

Non-destructive testing of nanomaterials by using subminiature eddy current transducers

S F Dmitriev¹, A V Ishkov², A O Katasonov¹, V N Malikov¹, A M Sagalakov¹ and L I Shevtsova³

¹ Altay State University, 61, Lenin str., Barnaul, 656049, Russia

² Altay State Agrarian University, 98, Krasnoarmeyskiy ave., Barnaul, 656049, Russia

³ Novosibirsk State Technical University, 20, Karka Marksa ave., Novosibirsk, 630073, Russia

E-mail: osys@mail.ru

Abstract. A sensor for studying nanomaterials has been developed on the basis of a transformer-type eddy-current transducer. The basic technical data are stated (the number of windings is 100-400 turns, and the value of the initial permeability of the core is $\mu_{\max} = 36000$). Measurements technique which allows high-precision measuring the electrical conductivity in thin film Ce-Nb. The electrical conductivity of Niobium-Selenium varies from $3.3 \cdot 10^5$ to $3.7 \cdot 10^5$ MS/m in the ratio of four from 4 to 60 nm.

1. Introduction

Advances in modern micro- and optoelectronics are inextricably linked with the intensive introduction of thin-film elements. The structural state and the topology of nanocrystalline materials, which include thin metal films, are one of the most important problems of modern solid-state physics. In connection with this, experimental studies of the similar materials are of fundamental importance for the understanding of the regularities of formation of nanostructures. Particularly relevant in this case is the development of new methods and devices, allowing the analysis of the structure and topology of thin films. T A de Assis and el study the effects of time-dependent substrate/film temperature in the deposition of a mesoscopically thick film using a statistical model that accounts for diffusion of adatoms without lateral neighbors which coefficients depend on an activation energy and temperature [1]. Effects of depositing atom kinetic energies and atomic composition are studied in order to predict the evolution of morphologies and the atomic structure of molecular dynamics (MD) grown thin films [2]. Recently, both the charge density wave and the superconductivity in monolayer NbSe₂ have been investigated [3-6]. In the construction industry, Nb plays an important role in the development of more resistant steels, which can contribute to reduction of total project costs of infrastructure, even if they become increasingly complex. On the other hand, the demand of the construction industry will continue to be increasingly growing due to urbanization, the population growth and replacement of obsolete infrastructure, so the need for lighter structures results in a greater use of high quality steel containing Nb [7, 8].

2. Specifications, material choices and design

While working on the improvement of the devices that implement the eddy current testing method [9, 10], subminiature eddy-current transducers which led to a significant increase in the localization of the



magnetic field were created. Reached locality allows the study of nanostructures, including nanometer thin films.

The subminiature eddy-current transducer (ECT) is connected to the sound board of a computer with special software that controls the voltage in the transformers exciting winding and also reads the voltage in the measuring winding (in arbitrary units). On the basis of preliminary calibration, these readings are converted to values of the electrical conductivity. The exciting winding (diameter $D_1 = 0.12\text{--}0.13\text{ mm}$) of the superminiature eddy-current transformer consists of ten turns of copper wire (cross-sectional area is $5\text{ }\mu\text{m}^2$). The measuring winding (diameter is $0.05\text{--}0.08\text{ mm}$) consists of 130 turns of copper wire (cross-sectional area is $20\text{ }\mu\text{m}^2$). To minimize the influence of the exciting winding on the final signal, the circuit includes a compensation winding that consists of 20 turns of copper wire (cross-sectional area is $5\text{ }\mu\text{m}^2$), connected to the measuring winding in such a way that the voltage of the exciting winding is subtracted from the result.

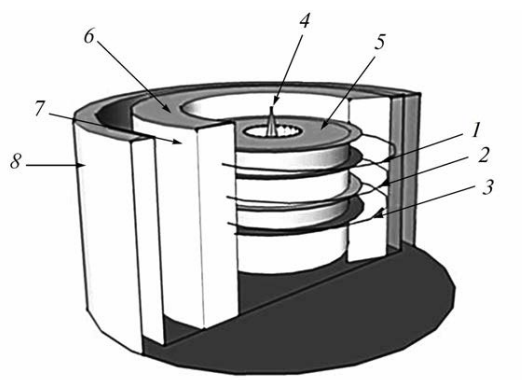


Figure 1. The scheme of the eddy current transducer.

