

REGULAR ARTICLE

SIMS Analysis of Copper-Nickel Thin Films Alloys

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The article presents the results of studying the elemental and isotopic composition of alloy films based on Cu and Ni films by the method of secondary ion mass spectrometric analysis (MS-7201 M secondary ion mass spectrometer). Films of alloys with a thickness of up to 130 nm were obtained on polished glass substrates with a pre-applied Al buffer layer by simultaneous separate evaporation of the components in a vacuum of 10^{-4} Pa. Copper was evaporated from a strip of tungsten foil with a thickness of 0.05 mm. Nickel was evaporated by the electron-beam method using an electron diode gun. The rate of condensation was 0.5-1.5 nm/s. The purity of evaporated metals was at least 99.98 %. Ar^+ ions with an energy of 5 keV were used as probing primary ions. The results of qualitative mass spectrometric analysis of secondary ions indicate the high purity of the films (absence of hydrides, oxides and carbides of Cu and Ni). The elemental composition of the films is represented by isotopes Ni^{58} , Ni^{60} and Cu^{63} , Cu^{65} . The ratios of isotopic intensities are $I_{\text{Ni}^{58}}/I_{\text{Ni}^{60}} = 2,6$ and $I_{\text{Cu}^{63}}/I_{\text{Cu}^{65}} = 2,3$, which corresponds to the natural distribution of nickel and copper isotopes. The ratio of isotopic intensities $I_{\text{Cu}^{63}}/I_{\text{Ni}^{58}}$ practically does not change over the entire thickness of the sample. It was shown that the quantitative analysis of the elemental composition of film alloys can also be carried out by the method of secondary ion mass spectrometry.

Keywords: Secondary ion mass spectrometric analysis, Thin films, Copper-nickel alloys.

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1. INTRODUCTION

Large-scale study of the physical properties of thin metal films have led to the emergence in recent decades of a new branch of solid state physics – thin film physics. Advances in the physics of thin films have found wide use in nanoelectronics, high-frequency technology, optoelectronics, optics and many other areas of modern science and technology.

Films of magnetically ordered metals Ni, Fe, Co and alloys based on them occupy a special place in the physics of thin films: solving the fundamental problems of “two-dimensional magnetism”, giant magnetoresistance, spin-polarized tunneling, colossal magnetoresistance, etc. These discoveries are widely used in nanoelectronics and spintronics for the development of miniature magnetoelectronic devices, new methods for recording and storing information, and new types of sensors [1-6].

In contrast to the thoroughly studied technologies for producing bulk alloys, the technologies for producing film alloys still remain not fully studied. A feature of technologies for producing film alloys in vacuum is the appearance of some uncertainty in the formation of a sample with a predetermined chemical composition. Studies of the chemical composition of film alloys using methods that are effective for bulk alloys cause certain difficulties due to the extremely small amount of the analyzed substance. The modern most widely used methods for studying the chemical composition of film

alloys are X-ray spectral microanalysis and secondary ion mass spectrometric microanalysis. Both methods have their advantages and disadvantages.

X-ray microanalysis of thin films of metal alloys [7] does not provide information about the presence of hydrides, oxides, nitrides and other chemical compounds of alloy components in their composition. These compounds can be formed as a result of the interaction of active gases of the residual atmosphere with the film material both during the preparation of samples and during their heat treatment. The formation of such compounds leads to a significant change in the electrical properties and structure of film samples.

One of the most sensitive modern methods for studying surfaces and thin films is the method of secondary ion mass spectrometry (SIMS), which is also often called ion microanalysis [8-9].

