METHOD OF STUDYING THE TENSORESISTIVE PROPERTIES OF CHROME FILMS AT RELATIVELY SMALL AND LARGE DEFORMATIONS

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UDC 621.315.592

An experimental method for studying the tensoresistive properties of thin Cr films in the range of elastic and plastic deformations has been developed. The method allows also diagnosing the start and degree of the cracking of a film in the course of deformation. Researches of the tensoresistive effect in a wide range of deformation had allowed the contribution of the internal size effect to the strain sensitivity factor to be determined. The dependence of the film resistance on time, which is connected to microplasticity, has been revealed at the opening intervals of the deformation curves.

1. Introduction

A large attention of researchers to film materials is caused by differences between the mechanical, electrical, optical, etc. properties of the latter and those of massive specimens. Despite the fact that the tensoresistive effect in thin films and film materials has been studying for a long time (see, e.g., [1]), this problem remains challenging at present. To be more accurate, we should indicate that researches of the tensoresistive effect at small deformations (up to 1%) in thin wires [1,2], single- and multilayer films, including film-shaped alloys [3–12], approach a state of certain completeness, while studies of the influence of the size effect on the strain sensitivity at a large static or dynamic deformation are only in an incipient state (for more details, see [13]). Here, the key issues concern the mechanism of film deformation, the peculiarities of film mechanical properties, the influence of film dispersity (the internal size effect) on the electromechanical properties. Recently, a number of works dealing with those subjects haa been published. In particular, the theoretical analysis of deformation effects at small and large deformations in thin and thick multilayers, taking into account the geometry of the layers, their plasticity, and the concentration gradient, has been carried out in work [14]. The authors of work [15] studied the shape memory and superelasticity effects in film alloys Ti+(48—51)at.%Ni. In work [16], the atomic force microscopy was used to study the mechanism of deformation, the amplitude of which did not exceed 2%, as well as a destruction of thin (the film thickness $d \approx 240$ nm) Ni films on a polycarbonate substrate. It has been marked that the method of researches allowed also the local stresses in Ni films and the adhesion of the latter to a substrate to be determined. The authors of work [17], using an original installation constructed on the basis of a photolithographic device and a laser, have carried out the researches of mechanical properties of free-standing Cu and Ag single-layer films and Ag/Cu multilayers, with the thickness of each layer being $d = 1.5$ nm – 1.5 μm. Basing on the $\sigma-\varepsilon$ charts (σ stands for stress and ε for strain), the authors of work [17] have determined the parameters σ_y, σ_0, k, and n in the Hall–Petch equation $\sigma_y = \sigma_0 + kL^n$, where σ_y is the yield stress, L is the average dimension of crystallites, σ_0, k, and n are constants, for Cu and Ag films and Ag/Cu multilayers. Similar researches have been carried out by the authors of works [18, 19] for Cu films with $d = 0.2 - 2$ μm and various degrees of dispersity. The difference between the results obtained consists in that, according to work [17], $n = -0.34$, whereas $n = -0.5$ in work [19] in agreement with the classical Hall—Petch law for massive specimens. The size effect for the mechanical properties of free-standing Al and Cu films with $d = 0.2, 0.3, 0.5$, and

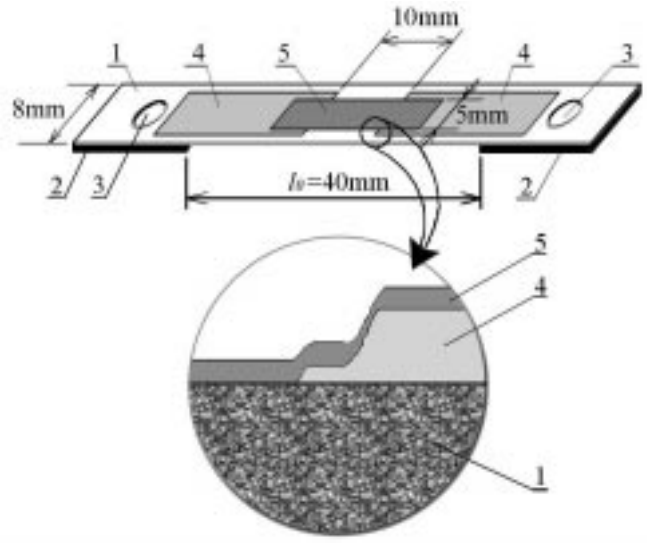


Fig. 1. Construction of the substrate: polished and cleared Teflon tape 0.3 mm in thickness (*1*), aluminum overlays (*2*), holes for fastening (*3*), copper step-like contact platforms (*4*), film or film system (*5*). The vertical cross-section of the substrate at the point of the junction between the contact platform and the film is shown in the inset

1 μm has been studied in [20]. The obtained $\sigma - \varepsilon$ charts have allowed Young's modulus to be determined and new features in mechanical properties to be discovered.

