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MECHANICAL PROPERTIES OF MICRON AND NANODIMENSIONAL METAL FILMS

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*Data concerning the mechanical and tensoresistive properties of bulk condensates and nanodimensional films under elastic and plastic deformation is analyzed. Investigation results of the tensoresistive properties of single-layer (*Fe, Cr, Pd *and* Pt*) and two-layer (*Fe/Cr, Cu/Cr *and* Pd/Fe*) films, in particular, dependence of the deformation for the transition from elastic to plastic deformation on the thickness, are presented.*

Keywords: *MECHANICAL PROPERTIES, BULK CONDENSATES, NANODIMENSIONAL FILMS, ELASTIC AND PLASTIC DEFORMATION.*

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1. INTRODUCTION

Unique physical and mechanical properties of nanocrystalline bulk and nanodimensional and nanocrystalline film samples are of great scientific interest. Investigations of the mechanical properties of fibrous crystals (the so-called "whiskers") [1] preceded this increased interest. The authors of [1] revealed a wide range of the elastic deformation, which was about some percents (for example, 3,8% (Cu, Cr) and 4,9% (Fe)) that exceeds some hundreds times the range for monocrystals; high coercivity and hardness, which is only 15% less than the theoretical value.

During the investigation of bulk and film nanocrystalline materials a great attention was paid to the study of their mechanical properties and the mechanism of plastic deformation [2-12]. Thus, it was shown in [2-6] that the initial stage of the plastic deformation of bulk nanocrystalline materials is conditioned by the micro-slippage along the grain boundaries and can not be described within the classical dislocation mechanism [3, 4]. The authors of Ref. [5, 6] performed the investigations of the deformation mechanism by the stretching of Cu bulk samples (the nanograin size is 40-60 nm) and of $Fe_{73,5}CuNb_3Si_{13,5}B_9$ alloy (10 nm) directly in the electron microscope column. They concluded that in this case the deformation is mainly realized due to the nanograin rotation (rotational deformation modes) and the defect interaction in the boundaries and the near-boundary zones. The authors of Ref. [7, 8] carried out "in situ" investigations of the mechanical properties and deformation mechanism by the example of thin Al films of micron thickness. It was established as well as in [2-4] that the mechanical characteristics of free films and bulk samples are considerably different that is explained by the local decrease in the grain thickness with the appearance of the internal stresses as a result of dislocation localization inside their volume. The authors

of Ref. [13] paid attention to the essential difference of the electrophysical properties of nanodimensional metal films in comparison with bulk materials. Measurements of the Young's modulus for multi-layer film system based on two-layer nanodimensional Cu/Ni fragments of the thickness from 1,6 nm to 12 nm are carried out in Ref. [14]. Obtained value of the Young's modulus E of multi-layer system was from 1,02 to 1,10 of the calculated value $E = c_1E_1 + c_2E_2$, where c_1 and c_2 are the concentrations of Cu and Ni atoms in separate fragment.

2. MECHANICAL AND TENSORESISTIVE PROPERTIES OF MICRON CONDENSATES AND BULK SAMPLES

The authors of [10, 11] obtained the diagrams of the deformation of the coarse-grained (grain size is more than 1,5 μm) and the fine-grained (grain size is less than 0,5 μm) Au films in the thickness range 0,2-2 μm. It was established that in the first case the mechanical properties do not substantially differ from those of bulk samples, except for the low film plasticity. The strong dependence of the mechanical properties on the deformation velocity was observed in the case of the fine-grained samples.

