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Electrophysical properties of Cu/Cr and Fe/Cr film systems within elastic and plastic deformation range

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Abstract We have investigated the electrophysical properties of metallic thin films based on Cu/Cr and Fe/Cr systems. We find that the longitudinal gauge factor of two-layer films is significantly greater as compared with one-layer films, which have the same thickness as the total thickness of a two-layer film. Interface and intensive grain–boundary electron scattering explain such an increase in the longitudinal gauge factor. We find that the longitudinal gauge factor increases in transition from elastic to plastic zone.

Introduction

There is a great interest in metallic thin film materials owing to their unique mechanical, electrical, and optical properties, which gives us opportunity to apply them widely as sensitive elements of different sensors [1–7]. Although, electrophysical properties of different materials have been studied for a long period (see, for example, [8]), nevertheless, the electrophysical, mechanical, and other properties within the field of inelastic deformation, are also of current interest. In particular, the study of longitudinal gauge factor (GF) under elastic deformation (up to 1%) of thin wires and one- and multi-layer films, including film alloys [8–11], has important consequences as strain sensor. Problems connected with the action of plastic deformation, influence of the dispersibility of films on their mechanical and electrophysical properties, role of the diffusion processes, and the phase transition in these phenomena are also addressed here.

We report the study of the electrophysical properties of two-layer films on the basis of Cu and Cr (they satisfy the condition of availability of two layers or "binary plate") as well as Fe and Cr (as solid solution is generated on basis of BCC Fe) within longitudinal deformation zone, $\varepsilon_l = 0$–2%. This interval ε_l includes elastic, quasi-elastic, and plastic deformation.

We used the following technique to get samples and study their electrophysical properties. We get two-layer films with total thickness up to 100 nm on polystyrene substrates using thermal film deposition in VUS-5 M—Vacuum Universal System (operating vacuum 10^{-3}–10^{-4} Pa). Thickness of one- and two-layer films was measured during the process of condensation, by means of a quartz resonator, and after condensation, by means of Linnik interferometry method, which allowed an accuracy of ±5%. Low-resistance contacts ($\sim$1-Ω resistance) were formed as step-plates on the basis of Cr and Cu films, according to the technique described in ref. [11].

Experimental details

The experimental setup reported in ref. [11] has been considerably modified. During the process of the experiment, and for the analysis of the results, we have used automatization (see ref. [12] for details). ADAM-4018—8-channel 16-bit sigma-delta ADC—was the base of the

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automated system using which we measured the sample resistance according to the four-point scheme; we also used USB → RS 232/422/485 ADAM-4561 interface converter, asynchronous motor, and Creative Labs Web-camera having resolution of 640 × 480 pixels. A program developed in LabVIEW with application of the LabVIEW Vision Development Module 8.2, machine vision module, controlled the experiment and data analysis.

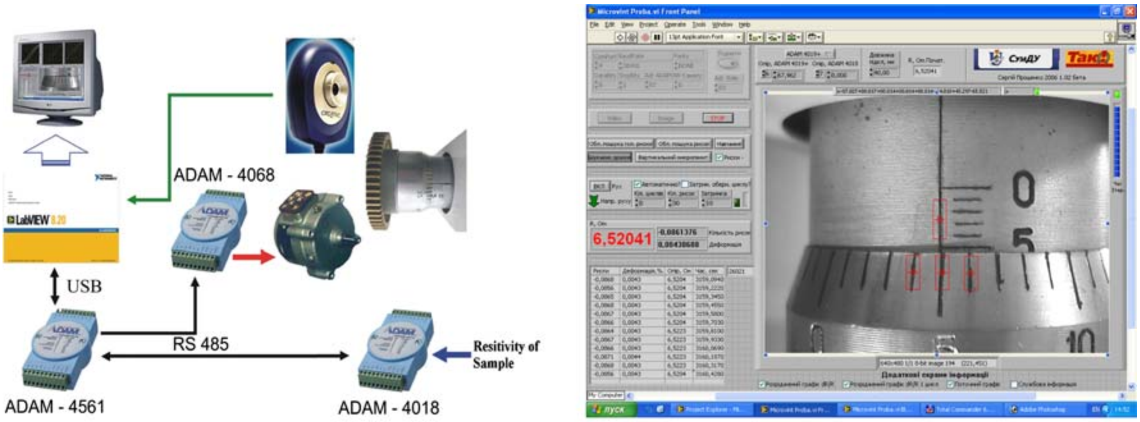
One end of the substrate with system of contacts and sample was fastened in place by bracket and the opposite end was fixed to the rod of a micro-screw (scale interval—0.02 mm) connected by means of a reducer to an electric motor, which can rotate either clockwise or anti-clockwise, depending on the control signals from ADAM-4068 relay-supply module. Web-camera was placed near the micro-screw and shot its images with frequency 10 frames/s. Simultaneously, with the process of micro-screw scale mark identification, readings of the sample resistance were also noted. In Fig. 1, we show a schematic of the automated system, which allows us to study the GF effect in dynamic or static modes, when the deformation speed varies from 0 up to 0.1%/s, within the interval $\varepsilon_l = 0$–10%. The system operation mode gives us opportunity to study working-life of film samples as possible sensitive elements of strain sensors.

