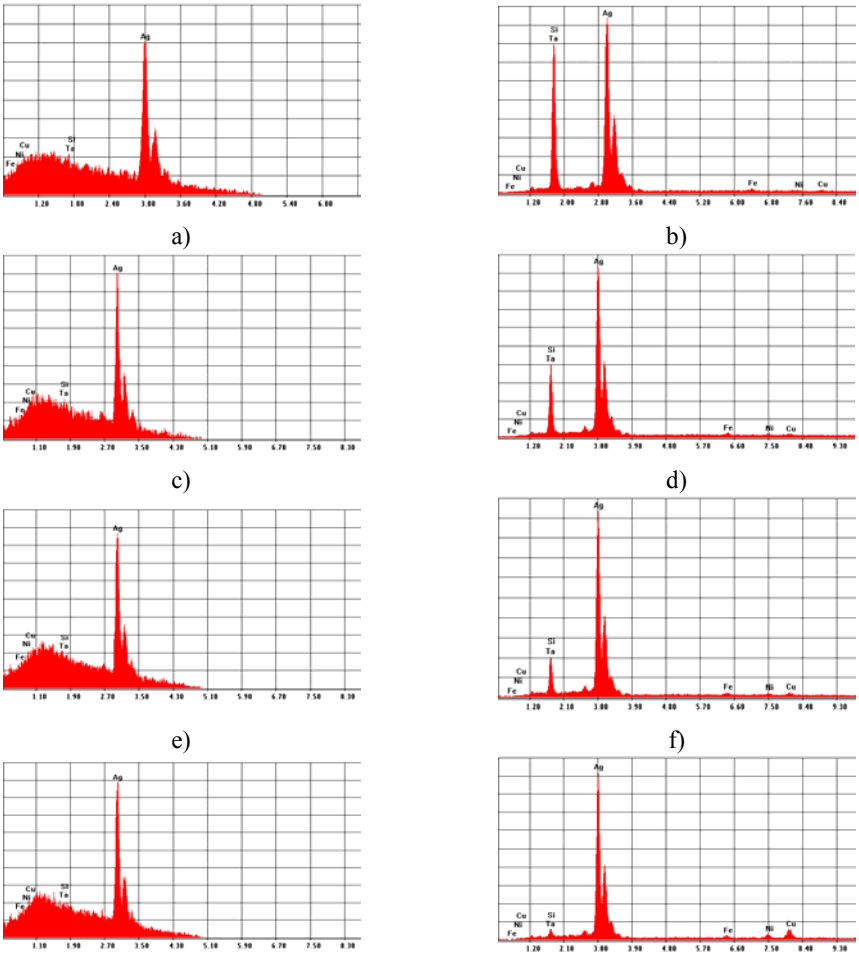

Fig. 5. X-ray emission spectra on Si, Cu (3, 5, 10, 50nm), Ni (3, 5, 10, 50nm), Fe (3, 5, 10, 50nm), Ta (3nm), and Ag500nm samples for layers having 3nm (a, b), 5nm (c, d), 10nm (e, f), 50nm (g, h), at 5kV (left side) and 30kV (right side).

The spectral analysis implied the X-ray emission measurements, which after processing and analysis provided, the compositional results are used to plot graphs of composition variation versus electron beam acceleration voltage. Due to

limited space, we will present in this paper only the spectra obtained with the extreme values of the electrons acceleration voltage of 5 and 30kV, respectively.

The use of high acceleration voltage enabled the excitation of K lines of Fe, Ni and Cu, this being impossible at voltages lower than 10kV. When using the acceleration voltage of 5kV only L and M excitation lines of the examined elements are observed. Due to very small thickness of the samples, the peaks characteristic to the elements' K lines are very low, and thus the lower energy lines signal summing was also necessary in order to obtain more accurate quantitative results.