The eddy-current transducer (Figure 1) is a transformer with measuring (1), exciting (2), and compensation (3) windings and a magnetic circuit (4), which is located inside cylindrical platform (5) with tracks intended for windings that are cut on the external side. The platform is impregnated with compound 6 at a temperature of 200°C to prevent the disintegration of the windings when ferrite screen 7, which is intended for the localization of the electromagnetic field on the tested object, is put in place. From the outside, the transducer is included in corundum washer 8, which protects core 4 from contacting the tested object.

The developed device allows, in particular, determining the electrical conductivity, which allows drawing some conclusions about the properties of the test films. The purpose of this study was to develop non-destructive testing methods for thickness, electrical conductivity and oxidative stability of nanofilms alloy Nb-Ce, intended for the use as contact and barrier materials in low-temperature superconducting electronic devices. Nb-Ce nanofilms were prepared by pulsed (200-250 ms), high-vacuum (10^{-3} - 10^{-4} Pa) deposition of alloy (0.005-0.008 g) at temperatures of $3000\text{--}3200^\circ\text{C}$. The alloy was evaporated from the surface of the Pt-Pd-strip-heater ($1\times 15\times 0.05\text{ mm}$) on the pre-annealed quartz or Pt-Pd-substrates in a vacuum chamber of the ALA-TOO installation of IMASH-275 type. Conductivity (σ) nanofilms found contactless eddy currents developed by us with the use of the subminiature eddy-current transducer.

Measurement of volume electrical resistivity of films (R_x) produced by a bridge on the E7-11 device using benchmark resistance class 1 accuracy. Resistance of samples (R_x), measured with device E7-11, was calculated by the formula:

$$R_x = R_N \cdot \frac{\alpha_{x_1} - \alpha_{x_{11}} + U_{x_1}}{\alpha_{N_1} - \alpha_{N_{11}} + U_{N_1} + U_{N_{11}}},$$

where: R_X – resistance of the sample, α_{X_1} , $\alpha_{X_{11}}$, α_{N_1} , $\alpha_{N_{11}}$ – indications on block AK of device E7 – 11, R_N – benchmark resistance of the sample made of copper M0, U_{X_1} – R_X voltage,

U_{N_1} – R_N voltage, $U_{N_{11}}$ – R_N voltage for a change in the direction of the current bridge, R_N , calculated according to formula $R_N = \frac{l}{\sigma \cdot S}$, where σ is a known value.

3. Experimental results

Results of measurements electrophysical characteristics of nanofilms alloys Ce: Nb - 1:4 and 4:1, obtained by pulsed high-vacuum sputtering of different amounts on the Pt-Pd-substrate are shown in Table 1.

Table 1. Electrophysical characteristics of nanofilms alloys Ce: Nb (N=5, P=0.95).

Composition of alloy, mass. %		Electrophysical characteristics	
Ce	Nb	R_x , Ohm	σ , MS/m
20	80	1.04 ± 0.03	2.2 ± 0.2
80	20	$(1.02 \pm 0.4) \cdot 10^{-2}$	$(3.5 \pm 0.2) \cdot 10^5$

Calculation of the film thickness (λ) of (2) based on the data in Table 1 was carried out using the resistance measurement circuit section assuming a constant specific volume resistance of the alloy:

$$l = \frac{\sigma \cdot S}{2} \cdot (R_x - R_{Lin.}), \quad (2)$$

where: R_x – resistance of the sample, S – the area of the film, σ – the film electrical conductivity, $R_{Lin.}$ – the reactance line including a resistance wire, resistance of the two Pt-Pd-substrates and the amount of contact resistance at boundaries Cu/Pt and Pt-Pd/Pt-Pd (for our installation, $R_{Lin.}$ was $(3.5 \pm 0.1) \cdot 10^{-3}$ Ohm).

The calculated thickness of the alloy film, Niobium-Selenium, in the ratio of four to one of 60 nanometers and the Niobium-Selenium alloy in the ratio of one to four nm.