Systematic investigations of the size effect in the plasticity of submicron (thickness is from 0,2 to 1 μm) Cu, Al and Au films are presented in Ref. [12]. Obtained deformation diagrams (Fig. 1 and 2) allowed to determine the Young's modulus and establish new features of the mechanical properties. Thus, for example, in Cu film with the thickness decrease from 1 to 0,2 μm the liquid limits increase from 160 to 345 MPa, and in Al films of the thickness of 0,2 μm the plasticity does not observed at all and their brittle failure occurs at the stress of 375 MPa. Transition from elastic to plastic deformation takes place at the longitudinal deformation $\varepsilon_{ltr} \cong 0{,}25\%$ (Al films), 0,1-0,2% (Cu films) and 0,05-0,15% (Au films) that corresponds to the same value obtained by the authors of [15] by the example of Pd films (Fig. 3) of the thickness of 4 μm ($\varepsilon_{ltr} \cong 0{,}25\%$) and of multi-layer film heterostructure based on Mo and Cu with the thickness of the separate layers up to 15 nm ($\varepsilon_{ltr} \cong 0{,}235$ %). The same results by the example of micron films were also obtained in [7, 16-18]. In particular, the author of Ref. [16] has studied the mechanical properties (limits of elasticity (ε_{tr}) and hardness (σ_m) and the Young's modulus) depending on the thickness using epitaxial, polycrystalline oriented and quasi-amorphous Au film of the thickness of 50-1500 nm. It was established that $\varepsilon_{tr} \leq 1\%$ and σ_m and E do not have a precise correlation with the film thickness, although in both cases the general tendency to their decrease with the increase in the thickness when the dependences pass through the maximum is observed. The author notes a significant increase in σ_m in comparison with bulk samples: 20-48 MPa (films), 10-15 MPa (annealed samples of bulk Au) and 20-30 MPa ("whiskers"). At the same time the Young's modulus has smaller value in comparison with bulk monocrystals: 4-8 GPa (films) and 4,2-11,4 GPa (bulk monocrystals). These conclusions completely agree with the results of [19, 20], where the mechanical properties of nanocrystalline Cr and Nb were studied by the molecular-dynamic simulation method.

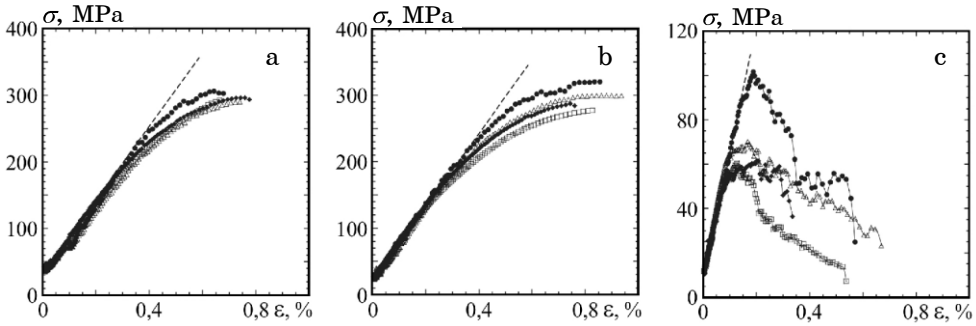

Fig. 1 *– Diagrams of the deformation for* Au *films of the thickness of* 0,3 *(a);* 0,5 *(b) and* 1,0 *(c) μm. Film width, μm:* 2,5 *(•);* 5,0 *(Δ);* 10 *(◆) and* 20 *(□). From [12]*

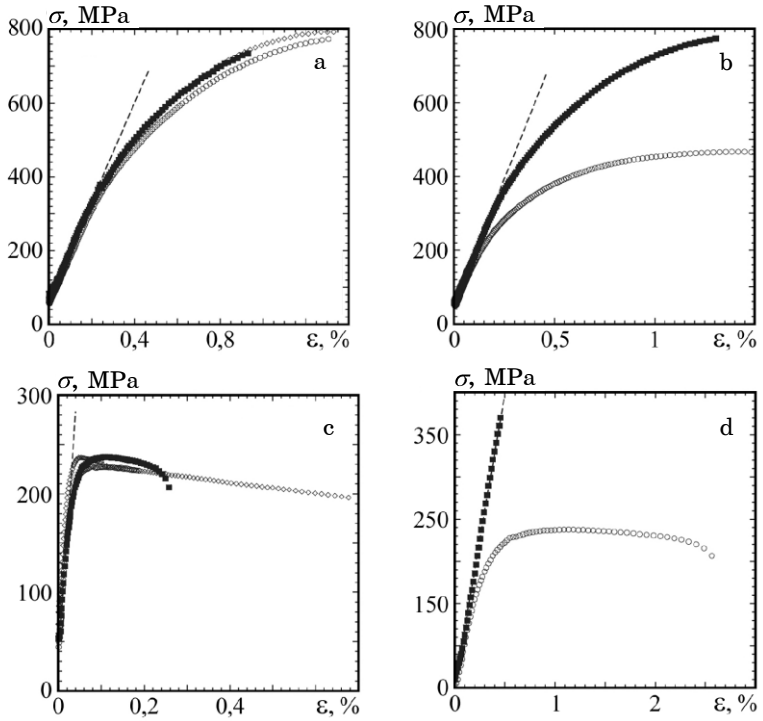

Fig. 2 *– Diagrams of the deformation for* Cu *(a, b) and* Al *(c, d) films of the thickness of* 0,2 *(■) and* 1,0 *(○) μm. Film width, μm:* 5 *(◇);* 10 *(■) and* 20 *(○). From [12]*

