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Influence of Plastic Deformation and Long-Term Natural Ageing on the Elastic Properties of Superplastic Eutectic Alloy Bi–43 wt.% Sn

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The study of changes in the dynamic Young's modulus of a typical model superplastic Bi–43 wt.% Sn alloy under the conditions of plastic deformation, under which polycrystalline materials are subjected in order to create a structural–phase state capable of manifesting the effect of superplasticity, is performed. Changes in the Young's modulus are also studied during long-term exposure at room temperature and normal atmospheric pressure, as a result of which the phenomenological indicators of the superplastic flow of the studied alloy are noticeably reduced, but the manifestation of the effect of superplasticity is observed. Acoustic measurements are carried out using the method of a two-component piezoelectric vibrator. An increase in the dynamic Young's modulus as a result of compression by ≅ 70% on a hydraulic press and in the ageing process is found in both cast and compressed samples. The obtained experimental data are analysed taking into account previously obtained data on changes in the phase composition of the alloy under experimental conditions. The results of the analysis show that the increase in the Young's modulus as a result of compression is caused by the appearance of internal stresses in the material. The increase in the Young's modulus during ageing is primarily related to the transition of the alloy from the initial metastable state to the phase state, which is in equilibrium at room temperature.

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On the kinetic dependences of the modulus of elasticity in both cast and compressed samples, there is an inhibition of its changes at the ageing stag, when the phase equilibrium in the alloy has not yet been established. This is explained by the change in the kinetics of the decomposition of the α(Sn)-phase (a supersaturated solid solution of bismuth in tin) caused by the appearance of phase stresses associated with the volume effect of phase transformation. As shown, such stresses have an inhibitory effect on the progress of decomposition.

Key words: eutectic alloy, superplasticity, plastic deformation, natural ageing, phase composition, dynamic Young's modulus, internal stresses.

Виконано дослідження змін динамічного модуля Юнґа типового модельного надпластичного стопу Bi–43 ваг.% Sn в умовах пластичної деформації, якій піддають полікристалічні матеріяли для створення структурно-фазового стану, здатного до прояву ефекту надпластичности. Також вивчено зміни модуля Юнґа в процесі тривалої витримки за кімнатної температури та нормального атмосферного тиску, у результаті якої феноменологічні показники надпластичної течії дослідженого стопу помітно понижуються, але прояв ефекту надпластичности спостерігається. Проведено акустичні міряння з використанням методи подвійного складеного п'єзоелектричного вібратора. Виявлено зростання динамічного модуля Юнґа в результаті стиснення на ≅ 70% на гідравлічному пресі та в процесі старіння як у литих, так і у стиснених зразках. Одержані експериментальні дані проаналізовано з урахуванням раніше одержаних даних про зміни фазового складу стопу в умовах експерименту. Результати аналізи свідчать, що збільшення модуля Юнґа в результаті стиснення зумовлено появою внутрішніх напружень у матеріялі. Зростання модуля Юнґа під час старіння пов'язане, перш за все, з переходом стопу з вихідного метастабільного до рівноважного за кімнатної температури фазового стану. На кінетичних залежностях модуля пружности і у литих, і у стиснених зразків спостерігається гальмування його змін на етапі старіння, коли фазова рівновага у стопі ще не встановилася. Це пояснюється зміною кінетики розпаду α(Sn)-фази (пересиченого твердого розчину Бісмуту в цині), зумовленою появою фазових напружень, пов'язаних з об'ємним ефектом фазового перетворення. Показано, що такі напруження чинять гальмівний вплив на перебіг розпаду.

Ключові слова: евтектичний стоп, надпластичність, пластична деформація, природне старіння, фазовий склад, динамічний модуль Юнґа, внутрішні напруження.

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1. INTRODUCTION

Superplasticity is a universal state of metallic materials that occurs after the creation of an ultrafine-grained microstructure in them and subsequent deformation under certain temperature and speed conditions. To obtain a small grain size, preliminary plastic deformation is widely used [1–5]. Manifestation of the effect of superplasticity of eutectic alloys is also associated with their fineness, which occurs under the conditions of crystallization and as a result of preliminary plastic deformation.

