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THE EFFECT OF NONEQUILIBRIUM STRUCTURE STATE ON THE CREEP AND SUPERPLASTIC BEHAVIOUR OF MATERIALS

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Mechanical behaviour at creep and superplasticity of large-grain and monocrystalline aluminum under torsion, large-grain molybdenum, fine-grain zinc alloy and amorphous cobalt alloy under tension are discussed from unified positions. It is shown that realization of their superplasticity requires fulfilment of structure-kinetic principle.

Introduction

At present the investigators pay still more attention to the nature of superplastic flow of metals and alloys. Usually, the ability of samples to a stable uniform plastic flow to anomalous high ductilities under a uniaxial tension with the constant strain rate is interpreted by superplasticity (SP). Here SP is characterized by a strong dependence of stress σ on strain rate $\dot{\varepsilon}$ determined by the parameter $m = \mathrm{d}\log\sigma/\mathrm{d}\log\dot{\varepsilon}$. It is related to the materials with a fine grain with the size of less than 10 μm and grain boundary sliding as the basic deformation mechanism. Often, SP with the mentioned features manifests itself on the materials with phase transformations.

However, lately there have appeared a lot of experimental data, showing that the mechanical behaviour typical of SP manifests itself on other different materials as well: large grained polycrystals, monocrystals, amorphous alloys. SP flow was observed at different kinds of deforming tension, torsion and other kinds of testing (deforming with a constant strain rate, creep). The investigation [1−10] can serve as an example.

The available experimental data make it possible to regard SP as an alternative to fracture in the course of deforming, when an optimum kinetic correspondence is reached between the processes resulting in deformation, on the one hand, and preventing from fracture, on the other hand. Under such conditions a stable uniform plastic flow appears as a result of maximum plasticity realization in each local microvolume of the sample, leading to the intensive delocalization of deformation over the whole sample[1] and, as a result, to the flow to exhausting the sample plasticity as a whole.

The more markedly are expressed the nonequilibrium of the structural or phase state and dynamic activity of structural elements, i.e. carriers of plastic deformation and/or relaxation of stresses, the more vividly the above correspondence and SP flow manifest themselves and the longer they are preserved in the course of deformation. On the whole, proceeding from the above, one can think that in order to realize SP it is necessary to fulfil a principle of structural-kinetic correspondence.

Results and discussion

SP of aluminum and its alloys at torsion[2] under a constant torque at 450°C illustrates this fact visually. A new stationary stage, i.e. stage of SP flow, which corresponded to the basic deformation of the sample and its high creep rate (Fig. 1) followed the ordinary observed three stages of creep. SP stage vanished and there came usual creep with the deviation from 450°C or decrease of stress. Four stages, adequate to those of creep, also corresponded to the stress-strain diagram at a constant strain rate. During loading and in the primary stage there was formed a structure of cells of approximately 4 μm in size with the tangle boundaries of dislocations of the density of more than 10^9 cm^{-2}. During the first steady state stage there were formed thin dense boundaries embracing dozens of cells, and their unstable boundaries were destroyed. At the third stage the process took place intensively and subgrains were formed[3]. During these three stages the grains grew

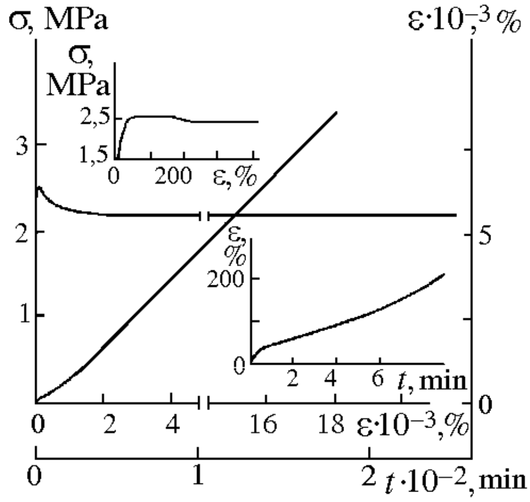

Fig. 1. The creep curve at σ = 2.5 MPa and the σ–ε-diagram at $\dot{\varepsilon} = 10^{-2}$ s^{-1} and T = 450°C. Al was annealed for 3 h at 500°C

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[1] At tension the decrease of the value m corresponds to the increase of the sample tendency to deformation localization, a decrease of a local sample cross-section with the formation of the neck and SP loss. At torsion deformation localization does not change the sample cross-section, the parameter *m* is unessential for SP.

[2] Al (99.92%) with the grains of 200 μm was mainly investigated. Deformation and stress were referred to the sample surface, recalculating torsion angle and torque in the approach to the ideal-plastic body. The equivalent results are obtained on tube and cylindric samples.

[3] In the centre of samples the structure corresponded to small deformations.

up to 600 μm.