2. METHOD OF SECONDARY ION MASS SPECTROMETRY

The SIMS method is based on the study of particles on the surface of a solid body, knocked out of it by a narrow beam of accelerated (probing) gas ions. For this purpose, both inert (most often Ar) and non-inert (most often H_2 and O_2) gases with energies of 1-20 keV are used. The probe ions are called *primary ions*. At such energies, primary ions penetrate into the substance under study to a depth of several nanometers, which corresponds to several atomic layers of the substance. To

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a first approximation, the interaction of primary ions with the surface of a substance can be considered as independent pair collisions “probing ion – surface atom”. The results of such collisions (Fig. 1) are the following processes [8-9]:

- 1) *heating of the target* – inelastic interaction with absorption of the energy of the probe ion;
- 2) *backscattering of primary ions* – elastic interaction with the reflection of the probe ion by the target surface;
- 3) *secondary ion emission* – knocking out atoms from the target surface in the form of positive and negative ions (surface sputtering);
- 4) *secondary atomic emission* – knocking out neutral atoms from the target surface (surface sputtering);
- 5) *secondary electron emission* – tearing out electrons from the target surface;
- 6) *secondary photon emission* – emission of light quanta from the target surface in a wide range of wavelengths (from infrared to X-ray).

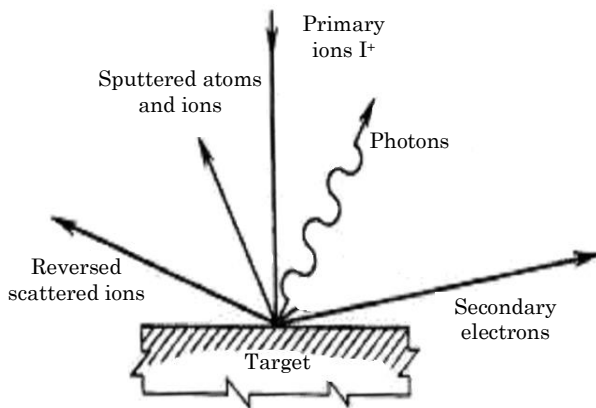


Fig. 1 – Scheme of interaction of probe ions with a target

All these types of emission from a target can, in principle, be used to analyze the composition of the target substance.

Typically, only about 5 % of the energy of the primary ions is used to knock out atoms and ions (surface sputtering). The rest of their energy goes to heating the target and is carried away by scattered (reflected) probe ions.

Some of the knocked-out target atoms escape from it in the form of positive ions, which are called *secondary ions*. They are used for mass spectrometric analysis of the chemical or isotopic composition of the target substance. This fact is reflected in the name of the method – SIMS. The process of producing secondary ions using the method described above is often called *ion etching* or *ion sputtering* of the target surface. Despite the fact that both of these names are not entirely successful, since they do not fully reflect the physical essence of the phenomenon, they are widely used in the scientific literature on secondary ion mass spectrometry.

As is known, the mass spectrometric method for analyzing the chemical and isotopic composition of a substance is based on measuring the mass of molecules (atoms) of this substance with special devices – mass spectrometers. Positive ions knocked out of the target surface are sent to the analyzer of the mass spectrometer, in which they are separated by mass

(mass numbers), after which the ion currents created by them are measured. At the same time, the masses of these ions are measured. The SIMS method can use negatively charged ions knocked out of the target and neutral atoms knocked out of the target after their additional ionization. But the easiest way to analyze the composition of the target surface is to use ready-made positive ions knocked out of the target. This significantly simplifies the design of the mass spectrometer due to the absence of an ion source.

To separate secondary ions by mass, you can use any of the existing mass analyzers, both magnetic static and dynamic (time-of-flight, quadrupole or monopole). Magnetic static mass analyzers have much better analytical characteristics than dynamic ones, but they are bulky, difficult to maintain and expensive. Because of this, dynamic mass analyzers are most often used in secondary ion mass spectrometers.

The features of the SIMS method allow to study both metals, semiconductors, and dielectrics, which makes this method a universal method of analysis.

The efficiency of the release of secondary ions from the target surface is characterized by the *surface sputtering coefficient* a_s (the number of secondary target ions knocked out by one probe ion). It has been experimentally established that the a_s coefficient is different for different substances and is a characteristic of the “probing ion – surface material” pair. For example, when bombarding a tungsten surface with Ar^+ ions, a_s is 2-3 times less than when bombarding an aluminum surface with the same Ar^+ ions.

