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BIR TOMONLAMA ISHORASI ALMASHINUVCHI TSIKLIK DEFORMATSIYALARGA UCHRAGAN G‘ OVAKLI (BixSb1-x)2Te3 PLYENKADA QARSHILIK O‘ ZGARISHINING GISTEREZISI

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Annotatsiya: *Ushbu ishda g‘ ovakli polikristall (Bi0,25Sb0,75)2Te3 plyonkalarning ishorasi almashinuvchi siklik mexanik deformatsiyalar ta’siridagi tenzorezistiv xususiyatlari tadqiq etilgan. Deformatsiya sikllari sonining plyonka elektr qarshiligi va tenzosezgirlik koeffitsientiga ta’siri tahlil qilindi. Kichik sikllar sonida qarshilikning sezilarli gisterezisi hamda deformatsiya olib tashlangandan keyin qoldiq qarshilik o‘ zgarishi kuzatilishi aniqlandi. Cho‘ zilish va siqilish jarayonlaridagi qoldiq qarshilik qiymatlarining farqlanishi plyonkadagi ichki mexanik kuchlanishlar mavjudligi bilan bog‘ landi. Sikllar soni ortishi bilan elektr qarshilikning ortishi, tenzosezgirlik koeffitsientining esa kamayishi va keyinchalik ularning o‘ zgarishi monoton xarakterga ega bo‘ lishi ko‘ rsatildi. Kuzatilgan hodisalar kristallitlar orasidagi dielektrik oraliqlarning o‘ zgarishi va elektr o‘ tkazuvchanlikning perkolyatsion mexanizmi asosida izohlandi. Olingan natijalar ushbu plyonkalarni konstruksiya elementlarida toliqish shikastlanishlarining to‘ planishini nazorat qiluvchi tenzosezgir elementlar sifatida qo‘ llash istiqbolini ko‘ rsatadi.*

Kalit so‘ zlar: *$(Bi_{0,25}Sb_{0,75})_2Te_3$ plyonka, polikristall plyonka, g‘ ovakli plyonka, tenzosezgirlik, elektr qarshilik, siklik deformatsiya, gisterezis, ichki mexanik kuchlanish, toliqish shikastlanishi.*

ГИСТЕРЕЗИС ИЗМЕНЕНИЯ СОПРОТИВЛЕНИЯ ПОРИСТОЙ ПЛЕНКИ (BixSb1-x)2Te3 ПОД ДЕЙСТВИЕМ ОДНОСТОРОННИХ ЦИКЛИЧЕСКИХ ЗНАКОПЕРЕМЕННЫХ ДЕФОРМАЦИЙ

Аннотация: *В работе исследованы тензорезистивные свойства пористых поликристаллических пленок $(Bi_{0,25}Sb_{0,75})_2Te_3$ при воздействии циклических знакопеременных механических деформаций. Проанализировано влияние числа циклов деформации на электрическое сопротивление и коэффициент тензочувствительности пленок. Установлено, что при малом числе циклов наблюдаются выраженный гистерезис сопротивления и остаточное изменение сопротивления после снятия нагрузки. Различие остаточных изменений сопротивления при растяжении и сжатии связано с наличием внутренних механических напряжений в пленке. С увеличением числа циклов электрическое сопротивление возрастает, а коэффициент тензочувствительности уменьшается, после чего изменение этих параметров приобретает более плавный монотонный характер. Наблюдаемые закономерности объясняются изменением диэлектрических зазоров между соседними кристаллитами и перколяционным механизмом переноса заряда через микроконтактирующие области. Полученные результаты показывают перспективность применения пористых пленок $(Bi_{0,25}Sb_{0,75})_2Te_3$ в качестве тензочувствительных элементов для контроля накопления усталостных повреждений в деталях и элементах конструкций.*

Ключевые слова: *пленка $(Bi_{0,25}Sb_{0,75})_2Te_3$, поликристаллическая пленка, пористая пленка, тензочувствительность, электрическое сопротивление, циклическая деформация, гистерезис сопротивления, внутреннее механическое напряжение, усталостное повреждение.*