The Bi–43 wt.% Sn eutectic alloy studied in this work is one of the typical superplastic (SP) model alloys, on which the authors first began a systematic study of changes in elastic and inelastic properties caused by previous plastic deformation, long-term natural ageing and in SP flow conditions [6, 7]. Interest in the study of these issues is largely determined by the lack of understanding of the role played by the disequilibrium of the phase state of alloys in the manifestation of the superplasticity effect. It is natural to expect, considering the well-known fact that superplasticity is observed only in rapidly hardened samples.

The experiments in [6, 7] were carried out using the method of acoustic spectroscopy, which is characterized by high sensitivity to changes in the structure and phase state of the material. This makes it possible to obtain new experimental data, which are important for further deeper understanding of the physical nature of the superplasticity effect.

Previously, during the study of the Sn–38% Pb eutectic alloy, the authors found that as a result of preliminary plastic deformation of cast samples by compression on a hydraulic press, the Young's modulus of the alloy increases by 70–75%. An increase in the Young's modulus is also observed during the ageing process of the cast samples. When studying compressed samples at the initial stage of ageing, a slight increase in Young's modulus was recorded. Then its value decreases, passes through a minimum and increases again during a rather long ageing time [6]. The established patterns of changes in the elastic properties of the Sn–38% Pb alloy were explained taking into account the data obtained by the authors on the disequilibrium of the phase state formed under the conditions of crystallization and its changes due to compression and long exposure at ambient conditions. Since these studies, as already mentioned above, were performed for the first time, there is a need for their further development, in particular, in the study of alloys of other systems which was the goal of this work. Ultimately, such studies contribute to the identification and establishment of the essence of phase changes that occur in multicomponent materials under conditions of superplasticity, and, therefore, to the successful resolution of the question of the role of phase transformation in the manifestation of this effect. This issue, which is important for further deeper understanding of the nature of the structural–phase state and mass transfer mechanisms under conditions of superplasticity, remains the subject of discussions for quite a long time.

2. EXPERIMENTAL DETAILS

The studied Bi–43 wt.% Sn alloy was produced in laboratory conditions by fusion of pure components followed by casting on a massive copper substrate. In separate experiments, it was established that the process of crystallization of the samples was carried out under conditions of supercooling which took place at ≅ 17°C. The resulting cast ingots were deformed by compression on a hydraulic press by ≅ 70%.

The experiments were carried out according to the methodology previously developed by the authors [7]. Young's modulus was determined based on the results of acoustic measurements using the two-component composite piezoelectric vibrator technique [8]. The measurements are done at room temperature at the frequencies of longitudinal oscillations ≅ 102 kHz and ≅ 113 kHz in the amplitude-independent region at the amplitude of ultrasonic deformation $\varepsilon_0 \sim 10^{-7}$.

The relative measurement error of Young's dynamic modulus did not exceed $\sim 1\cdot10^{-4}$, while the total absolute error was no greater than ≅ 0.5% measured quantity. The absolute error of determining the density of the samples did not exceed ≅ 2 kg/m^3.

3. RESULTS AND DISCUSSION

First of all, we note that in order to obtain as accurate data as possible, the temperature dependence of the dynamic Young's modulus of the Bi–43 wt.% Sn alloy was studied in the range of 60–302 K, with the help of which the influence of temperature on the change of the Young's modulus was taken into account. This dependence for the case of an increase in the temperature of the sample during the measurement process is shown in Fig. 1.

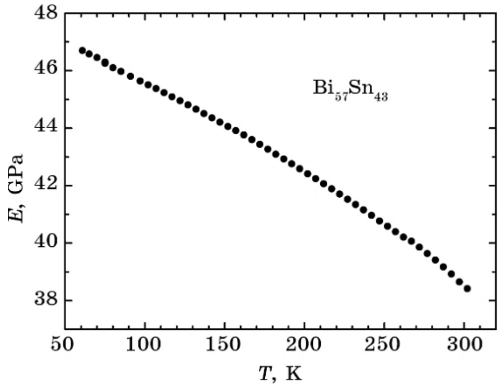

Fig. 1. Temperature dependence of the dynamic Young's modulus E of the Bi–43 wt.% Sn alloy. Freshly compressed sample.

In the course of the experiments, it was also established that there were no significant changes in the density ρ of the alloy in the cast and compressed states during ageing. The density was determined using the absolute method of hydrostatic weighing [9].

Figure 2 shows the dependence of the value ρ of the deformed samples on the ageing time *t*.

Figure 3 shows the time dependences of the dynamic Young's modulus *E* of the Bi–43 wt.% Sn alloy both in cast and compressed samples.