The surface sputtering coefficient a_s also depends on the kinetic energy, mass, electronic configuration and angle of incidence of the primary ions, as well as on the crystal structure of the target, the binding energy of the target atoms and the state of its surface (cleanliness of processing). As a rule, the temperature of the surface under study does not play a significant role [8-10].

It was also established experimentally that the surface sputtering coefficient a_s periodically changes with increasing atomic number of the elements of the target substance.

The interaction between primary ions and target atoms is complex and not fully understood. The interaction of primary ions and target atoms is described most simply, according to the type of classical interaction of billiard balls, if the bombarding ions are ions of inert gases (He^+ , Ar^+ , Kr^+), and the targets are noble metals (Ag, Au). In the case of using ions of non-inert gases and targets with complex composition (alloys, dielectrics, semiconductors), the situation becomes significantly more complicated.

Another parameter characterizing target sputtering is the *sputtering rate* v_s of the material of the target being analyzed (the amount of sputtered target material per unit time). The sputtering rate v_s depends on the sputtering coefficient a_s , the magnitude of the ion current of primary ions I^+ and can be given by an empirical formula in the form [10]:

$$v_s = 6 \cdot 10^{-2} \cdot \frac{\alpha_s I^+ A}{d}, \quad (2.1)$$

where A is the atomic or molecular mass of the target substance; d is the density of the target material.

Consequently, by bombarding the target surface with primary ions of certain energies, it is possible to etch the target layer by layer and simultaneously analyze the chemical composition of the ions knocked out of the target substance using a mass spectrometer. In this way, it is possible to obtain information about the distribution of elements both on the surface and throughout the depth of the target. The area of the surface from which sampling is carried out for mass spectrometric analysis (probe size) in relatively simple devices has a round shape with a diameter of 1-2 mm. The current strength of the primary ion beam is 10^{-5} - 10^{-6} A. Under such conditions, the current of secondary ions is 10^{-11} - 10^{-16} A.

In devices of a higher complexity group, designed to analyze the distribution of microimpurities both over the surface of the sample and in its depth (semiconductor technologies, nanotechnologies, etc.), probe ions focused into a very narrow beam scan the target surface in a certain order. The diameter of their ion probe is about one micron. By scanning with such a probe a microsection of the surface of a sample with an area of, for example, $100 \times 100 \mu\text{m}^2$ or $500 \times 500 \mu\text{m}^2$, it is possible to simultaneously analyze the composition of the surface of this area and observe the morphology of the sputtered surface using electrons ejected from it as a result of secondary electron emission.

To summarize the above, we note the most important features of the secondary ion mass spectrometric analysis method:

- high sensitivity (for modern instruments, the consumption of the substance for analysis is $\sim 10^{-14}$ - 10^{-15} g, which corresponds to 1-2 surface monolayers);
- the possibility of isotopic and chemical analysis of the composition of the target in a wide range of atomic masses (all elements from hydrogen to transuranium are recorded);
- the method is close to directly analyzing the surface of a solid body itself, since the main information about the composition of the material comes from the near-surface region with a thickness of 10-20 Å;
- not only individual chemical elements are identified, but also their chemical compounds (hydrides, carbides, oxides, etc.).

At the same time, the method of secondary ion mass spectrometric analysis has some disadvantages:

- difficulties in accurately quantitatively determining the elemental composition of a sample, due to the dependence of the probability of ions leaving the sample on their environment;
- destruction of the sample under study during the analysis process, as a result of which uncontrolled mixing and segregation of atoms on its surface is observed (the method of secondary ion mass spectrometric analysis is a destructive method).

In conclusion, the following should be noted. The method of secondary ion mass spectrometry has been further developed in recent decades in the direction of expanding methods for the formation of secondary ions. To excite secondary ions, beams of molecules and clusters began to be used (molecular and cluster secondary ion mass spectrometry), which made it possible to use the method of secondary ion mass spectrometry in biology and medicine [11-12].

