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STRAIN-RESISTIVE PROPERTIES OF $(Bi_{0.25}Sb_{0.75})_2Te_3$ FILMS AT ONE-SIDED CYCLIC ALTERNATING STRAINS

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The results of a study of the deformation characteristics of polycrystalline films from the $(Bi_{0.25}Sb_{0.75})_2Te_3$ solid solution at one-sided cyclic alternating mechanical stresses is presented. The films were obtained on a polyamide substrate by the method of thermal vacuum condensation of molecular beams and had a columnar porous structure with the dimensions of individual crystalline grains of 2.0-2.5 μm. The effect of static and cyclic deformations on the electrical resistance and volt-ampere characteristics of strain gauge films was studied in order to manufacture strain gauges for fatigue damage accumulation on their basis. It was shown that at room temperature such films have an abnormally high static strain sensitivity $G \approx 10^3$ arb. units and a significant hysteresis of their resistance change was detected at small numbers of alternating deformation cycles. As a result of $N = 5 \cdot 10^5$ deformation cycles, the linear section of the volt-ampere characteristic expands from (0-5) V at $N = 0$ to (0-12) V. And the temperature coefficient of resistance in the range of 293 K-T_{min} changes from $\alpha = -5.6 \cdot 10^{-3} K^{-1}$ to $\alpha = -2.5 \cdot 10^{-4} K^{-1}$. Thecharacteristic value of T_{min}, at which $\alpha = 0$, increases with the growth of N. The studied strain gauge films can be successfully used as a sensor of fatigue stress accumulation in the temperature range of T = 273-413 K and the value of $N = 0 – 5 \cdot 10^5$.
Keywords: *Narrow-gap semiconductor $(Bi_xSb_{1-x})_2Te_3$; Porous polycrystalline films; Strain sensitivity; Cyclic alternating strain; Hysteresis of changes in electrical resistance with strain; Strain gauge of fatigue damage accumulation*
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INTRODUCTION

The strain gauge method for studying the properties of semiconductor materials is one of the most common and informative methods for studying thin-film structures, which are widely used to manufacture electrical sensors [1-4], pressure and displacement sensors [5-7]. From the point of view of the technical application of thin semiconductor films as a strain-sensitive element, the current-voltage characteristic (CVC) plays a special role, in particular, it allows us to judge their energy capabilities and suitability for strain gauge measurement. An important parameter is the maximum permissible power consumption of the strain gauge under specified operating conditions. This is due to the fact that semiconductor films are very sensitive to various types of radiation and temperature changes, and when passing high currents through film samples, their excess Joule heating may occur, which is reflected accordingly in their operating parameters. Thus, it can lead to a nonlinear CVC and, thus, to an increase in the error in the instrument readings. In addition, to ensure safe operation of structures such as aircraft and prevent their destruction during operation, it is necessary to know the number of deformation cycles that a given structure has experienced. In this regard, film strain gauges of fatigue damage accumulation (FDA) based on narrow-band semiconductors [5-10] are successfully used, changing their characteristics with an increase in the number of deformation cycles acting on them.

As is known [7-11], the main electrophysical characteristics of strain gauges are the initial values of electrical resistance R_0, strain sensitivity G_0 and their changes under the influence of external factors, such as cyclic mechanical deformation ε, temperature T, frequency of the supply alternating voltage f etc. Recently, due attention has been paid to the study of the features of low- and high-cycle fatigue of strain gauges made of low-dimensional structures at different deformation amplitudes [12-16]. It should be noted that the authors and their colleagues have recently obtained interesting results in the study of the strain gauge properties of thin polycrystalline films of narrow-band semiconductors [17-21]. Although it has been shown [9-11] that the nature of the anomalously high strain sensitivity $G \approx 10^4\ arb.units.$ of porous films $(Bi_xSb_{1-x})_2Te_3$ is associated with the formation of microcracks at high deformation amplitudes $\varepsilon \approx 10^{-3}$, however, the mechanisms of cyclic deformations at arbitrary amplitudes and amounts of stresses still remain unclear.