As can be seen from this figure, in the process of ageing of the cast alloy for ≅ 1 month, a noticeable increase in the Young's modulus *E* is observed, and then its value practically does not change in the investigated time interval (Fig. 3, curve *1*). Because of plastic deformation by

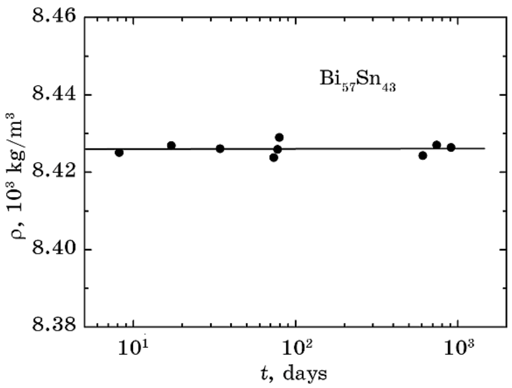

Fig. 2. Dependence of the density ρ of plastically deformed samples of the Bi–43 wt.% Sn alloy on the time of natural ageing *t*.

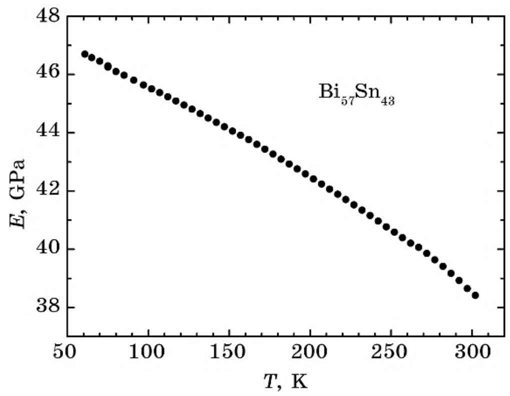

Fig. 3. Dependences of the dynamic Young's modulus *E* of the Bi–43 wt.% Sn alloy on the ageing time *t*. *1*—in the cast state; *2*—after plastic deformation by compression by ≅ 70%.

compression, the Young's modulus of the alloy also increases. During further ageing of the deformed samples in ambient conditions during the above-mentioned period, the value of E continues to increase, and then, remains practically constant too (Fig. 3, curve *2*).

The detected changes in the elastic properties of the studied alloy Bi–43 wt.% Sn were associated, first of all, with the peculiarities of the phase state in which the alloy is immediately after casting, and transformations of its phase composition during subsequent mechanical processing and long-term natural ageing.

Earlier in Ref. [10], it was established that under the chosen conditions of crystallization of the Bi–43 wt.% Sn alloy a significant excess of the relative amount of the α(Sn)-phase (a solid solution of bismuth in tin) is recorded in the initial ingots compared to its amount corresponding to the phase equilibrium both at room and eutectic temperatures. During exposure at ambient conditions, the α(Sn)-phase supersaturated with bismuth disintegrates. However, the disintegration process is very slow. The experimental data obtained in Ref. [10] indicate the stimulating effect of plastic deformation on the disintegration of the α(Sn)-phase. However, even after ageing for more than one year, the equilibrium phase state of the alloy at room temperature is not reached. It was also established that in the compressed state immediately after casting, the alloy exhibits quite bright SP properties. Elongation to failure ε reaches almost 800%. During ageing, the value of ε and the rate of SP deformation decrease but the alloy remains superplastic [11].

As it turned out, the obtained kinetic curves in the time interval investigated (see Fig. 3) are quite well approximated by the well-known Avrami relation, which is used to describe the kinetics of isothermal transformations controlled by the processes of nucleation and growth of particles of a new phase (see, for example, [12]):

$$E_{\mathrm{i}}(t) = E_{\mathrm{i}}(0) + \Delta E_{\mathrm{imax}} \left\{ 1 - \exp\left[-\left(\frac{t}{\tau_{\mathrm{i}}} \right)^{n} \right] \right\}, \quad (1)$$

where ΔE_{imax} is the maximum change of the measured value, τ_{i} is the relaxation time. Information about the numerical values of the parameters in this equation is given in Table 1.