The short analysis of the results of previous works testifies that the basic attention in them has been paid to the study of the interrelations between mechanical properties and a structural state of the specimen. But researches of the tensoresistive effect in metal films in the range of their relatively large deformation (about 10%) are almost absent. Just this circumstance has determined the purpose of our investigations, namely, the development of a method for studying the tensoresistive properties of thin Cr films in the range of both elastic and plastic deformations.

2. Method and Experimental Technique

Films of Cr with the thickness $d = 30 \div 50$ nm were fabricated by thermal evaporation in vacuum of 1×10^{-3} Pa at a VUP-5M installation at room temperature onto a polished Teflon substrate with contact platforms (Fig. 1). The step-like geometry of the latter was formed making use of masks and following the method described in work [12]. The structure of a junction between the film and the contact shown in the inset in Fig. 1 ensured a reliable electric connection with low electric resistance. Aluminum overlays *2* prevented from the concentration of stresses in holes for fastening *3* and confined the substrate section that underwent deformation. Researches of resistive properties of continuous thick Cu films evidenced for their weak sensitivity to deformation (the strain sensitivity coefficient is about unity). So, if the thickness of the contact platforms is large, one may neglect the variation of their resistance, which amounts to about 1 Ω in a non-deformed state of the platform.

The thickness of the films was measured by two methods, namely: (i) in the course of deposition, by the method of quartz resonator, which allowed us to select the necessary rate of condensation (about $0.1 - 1$ nm/s), and (ii) after deposition, by the Linnyk method of optical interferometry on a MII-4 device, which ensured the error of measurements of no more than 5%.

To determine the value of deformation, one of the ends of the Teflon substrate with initial length l_{init} (Fig. 1) was rigidly fixed on the deformation table located inside the vacuum installation. The other end was joined to a mobile rod of the microscrew, the minimal step of which corresponded to a longitudinal stretching of the substrate by 0.02 mm or to the minimal step of deformation of 0.05%.

The system "film/substrate" was tested for stress relaxation [21]. In so doing, contrary to the load curves stress–strain where the stress was kept constant, we held the strain to be constant. The strain was calculated according to the relation [21] $e_l = \ln(l/l_{\text{init}}) = \ln(1+\varepsilon_l)$, where e_l is the longitudinal strain; l and l_{init} are the current and initial lengths , respectively; $\varepsilon_l = \Delta l/l_{\text{init}}$ is the relative stretching and $\Delta l = l - l_{\text{init}}$ is the absolute one. At small $e_l \sim 0.01$, one can use the approximation $e_l \approx \varepsilon_l$.

When considering plasticity, the strain e_l can be presented as two terms: $e_l = e_{l0} + e_{l1}$, where e_{l0} is the elastic component and e_{l1} the plastic one.

The return stage of the plastic deformation cycle takes place only at the expense of a reduction of the e_{l0} value. At the moment when the elastic component achieves the value $e_{l0} = 0$, a squeezing force F begins to act on the substrate in the direction of its longitudinal axis, which results in a bending of Teflon tape *2* (Fig. 2,*a*). Light beam *1*, whose diameter is about 2 mm, strikes the horizontal substrate *2* at an angle α and, being reflected from it completely, arrives at a sensitive element of photodetector *3*. If substrate *2* has become bent, the angle of reflection changes and the area of overlapping *6* starts to decrease, which results in an abrupt diminishing of the signal I registered by millivoltmeter *4*. The residual deformation of the

substrate e_{l1} is determined in accordance with a sharp reduction of this signal and using the corresponding value of the shortening of the substrate, which is read out on the microscrew scale (Fig. 2,*b*).

The electroresistance of the films in our experiments amounted to about 100 Ω. In order to determine it, we used a point-to-point metering circuit (an APPA109 digital device).