HYSTERESIS OF RESISTANCE CHANGE IN A POROUS (BixSb1-x)2Te3 FILM SUBJECTED TO UNILATERAL CYCLIC ALTERNATING DEFORMATIONS

Abstract: *The strain-resistive behavior of porous polycrystalline $(Bi_{0,25}Sb_{0,75})_2Te_3$ films subjected to cyclic alternating-sign mechanical deformation is investigated. Particular attention is paid to the influence of the number of deformation cycles on the electrical resistance and strain sensitivity coefficient of the films. At a small number of cycles, a pronounced resistance hysteresis and a residual resistance change after unloading are observed. The difference between the residual resistance values obtained under tensile and compressive deformation is attributed to internal mechanical stresses in the film. With increasing number of deformation cycles, the electrical resistance increases, whereas the strain sensitivity coefficient decreases, followed by a transition toward a smoother monotonic variation. The observed behavior is interpreted in terms of changes in the dielectric gaps between neighboring crystallites and a percolation-type mechanism of charge transport through microscopic intercrystallite contacts. The obtained results demonstrate the*

potential of porous $(Bi_{0,25}Sb_{0,75})_2Te_3$ films as strain-sensitive elements for monitoring the accumulation of fatigue damage in structural components.

Keywords: *$(Bi_{0,25}Sb_{0,75})_2Te_3$ film, polycrystalline film, porous film, strain sensitivity, electrical resistance, cyclic deformation, resistance hysteresis, internal mechanical stress, fatigue damage.*

Introduction

To ensure the safe operation of engineering structures and to prevent their failure during service, it is essential to determine the number of deformation cycles experienced by a given structural element. For this purpose, fatigue-damage accumulation sensors can be employed. Such sensors, fabricated on the basis of semiconductor films, exhibit changes in their characteristics as the number of applied deformation cycles increases. As is well known [1-4], the principal electrophysical parameters of such strain-sensitive elements are the initial resistance R_0, the initial strain sensitivity K_0, and their variations under the influence of external factors such as temperature T, mechanical strain ε, frequency f of the applied alternating voltage, and other parameters. Recent developments further confirm the suitability of bismuth-telluride-based films for electromechanical sensing: p-type Bi-Sb-Te coatings have demonstrated durable piezoresistive response under repeated vibration/loading, while flexible Bi-Sb-Te architectures have been successfully employed in pressure-sensitive devices [5,8].

Therefore, the present study investigates the effects of the number of deformation cycles N and the strain amplitude ε under alternating loading on the characteristics of $(Bi_{0,25}Sb_{0,75})_2Te_3$ films. Particular attention is paid to variations in electrical resistance and the strain sensitivity coefficient (gauge factor), defined as

$$K = \frac{\Delta R}{R_0 \cdot \varepsilon}, \qquad (1)$$

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

The investigated $(Bi_{0,25}Sb_{0,75})_2Te_3$ films were deposited on polyimide substrates using the MP-I vacuum molecular-beam condensation method from the vapor phase, as described in detail in [1,2]. To determine the deformation characteristics, the films were bonded to an equal-strength beam made of a titanium alloy. Film deformation was produced by bending the beam. The relative strain was calculated from the bending of a cantilever-fixed beam and did not exceed $\varepsilon = \pm 1 \times 10^{-3}$ in relative units.

Low-N Region. Experimental Results and Discussion

This section presents the results of studying the absolute $\Delta R = R(\varepsilon) - R_0$ and relative $\Delta R(\varepsilon)/R_0$ changes in the electrical resistance of as-prepared samples at a small number of loading cycles, with the strain varied both in magnitude and sign. In the first stage, the tensile-strain region from $\varepsilon = 0$ to $\varepsilon = \varepsilon_0$ was investigated. In the second stage, the strain was gradually removed, i.e., ε was varied from ε_0 to 0. In the third and fourth stages, the compression region was examined: first from $\varepsilon = 0$ to $\varepsilon = -\varepsilon_0$, and subsequently from $\varepsilon = -\varepsilon_0$ back to $\varepsilon = 0$. These four stages constituted one complete cycle of alternating-sign deformation.