Experimental data presented in Fig. 3, were analysed taking into account the information obtained in [10] about the volumetric ratio of α(Sn)- and β(Bi)-phases (the second phase is a solid solution of tin in bismuth) $V_{\mathrm{Sn}}/V_{\mathrm{Bi}}$ and the results of a theoretical assessment of the change in Young's modulus during its transition from the initial metastable to the equilibrium state at room temperature. Such a numerical estimate was made under the assumption that in the process of ageing, the volume ratio of the phases changes from the value corresponding to their ratio, which is in equilibrium at the eutectic temperature, to the value in equilibrium conditions at room temperature. Data on the modulus of elasticity of tin-based and bismuth-based phases, which are structural components of the alloy, and on the volume ratio of these phases were used. Numerical estimates of the volume ratio of the V_{Sn}/V_{Bi} phases in the alloy for these states were previously performed in [10]. Data on Young's moduli of phases were taken from [13]. Due to the negligibly small solubility of tin in bismuth, the Young's modulus value of the β(Bi)-phase was considered unchanged and equal to the Young's modulus of pure bismuth. Young's modulus of the alloy was determined according to the mixture rule using the formula [14]:

$$\overline{E} = \sum_{j} c_j^{vol} E_j , \quad (2)$$

where c_j^{vol} and E_j are, respectively, the relative volume and the Young's modulus of each phase. The calculations were carried out in the Voigt (E_V [15]) and Reuss (E_R [16]) approximations. The results of the calculations are given in Table 2.

The data presented in the table indicate that the transition of the studied alloy Bi–43 wt.% Sn to the equilibrium state should be accompanied by an increase in the Young's modulus. At the same time, the

TABLE 1. Numerical values of the parameters of the Avrami equation for the investigated states of the alloy Bi–43 wt.% Sn.

Parameter	Cast state (i =1)	After plastic deformation (i = 2)
E_i (0), GPa	38.56 ± 0.045	39.16 ± 0.047
ΔE_i, GPa	0.968 ± 0.043	0.634 ± 0.046
τ_i, days	14.26 ± 1.62	8.737 ± 1.37
n	1	1

TABLE 2. Theoretical estimation of the value of the Young's modulus E of the Bi–43 wt.% Sn alloy for different volume ratios of phases.

Alloy Bi–43 wt.% Sn	V_{Sn}/V_{Bi}	E_V, GPa	E_R, GPa	E_H, GPa
The equilibrium phase ratio corresponding to room temperature	1	43.5	41.4	42.5
The equilibrium phase ratio corresponding to the eutectic temperature	1.5	41.5	40.5	41.0

change in its average arithmetic value E_H (Hill's approximation), which, according to Ref. [17], corresponds well with the effective values of Young's modulus of isotropic composite materials, should be at least 3.7%. This gives grounds for asserting that the observed increase in Young's modulus is primarily related to the disequilibrium of the initial phase state of the alloy and its transition to an equilibrium state at room temperature.

In Ref. [10], it was found that after compression, the relative number of phases in the surface layers of the samples remains unchanged. This indicates that under conditions of actual plastic deformation, the supersaturated α(Sn)-phase does not disintegrate. In this regard, the increase in E as a result of compression should be associated with the appearance of internal stresses in the material. As it was shown in [18] on the basis of the analysis of the changes in the crystal lattice parameters of the phases, such stresses occur in the Sn–38 wt.% Pb alloy after deformation under the same conditions as for the Bi–43 wt.% Sn alloy.

The appearance of significant internal stresses in the Bi–43 wt.% Sn alloy as a result of deformation is evidenced by the macroscopic cracking of the ingots, which occurs in this case. Figure 4 shows an image of the cast ingot surface after its compression on a hydraulic press.

Internal stresses under conditions of compression deformation thus reach the limit of the strength of the material. In undamaged parts of the ingot, which, as already mentioned, show rather high SP properties, elastic stresses certainly decrease after cracking and relaxation, but there is no reason to believe that they disappear completely.