3. EXPERIMENTAL METHODOLOGY AND TECHNIQUE

To obtain films of copper-nickel alloys with different concentrations of components, we used the method of forming film samples directly on the substrate by separate evaporation of components, described in detail in [13].

To study the chemical and isotopic composition of copper-nickel alloy films, an MS-7201 M secondary ion mass spectrometer was used, the schematic illustration of which is shown in Fig. 2.

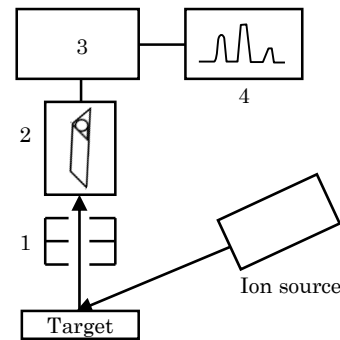


Fig. 2 – Schematic illustration of MS-7201 M

Ar^+ ions obtained in the ion source (Fig. 2), accelerated to energies of 5 keV, were used as probing primary ions. Secondary ions knocked out of the film target are collected by a system of electrostatic lenses (1), focused into a narrow beam and sent to a monopole mass analyzer (2). In a mass analyzer, ions are temporarily separated depending on the ratio of their mass m to charge q . Ions with the same m/q ratio create an ion current in the detection system (3), determined by the expression [10]:

$$J_A^+ = h_A S_A^+ J_T, \quad (3.1)$$

where J_A^+ is the ion current of monoisotopic element A ; h_A is the detection efficiency of ions of this isotope in a given device; S_A^+ – coefficient of secondary ion emission of element A in the sample matrix; J_T is the total current of the primary ion beam incident on the sample.

After amplification, the ion currents are recorded by a recording potentiometer (4) in the form of a mass spectrum.

Films of copper-nickel alloys for studying the elemental composition by SIMS were deposited on polished glass substrates with an Al buffer layer. A conductive layer of aluminum is necessary to remove the electrostatic charge from the sample under study when it is bombarded with argon ions.

Interpretation of the mass spectra of secondary ions obtained as a result of sputtering of samples with primary Ar^+ ions was carried out according to the method described in [10].

As a rule, interpretation of the mass spectrum occurs in three stages. At the first stage, the presence of mass spectrometric peaks is established and their list is compiled depending on the mass number $M = m/q$. At the second stage, peaks are identified based on the isolation of monatomic ions (and their isotopes), molecular ions and their fragments depending on the mass number M . At the third stage, a list of identified elements and compounds is

compiled in accordance with their atomic concentrations (taking into account the isotopic composition of the elements).

The first two stages form the essence of qualitative analysis. If it is necessary to conduct a quantitative analysis, it is necessary to take into account the dependence of the secondary ion emission coefficients S_A^+ on a number of factors: the state of the surface, the chemical nature and crystal structure of the sample matrix, etc. As a rule, the secondary ion emission coefficients are determined using a standard, having a chemical composition close to the sample under study. To determine S_A^+ you can use the following relation [10]:

$$S_A^+ = S g_A^+ C_A, \quad (3.2)$$

where S is the total sputtering coefficient of the material (the number of atoms per primary ion); g_A^+ – the ratio of the number of secondary positive ions of element A to the total number of neutral and charged sputtered particles of the element; C_A is the atomic concentration of a given element in the sample.

The values of g_A^+ and S are determined by the composition of the sample matrix (the binding energy of the atoms of the solid) and will also be determined experimentally.

Combining (3.1) and (3.2) for the sample and standard, we obtain the relation:

$$C_A^+ = \frac{J_A^+}{J_{ST}^+} C_{ST}^+ = \gamma C_{ST}^+, \quad (3.3)$$

according to which it is possible to calculate the composition of the sample under study. In this case, the standard must have a similar chemical composition to the sample and the same structural state. For the analysis of film samples, the standard must also be in the form of a film.