The strain-sensitive $(Bi_{0,25}Sb_{0,75})_2Te_3$ films were capable of withstanding a sufficiently large number of cyclic alternating-sign deformation cycles. Therefore, it is of particular interest to investigate the deformation characteristics of the films both at extremely small and at large values of the cycle number N. Repeated-bending studies on bismuth-telluride-based films supported by polymer substrates have likewise shown that electrical functionality during cyclic deformation is strongly governed by substrate compliance, film integrity, and microstructural design [7,9].

Figure 1 presents the deformation characteristics at small values of N. Initially, changes in the film resistance R were studied during four tensile-deformation cycles ($0 \le \varepsilon \le 0.9 \times 10^{-2}$), followed by four compression-deformation cycles ($-0.9 \times 10^{-2} \le \varepsilon \le 0$). It can be seen that, when the applied strain is first gradually increased to ε_0 and then smoothly reduced back to zero, a residual change in the relative resistance, $\Delta R^0_{re}(N)/R_0$, is observed. The magnitude of this residual resistance change differs

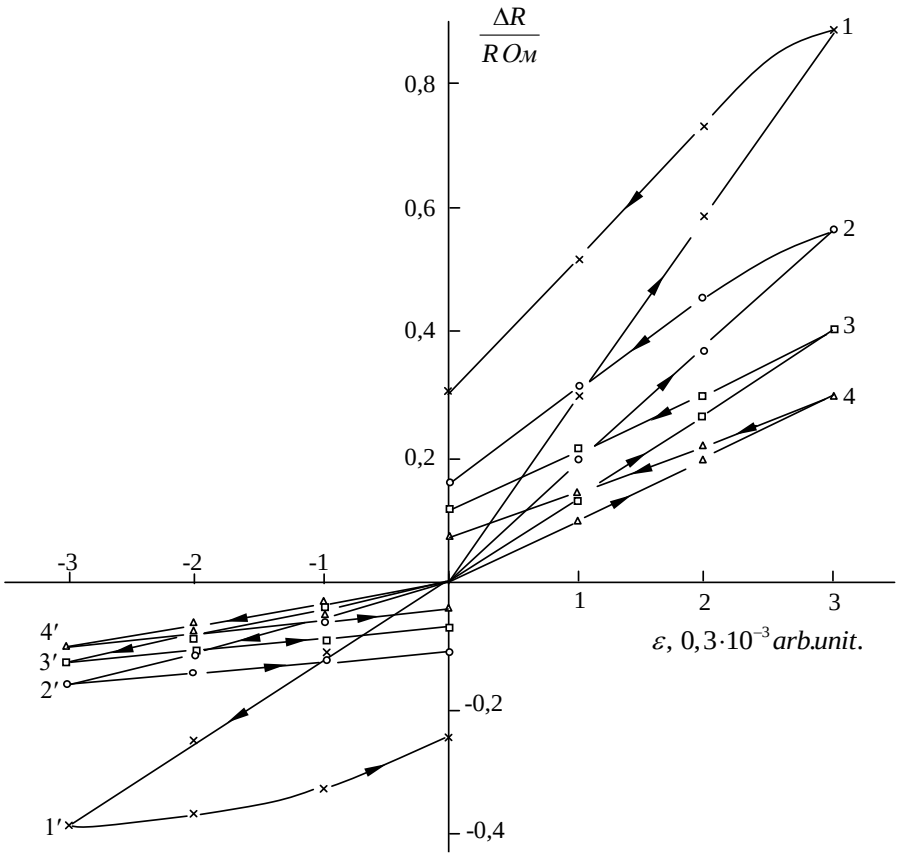

Fig. 1. Deformation characteristics of the as-prepared film at room temperature: **tensile deformation (curves 1–4) and compressive deformation (curves 1′–4′).**

for tensile and compressive deformation, indicating the presence of internal mechanical stress in the film. In the present case, the internal mechanical stress is negative, corresponding to a compressive state ($\varepsilon_0 < 0$ [2]). Moreover, its magnitude decreases monotonically with increasing N.