As it has been intended to make the deposition of nanofilms of Ce:Nb alloys, state, adhesion and stability of which considerably depend on the condition of the substrate surface, then the process of their vacuum annealing followed by atmospheric gases and vapors adsorption has been previously studied. Substrates annealing has been carried out by a passage of current through them from 10 to 50 A at a voltage of 4 V during 2-3 s to red heat, and then from 50 to 100 A during 250 ms up to white heat of platinum (3000-3200°C). This mode of substrates annealing has caused reduction of their weight from 1.7 to 4% depending on the initial mass of a heater (in absolute terms, the greatest reduction in weight of the substrate – 0.0012 g, and the smallest reduction – 0.0003 g) that is attributable to removal of a layer of adsorbed gases, vapors and dust-like particles from chemically inert surface that would prevent further sedimentation of a nanofilm.

Investigation of the kinetics oxidation of nanofilms oxygen in the air at 25 °C was carried out through the contactless visually-optical method by photographing them at different time intervals and determining the decreasing area of the film. Then, the conversion substance (α) can be calculated from the formula (3) for reducing the area of the alloy film:

$$\alpha = \frac{S_0 - S_t}{S_0}. \quad (3)$$

Here, S_0 and S_t – the original areas of the film and the film in time t .

The film area was determined with the help of the programs for ECM Analyzer и FracDim, allowing the users to search for and to identify the edges in bitmap images, to perform their black-and-

white contrasting and to calculate the closed loops areas and the fractal dimension of the edges in the images. The kinetic data processing was conducted according to the Kolmogorov-Erofeev equation (4)

$$\alpha = 1 - \exp(-kt^n), \quad (4)$$

where k - the kinetic constant; t - the decomposition time; n - the kinetic parameter.

The kinetic oxidation curves of two Ce-Nb 1:4 alloy nanofilms, differentiated by the specific electrical conductivity, in the air at temperature of 25°C are shown in Figure 2. 1 – film with electroconductivity 3.3×10^5 MS/m, 2 – film with electroconductivity 3.7×10^5 MS/m. As an example, in the same place, original video frames of the second film, oxidizing in the air during 3, 10, 20 and 60 minutes, respectively, are provided. Similar kinetic curves were obtained for Se-Nb 1:4 as well. The kinetic equation parameters (4) presented in Table 2 were determined from the kinetic curves linear anamorphoses by the OLS method. For films having different conductivity and the composition, kinetic constant k differed by more than 100 times, the kinetic parameter n – almost 4 times.

The obtained results testify the quickest oxidation and, consequently, the least stability of the Ce 80%: Nb 20% alloy nanofilms in the air, the film characterized by higher resistance and less thickness oxidizes quicker than a thicker one does. The Ce 20% : Nb 80% alloy films oxidize 10 to 100 times slower with the common pattern being broken; a thinner film (with higher resistance) oxidizes slower than a thicker one. Depending on the thickness and the nature of the film, the process mechanism changes as well, which is manifested by the alteration in the topochemical reaction order.

Table 2. Kolmogorov-Erofeev equation of kinetic equation parameters

Composition of alloy, mass. %		σ , MS/m	Kinetic equation parameters	
Ce	Nb		$k \cdot 10^{-2}$	n
20	80	$3.3 \cdot 10^5$	0.07	0.98
		$3.7 \cdot 10^7$	3.2	0.48
80	20	2.1	12.5	0.26
		2.5	0.6	0.56

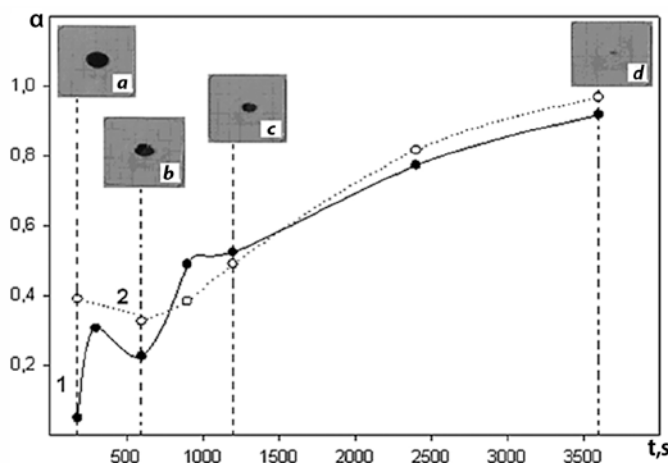


Figure 2. Kinetics of alloy nanofilms Ce-Nb.