Carrying out a quantitative analysis using relation (3.3) involves a sequential analysis of the composition of the standard and sample with constant parameters of the primary ion beam (composition, energy, beam current). When studying a binary copper-nickel alloy, to determine the coefficient γ , both direct ratios J_{ST}^{Ni}/J_{ST}^{Ni} (J_{ST}^{Cu}/J_{ST}^{Cu}) and cross ratios J_{ST}^{Ni}/J_{ST}^{Cu} (J_{ST}^{Ni}/J_{ST}^{Cu}) can be used equally effectively.

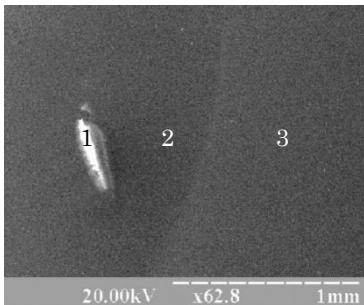


Fig. 3 – Micrograph of a section of the film after SIMS analysis: 1 – crater bottom (substrate); 2 – crater slope; 3 – area outside the analysis zone

As already noted, the method of secondary ion mass spectrometry is destructive. The primary beam of ions, bombarding the surface of the sample, pulls out atoms and molecules from it, creating a crater. In Fig. 3 shows, as an

illustration, an electron microscopic image of a section of a CuNi alloy film with a thickness of $d = 130$ nm after secondary ion mass spectrometric analysis of its composition. The target was sputtered with a primary beam of Ar^+ ions for 10 min at a sputtering rate $v_s \approx 15$ nm/min. The left side of the image shows part of a drop-shaped crater (2), at the bottom of which the substrate (1) is visible. As a rule, there is a small electron microscopic contrast between areas of the film subjected (2) and not subjected (3) to bombardment by argon ions, which can be observed in the secondary electron mode. This allows you to estimate the shape and size of the analysis zone.

4. RESULTS AND DISCUSSION

In Fig. 4 shows the mass spectrum of secondary ions from the surface of the studied film alloy with a thickness of $d \approx 100$ nm ($C_{Cu} = 55$ at.%) on a glass substrate with an intermediate layer of aluminum.

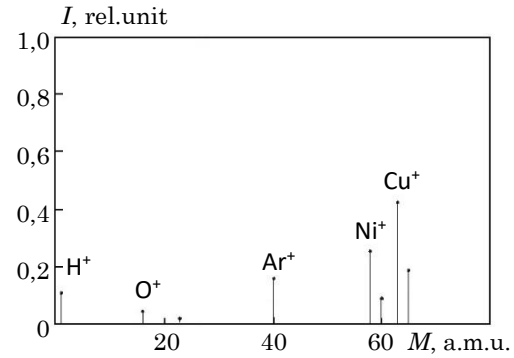


Fig. 4 – Mass spectrum of secondary ions from the CuNi film surface

From the mass spectrum it is clear that the film is quite clean: there are no oxides, nitrides and other chemical compounds based on Cu and Ni. Peaks H^+ , O^+ , and sometimes H_2O^+ are usually present in the mass spectra of secondary ions of any samples. The reason for their appearance is the presence of water molecules on the surface of the film, sorbed during exposure of the sample to atmospheric air. The Ar^+ peak corresponds to the composition of the reflected primary ion beam. The intensities of all these peaks vary randomly from sample to sample and do not provide information about its true chemical composition. The Ni^+ and Cu^+ peaks, represented by two isotopes (Ni^{58} , Ni^{60} and Cu^{63} , Cu^{65}), reflect the elemental composition of the film sample.

Analysis of the obtained mass spectra showed that in CuNi film alloys, as a rule, the isotope ratios are $I_{Ni}^{58}/I_{Ni}^{60} = 2.6$ and $I_{Cu}^{63}/I_{Cu}^{65} = 2.3$, which is in good agreement with the tabulated data ($I_{Ni}^{58}/I_{Ni}^{60} = 2.59$; $I_{Cu}^{63}/I_{Cu}^{65} = 2.24$ [13]) of the natural abundance of nickel and copper isotopes.