The pronounced evolution of *R a*nd *K* with cycle number is also consistent with recent evidence that grain-boundary structure and defect-mediated deformation can strongly influence the mechanical durability and electrical stability of Bi_2Te_3 thin films under repeated bending [6].

Thus, the $(Bi_{0,25}Sb_{0,75})_2Te_3$ films exhibit pronounced hysteresis in the resistance dependence $R_N(\varepsilon)$ during a single cycle of alternating-sign deformation, with the hysteresis loop remaining open. Each subsequent hysteresis loop directly continues the preceding one and is shifted toward higher resistance values *R* (Fig. 2).

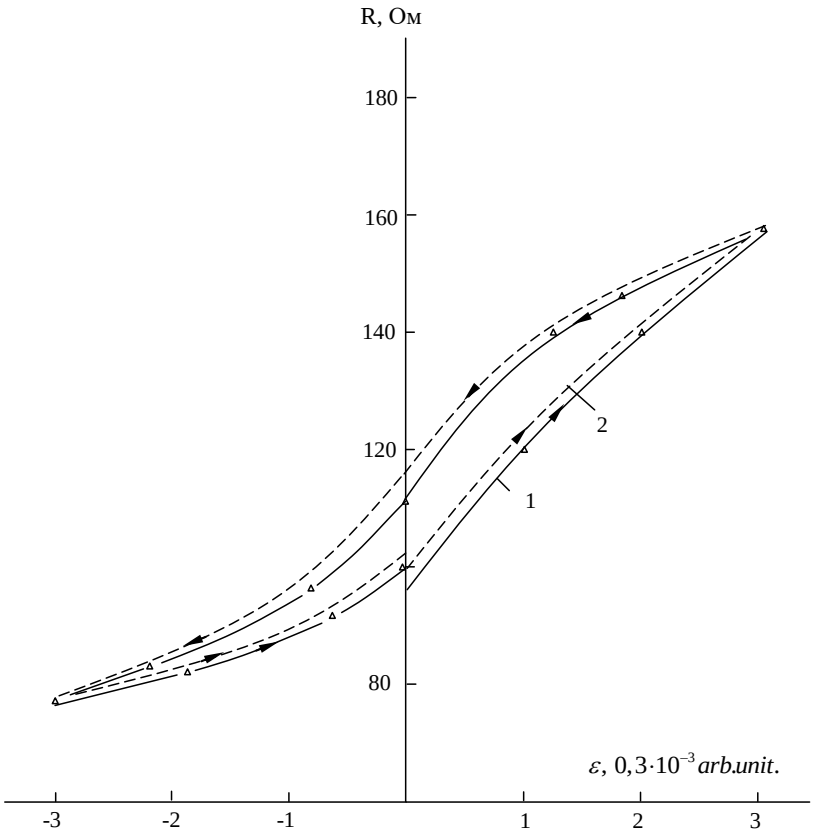

Fig. 2. Hysteresis of the resistance variation in a (BixSb1-x)2Te3 film under cyclic alternating-sign deformation. The open hysteresis loop shown by dashed lines corresponds to the second deformation cycle (N=2).

Figure 3 shows the dependence of the resistance variation and the strain sensitivity coefficient on the number of cyclic alternating-sign deformation cycles in the range $1 \le N \le 4$. It is evident that, at small N, the coefficient K decreases sharply, whereas the resistance R increases with the number of deformation cycles. With a further increase in N, the changes in these parameters become smoother and approach a monotonic regime. Apparently, the initial portions of the $R_\varepsilon(N)$ and $K_\varepsilon(N)$ dependences are governed by strongly nonequilibrium internal mechanical stresses present in the non-annealed $(Bi_{0,25}Sb_{0,75})_2Te_3$ films.