Measuring wires were soldered to the contact platforms. In the course of deformation, the resistance was registered with a time interval of 0.5 or 1 s. The relative error of the device was 0.3%. The resistance of measuring wires was determined and subtracted from the total resistance. To prevent the influence of the environment, all experiments were carried out *in situ*, immediately in the working volume of the vacuum chamber.

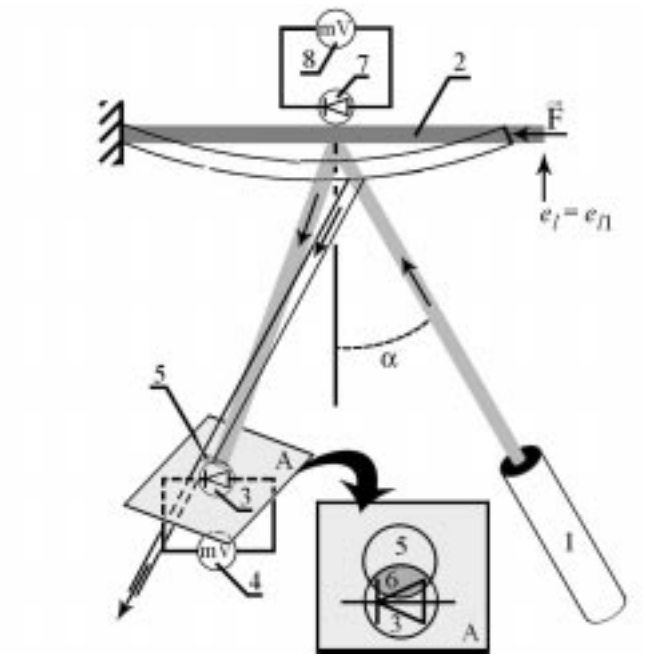
3. Monitoring of the Structural State of a Film

Studies of the tensoresistive effect at large deformations have reason only provided the structural integrity of the specimen. The gradual cracking of the film at rather large relative stretching stimulates a smooth nonlinear increase of the film resistance. The monitoring of the structural state in the course of experiments was carried out by the optical method. We analyzed the strain dependences of the intensity of the beam that had transmitted through a film/substrate system. The optical signal was registered by photocell *7* (Fig. 2,*a*) located behind substrate *2*, and corresponding millivoltmeter *8*.

Using the Bouguer law, it can be shown that, for a "continuous film/substrate" system, the relation $I/I_0 = \exp(-ke_l)$ holds good, where I and I_0 are the intensities of light that has passed through the system in a deformed state ($e_l \neq 0$) and a non-deformed ($e_l = 0$) one, respectively; $k = \chi_f d_{f0}\mu_f + \chi_s d_{s0}\mu_s$; χ_f and χ_s are the absorption coefficients, d_{f0} and d_{s0} the thicknesses in the non-deformed state, and μ_f and μ_s are Poisson's ratios of the film (the subscript f) and the substrate (the subscript s), respectively. We note that the intensity I of optical signals in our experiments was measured in Volts, i.e. in terms of voltage units U across the *p-n* junctions of photodiodes. In this case, one may write down that

$$\ln U = ke_l + \ln U_0 \,. \qquad (1)$$

One can see that this dependence is linear in the $\ln U$ versus e_l coordinates, which allows the coefficient k to be determined from the experimental $U(e_l)$ data.