The presence of residual internal stresses in the Bi–43 wt.% Sn alloy

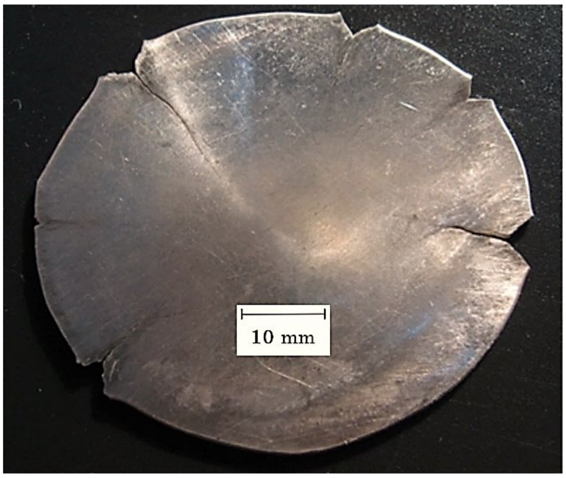

Fig. 4. Macroscopic cracking of the Bi–43 wt.% Sn alloy ingot after plastic deformation by compression by ≅ 70%.

is also evidenced by the joint analysis of the above data on the change in Young's modulus during the ageing process and the data on the change in the relative phase ratio V_{Sn}/V_{Bi} that accompanies this process. As established in Ref. [10], the values of V_{Sn}/V_{Bi} in cast samples are 3.8 after ageing for 8 days and 3.1 after ageing for 23 months. At the same time, in plastically deformed samples, the V_{Sn}/V_{Bi} ratio is equal to 2.7 and 1.9 at the indicated sample holding times, respectively. Taking this one into account, one should expect a more significant change in the Young's modulus in deformed samples compared to cast ones during the ageing process. However, as evidenced by the data of Fig. 3 and Table 1, in the cast alloy, the maximum change in the elastic modulus during the ageing process, at its investigated stage, is greater than in the plastically deformed samples. This allows us to conclude that in the deformed Bi–43 wt.% Sn alloy, the change in Young's modulus during ageing is due, on the one hand, to its increase due to the change in the phase composition of the alloy, and to its decrease due to the relaxation of internal elastic stresses, on the other hand. At the same time, the process of relaxation to the thermal equilibrium state in the cast samples is slower compared to the compressed samples, which reflects the stimulating effect of plastic deformation on the decomposition of the supersaturated α(Sn)-phase.

As well known, plastic deformation leads to an increase in the density of dislocations and the concentration of point defects, which arise from their non-conservative movement in crystals. As a result, the looseness of the crystal lattice increases, places with weakened interatomic interaction appear, which leads to a decrease in the modulus of elasticity [19]. The action of this factor, thus, can level out the increase in the value of E of the studied alloy, which is associated with the appearance of internal elastic stresses after compression of the samples. Relaxation processes in the dislocation subsystem during sample exposure in an unloaded state may be accompanied by a decrease in the density of dislocations and, accordingly, contribute to an increase in the elastic modulus. Then, this will indicate a more significant contribution of the processes that ensure the relaxation of internal elastic stresses to the resulting change in Young's modulus in deformed samples under ageing conditions, which is observed in the experiment.

The influence of another factor caused by the dislocation nature of plastic deformation is associated with an increase in the contribution of dislocation inelasticity to the effective values of elastic moduli [19]. As evidenced by the analysis of literature data, a decrease in the modulus of elasticity caused by dislocation inelasticity is observed in the early stages of plastic deformation. At this stage, the density of fresh dislocations, which are not surrounded by the formed Cottrell atmospheres, increases in the material. The inelasticity associated with such dislocations leads to the appearance of a modulus defect. In steel, for example, such effects are manifested at deformations that are only 2.5–3% [19]. With further deformation, the modulus defect usually reaches saturation, and then its decrease is observed. In addition, taking into account the large value of the compression deformation of the samples (≅ 70%), as well as the small value of the amplitude of the ultrasonic deformation (≅ 10^{-7}) during the measurements, the effect of dislocation inelasticity on the dynamic modulus of the studied alloy can be neglected.

A joint analysis of the above and previously obtained experimental results allows us to come to a conclusion about the importance of internal stresses for the emergence of the SP state in the studied material. As shown in Ref. [18] on the example of the Sn–38% Pb alloy, the level of internal stresses arising as a result of compression is sufficient to activate the additional Frank–Read dislocation sources and ensure a noticeable increase in the dislocation density of under the action of external mechanical stress. As indicated in Ref. [20], the presence of significant internal stresses can be the reason for the manifestation of the hydrodynamic mode of deformation in conditions of superplasticity, which was first discovered by the authors.

One of the important reasons for the change in the elastic characteristics of polycrystals is the appearance or changes in the texture of the material. The analysis of the results of the x-ray diffractometric studies published in Ref. [21] shows that there are no significant changes in the texture of the investigated Bi–43 wt.% Sn alloy due to compression.