Results and discussions

In this article, we describe researches of tensoresistance in Cu, Cr, Fe one-layer films, and Cu/Cr/S and Fe/Cr/S (S-substrate) two-layer film systems under static–dynamic, and under dynamic longitudinal deformations within $\Delta\varepsilon_{l1} = 0$–1% and $\Delta\varepsilon_{l2} = 0$–2% intervals, when I–VII were “loading–unloading” cycles. The samples having been deformed within $\Delta\varepsilon_{l1}$ interval were later deformed within $\Delta\varepsilon_{l2}$ interval. In order to stabilize the micro-plastic processes, we stopped deforming each time $\Delta\varepsilon_l = 0.05\%$ for 10 s. Moreover, we studied the influence of deformation speed on the GF value and, for this purpose, during several experiments, deformation cycles were performed under different film deformation speeds in the dynamic mode. The automated system allowed carrying out tests for four different film-deformation speeds.

As it makes sense to study the GF effect only on the condition of structural integrity of the sample, we monitored its structural state during the experiment according to the method described in ref. [11]. The essence of this method consists in registration of the relative intensity (I) of light beams passed through the system of sample/substrate using photoelectric detector (voltage $U \sim I$ at p–n-junction of photodiode was measured). As it was shown in ref. [11], the dependence of I on ε_l has linear character in the range of elastic or quasi-elastic deformation and becomes nonlinear with transition to plastic deformation; when microcracks are generated in the samples (which can be revealed using a scanning electron microscope or AFM), the dependence deviates from the linear one. In addition, as it is shown below, change of resistance (R) and the value of the instantaneous coefficient of longitudinal GF, $\gamma_{lM} = \frac{1}{R_i} \cdot \frac{\Delta R_i}{\Delta \varepsilon_{li}}$ (i is the number of $\Delta\varepsilon_l$ interval), depending on ε_l, is to be correlated with the type of deformation. In Fig. 2, illustrations of deformation dependences R, $\Delta R/R_i$ and U for two $\Delta\varepsilon_l$ intervals are represented (index “i” is mark of initial value of R and U).

Linear character of R and $\Delta R/R_i$ dependences on ε_l, within the interval up to 1% for II–VII deformation cycles, can be explained by elastic (up to $\varepsilon_l \cong 0.25\%$) or quasi-elastic (within $\varepsilon_l = 0.25$–0.90% interval) deformation of Cr film. Sharp distinction of I deformation cycle from the other cycles can be explained by different relaxation



(a) (b)

Fig. 1 Structural representation of the automated system (**a**) and interface of the developed software (**b**)