The authors of [7, 17] by the example of Al and Al-1,5%Ti and Ti-(48-51)%Ni films obtained the diagrams of the deformation and established that $\varepsilon_{tr} \cong 0{,}17$ and 0,30% and 1,0 and 2,0%, respectively. The authors of [18] studied the dependence of ε_{tr} on the thickness of Cu films obtained by the ion or magnetron deposition. It was established that during ion deposition $\varepsilon_{tr} \cong 0{,}40\%$ at $d \cong 0{,}5\text{-}1{,}5$ μm and during magnetron deposition $\varepsilon_{tr} \cong 0{,}1\text{-}0{,}3\%$ at $d \cong 1{,}5\text{-}2{,}5$ μm.

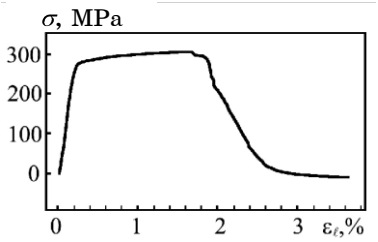

Fig. 3 *– Diagram of the deformation for* Pd *film of the thickness of* 4 *μm. From [15]*

Analysis of cited publications indicates that the main attention is devoted to the investigation of the correlation between the mechanical properties and structural state of the sample. In this case, as we mentioned in [21, 22], the study of the tensoresistive effect in metal films in the range of severe deformations (up to 2%) involving elastic, quasi-elastic and plastic deformations is almost absent.

From the most general considerations using relation for the averaged (in the measured deformation ranges) longitudinal gauge factor

$$\gamma_l = \gamma_l^\rho + 1 + 2\mu,$$

where $\gamma_l^\rho = \frac{1}{\rho} \cdot \frac{\partial \rho}{\partial \varepsilon_l}$, ρ is the resistivity (index "ρ" denotes that γ_l is expressed in terms of the resistivity), μ is the Poisson's ratio (which is equals to 0,5 in the case of the plastic deformation). The author of [23] with the assumption that at the grain-boundary slippage of the grains their bulk resistance does not change ($\gamma_l^\rho \cong 0$) concludes that $\gamma_l \cong 2$ for the plastic deformation. This result is somewhat confirmed in the case of bulk macrocrystalline samples. For example, in accordance with our data [24] obtained for Cu, Ni and Mo wires (the diameter D is from 0,06 to 0,44 mm; the length is from 7 to 50 cm) $\gamma_l^\rho \cong -$ (0,2-0,3) at $\mu \cong$ 0,4-0,5 (Cu); (0,25-0,50) at $\mu \cong$ 0,35-0,40 (Ni) or 0,75 at $\mu \cong$ 0,37-0,40 (Mo), respectively (as an example in Fig. 4 we present the data for thin Cu wires), that agrees with the data of [19, 20] for μ. Note, that extension of the samples lasted until the rupture of the wires and this allowed to determine their failure deformation: $\varepsilon_{l\max}^{Cu} \cong 0{,}8\%$ or 1,6% (D = 0,06 or 0,12 mm); $\varepsilon_{l\max}^{Ni} \cong 0{,}5\%$ (D = 0,44 mm) and $\varepsilon_{l\max}^{Mo} \cong 1{,}06\%$ (D = 0,21 mm).

In film materials the grain-boundary electron scattering (see, for example, [22, 25]) makes a substantial contribution to the gauge factor, that is conditioned the value of γ_l up to some tens of units [21, 25]. Obviously, this scattering mechanism, which leads to the decrease in the mean free path and its corresponding increase due to the increase in the crystalline size, conditions the result value of $\gamma_l^\rho < 0$ (Cu and Mo wires) and $\gamma_l^\rho > 0$ (Ni wire).