This work is devoted to the study of the influence of the number of cycles N and the amplitude ε of alternating deformation on the characteristics of films from a solid solution $(Bi_xSb_{1-x})_2Te_3$ according to the change in the value of its electrical resistance and the strain gauge coefficient (SGC), determined by the expression

$$G = \frac{\Delta R}{R_0 \cdot \varepsilon}, \tag{1}$$

where $\varepsilon = \Delta\ell/\ell_0$ and $\Delta\ell = \ell(\varepsilon) - \ell_0$ are the values of relative and absolute strain, $\Delta R = R(\varepsilon) - R_0$, and R_0 are the electrical resistance of the sample at $\varepsilon = 0$.

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The current-voltage characteristic and temperature dependence of the resistance of $(Bi_{0.25}Sb_{0.75})_2Te_3$ strain gauge films under the action of static and cyclic alternating deformations (CAD) are considered with the aim of manufacturing FDA based on them. It is shown that as a result of the action of $N = 5\cdot 10^5$ strain cycles, the linear section of the current-voltage characteristic expands from (0-5) V at $N = 0$ to (0-12) V. The studied films can be successfully used as FDA in the temperature range of $T = 273-413\ K$ and the value of $N \leq 5\cdot 10^5$.

TECHNOLOGY AND MEASUREMENT METHODS

Polycrystalline films of $(Bi_xSb_{1-x})_2Te_3$ with an area of $5\times 20\ mm^2$ were obtained by thermal evaporation in a vacuum with a residual vapor pressure of $(1-3)\cdot 10^{-2}\ Pa$ from a mixture of powders Bi_2Te_3 and Sb_2Te_3 in a ratio of x и $(1-x)$ mol %. The temperature of the substrate made of polyamide PM-1 varied in the range of $T_S = 323-423\ K$, and the growth rate of the films was $-\ W = 150-450\ \overset{0}{A}/c$. The most strain-sensitive ($G \approx 10^3\ arb.units.$) films with optimal performance characteristics were obtained at thicknesses of $d \approx 3-5\ \mu m$, $T_S = 363\ K$, $W = 200\ \overset{0}{A}/c$ and at a value of $x = 0.25$. The method for measuring the deformation characteristics of the films was chosen in the same way as in [1, 7-9]. The samples were not subjected to preliminary heat treatment. The freshly prepared films had an unstable SGC, caused by the presence of strong nonequilibrium internal mechanical stresses (IMS) [11]. With an increase in the N number of CAD, the value of G decreases monotonically, which indicates the possibility of using the manufactured films as FDA. Electron microscopic and X-ray structural studies [3, 7] showed that the grown $(Bi_{0.25}Sb_{0.75})_2Te_3$ layers had a polycrystalline columnar and porous structure. The sizes of individual crystalline grains were $2-2.5\ \mu m$.

To measure the deformation characteristics (DC), the studied films from $(Bi_{0.25}Sb_{0.75})_2Te_3$ were glued to a beam of equal resistance made of titanium alloy. The deformation of the films was carried out by bending the beam. In this case, the value of the relative deformation ε was calculated by the magnitude of the bending of the cantilever-fixed beam according to the known expression [1]

$$\varepsilon = 3ab\Delta x/\ell^3, \tag{2}$$

where a is the distance from the neutral axis of the cantilever beam to the film, b is the distance from the point of application of the force to the middle of the film sample, Δx is the bending of the free end of the plate at the point of application of the force, ℓ is the length of the plate between the support point and the point of application of the force. The deformation value varied in the range from $\varepsilon = +2\cdot 10^{-3}$ to $\varepsilon = -2\cdot 10^{-3}$ arb. units.

Strain measurements were made at different temperature ranges of the environment. In order to reduce the measurement error, it is necessary to manufacture strain gauges with minimum temperature coefficients of resistance and strain sensitivity.

$$\alpha = dR/R^0dT,\ \alpha' = dG/G^0dT, \tag{3}$$

where R^0 and G^0 are the resistance and SGC at a temperature of $T_0 = 273^0\ K$ in a given mechanical state of the films. The study of these parameters of semiconductor strain gauges will to a certain extent help to explain the nature of the physical processes occurring in such a heterogeneous structure as a porous polycrystalline film subjected to mechanical deformation [7, 8].

The temperature dependence of the tensometric parameters of the films $(Bi_{0.25}Sb_{0.75})_2Te_3$ produced was investigated in the range 293-455 K. It turned out that at high substrate temperatures $T_s \approx 413\ K$ and high condensation rates $W \approx 400\ \overset{0}{A}/c$ denser films with a small SGC are obtained. The resistance of such films has a metallic dependence with temperature ($\alpha = 0.85\cdot 10^{-4}K^{-1}$) and it remained practically unchanged after exposure CAD.