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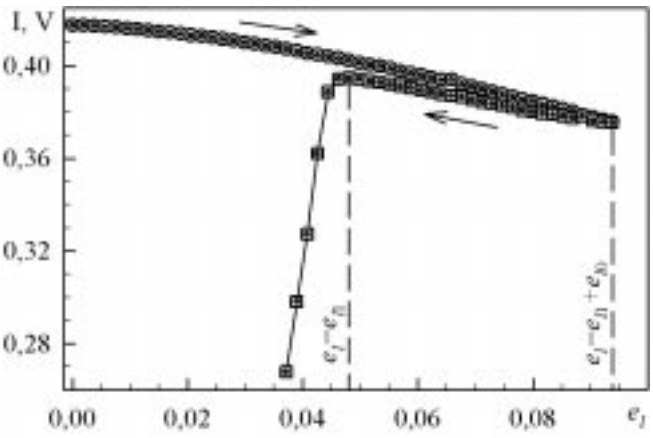
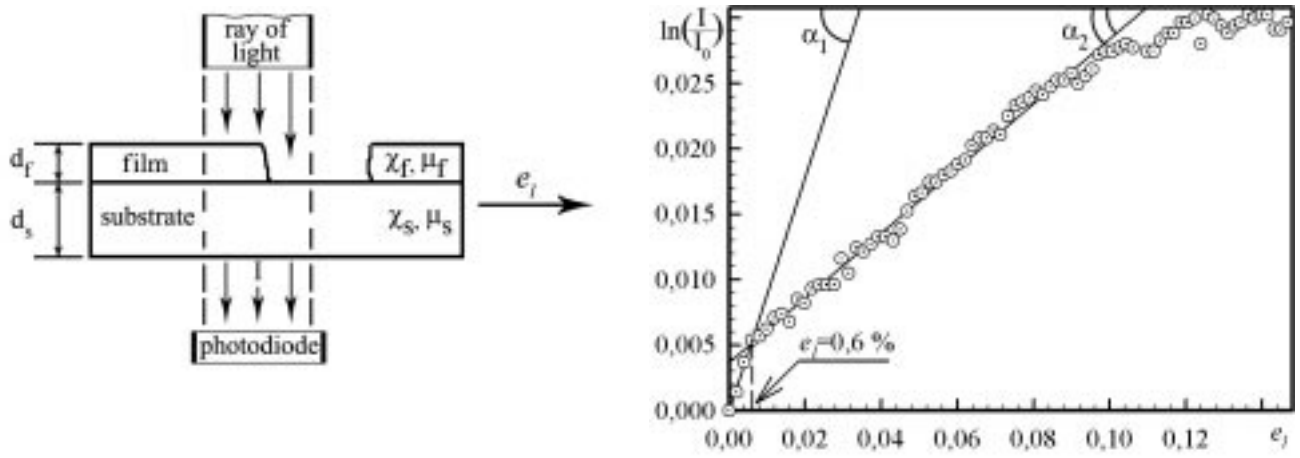


b

Fig. 2. (*a*) Diagram of registration of the residual deformation: (*1*) light source, (*2*) Teflon substrate, (*3,7*) FD256 photodiodes, (*4,8*) UT70B millivoltmeters, (*5*) cross-section of the reflected beam in the plane A of the photodetector *3* sensitive element, (*6*) the region of the overlapping of a sensitive surface of photodiode *3* and beam *5*; (*b*) the experimental dependence of the intensity I of a beam reflected from the substrate on strain e_l

In the case of the "cracked film/substrate" system (Fig. 3,*a*), the relation analogous to Eq. (1) can be written as

$$\ln U = \ln U_0 - \ln S + \chi_f d_{f0} + \chi_s d_{s0}\mu_s e_l + \\ + \ln[S + S_f(\exp(\chi_f d_{f0}(\mu_f \cdot e_l - 1)) - 1)] \,, \qquad (2)$$



a **b**

Fig. 3. (*a*) Schematic view of the vertical cross-section of the "cracked film/substrate" system; (*b*) the experimental dependence $\ln(I/I_0)$ versus e_l for a system Cr(d = 38 nm)/substrate

where $S = S_f + S_s$ is the area of the beam projection onto the substrate, and S_s and S_f are the total area of cracks and the substrate covered with the film within the limits of the area S, respectively. Equation (2) is linearized in the $\ln U$ versus e_l coordinates only if the film is continuous ($S_f = S$); in this case, it acquires the view of Eq. (1).

The experimental dependence $\ln(I/I_0)$ versus e_l for a Cr film is shown in Fig. 3,*b*. These results testify to that, at the transition from elastic (quasi-elastic) to plastic deformation (at $e_l = 0.006$), the parameter k changes. After the slope angle has changed from α_1 to α_2, the dependence remains linear, which is in agreement with Eq. (1) and, therefore, evidences for the film integrity. If the relative stretching reaches the value $e_l \approx 0.1$, the experimental dependence deviates from linearity and obeys Eq. (**??**), which evidences for the film cracking.

The researches of the film surface structure were carried out by the methods of optical scanning electron (SEM) and atomic force (AFM) microscopies. The capabilities of a МИ-4 optical device, which was used in our studies, are limited by 490-times magnification, so that this method allows the structural modifications to be registered only at the stage of a rather extensive cracking. From this point of view, more informative results can be obtained using the SEM and AFM methods.