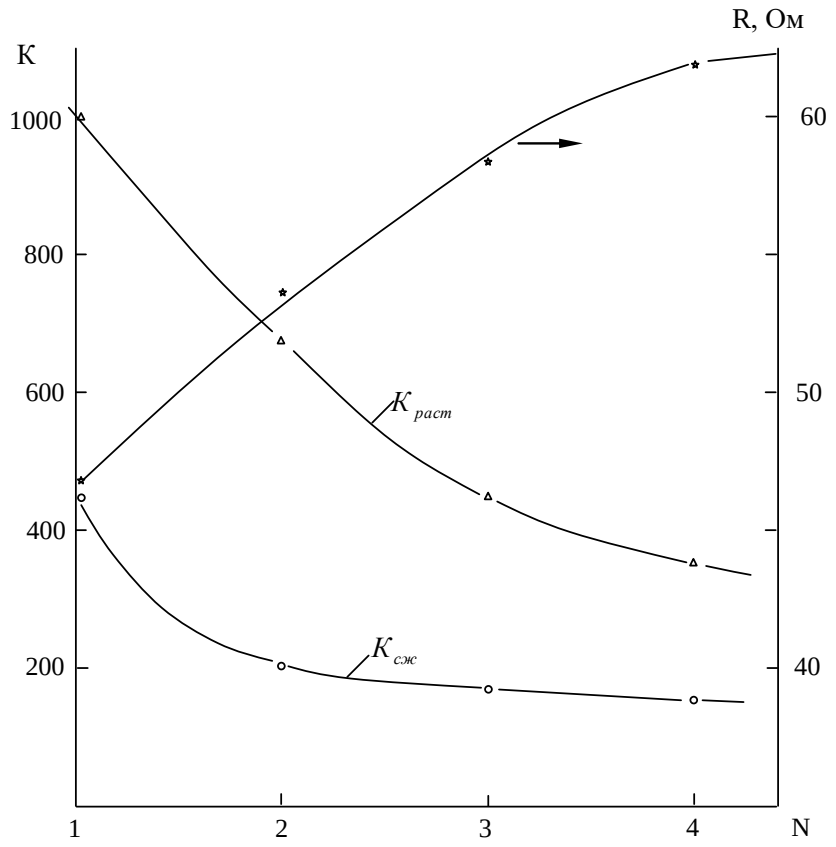

Fig. 3. Variation in the resistance and strain sensitivity coefficient of a film with increasing number of cyclic alternating-sign deformation cycles in the range. $\boldsymbol{RK(Bi_xSb_{1-x})_2Te_3 1 \leq N \leq 4}$

The observed hysteresis can be considered a consequence of the fact that the electrical state of a porous polycrystalline film is not determined solely by the instantaneous value of the externally applied strain. During loading and unloading, the microstructure undergoes a sequence of reversible and partially irreversible changes. In particular, deformation modifies the distances between neighboring crystallites and the effective area of microscopic contacts through which charge carriers pass. Because these structural changes do not necessarily return immediately to their initial configuration after removal of the external load, the resistance measured at the same strain can depend on the previous deformation history. This provides a physical basis for the open hysteresis loops observed in the present experiment.

The porous character of the film is especially important for this behavior. In a dense continuous film, the conducting path is distributed over a comparatively large number of direct contacts, whereas in a porous polycrystalline layer the current is concentrated through a limited network of intercrystallite conducting regions. A small change in the separation of neighboring crystallites can therefore produce a comparatively large change in the resistance. The deformation response is consequently amplified by the geometrical sensitivity of the microscopic conducting network. At the same time, this structure makes the electrical response sensitive to microstructural rearrangements accumulated during repeated deformation.

The transition from a pronounced cycle-dependent response at small N toward a smoother variation at larger N indicates that the film passes through an initial structural adjustment stage. During the first cycles, internal stresses and nonequilibrium configurations contribute substantially to the measured resistance. Repeated loading progressively redistributes these stresses and modifies the most weakly connected crystallite contacts. Once the principal microstructural rearrangements have occurred, subsequent deformation produces a more reproducible response. Thus, the reduction in the rate of change of K and the gradual increase in R should not be regarded as independent effects; they can be interpreted as two electrical manifestations of the same evolution of the conducting network.