Based on the presented data, the aim of our investigation can be stated as follows: to study the features of the tensoresistive effect in single-layer (Fe, Cr, Pd and Pt) and two-layer (Fe/Cr, Cu/Cr and Pd/Fe) films. In particular, to obtain the dependence of ε_{ltr} on the thickness by the tensoresistive method.

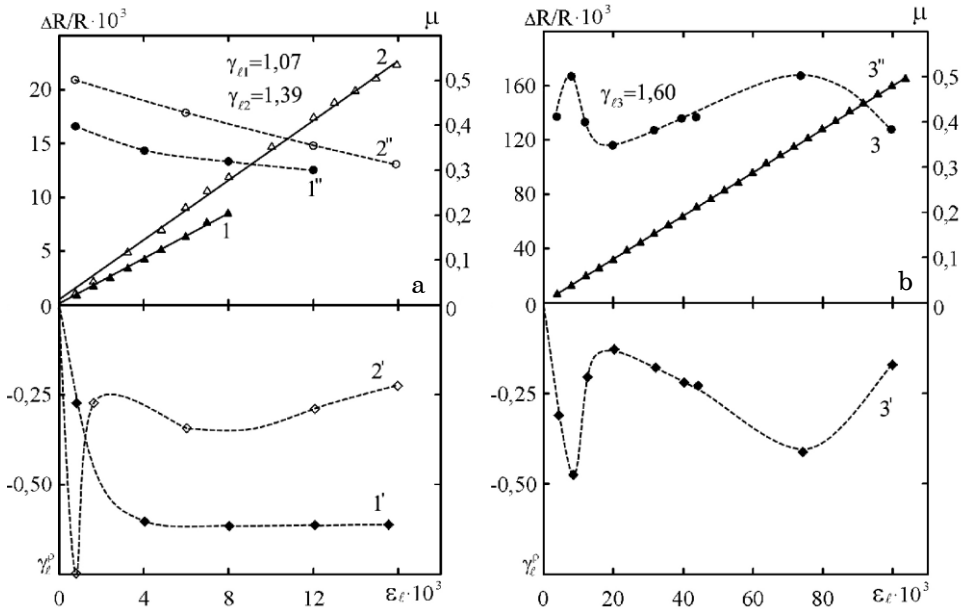

Fig. 4 *– Dependence of the relative resistance change ΔR/R (1-3); γ_l^ρ (1′-3′) and calculated μ (1″-3″) for* Cu *wires. Diameter, mm:* $6 \cdot 10^{-2}$ *(1),* $12 \cdot 10^{-2}$ *(2) (a) and* $28 \cdot 10^{-2}$ *(3) (b)*

3. CORRELATION BETWEEN THE ELECTROPHYSICAL PROPERTIES OF NANODIMENSIONAL FILMS AND THEIR LONGITUDINAL DEFORMATION

Single- and two-layer films (thickness of the separate layers is from 10 to 80 nm) were obtained by the thermal evaporation method in the chamber of the plant VUP-5M (pressure of the residual gases is ~ 10^{-4} Pa) on the polystyrene substrate, which was beforehand prepared by the contact pads [21, 25]. The advantage of such a substrate in comparison with the fiberglass and fluoroplastic ones or nickel foil substrate coated by dielectric we used before is that it has elastic properties in the deformation range up to 2%. We have especially to note that high adhesion of metal films to polystyrene leads to measuring the tensoresistive properties on the system film/substrate. In this case since the elasticity limit of metal films ε_{ltr} is less than 2%, all measurements were performed in the conditions of elastically or plastically deformed film and elastically deformed substrate. It is necessary to note, that the given technique of measuring the electrophysical properties of the relatively thin films (up to 100 nm) is used by other authors as well (see, for example, [26]). We were guided by the results of Ref. [10-15] while determining the value of ε_{ltr} by the indirect method (using the dependence of the resistance R on ε_l).

Investigation of the tensoresistive properties was carried out using the developed automated system [21, 25] that gave the possibility to realize many deformation cycles “stress-strain” under static and dynamic conditions at the deformation speed from 0 to 0,1 %/s. Hardware of this system consists of the frequency meter; 8-channel 16-bit sigma-delta ADC ADAM-4018 using

which the sample resistance is measured; relay module ADAM-4068 (which is the source of driving voltage of the electric motor); USB → RS232/422/485 interface converter ADAM-4561; asynchronous capacitor motor and web-cam Creative Labs. The experiment control and result processing were performed based on the software developed in LabVIEW 8.50 using the machine vision module LabVIEW Vision Development Module 8.2.