According to the results of researches, the surface of the film in the initial state has no prominent inhomogeneities. As the deformation grows, the crystal structure of the specimen becomes apparent more clearly. Above the value $e_l = 0.1$, the cracks become visible in microphotos, being well-detected at $e_l = 0.15$. For example, for the 20-nm Cr film, the average length and width of cracks are 2.2 and 0.27 μm, respectively, at the strain $e_l = 0.15$ (Fig. 4). The results obtained making use of the SEM and AFM methods are in qualitative agreement with each other.

4. Tensoresistive Properties of Cr Films

A great number of both theoretical and experimental works [1–6] evidences for a low strain sensitivity of continuous single-layer metal films in the range of elastic deformation. However, the situation changes when plastic deformation is taken into consideration.

The ability of the material to change the absolute, R, or the specific, ρ, electric resistance under the longitudinal stretching is characterized by either the coefficient of longitudinal tensoresistive sensitivity γ_l^R or the coefficient of longitudinal strain sensitivity γ_l^ρ, respectively, which are defined by the relations

$$\gamma_l^R = \frac{1}{R_{\text{init}}}\frac{\partial R}{\partial e_l}, \qquad \gamma_l^\rho = \frac{1}{\rho_{\text{init}}}\frac{\partial \rho}{\partial e_l}, \tag{3}$$

where R_{init} and ρ_{init} are the initial values of the resistance and specific resistance, respectively. In the case, where the dependence $R(e_l)$ remains linear (the opening section of the dependence in Fig. 5,*a*), i.e. $R = R_{\text{init}}\gamma_l^R e_l + R_{\text{init}}$, the parameters R_{init} and γ_l^R determine the equation of the straight line unambiguously.

In the range of the transition to plastic deformation, the experimental dependence deviates from the preceding linear one possessing the slope parameter

$\tan\alpha_1$. In this range, $\tan\alpha = \partial R/\partial e_l = R_{\text{init}}\gamma_l^R \neq$ const, because the magnitude of the angle α and, accordingly, the value of the coefficient γ_l^R grow with e_l. Moreover, when e_l decreases in the course of the return stage of the cycle, the value of the film resistance R does not return to its initial value R_{init} (Fig. 5,*b*), which is caused by a residual deformation. It should be also noted that the method of processing of experimental data essentially affects the resulting values that are determined for the coefficients of tensoresistive sensitivity; in particular, it is true for the manner to evaluate the derivative $\partial R/\partial e_l$. If the dependence $R(e_l)$ is linear, the experimental data are approximated by a straight line making use of the least-squares method, and the derivative $\partial R/\partial e_l$ is determined unambiguously by the angular factor of the line obtained.

To determine the derivative $\partial R/\partial e_l$ and the coefficients of tensoresistive sensitivity in the range where the dependence $R(e_l)$ is nonlinear, we used the method of analytical differentiation, the essence of which is as follows:

— First, in order that the experimental dependence can be fitted as precisely as possible, the interval of measuring was split, according to the experimental data, into several characteristic subintervals. Using the least-squares method, the experimental data within each subinterval were approximated by a relevant polynomial; in essence, we found an analytical piecewise polynomial approximation of them.

— The derivative $\partial R/\partial e_l$ was calculated by differentiating the obtained functions, and the coefficient of tensoresistive sensitivity γ_l^R was determined according to Eq. (3) (see Fig. 5,*c*).

From Fig. 5,*c*, one can see that the value of the coefficient γ_l^R is almost constant at $e_l < 1.7\%$. At the boundary between the elastic and plastic ranges, the sensitivity of the film starts to grow. Such a behavior can be explained by the activation of plastic deformation mechanisms. In particular, the dislocation mechanism and grain-boundary microsliding, the latter being typical of the nano-crystalline state, should result in an increase of the concentration of crystal structure defects and in a variation of the conditions of the conduction electron scattering by grain boundaries, which, in turn, should cause the growth of the specific resistance of the material owing to the internal size effect. Such an explanation does not contradict the microscopic model of strain sensitivity [5]. The modification of the crystal structure in the course of plastic deformation, which has been