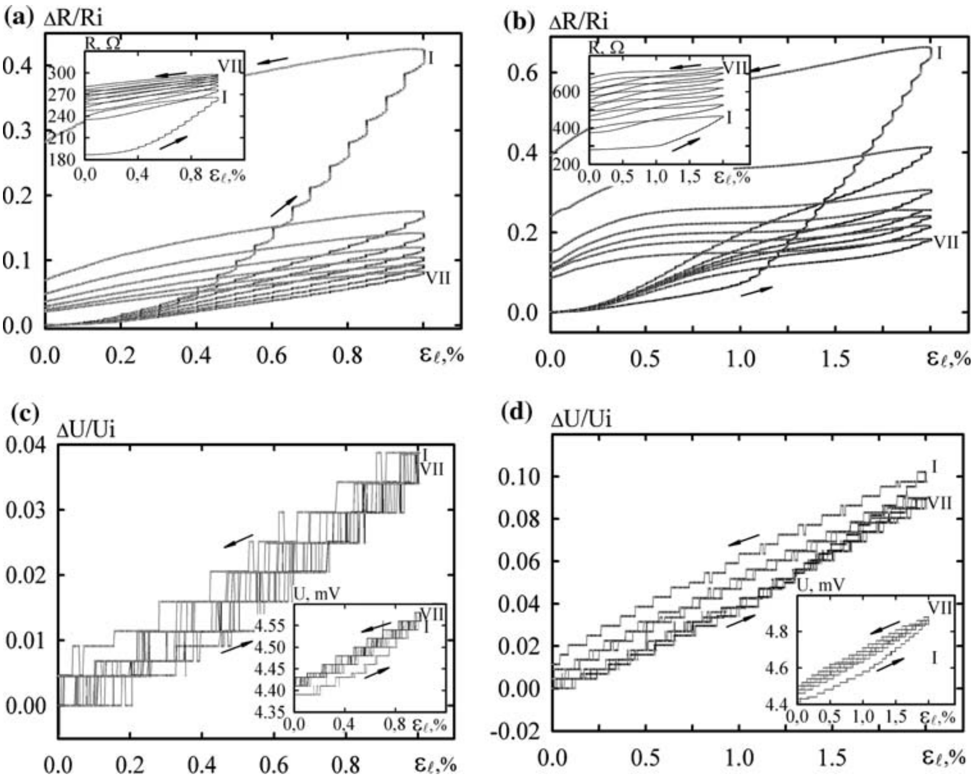


Fig. 2 Deformation dependence of relative change of resistance and resistance (**a**, **b**), of relative change of voltage and voltage at p–n-junction of photodiode (**c**, **d**) for Cr(30.6)/S film. Thickness in nanometer is given in parentheses. I–VII are the numbers of deformation cycles

processes, which take place there (such as partial turn of grains, microplastic deformation, re-allocation, and motion of imperfections of crystalline structure and foreign atoms, etc.). When increasing the longitudinal deformation up to 2% (Fig. 2b), we transit to the range of plastic deformation as can be inferred from the nonlinear dependences of R and $\Delta R/R_i$ on ε_l. Moreover, we can note that the dependences of U and $\Delta U/U_i$ on ε_l have linear character and this fact proves the structural integrity of the film (the differential dependence of $\Delta U_i/U_i$ on ε_l is given by the horizontal line, which lends support to our conclusion about the absence of micro- and macro-cracks in the film sample). Increase of intensity of light beam's transmission through the sample/substrate system with increase of deformation can be explained according to the data given in article [9], where the mechanism of longitudinal deformations up to 5% has been studied by electron microscopy in chemically precipitated Al films with 2 μm thickness and mean grain-size in the range from 1.1 (immediately after precipitation) up to 2.3 μm (after annealing at 720 K). Using stress–strain diagrams, the authors of ref. [9] found a strong difference between mechanical properties of bulk and film samples. In particular, at the limiting value of elastic deformation for Al films, $\varepsilon_l \cong 0.15\%$, there takes place a high yield point equal to 92–125 MPa for films and/compared with the yield point equal to 10 MPa for the bulk samples. Besides, films are characterized by their 'low' plasticity. These results can be explained by the local decrease of the separate grain thickness and their boundary thickness as a result of penetration of dislocations into the grains.

Test results of one-layer Cr-films and Cu-films are similar and agree with the data presented in ref. [11]. In both cases, for Cu and Cr films, the limiting transition from elastic (quasi-elastic) to plastic deformation decreases when the thickness of the film increases (for instance, when Cr film thickness $d \cong 35$ and 75 nm, the limit values ε_l are 0.6 and 0.4% respectively).