The difference between tensile and compressive branches is also consistent with the asymmetric response of a porous contact network. Under tensile deformation, some intercrystallite gaps may increase and selected conducting paths may become less effective. Under compression, other contacts can approach each other, while local stress concentrations can simultaneously modify the contact geometry. Consequently, equal absolute values of positive and negative strain do not necessarily produce identical resistance changes. The residual difference after unloading provides additional information about the internal mechanical state of the film and its interaction with the polymer substrate.

From the viewpoint of strain-sensor operation, the strain sensitivity coefficient K provides information not only about the magnitude of the instantaneous electromechanical response but also about the state of the film after previous mechanical loading. The strong decrease of K during the initial cycles suggests that calibration performed only on a completely as-prepared sample may not represent the response after mechanical conditioning. For practical sensor design, an initial conditioning stage may therefore be useful before quantitative measurements of accumulated deformation are performed. At the same time, the residual resistance change can serve as an additional state parameter because it retains information about the deformation history.

The results are relevant to the development of sensors intended to monitor fatigue accumulation rather than merely to measure instantaneous strain. A conventional strain gauge is generally calibrated to reproduce the applied strain, whereas the present porous film exhibits a measurable change in its electrical state as the number of deformation cycles increases. This difference creates the possibility of using the film as a history-dependent sensing element. In such an approach, the resistance measured at zero external strain can be compared with the initial resistance R0, while the evolution of K can provide complementary information about changes in the deformation-sensitive conducting network.

Several factors should be considered when extending the present results to practical devices. The response of a porous semiconductor film may depend on temperature, film thickness, porosity, crystallite-size distribution, substrate elasticity, deformation amplitude, loading frequency, and the number of cycles. The present experiment was performed at room temperature and at a limited strain range; therefore, the obtained trends should be interpreted within these experimental conditions. In particular, separating purely mechanical effects from possible temperature-induced resistance variations would require simultaneous temperature monitoring during long-term cyclic tests. Additional experiments over a wider range of strain amplitudes and cycle numbers would also help determine the limits of the observed monotonic regime.

On the basis of the experimental observations, the deformation process can be represented schematically as a sequence of microstructural and electrical transformations. The initial film contains crystallites separated by microscopic dielectric or weakly conducting gaps. Application of strain changes these gaps and the geometry of contacting regions, thereby modifying the probability that a continuous conducting path is formed through the film. During repeated cycles, the most unstable contacts are progressively rearranged and internal stresses are redistributed. The resulting conducting network becomes less sensitive to further small rearrangements, which is reflected experimentally by the smoother evolution of resistance and the decrease in strain sensitivity. This interpretation is consistent with a percolation-type model of transport through a heterogeneous polycrystalline medium.

For practical implementation, the resistance response may be divided into two characteristic quantities: the instantaneous resistance change during deformation and the residual resistance change measured after unloading. The first quantity characterizes the current mechanical state, whereas the second contains information about the previous loading history. Recording both quantities during repeated cycles would allow the sensor response to be described by a two-parameter calibration rather than by a single resistance-strain curve. Such an approach could improve the identification of progressive changes in the film and provide a more informative indicator for fatigue-monitoring applications.

The combined analysis shows that the electrical response of the porous $(Bi_{0,25}Sb_{0,75})_2Te_3$ film contains both an instantaneous strain-dependent component and a history-dependent component. The latter is manifested by residual resistance and by the progressive stabilization of the hysteresis behavior with repeated deformation. Therefore, monitoring resistance at zero strain together with the strain sensitivity coefficient can provide complementary indicators of microstructural evolution during cyclic loading.

Conclusion

The observed changes in the resistance and strain sensitivity coefficient of the films with increasing number of deformation cycles can be attributed to an increase in the dielectric gaps between individual crystallites within the film. A model describing electrical transport through microscopic contacting regions between crystallite surfaces, based on percolation theory, accounts for both the high strain sensitivity of the films and the nonlinear character of their static deformation response. Porous $(Bi_{0,25}Sb_{0,75})_2Te_3$ films fabricated by thermal evaporation in vacuum under the optimal technological conditions described above can therefore be considered promising sensing elements for monitoring the accumulation of fatigue damage in components and structural elements.

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