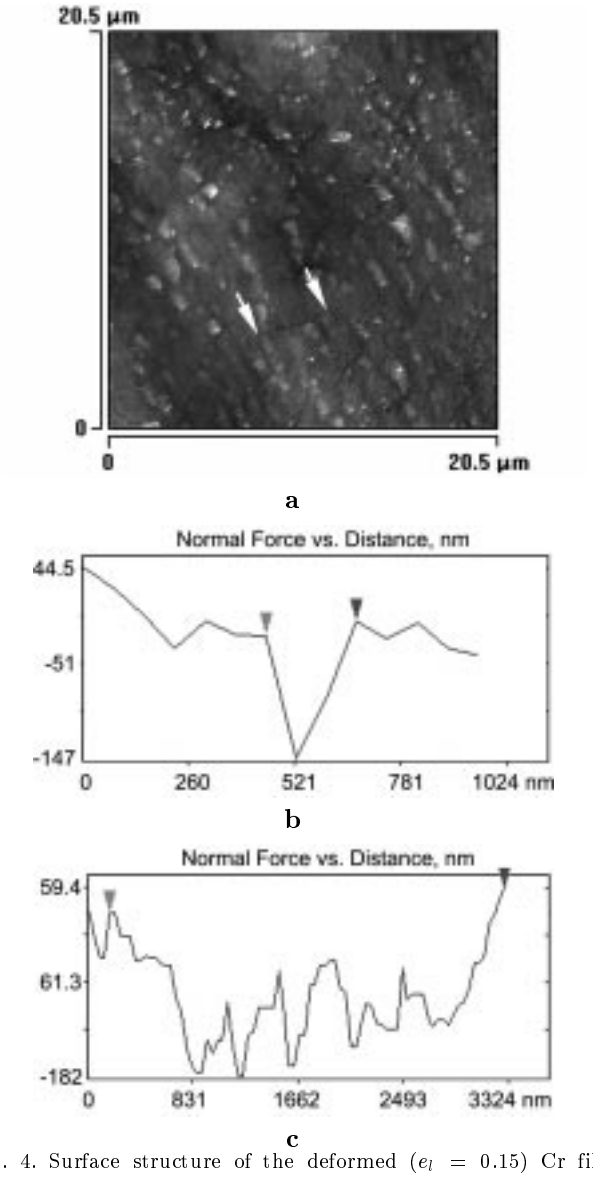


Fig. 4. Surface structure of the deformed ($e_l = 0.15$) Cr film ($d = 20$ nm) deposited onto a substrate revealed by SEM: (*a*) surface topology, (*b*) the profile of one of the cracks (indicated by markers) in the transverse direction with respect to the crack, (*c*) the same as in panel (*b*) but for the longitudinal direction

studied in works [22–24], evidences for an increase of the volume fraction of the intergrain phase and a reduction of the average size of crystallites, which leads to a diminishing of the mean free path of electrons.

In addition, the calculation of the coefficients of tensoresistive sensitivity becomes more complicated, because the initial resistance R_{init} grows in every successive cycle, which is connected to the residual deformation (Fig. 5,*b*). The values of γ_l^R at the