Investigation of the crystal structure and phase composition was carried out by the transmission electron microscopy and the electron-diffraction methods. In accordance with the obtained results Cr and Fe films have the bcc- and Cu, Pd and Pt have the fcc-lattices with the parameters close to those for bulk samples. Phase composition of two-layer Fe/Cr and Pd/Fe films corresponds to the bcc or fcc of (α-Fe, Cr) and (Pd, Fe) solid solution, and of Cr/Cu films – to the bcc-Cr + fcc-Cu (biplate). Structural state of film samples before their failure was controlled by the optical method using light-emitting and photodiodes placed above and below the substrate, respectively.

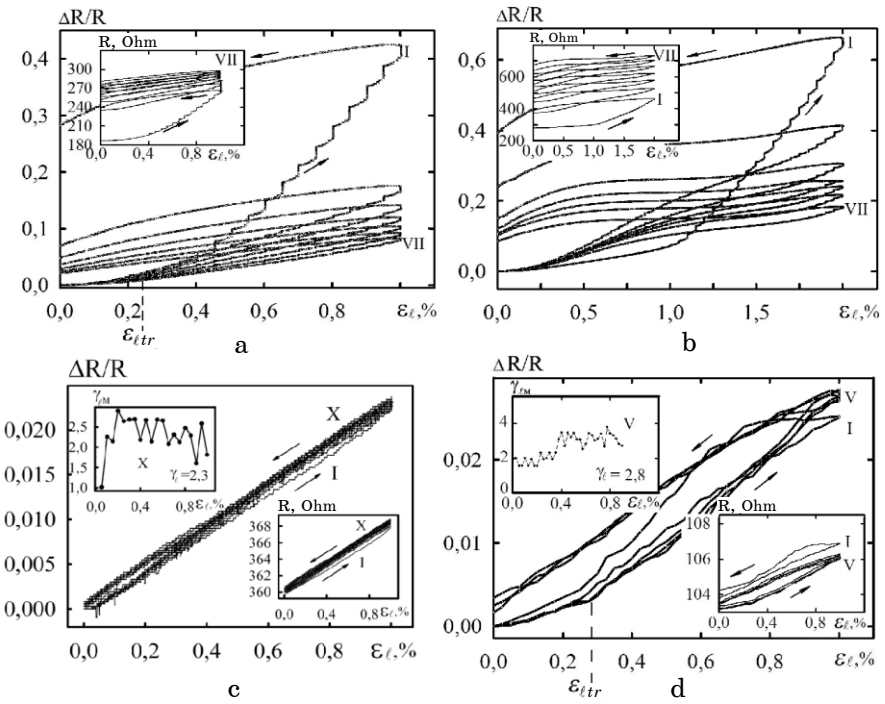

Fig. 5 *– Dependence of the relative resistance change ΔR/R, resistance R and instantaneous gauge factor γ_{lm} on ε_l for* Cr *(30) (a, b),* Pt *(38) (c) and* Pd *(30) (d) films (thickness in nm is given in round brackets). On the insets: dependences of R and γ_{lm} on ε_l for different deformation cycles (they are denoted by the Roman numerals)*

In Fig. 5 we present the examples of deformation dependences for single- and two-layer films for two deformation ranges $\Delta\varepsilon_{l1}$ = (0-1)% and $\Delta\varepsilon_{l2}$ = (1-2)%. Their features are the following: after the transition from elastic to plastic deformation during the first deformation cycle (for example, at $\varepsilon_{ltr} \cong$ ≅ 0,2% in Fig. 5a) the sample exists in a plastic state during next defor-

mation cycles, and the change $\Delta R/R = [R(\varepsilon_l) - R(0)]/R(0)$, where $R(0)$ is the initial resistance (for example, at $\varepsilon_l \cong 1{,}0\%$ in Fig. 5b), is conditioned, to our opinion, by the different mechanisms of the plastic deformation [4-8, 12], namely, by the dislocation movement to the grain boundaries, rotation and thinning and by the grain-boundary slippage. Note, that in Fig. 5a,b we present the deformation dependences for one and the same sample, which firstly was deformed during seven cycles in the range $\Delta\varepsilon_{l1} = (0\text{-}1)\%$ and afterwards – in the range $\Delta\varepsilon_{l2} = (1\text{-}2)\%$.