It is found that the substance oxidation level is considerably changed in the process of oxidation. Upon that, the time it takes for achievement of a certain oxidation level considerably changes depending on conductivity of a substance and a proportion of elements.

For the first alloy with a Ce:Nb 1:4 proportion for the first film with specific electrical conductivity of $3.3 \cdot 10^5$ MS/m, the oxidation level is: for 3 minutes of oxidation – 5%, for 10 minutes of oxidation – 24%, for 20 minutes of oxidation – 50%, for 60 minutes of oxidation – 90%. The kinetic constant is $0.07 \cdot 10^{-2}$, the kinetic parameter is of 0.98.

For the second film with specific electrical conductivity of $3.7 \cdot 10^5$ MS/m, the oxidation level is: for 3 minutes of oxidation – 39%, for 10 minutes of oxidation – 40%, for 20 minutes of oxidation – 51%, for 60 minutes of oxidation – 92%. The kinetic constant is $3.2 \cdot 10^{-2}$, the kinetic parameter is of 0.48.

For the second alloy with a Ce:Nb 4:1 proportion for the first film with specific electrical conductivity of 2.1 MS/m, the oxidation level is: for 3 minutes of oxidation – 38%, for 10 minutes of oxidation – 48%, for 20 minutes of oxidation – 54%, for 60 minutes of oxidation – 65%. The kinetic constant is $12.5 \cdot 10^{-2}$, the kinetic parameter is 0.26.

For the second film with specific electrical conductivity of 2.5 MS/m, the oxidation level is: for 3 minutes of oxidation – 10%, for 10 minutes of oxidation – 19.5%, for 20 minutes of oxidation – 27%, for 60 minutes of oxidation – 44.5%. The kinetic constant is $0.6 \cdot 10^{-2}$, the kinetic parameter is of 0.56.

4. Conclusions

While alloying the Ce and Nb mixtures within a wide range of the PT mixing ratio in full vacuum vials, the alloys are formed; the latter have the form of solid solutions of γ Ce in niobium, which is verified by the chemical and X-ray diffraction analysis data. The pulsed high-vacuum deposition of the alloys with the Ce:Nb 1:4 and 4:1 (Ce 20% : Nb 80% and Ce 80% : Nb 20%) mixing ratio onto pre-annealed Pt-Pd-templates led to the obtainment of the nanofilms equal to 4 and 60 nm, respectively. The specific electrical conductivity of the nanofilms amounted to 2.1 – 2.5 (for the Ce:Nb 1:4 alloy) and $(3.3-3.7) \cdot 10^5$ (for the Ce:Nb 4:1 alloy) mS/m. The air oxidation of the nanofilms obtained in the Ce-Nb system at the temperature of 25°C studied by means of a contactless optical method is a topochemical reaction, described by the Erofeev-Kolmogorov equation with the following constants: $k (0.07-12.5) \cdot 10^{-2}$ and $n 0.26-0.98$ depending on the film specific electrical conductivity.

References

- [1] T A de Assis and F D A Aarão Reis 2013 *J. of Statist. Mech.: Theory and Exper.t* **92** 1-10
- [2] Xie L and Bedra L 2014 *J. of Physics D: Appl. Phys.* **47**(22) 1-17
- [3] Xi X and Mak K F 2015 *Nature Nanotechnol.* **10** 765-769
- [4] Ugeda M M, and Riss A 2016 *Nature Phys.* **12** 92-97
- [5] Xi X and Mak K F 2016 *Nature Phys.* **12** 139-143
- [6] Cao Y and Khestanova E 2015 *Nano Lett.* **15** 4914-4921
- [7] Grill R and Gnadenberger A 2006 *Int. J. of Refr. Metals & Hard Mat.* **24**(4) 275–282
- [8] Tither G 2001 *The International Symposium Niobium* (Orlando, Florida) vol 1 p 845
- [9] Malikov V N and Sagalakov A M 2015 *Int. Conf. on Mechan. Eng., Automation and Control Systems* (Tomsk) vol 1 (IEEE) pp 1-4
- [10] Malikov V N and Sagalakov A M 2016 *Russian Eng. Res.* **8** 626-629