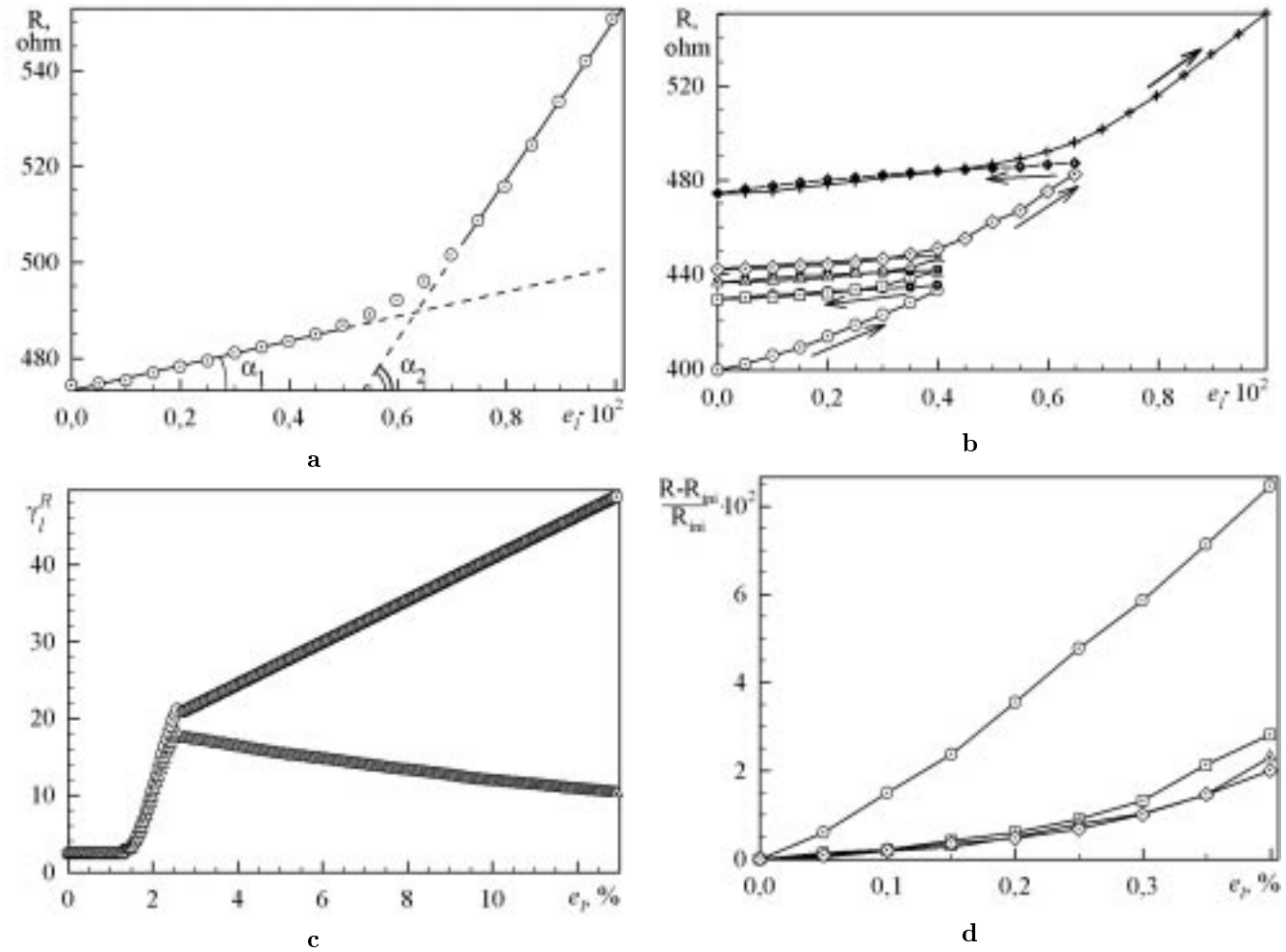


Fig. 5. Experimental dependences for a film system Cr(d = 36 nm)/substrate: (a) transition from elastic (quasi-elastic) to plastic deformation; (b) the dependences of the resistance on the strain for the first five "stretching–relaxation" cycles (circles, squares, triangles, diamonds, and crosses, respectively); (c) the dependences of the average (circles) and instantaneous (triangles) coefficients of tensoresistive sensitivity on the strain for the 9-th deformation cycle; (d) the relative variation of the film resistance as a function of the strain (curve labeling is the same as in panel b)

return elastic stages of the cycles are considerably smaller than those at the forward inelastic ones. The influence of this circumstance was minimized in our works by calculating the instantaneous values of the strain sensitivity coefficients $\gamma^R_{l,\mathrm{inst}}$ and $\gamma^\rho_{l,\mathrm{inst}}$, i.e. the deformation sensitivity of the material with respect to the current, instantaneous value of the resistance R_0 or the specific resistance ρ_0, respectively, rather than to the initial values R_{init} and ρ_{init} (see Eqs. (3)). To calculate the instantaneous values of both the longitudinal strain sensitivity coefficients, the corresponding equations can be written down as (cf. Eqs. (3))

$$\gamma^R_{l\mathrm{inst}} = \frac{\partial \ln R}{\partial \ln l}, \qquad \gamma^\rho_{l\mathrm{inst}} = \frac{\partial \ln \rho}{\partial \ln l}. \tag{4}$$

An extremely important parameter of strain sensors is the stability of their characteristics [10, 11]. Under plastic deformations, such a stability cannot be obviously achieved, but the activation of low-energy mechanisms of plasticity makes it possible to obtain better a reproducibility and recurrence of results at further elastic cycles "stretching–relaxation". To explain this statement, let us return back to Fig. 5,b, from which one can see that it is impossible to precisely detect a transition to plasticity at the first cycle of the dependence $R(e_l)$. Such a behavior can be connected to the microplastic deformation which was observed in [25] at a very small relative stretching $e_l = 10^{-6} - 10^{-3}$. An evidence for the gradual