In Figs. 3, 4, there are examples of different deformation dependences for Cu/Cr/S and Fe/Cr/S two-layer films. Figure 3 shows that there occurs a peculiar stabilization of microdeformation processes, when a large amount of deformation cycles takes place (polycrystal non-elasticity effect [13]), and this fact allows us to find a certain analogy with thermal stabilization of resistive properties of film materials. Dependences of $\Delta R/R_i$, obtained during VIII–XI deformation cycles (Fig. 3a) under different deformation speeds ($1\%/\tau = 140$, 67, 30 and 20 s, respectively), allow us to draw the conclusion about weak dependence of $\overline{\gamma_l}$ on deformation speed: $\overline{\gamma_l} \cong 2.30$ when $\tau = 140$ s and 2.50 when $\tau = 20$ s. Transition from elastic deformation to plastic deformation exerts much more influence on the GF value than the change of the deformation speed (Fig. 3a, b). At the same time, the proximity of γ_{lM} and $\overline{\gamma_l}$ values and similar character of the dependences of $\Delta R/R_i$ and γ_{lM} for Fe/Cr/S film system (Fig. 4) allow one to assert that in this case the plastic deformation takes place starting with $\varepsilon_l \cong 0.4$.

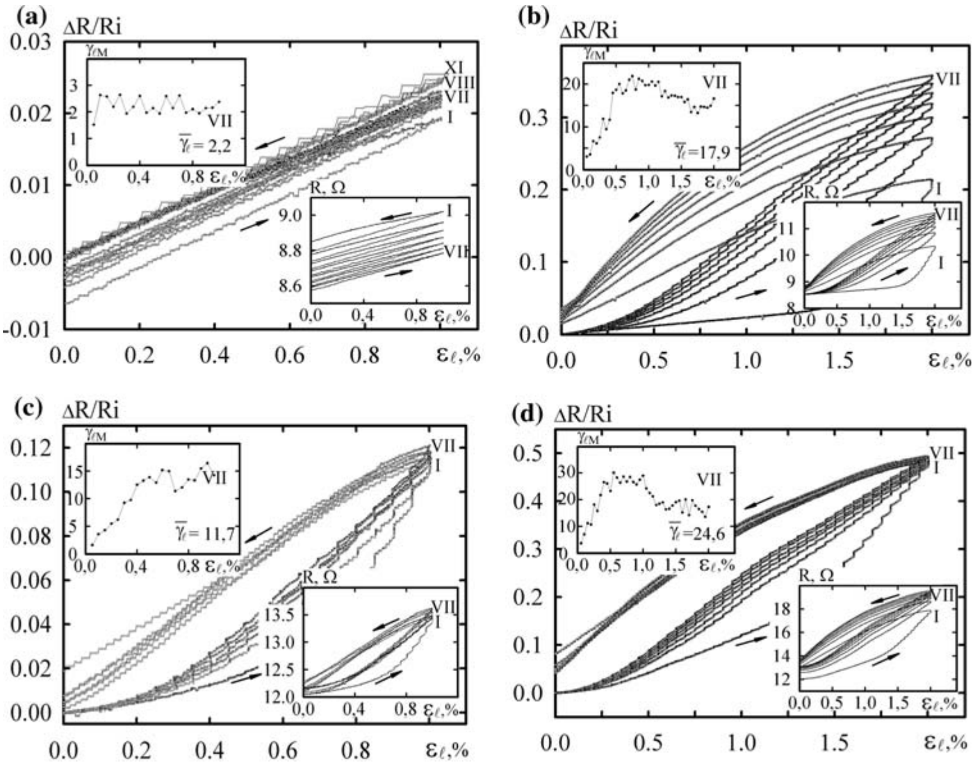


Fig. 3 Dependences of R, $\Delta R/R_\mathrm{i}$ and $\gamma_{l\mathrm{M}}$ on ε_l for Cu(20)/Cr(14)/S film systems (**a**, **b**) and Cu(20)/Cr(30)/S (**c**, **d**), where $\overline{\gamma_\mathrm{l}}$ is mean value of GF