Finally, let us turn once again to the dependence of the dynamic Young's modulus of the Bi–43 wt.% Sn alloy on the duration of ageing shown in Fig. 3. As already mentioned above, significant changes in the value of E in both states of the alloy are observed only at the initial stage of ageing. At the same time, the relative increase in Young's modulus in the ageing process does not reach its value, which may be due to the decomposition of the supersaturated α(Sn)-phase. This indicates that at the stage of a significant decrease in the growth rate of E, there are reasons that slow down the disintegration of the solid solution of bismuth in tin. One such reason could be the internal stresses associated with the bulk effect of this phase transformation.

As known, the structure of alloys of the type investigated in this work is characterized by the presence of continuous skeletons of grains of both phases, and each of them reacts in one way or another to volumetric changes in the other skeleton. Let us evaluate the volumetric changes that should occur in the α(Sn)-phase as a result of the transition to an equilibrium state at room temperature. Such an estimate can be made based on the data on the x-ray density $\rho_{\text{x-ray}}$ of the two-phase mixture, which is formed as a result of decomposition, and the relative number of phases.

Calculations performed using known formulas [10] made it possible to establish the following. At the concentration of the solid solution of bismuth in tin, which corresponds to the eutectic temperature, the x-ray density of the α(Sn)-phase is of 7.7719 g/cm³. According to the state diagram of the Sn–Bi system, this solution at room temperature turns into a mixture of a solid solution of bismuth in tin with a concentration of 1.3 wt.% Bi and practically pure bismuth. The quantitative phase ratio is 80.04 to 19.96, respectively. The calculated x-ray density of such a mixture is equal to 7.7032 g/cm³. Thus, during the decomposition of the α(Sn)-phase, the specific volume of the material increases. The rigid skeleton of β(Bi)-phase grains certainly has an inhibitory effect on the kinetics of this transformation.

4. CONCLUSIONS

For the first time, a study of the effect of preliminary plastic deformation, which ensures the emergence of a structural phase state capable of manifesting the effect of superplasticity, and long-term natural ageing on the dynamic Young's modulus of the superplastic eutectic alloy Bi–43 wt.% Sn was carried out. The experiments were carried out using the acoustic method of the two-component composite piezoelectric vibrator.

It is shown that the detected changes in the elastic properties of the superplastic alloy Bi–43 wt.% Sn are primarily related to the disequilibrium of the phase state, which is formed under conditions of rapid crystallization, the effect of plastic deformation on the phase composition of the alloy, as well as the occurrence of internal stresses in as a result of plastic deformation.

The increase in the Young's modulus of the Bi–43 wt.% Sn alloy during the ageing process in both cast and compressed samples is due to its transition to an equilibrium phase state, namely, the decomposition of the α(Sn)-phase—a supersaturated solid solution of bismuth in tin. At the same time, on the kinetic dependences of the modulus of elasticity in both cases, there is an inhibition of its changes at the stage of ageing when the phase equilibrium in the alloy has not yet been established. This is explained by the difference in decomposition kinetics at the initial and later stages of ageing due to the appearance of phase stresses associated with the volume effect of phase transformation. Such stresses have an inhibitory effect on the progress of decomposition.

The increase in the Young's modulus of the alloy as a result of plastic compression deformation is mainly determined by the internal stresses, which arise.

The change in Young's modulus during the ageing process in samples previously deformed by compression is due, on the one hand, to its increase in connection with the transition of the alloy to a more equilibrium structural–phase state and to a decrease associated with the relaxation of internal stresses remaining after compression, on the other hand.

The patterns of changes in the Young's modulus of the superplastic alloy Bi–43 wt.% Sn, which are caused by previous plastic deformation and natural ageing, well reproduce the previously discovered patterns of changes in the elastic characteristics of the superplastic alloy Sn–38 wt.% Pb under similar conditions of external influence. They agree with the new data obtained by the authors earlier about the characteristic features of the phase state of superplastic eutectic alloys, which is formed under the conditions of crystallization, and its changes under the influence of plastic deformation and ageing.

The results of the conducted research are important for a further deeper understanding of the nature of the structural–phase state of multicomponent metallic materials that have the ability to manifest the effect of superplasticity, and the relationship between phase transformations and mass transfer mechanisms under conditions of superplastic flow.

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