activation of plastic mechanisms of deformation and structural modifications in the specimen is a gradual increase of the tensoresistive sensitivity coefficient γ_l^R. The return stage of the cycle is realized only due to elastic processes in the film, so that the coefficient of tensoresistive sensitivity has a considerably smaller value at this stage as compared to the value at the preceding forward stage. The dependence $R(e_l)$ at the stage of specimen stretching almost completely coincides with that at the return stage of the preceding cycle. But if the deformation comes closer to the maximal value of e_l achieved in the preceding cycles, higher-energy mechanisms of plasticity are activated, which results again in further structural modifications of the material of the film and increases its specific resistance. The range of elastic deformation can be extended by applying a cyclic loading, according to the theory put forward by Orowan (see, e.g., [21]). The indicated method of loading is realized rather effectively within the range of elastic deformation of the Teflon substrate. In this case, after reaching the value of e_l that corresponds to the residual deformation of the metal film, the latter undergoes the action of squeezing forces at the relaxation stages. It is owing to these forces that the return stages of cyclic plastic deformations variable by direction become possible. If the previously achieved value is not exceeded in the course of the further film stretching, the character of deformation will remain elastic due to a lack of the energy needed to activate new higher-energy mechanisms of plasticity. From such a point of view, it is possible to explain also the gradual stabilization of tensoresistive characteristics after several stretching–relaxation cycles (Fig. 5,*d*) within the same interval of the e_l values.

One of the ways to extend the range of elastic deformation is the use of nano-crystalline materials, which is illustrated excellently by the results of work [20]. As follows from the Hall–Petch relation, a reduction of the average size L of the crystalline body's grains leads to the growth of the yield stress σ_y. But this manner is also confined to a temperature interval, the upper limit of which is governed by either enhancement of recrystallization and diffusive processes or phase transitions. From this point of view, fine-grained films of refractory metals seem perspective for the creation of strain sensors.

5. Conclusions

The method proposed in this work for researching the tensoresistive properties of thin Cr films has made it possible to reveal some peculiarities of the tensoresistive effect.

At the transition from elastic to plastic deformation, an increase of the tensoresistive sensitivity coefficient is observed from $\gamma_l^R \sim 1$ to $\gamma_l^R \sim 10$, which is caused by structural modifications at a microplastic level.

The magnitude of deformation, above which plasticity starts to manifest itself, can be determined for a metal film using the experimental dependence $R(e_l)$ obtained when testing for strain relaxation.

Structural modifications that occur on the film surface in the course of its deformation, in particular its cracking, can be monitored optically by measuring the variation of the intensity of the light beam transmitted through the film/substrate system.

The choice of a substrate, in particular its elastic properties, deserves a special attention, while studying the tensoresistive effect. The inelastic deformation of the substrate complicates the interpretation of experimental results. Moreover, the creep of the substrate material, which manifests itself in the course of researches of the tensoresistive properties of the deposited film, results in a poor reproducibility of experimental results. The further development of researches on the basis of the experimental method described above can be carried out in the following directions:

— Study of the mechanism of deformation and the tensoresistive effect in single-layer films with different degrees of dispersion.

— Transition to multilayer film systems fabricated on the basis of metals with low mutual solubility (e.g., Cr, Cu, Sc, etc.).

The authors express gratitude to post-graduate student S.I. Protsenko for carrying out the researches by the AFM method.

The work was executed under the financial support of the Ministry of Education and Science of Ukraine (Budget project No. 0103U000773 and mutual Ukrainian-Polish project No. M/18-2004).

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Received 09.09.04.

Translated from Ukrainian by O.I.Voitenko

МЕТОДИКА ВИВЧЕННЯ ТЕНЗОРЕЗИСТИВНИХ ВЛАСТИВОСТЕЙ ПЛІВОК ХРОМУ ПРИ ВІДНОСНО МАЛИХ І ВЕЛИКИХ ДЕФОРМАЦІЯХ

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Р е з ю м е

Розроблено методику експериментального вивчення тензорезистивних властивостей тонких плівок Cr в області пружної та пластичної деформацій. Методика дозволяє також діагностувати початок та ступінь розтріскування плівки в процесі деформацій. Дослідження тензорезистивного ефекту в широкому діапазоні деформацій дозволяє виділити внесок внутрішнього розмірного ефекту в тензочутливість. На початкових ділянках деформаційних кривих виявлено залежність опору від часу, що пов'язано із мікропластичністю.