RESULTS OF THE EXPERIMENT AND THEIR DISCUSSIONS

1. The region of small values of N.

Here we first present the results of the study of the absolute $\Delta R = R(\varepsilon) - R_0$ and relative $\Delta R(\varepsilon)/R_0$ changes in the resistance of freshly prepared samples with a small number of cycles of mechanical loading with a change not only in value but also in sign. For example, at the first stage, we will consider the region of tensile deformation from $\varepsilon = 0$ to $\varepsilon = \varepsilon_0$, at the second stage we will obtain in the direction of deformation removal, i.e. when changing ε from ε_0 to 0, at the third and fourth stages we radiate the region of compression deformations, first from $\varepsilon = 0$ to $\varepsilon = -\varepsilon_0$, and then from $\varepsilon = -\varepsilon_0$ to $\varepsilon = 0$ and complete one cycle of alternating deformations. The strain-sensitive films made from $(Bi_{0.25}Sb_{0.75})_2Te_3$ withstood quite large numbers CAD before noticeable mechanical destruction. It is obviously of interest to study DC films in extremely small and large quantities N CAD.

Fig. 1 shows DC at small values of N, and here the change in R film was first studied only at 4-x cycles of tensile deformation ($0 \leq \varepsilon \leq 0.9\cdot 10^{-2} arb.units.$), and then at 4-x cycles of compressive deformation ($-0.9\cdot 10^{-2} \leq \varepsilon \leq 0$). It is seen that when we first smoothly increase the load to ε_0 and then also smoothly remove it to 0, we observe a residual change in the relative resistance of $\Delta R^0_{res}(N)/R_0$. This value is different for stretching and compression, which reflects the presence of IMS in the film. It is seen that in this case IMS is negative (compressed $\varepsilon_0 < 0$), in addition, it decreases monotonically with the growth of *N*.

Thus, in films $(Bi_{0.25}Sb_{0.75})_2Te_3$, a significant hysteresis of resistance change $R_N(\varepsilon)$ is observed with one CAD $(N = 1)$ with an unclosed end. The following hysteresis loops directly continue the previous one and shift to the region of large values R, and the absolute value of the vertical displacement ΔR for the next loop decreases monotonically (Fig. 2).

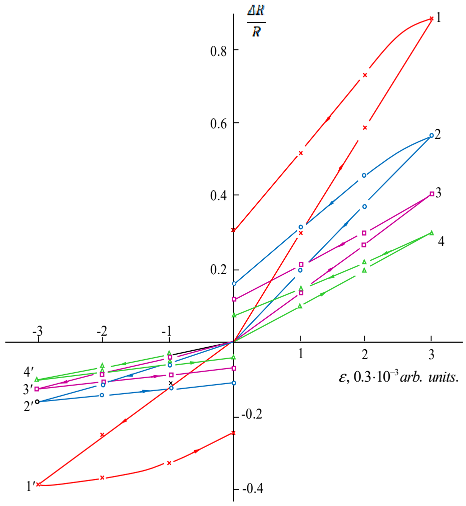

Figure 1. Deformation characteristics of freshly prepared film $(Bi_{0.25}Sb_{0.75})_2Te_3$ under 4-x cycles of tensile deformation (curves 1-4 with arrows indicating the direction of loading) and compression (curves 1′-4′) at room temperature

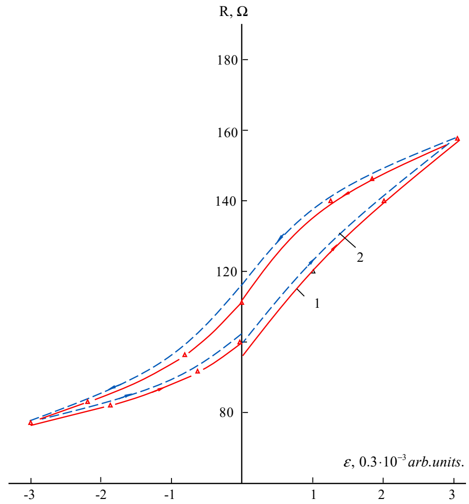

Figure 2. Hysteresis of the change in resistance of the film from $(Bi_{0.25}Sb_{0.75})_2Te_3$ under the influence of CAD. The open hysteresis with dashed lines corresponds to the second CAD (*N*=1)

Fig. 3 shows the dependence of the change in resistance and SGC on the number CAD in the region $1 \le N \le 4$. It is evident that the value of G decreases sharply, and R increases, with an increase in the number CAD at low N, and then passes to a smooth monotonic decline. Apparently, the initial sections of the dependence $R_\varepsilon(N)$ and $K_\varepsilon(N)$ are due to strongly nonequilibrium IMS films $(Bi_{0.25}Sb_{0.75})_2Te_3$ without preliminary heat treatment.