It should be noted that as the film of the CuNi alloy was sputtered with Ar^+ ions, we did not observe a redistribution of the intensities of the lines of the corresponding Cu^+ and Ni^+ isotopes over depth (Fig. 5). This allows us to speak about the sufficient homogeneity of the composition of film alloys obtained by the method of simultaneous separate condensation of components in terms of thickness. As can be seen from the figure, the ratio

of the intensities of the isotope peaks $I_{\text{Cu}}^{63}/I_{\text{Ni}}^{58}$ practically does not change as the crater deepens. The results we obtained are in good agreement with the known literature data [10], according to which Cu and Ni, when used for analysis of a primary beam of Ar^+ ions, have close values of sputtering coefficients ($\alpha_0^{\text{Cu}}/\alpha_0^{\text{Ni}} = 1.16$).

The MS-7201 M secondary ion mass spectrometer we use, due to its analytical characteristics, is intended for high-quality express control of the chemical and isotopic composition of the surface of a solid. The sensitivity of this device (the amount of substance for a confident analysis of the composition of the target is several picograms) allows, using film standards, to obtain quantitative results for determining the composition of the film in a small range of component concentrations. The use of standards is mandatory in such measurements, since there is no reliable data in the literature on the yield coefficients of elements from samples in the form of films as a result of interaction with the primary beam of argon ions.

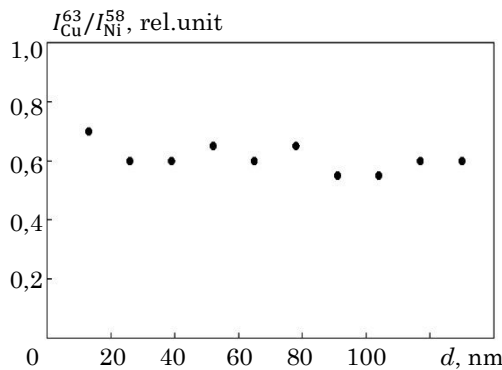


Fig. 5 – Change in the $I_{\text{Cu}}^{63}/I_{\text{Ni}}^{58}$ ratio during SIMS analysis of the film ($d = 130$ nm, $C_{\text{Cu}} = 40$ at.%)

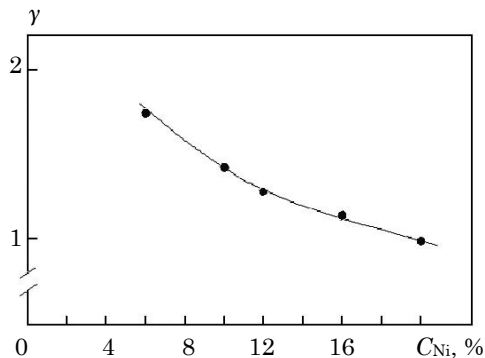


Fig. 6 – Calibration curve for CuNi films

To carry out a quantitative analysis of the composition of films of copper-nickel alloys, the mass spectrometer was calibrated. Film samples with a known concentration of components, determined by X-ray microanalysis [14], were used as a standard. For this purpose, a series of samples of CuNi film alloys with different Ni concentrations in the range $C_{\text{Ni}} = 6-20$ at.% was obtained. Based on the results of mass spectrometric studies, a

calibration curve was constructed – the dependence of the coefficient $\gamma = I_{\text{Cu}}^{63}/I_{\text{Ni}}^{58}$ (I_{Cu}^{63} and I_{Ni}^{58} are the mass spectrometric intensities of the peaks of the Cu^{63} and Ni^{58} isotopes, respectively) on the nickel concentration (Fig. 6).

In Fig. 7 shows fragments of the mass spectra of secondary ions from CuNi film alloys in the mass number range of 58-65 a.m.u. From the obtained mass spectra, the values of the ratio $I_{\text{Cu}}^{63}/I_{\text{Ni}}^{58}$ were found and the concentration of Ni in the film sample was determined using a calibration curve.