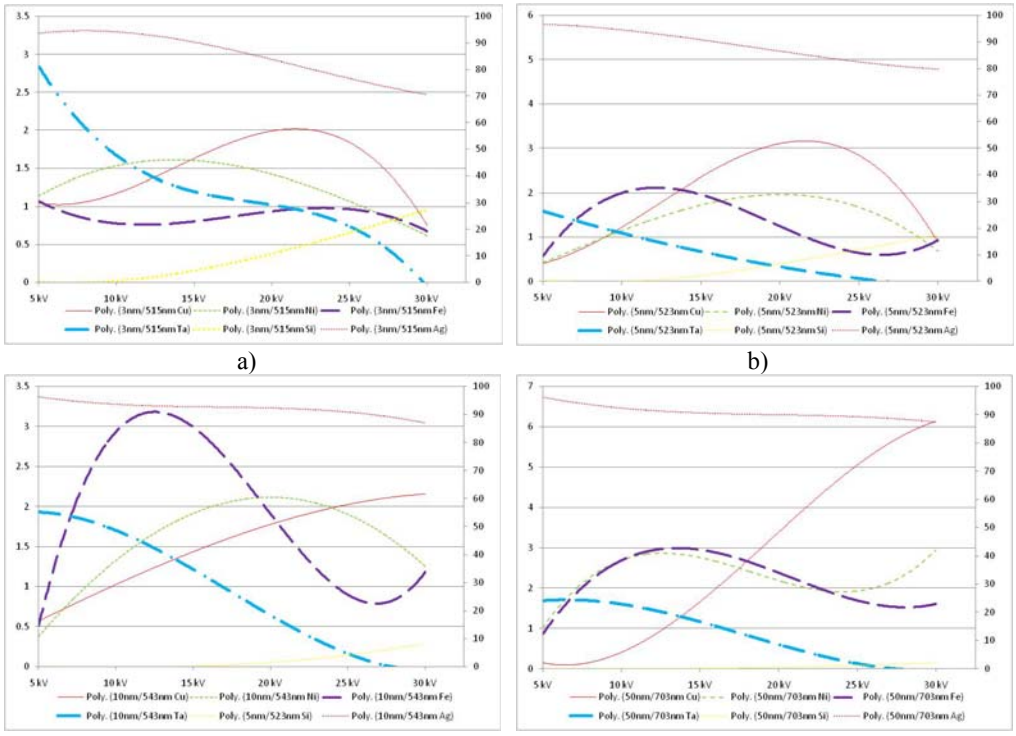

Fig. 6. Mass concentration variation of Si, Cu (3, 5, 10, 50nm), Ni(3, 5, 10, 50nm), Fe(3, 5, 10, 50nm), Ta(3nm), and Ag500nm samples, for the layers having 3nm (a), 5nm (b), 10nm (c) and 50nm (d).

For the first nanostructure, with individual layers thickness of 3nm and a total of 515nm, it has been observed the increase of Si concentration in substrate from nearly 0% for 5kV, 4% for 15kV, and 27% for 30kV, respectively. So, the high voltage energy is big enough to penetrate all layers, and the signal showing the characteristic X-rays emerged from the substrate. In contrast, the Ta layer situated just under the Ag deposition, has played the role of X-ray signal barrier

corresponding to the drop in concentration from 3% for 5kV, 2% for 15kV, and 0% for 30kV, respectively; so, the energy required for atoms excitation was too large considering the 500nm magnitude of Ta layer. Unlike Ta, in this case, Fe layer gave a signal approximately equal, regardless of the voltage acceleration of around 1%, with a slight decrease for 30kV. The displayed signal of the Ni layer, deposited between two layers of copper, ranged from 1% for 5kV, about 2% for 10, 15 and 20kV, followed by a decrease of 0.5% from 25 to 30kV, so the energy needed for the atomic excitation of the elements of interest layers are found between 10 and 20kV; up to 10kV the K lines of atoms were not excited. Cu reproduced a constant signal around 1% for the corresponding energies of 5, 10 and 15kV to 20kV, observing a threefold increase in the concentration, followed by a reduction of 1% for 25 and 30kV, which may be the appropriate excitation of the Cu layer around 20kV.

Increasing the thickness of individual layers to 2nm has reduced the Si concentration of substrate about 10% for the big accelerating voltages, at 5 and 10kV voltage being zero. The displayed signal from the thin layer of Ta was around 2% for 5 and 10kV, decreasing gradually to zero over 15kV, so, in this case, the small voltages are sufficient to excite the Ta lower lines, at depths of 500nm. After a value of 5% for 5kV, Fe showed a concentration of 2% for 10kV, gradually decreasing to 1% for 30kV. As the concentration of Ta, Fe showed the maximum value for 10kV; it is possible that this energy to be sufficient to excite the atoms of this layer, but only on the L line. Unlike Ta and Fe, Ni presents the maximum concentration of 5% for 20kV, increasing with 0.5% from 5 to 15kV; for 20kV, the concentration value decreases in the same way than being dealt with obvious excitement level at the 20kV voltage.