2. The region of large values of the number of deformation cycles. Stabilization of the strain gauge parameters of films.

Figure 4 shows the static deformation characteristics of the films before and after exposure to cyclic alternating loads in large quantities $(N \gg 1)$. It can be seen that before exposure to deformation cycles, the film resistance increases almost linearly under the action of tensile deformation, while the dependence of the resistance on compressive deformation is nonlinear, and the value of the strain-sensitivity coefficient under compressive deformation is noticeably less than under tensile deformation (curve 1).

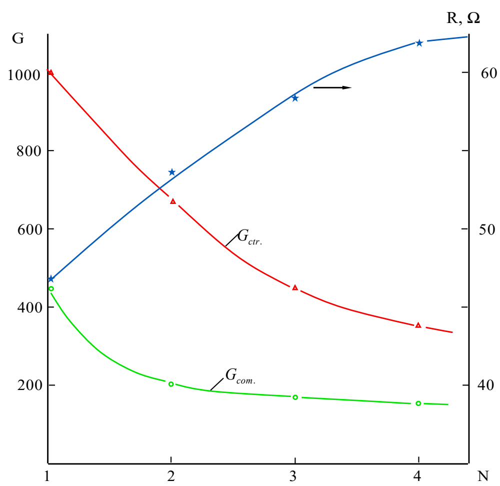

Figure 3. Change in resistance *R* and SGC with the growth of the number CAD in the area of $1 \le N \le 4$

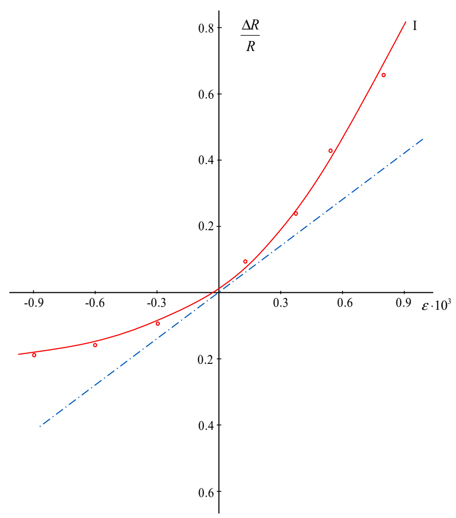

Figure 4. Deformation characteristic of the relative change in resistance of films from $(Bi_{0.25}Sb_{0.75})_2Te_3$ before (curve 1) and after (2) cyclic deformation. For curve $N = 5 \cdot 10^5$

After exposure to deformation cycles ($N \cong 5 \cdot 10^5$), the nonlinearity of the static DC film decreases, its strain sensitivity increases during compression deformation, and decreases during tension (curve 2), i.e. the asymmetry practically disappears.

Fig. 5 and Fig. 6 show the dependence of the relative change in resistance $\Delta R/R_0$ and the strain-sensitivity coefficient G on the number of deformation cycles at different deformation amplitudes ε. It is evident that with an increase in the number of alternating deformation cycles and its amplitude, the relative change in resistance increases, while their strain-sensitivity G decreases, and at values ($N = 5 \cdot 10^5$) in the dependences $R(N), G(N)$, a tendency toward saturation is observed.