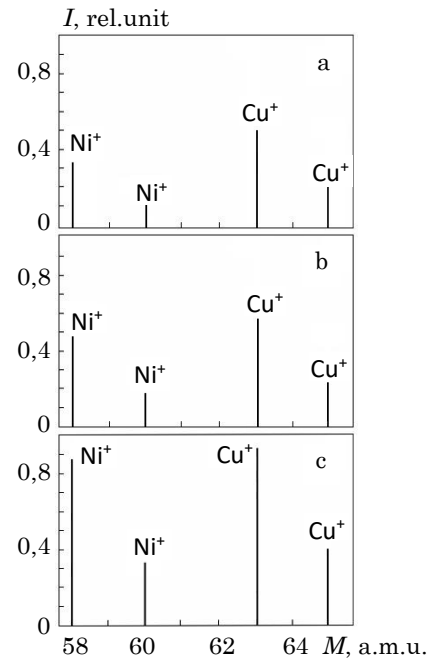


Fig. 7 – Mass spectra of secondary ions from the surface of CuNi films: Ni concentration, at. %: 8.8 (a); 15.0 (b); 19.0 (c)

A comparison of the results obtained in this way with the composition determined by X-ray spectral analysis [14] shows that the error in determining the quantitative composition of CuNi alloy films by secondary ion mass spectrometry is 5-7 %.

Thus, using the SIMS method it is possible to carry out not only a qualitative analysis of film alloys, but also their quantitative analysis with fairly good accuracy, with the obligatory presence of film standards.

5. CONCLUSIONS

1. The results of studying the elemental composition of film alloys using secondary ion mass spectrometry indicate the high purity of films of copper-nickel alloys and the uniformity of their composition over thickness.

2. A comparison of the results of determining the composition of film alloys using the SIMS method with the results of X-ray spectral analysis shows that the error in determining the quantitative composition of films of CuNi alloys using secondary ion mass spectrometry is 5-7%.

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ВІМС аналіз тонких плівок мідно-нікелевих сплавів

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У статті наведено результати дослідження елементного та ізотопного складу плівок сплавів на основі Cu і Ni методом вторинно-іонного мас-спектрометричного аналізу (вторинно-іонний мас-спектрометр MS-7201 M). Плівки сплавів товщинами до 130 нм були отримані на скляних полірованих підкладках з попередньо нанесеним буферним шаром Al одночасним роздільним випаровуванням компонент у вакуумі 10^{-4} Па. Мідь випаровувалася зі стрічки з вольфрамової фольги товщиною 0,05 мм. Нікель випаровувався електронно-променевим способом за допомогою електронної діодної гармати. Швидкість конденсації становила 0,5-1,5 нм/с. Чистота випаровуваних металів становила щонайменше 99,98%. В якості зондуючих первинних іонів використовувалася іони Ar^+ з енергією 5 кеВ. Результати якісного мас-спектрометричного аналізу вторинних іонів свідчать про високу чистоту плівок (відсутність гідридів, оксидів і карбідів Cu та Ni). Елементний склад плівок представлений ізотопами Ni^{58} , Ni^{60} та Cu^{63} , Cu^{65} . Відношення ізотопних інтенсивностей складають $I_{Ni^{58}}^{58}/I_{Ni^{60}}^{60} = 2,6$ та $I_{Cu^{63}}^{63}/I_{Cu^{65}}^{65} = 2,3$, що відповідає природній поширеності ізотопів нікелю та міді. Відношення ізотопних інтенсивностей $I_{Cu^{63}}^{63}/I_{Ni^{58}}^{58}$ практично не змінюється по всій товщині зразка. Було показано, що методом вторинно-іонної мас-спектрометрії можна проводити також і кількісний аналіз елементного складу плівкових сплавів.

Ключові слова: Вторинно-іонний мас-спектрометричний аналіз, Тонкі плівки, Мідно-нікелеві сплави.