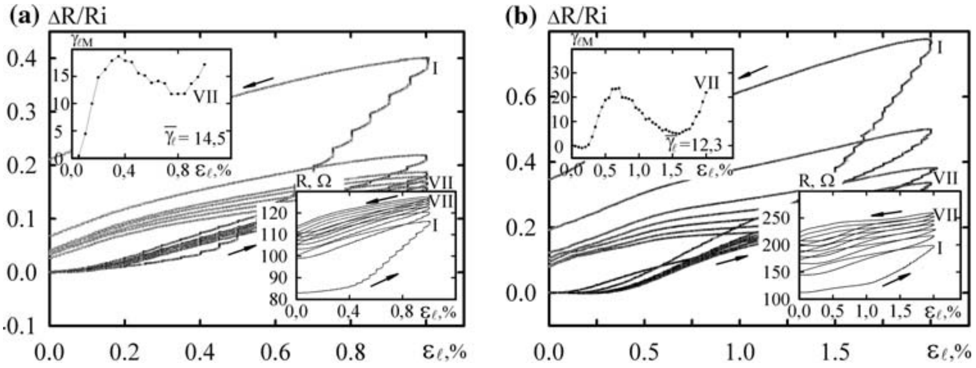


Fig. 4 Dependences of R, $\Delta R/R_\mathrm{i}$ and $\gamma_{l\mathrm{M}}$ on ε_l for Fe(21)/Cr(31)/S film system in range $\Delta\varepsilon_{\mathrm{l}1} = 0$–1% (**a**) and $\Delta\varepsilon_{\mathrm{l}2} = 0$–2% (**b**)

Relatively large rate of increase of resistance under deformation may be an indirect proof for this assertion.

In view of the fact that $\overline{\gamma_\mathrm{l}}$, in the range of plastic deformation, has relatively large values for both systems, we focus our attention on a certain contradiction between this result and the data for bulk samples. Thus, according to ref. [14], in the range of plastic deformation of bulk electrical resistance strain gauge, the relation between $\gamma_{0\mathrm{l}}$ and the coefficient of longitudinal GF can be written as:

$$\gamma_{0\mathrm{l}} = \frac{\mathrm{d}\rho}{\rho\mathrm{d}\varepsilon_\mathrm{l}} + 1 + 2\mu = \gamma_{0\mathrm{l}}^{\rho} + 1 + 2\mu \tag{1}$$

where ρ is the resistivity and μ is the Poisson's ratio.

Since in this case the grain–boundary slippage takes place, we can apply the conclusion to ref. [14], according to which the first term in Eq. 1 is to be equal to zero, and, when $\mu \cong 0.5$, then $\gamma_{0\mathrm{l}} \cong 2$. The data we obtained for the film samples allow to conclude that a significant contribution (more than 10 units) of grain–boundary electron scattering to the total GF value. Size dependences of $\overline{\gamma_\mathrm{l}}$ on the thickness of one of the layers, when thickness of the other layer is specified, which are represented here in Fig. 5, indicate that $\overline{\gamma_\mathrm{l}} \cong 2$ most probably takes place in the range of elastic deformation. The results obtained for the range of elastic deformation are in agreement with the calculated results obtained from the so-called macroscopic model for the "binary plate" type film system [15]:

$$\begin{aligned}\gamma_\mathrm{l}^{\rho} = {} & \gamma_{\mathrm{l}1}^{\rho} + \gamma_{\mathrm{l}2}^{\rho} - \frac{\mathrm{d}_1\mu_{\mathrm{f}1} + \mathrm{d}_2\mu_{\mathrm{f}2}}{\mathrm{d}_1 + \mathrm{d}_2} \\ & - \frac{\gamma_{\mathrm{l}1}^{\rho}\rho_1\mathrm{d}_2 - \rho_1\mathrm{d}_2\mu_{\mathrm{f}2} + \gamma_{\mathrm{l}2}^{\rho}\rho_2\mathrm{d}_1 - \rho_2\mathrm{d}_1\mu_{\mathrm{f}2}}{\rho_1\mathrm{d}_2 + \rho_2\mathrm{d}_1}\end{aligned} \tag{2}$$