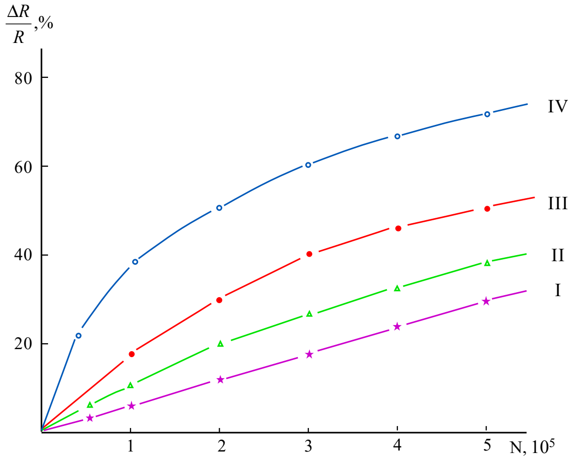

Figure. 5. Relative change in the resistance of strain films $(Bi_xSb_{1-x})_2Te_3$ from the number of cycles of alternating deformations N, at different deformation amplitudes $\varepsilon \cdot 10^{-3}$: I - ± 0.25, II - ± 0.5, III - ± 0.75 and IV - ± 1.0

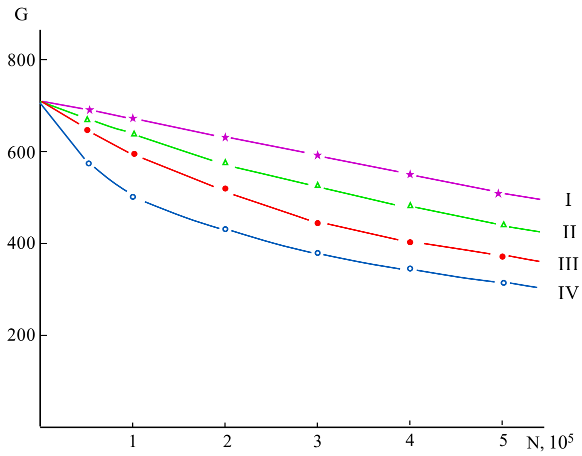

Figure 6. Dependence of G films on the number of deformation cycles at relative deformation amplitudes of $\varepsilon \cdot 10^{-3}$: I - ± 0.25, II - ± 0.5, III - ± 0.75 and IV - ± 1.0.

In our opinion, the experimental facts obtained here can be explained on the basis of a model of the film as a system of microcontacting conducting grains, the dielectric gap between which changes with deformation. Indeed, after the action of the required number of cycles of alternating deformation, the width of the gap between the crystalline grains increases as a result of abrasion of the contacting surfaces of the grains, leading to an increase in the film resistance and the removal to one degree or another of the preliminary IMS. The latter, in turn, determines the tendency toward a linear and symmetrical form DC of the film under the action of deformation cycles.

Therefore, it can be assumed that in the manufactured films $(Bi_{0.25}Sb_{0.75})_2Te_3$, high values of resistance and strain-sensitivity coefficient are correlated with the value of IMS, the dielectric gap and the size of the crystallites. The action of CAD leads to an increase in the change in the value of resistance and strain-sensitivity coefficient under compression, and to a decrease in the value of G under tension, as well as a change in the shape of the static deformation characteristic.

3. Effect of cyclic deformations on the temperature dependence of film resistance.

The temperature dependence of the electroresistance of the polycrystalline film $(Bi_{0.25}Sb_{0.75})_2Te_3$ and the influence of the current CAD can be qualitatively described on the basis of the linear model of microcontacting [7,8,11,21], according to which the resistance of the film is represented as the sum of the connected resistance of the intercrystalline interface $R_{por.i}$ and the individual crystalline levels $R_{cr.i}$

$$R_{film} = R_{cr} + R_{por} = \sum_i \left(R_{cr.i} + R_{por.i} \right) \ . \tag{4}$$

Here R_{cr} is the film resistance due to the volume of crystalline grains and depends on the temperature as

$$R_{cr} = R_{cr}^0 \left[1 + \alpha_{cr} \left(T - T_0 \right) \right] , \tag{5}$$

characteristic of metal resistors made of massive monocrystalline material, R_{cr}^0 - the resistance of a dense film at $T_0 = 273\ K$. We believe that the film resistance, caused by the presence of pores (interface potential barriers), will change with temperature as

$$R_{por} \approx R_{por}^0 \cdot exp\left(E_{por} / kT \right) \ , \tag{6}$$

where R_{por}^0 is some characteristic resistance of the porous film, weakly dependent on temperature, E_{por} is the height of the micropotential barrier, k is the Boltzmann constant.