Doubling the thickness of the nanodimensional layers there was obtained a significant reduction in the concentration of Si, from 0% for 5 and 10kV to a maximum of 8% for 30kV, which represents one half of the concentrations, compared with the situations previously analyzed. However, the same trend was revealed for the Cu layer, starting from 0% for 5kV, and reaching a constant value of 2% for 25 and 30kV; in this range there is enough energy for the excitation of the elements for 540nm layers. The analysis of Fe presents maximum concentrations for 10 and 15kV, around 3% concentration, increasing from 0.5% for 5kV, followed by a decrease of 1% for 20, 25 and 30kV. So, we may conclude that this layer is sufficiently excited at voltages in the range 10–15kV, which allows the excitation of K lines. Ni reaches the maximum concentration of 3% for a 20kV accelerating voltage, after a 0.5% increase of every 5kV, and 25 and 30kV showing concentrations of approximately 1.5%. In this situation, it could be observed a direct correlation between the depth at which the signal had been detected by X-rays and the acceleration voltage of the electron beam.

By increasing individual layer thickness up to 50nm, reaching a total thickness of 703nm, there was an increase of Si concentration with 0.5% each 5kV; above the 20kV threshold the concentration was zero, so the electron beam energy was not sufficient to penetrate the nanolayers. The Ta concentration was maximum (2%) for 5kV and decreased gradually until it was canceled over 25kV. The Cu layers attended maximum concentrations for 30kV voltage, increasing by 1% every 5kV, so the maximum beam energy was enough to excite elements from all layers. The Ni layer, deposited between two layers of copper, gave a maximum signal for 25kV, at around 2.5% and 4% Fe layer for 15kV. As well as the previously analyzed structure, the thicker layers confirmed the intensity increase of the X-ray signal in the same time with both the increase of the depth of the deposited layer and of the energy intensity electron beam.

4. Conclusions

Using *Energy by Position* distribution function, variations in the characteristic energy emission levels of different EDS types of signals have been highlighted. It may be noted that, predominant X-ray signals used in EDS microanalysis comes from the proximity of the characteristic lines of 10% energy contour. This distribution function showed that when low accelerating voltages (5-10kV) are used, the volume of interaction and, consequently, the X-ray emission area covers also the deposited layers, which confirms the achievement of the L and M lines peak emission for small energies.

In cases of layers covered by 500nm Ag, it was observed, especially at high accelerating voltages, the fact that the concentrations relationship of Cu = Ni + Fe is followed in most cases, except the thinnest layers of 3nm, at which it has established an intermediate relationship of the form 1.5 Cu = Ni + Fe. From 5nm thick layers, the relationship of proportionality was revealed initially at 30kV, the layers of 10nm to 25 and 30kV, and the thick layers from 20kV.

By using additional coating layer of 500nm Ag nanostructures, it has become possible to analyze the nanodimensional multilayers at the energy electron beam acceleration levels of 25 and 30kV, which allows the excitation of K lines of heavy elements and determines a proper proportion between the factors used for deposition. There is the disadvantage of higher possibility of errors when calculating the full disposal of surface sites occupied by peaks corresponding to Ag, because, in this case, the background correction signal is less precise, and peak area sites allocated to the items of interest is lower. From data analysis, it can be concluded that, depending on the chemical elements analyzed and thick layers that they form, additional coatings can be used, with hundreds of nanometers thick, of various components, to study quite precisely the proportion

of chemical elements submitted as nanolayers and depth to which they are located.

From Monte Carlo simulations it can be seen that the layer's atomic number strongly affects the distribution of X-ray radiation generated in the sample. The effects are even more complex when several layers are considered. Based on the above experimental results and simulations, the direct influence of accelerating voltage (electron beam energy) and atomic number on the detected X-ray quantity, was highlighted. When the acceleration voltage is too low, X-rays will not be released because the overvoltage was not reached. As the accelerating voltage increases, both the penetration depth and the size of interaction volume increases, resulting in an increased detected signal. Among other things, the excitation volume defines the area from which X-ray radiation comes.

The metal layer thickness is in an almost linear relationship with the energy required for electron beam penetration. Based on the experimental results and mathematical models applied in Cu-Ni-Cu-Fe-Ta-Ag multilayers study, relations between the detected signal intensity function of the incidence electron beam acceleration voltage were established. If high primary electrons energy is applied, there will be more inelastic collisions, and the penetration depth and lateral spreading will be higher. Inelastic scattering probability decreases with increasing atomic number, thus the penetration depth and spreading are lower for heavier elements. The amount of interaction was simulated and determined using a Monte Carlo model.

The original simulation results are in good agreement with experimental data. The combined model, simulation – experiment, proposed in this paper and the type of coverage the nanolayers with thicker layers of chemical elements with high possibilities to mitigate the X-ray beams energy provides important data and constitutes an early stage of a field with real development opportunities. Further systematic experimental studies on different multilayer models are required for a better understanding of the X-ray production and detection phenomena in EDS microanalysis.

Acknowledgements

Authors recognize financial support from the European Social Fund through POSDRU/89/1.5/S/54785 project: "Postdoctoral Program for Advanced Research in the field of nanomaterials".

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