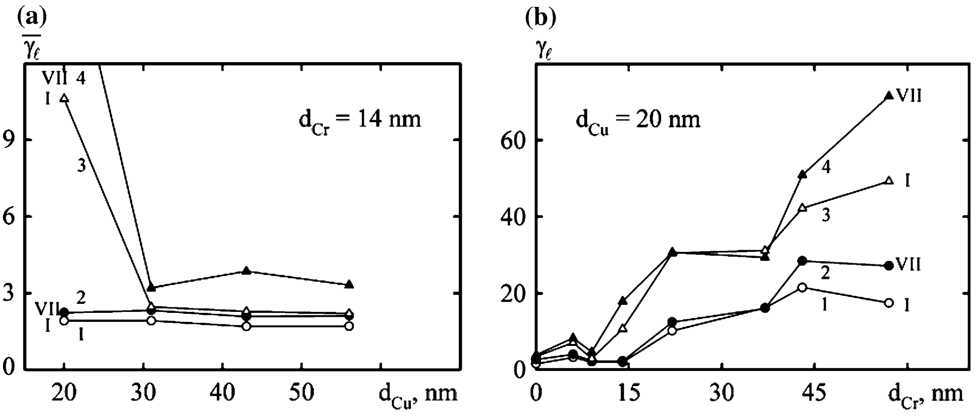


Fig. 5 Size dependences of $\overline{\gamma_l}$ for Cu/Cr(14)/S (**a**) and Cu(20)/Cr/S film systems (**b**) 1, 2—$\Delta\varepsilon_l = 1\%$; 3, 4—$\Delta\varepsilon_l = 2\%$

where, as in Eq. 1, $\gamma_l^\rho = \frac{d\rho}{\rho d\varepsilon_l} = \gamma_l - 1 - 2\mu_f$, μ_f is the Poisson's ratio of film material (for the purpose of calculation, we have considered μ for the substrate, as film system do not slip along the substrate during deformation).

It should be noted that experimentally obtained results for Fe/Cr/S system do not agree with Eq. 2, as in this case, the results were obtained not for a two-layer film system, but for a one-layer two-component film, phase composition of which represents a solid solution (α-Fe, Cr). Such a conclusion is proved well by calculations made according to the relation for film alloy:

$$\gamma_l^\rho \cong \frac{\gamma_{l1}^\rho}{1 + c_2\rho_2/c_1\rho_1} + \frac{\gamma_{l2}^\rho}{1 + c_1\rho_1/c_1\rho_2} \tag{3}$$

where c is the total concentration of atoms in the film system.

Calculated data obtained according to relation 3 agree with the experimentally obtained data for Fe/Cr/S system with an accuracy of 15–20%, whereas data obtained according to relation 2 for "binary plate" agree with an accuracy of 30–40% only.

Conclusions

Our investigations show that film systems of Cu and Cr or Fe and Cr can be used as sensitive elements of strain sensors. If we compare the γ_l value of the above-mentioned film systems with the one of different film materials, which have been analyzed by the authors in refs. [2, 6, 16–19], as sensitive elements of strain sensors, we can state that in certain cases Cu/Cr and Fe/Cr two-layer films have advantage in respect to $\overline{\gamma_l}$ value. Thus, in Ta oxynitride films, which have been analyzed in ref. [16] as high-temperature sensors, $\overline{\gamma_l} \cong 3.0$–$3.5$ units; in diamond polycrystalline films [17], $\overline{\gamma_l} \cong 40$–$50$ units; in TaN_x films ($x = 0.04$–0.20) $\overline{\gamma_l}$ varies from 3.4 ($x \cong 0.04$) to 6 ($x \cong 0.12$) units, and after $TaN_{0.08}$ film annealing from 870 up to 1,270 K, value of $\overline{\gamma_l}$ reduces from 4.25 to 4.05 units [18]; in Si nanocrystalline films, $\overline{\gamma_l} = 10$–25 (p-type) and $\overline{\gamma_l} = -(10$–$20)$ units (n-type) [19]. Therefore, our results are, up to some degree, in good agreement with the reported results in the literature for the strain sensitivity requirements.

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