Then, for the temperature coefficient of electrical resistance of the porous film, from (3-6) we obtain the phenomenological expression

$$\alpha_{film} = \frac{\alpha_{cr} \cdot R_{cr}^0 - \frac{E_{por}}{kT^2} \cdot R_{por}(T)}{R_{cr}^0 + R_{por}^0} . \quad (7)$$

From this it is clear that if at the value $T = T'$ the condition $\alpha_{cr} R_{cr}^0 < \frac{E_{por}}{kT'^2} \cdot R_f(T')$ is satisfied, then the porous film initially has a negative value of the temperature coefficient of resistance and at some $T = T_{\min} > T'$ it turns to zero, and then changes sign (i.e. acquires a metallic character of electrical conductivity). From (7) we find that $T_{\min}$ can be roughly estimated by the formula

$$T_{\min} = \sqrt{\frac{E_{por}}{k\alpha_{cr}} \cdot \frac{R_{por}(T_0)}{R_{cr}^0}} . \quad (8)$$

In practice, it can be assumed that E_{por} , α_{cr} , R_{cr}^0 weakly depend on CAD, and the interface resistance $R_{por}(T_0)$ increases with increasing *N*, then in accordance with formula (8), the value of $T_{\min}$ grows like $T_{\min} \sim \sqrt{R_{por}(T_0, N)}$ and at large $N \sim 5 \cdot 10^5$ experiences a tendency to saturation, which is observed in the experiment [11].

4. Effect of cyclic deformations on the current-voltage characteristics of films.

The effect of static deformation on CVC films was studied by us [13] in the range of relative deformation values up to $0.9 \cdot 10^{-3} arb.units.$, the corresponding curves are shown in Fig. 7. They show that CVC of the samples is linear in the region of low stresses. With increasing stress, the linearity of the current-voltage dependence is disrupted, which occurs, for example, in the undeformed state at *U=5 V* (curve 4). This stress, at which nonlinearity CVC occurs, depends significantly on the sign and level of deformation.

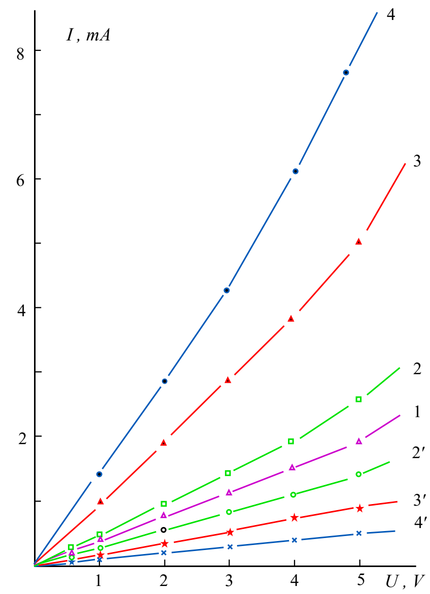

Figure 7. Volt-ampere characteristic of film $(Bi_{0.25}Sb_{0.75})_2Te_3$ on polyimide substrate ПМ-I, under the action of static deformation: ε=0 (curve 1), ± 0.3·10⁻³ (2,2′), ± 0.6 (3,3′), ±0.9 (4,4′). Curves 2-4 were taken under compression, and 2′-4′- under tension

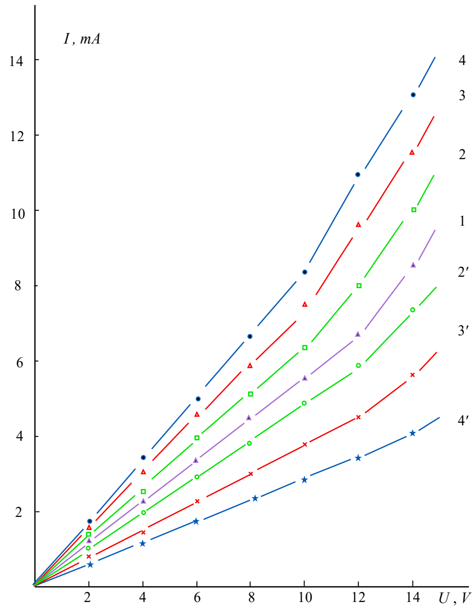

Figure 8. Volt-ampere characteristic of film $(Bi_{0.25}Sb_{0.75})_2Te_3$ after exposure to $N = 5 \cdot 10^5$ cycles of alternating loading. Curves 1-4 and 2′-4′ correspond to curves 1-4 and 2′-4′.