We should point to the rather wide range of the elastic deformation of Pt films (Fig. 5c). In the case of two-layer films (Fig. 6) the dependences have almost the same behavior.

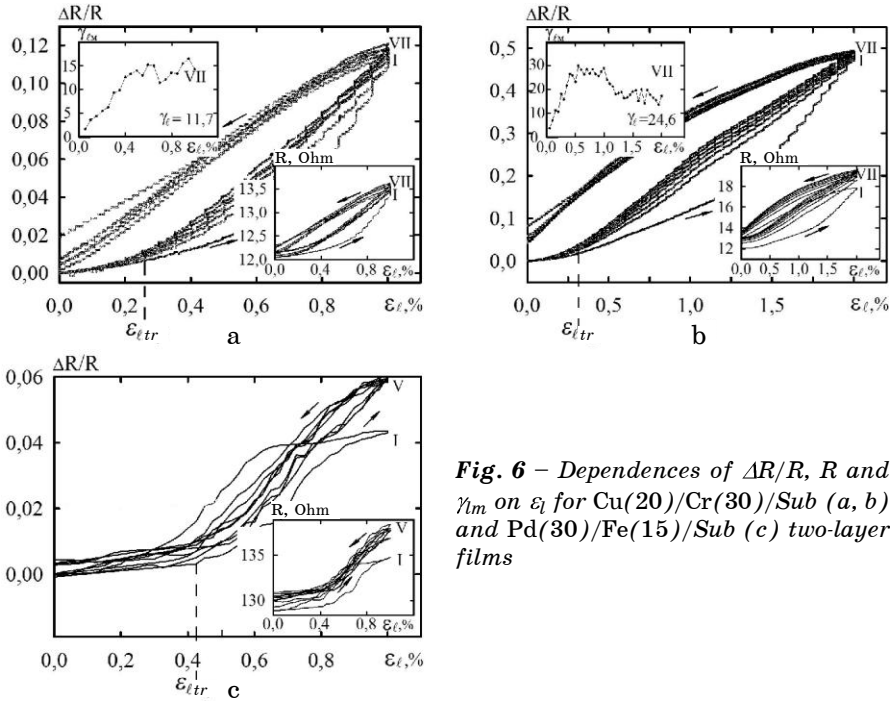

Fig. 6 *– Dependences of $\Delta R/R$, R and γ_{lm} on ε_l for* Cu*(20)*/Cr*(30)*/*Sub (a, b) and* Pd*(30)*/Fe*(15)*/*Sub (c) two-layer films*

Based on the obtained deformation dependences we calculated the averaged (over the whole deformation range) values of the gauge factor γ_l. Generalization of the results of the tensoelectric study, which agree with the results for micron films [12, 15, 16], are presented in Table 1.

Table 1 *– Dependence of ε_{ltr} on the total film thickness*

Film	Cr		Pd		Fe/Cr		Cu/Cr		Pd/Fe
Thickness, nm	30	75	18	100	Fe(20)/ Cr(30)	Fe(40)/ Cr(30)	Cu(20)/ Cr(20)	Cu(20)/ Cr(35)	Pd(10)/ Fe(20)
ε_{ltr}, %	0,20	0,10	0,52	0,25	0,40	0,30	0,40	0,25	0,48

As seen from Fig. 6 the value of γ_l in the case of the plastic deformation is equal to some tens of units. Note, the authors of Ref. [26] also obtained that in the range of elastic or plastic deformation of Au films ($d \cong 100$ nm) $\gamma_l \cong 1$ or 90, respectively, and this agrees with our results and does not correspond to the general considerations of the author of [23].

4. CONCLUSIONS

1. Analysis of the publications shows that such mechanical characteristics like the limits of elasticity and hardness in nanodimensional and micron films several times more than those for bulk polycrystals, although the values of the Young's modulus are approximately equal.
2. In thin single- and two-layer films based on Fe, Cr, Cu and Pd the elasticity limit is $\varepsilon_{ltr} \cong 0{,}2\text{-}0{,}5\%$, while in Pt films it is more than 1%.
3. In the case of the elastic deformation of nanodimensional films the longitudinal gauge factor is about 1, and in the case of the plastic deformation it is equal to some tens.

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