Studies of the current-voltage characteristics (CVC) of films $(Bi_{0.25}Sb_{0.75})_2Te_3$ subjected to CAD are shown in Fig. 8. It is evident from the figure that the CVC of the film change significantly after exposure to cyclic deformations $(= 5 \cdot 10^5)$. All curves taken at different values of relative deformation ε clearly reflect increases in film resistance. The action of $N = 5 \cdot 10^5$ cycles of alternating deformations leads to an increase in the voltage drop across the film with a constant source of almost 10 V, i.e., two-fold.

Since the distances between them increase under the action of cyclic deformations as a result of abrasion of the contacting surfaces of the crystalline grains, this leads to the expansion of the linear section CVC of the film. It is known that strain gauge measurements must be made in the linear region CVC. In this case, it is necessary to take into account the possibility of an error associated with the fact that a fairly large current can flow in the devices, which heats up the strain gauge and also causes the appearance of a nonlinear section CVC. In this case, not the nominal resistance is measured, but the static resistance corresponding to a certain point CVC. To reduce the current through the strain gauge, an additional resistance can be included, previously measured with sufficient accuracy. The measuring current through the strain gauge in this case should be an order of magnitude lower than the current causing a change in the electrical conductivity mechanism in the film or heating of the strain gauge body. The magnitude of this current can be roughly determined by CVC. Usually this is the current value where CVC begins to deviate from the linear dependence.

CONCLUSIONS

The changes in the resistance value of SGC porous films with increasing number of deformation cycles can be explained by the growth of the dielectric gap between crystallites and micro-wear of their contacts. The model describing the electrical conductivity through micro-contacting surfaces of crystallites, based on the theory of percolation, explains the high values of SGC films and the nonlinearity of their static deformation characteristics. The polycrystalline films of narrow-gap ternary compound $(Bi_{0.25}Sb_{0.75})$ studied here, obtained by thermal evaporation in a vacuum at the above-mentioned optimal process parameters, can be successfully used as FDA for testing and monitoring the parameters of aircraft structures in the temperature range of $T = 273 - 413\ K$ and the value of CAD $N \le 5 \cdot 10^5$.

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ТЕНЗОРЕЗИСТИВНІ ВЛАСТИВОСТІ ПЛІВОК $(Bi_{0.25}Sb_{0.75})_2Te_3$ ПРИ ОДНОСТОРОННІХ ЦИКЛІЧНИХ ЗНАКОЗМІННИХ ДЕФОРМАЦІЯХ

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Наводяться результати дослідження деформаційних характеристик полікристалічних плівок з твердого розчину $(Bi_{0,25}Sb_{0,75})_2Te_3$ при односторонніх циклічних знакозмінних механічних напруг. Плівки виходили на поліамідній підкладці методом термовакуумної конденсації молекулярних пучків і мали стовпчасту пористу структуру з розмірами окремих кристалічних зерен 2,0-2,5 мкм. Вивчався вплив статичних та циклічних деформацій на електричний опір та вольт амперні характеристики тензорезистивних плівок з метою виготовлення на їх основі тензодатчиків накопичення втомних ушкоджень. Показано, що при кімнатній температурі такі плівки мають аномально високу статичну тензочутливість $G \approx 10^3$ відн. од. і виявляється суттєвий гістерезис зміни їхнього опору при малих кількостях циклів знакозмінних деформацій. В результаті дії $N = 5\cdot10^5$ циклів деформацій лінійна ділянка вольтамперної характеристики розширюється від (0-5) при $N = 0$ до (0-12) В. Температурний коефіцієнт опору в інтервалі 293 K-T_{min}змінюється від $\alpha = -5{,}6\cdot10^{-3}K^{-1}$ до $\alpha = -2{,}5\cdot10^{-4}K^{-1}$. Характерне значення T_{min}, при якому $\alpha = 0$, збільшується зі зростанням N. Досліджені тензорезистивні плівки з успіхом можуть бути використані як датчик накопичення втомних напруг в інтервалі температур $T = 273 - 413\ K$ і значенні $N = 0 - 5\cdot10^5$.

Ключові слова: *вузькозонний напівпровідник $(Bi_xSb_{1-x})_2Te_3$; пористі полікристалічні плівки; тензочутливість; циклічна знакозмінна деформація; гістерезис зміни електричного опору з деформацій; тензодатчик накопичення втомних ушкоджень*