The structure of subgrains of about 70 μm in size, misoriented up to 7° and including the structure elements inherent in a different way in the previous stages, corresponded to the SP flow. Subgrains increased with SP deformation and with the decrease of stress or creep rate. Subboundaries and boundaries of the cells were essentially nonequilibrium and dynamic. While observing them in the electron microscope they often changed, turned, migrated and destroyed. If the samples underwent SP flow, the peak of the internal friction at 450°C appeared on them. Hardening of samples under the load resulted in the formation of numerous dislocation loops. The boundaries of the grains were also nonequilibrium and active, sliding over them and their migration were observed. On the surface of samples in grains, over their boundaries and subboundaries there were small cracks, inside the samples there were small voids and cracks. The density of the metal at SP flow decreased. The length of the samples did not affect the ductility value. A stable uniform SP flow along the whole length of the samples was observed.

Temperature deviation from the optimal one and decrease of the deformation rate or stress resulted in the formation of other structures, more intensive decompaction and appearance of deformation localization in the flow process, a decrease in ductility ψ, its dependence on the length of the sample and SP vanishing (Fig. 2). Al alloying also decreased the height and width of the peak ψ. Outside the peak at decreased temperatures the structure consisted of fragments and the samples were fractured due to the nucleation and propagation of cracks. At elevated temperatures subgrains were commensurable with grains, subboundaries were low-angled, fracture occurred due to the nucleation and growth of voids on the subboundaries and grain boundaries.

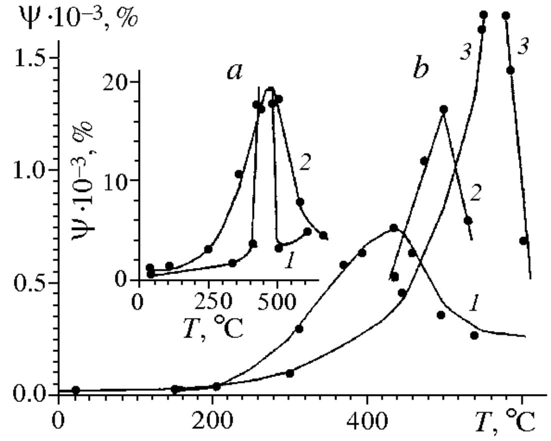

Fig. 2. The temperature dependences of Al ductility: *a* – a single crystal, $\dot{\varepsilon}$ = 0.2 s^{-1}, torsion axis ⟨100⟩ (*1*) and a polycrystal, $\dot{\varepsilon}$ = 0.2 s^{-1} (*2*); *b* – a polycrystal, tension rate was $8.3\cdot10^{-4}$ (*1*), $1.7\cdot10^{-2}$ (*2*), 1.7 mm·s^{-1} (*3*)

The analysis has shown that SP flow is due to in-situ dynamic recrystallization at the level of subgrains. It consists in cycle continuously repeated in the course of the flow, including sequentially a generation of dislocations, their interaction and formation of cells, rearrangement of dislocations in their boundaries, destruction and migration of the latter, formation of subboundaries, their reorganization, destruction and migration with the appearance of new volumes free from dislocations where the cycle is repeated, i.e. in a continuous balanced repetition of the processes, occurring at three stages up to SP flow and causing strengthening and disstrengthening of the metal. The continuity of the cycle stages, their mutual balanced imposing and simultaneous realization in the whole volume of the sample provide a dynamic equilibrium in the structure under the conditions of high non-equilibrium and mobility of all structural elements, especially, dislocation collectives. Migration of grain boundaries and sliding over them make their contribution into polycrystals.

Reaching of the anomalous ψ at SP flow is caused by the fact that under the conditions of high-mobile dynamic structure the dislocation non-diffusion fracture processes, related to cracks (pronounced at $T < 400°C$), are markedly hindered due to the realization of a great number of degrees of freedom for the relaxation of stresses. Meanwhile, diffusional processes of fracture, related to voids (pronounced at $T > 500°C$) are not provided kinetically, since diffusion rate is not enough to ensure nucleation and growth of voids on rapidly moving elements of the structure, especially on subboundaries and grain boundaries.

The peak ψ of SP flow manifested itself most vividly for the local elongation in the neck of Al samples at tension and at torsion of Al (99.999%) monocrystals, Fig. 2.

The samples having undergone SP flow and with the nonequilibrium structure, manifested a considerably larger plasticity and a later deformation localization at a room temperature than the ones annealed or deformed under the conditions not corresponding to SP (Table). The values of the local deformations ε_l and their segments in the process of deformation vary relative to the

Table

Dependence of the value of torsional strain, $T = 20°C$, corresponding to the onset of stable local deformation φ and to fracture ψ for Al (99.92%) samples preannealed for 1 h at 660°C and subjected to torsion to deformation ε at rate $\dot{\varepsilon}$ and temperature T

Preliminary deformation regime							
T,°C	350	450	600	350	450	600	600
$\dot{\varepsilon}$, s^{-1}	0.001	0.001	0.001	0.1	0.1	0.1	0
ε, %	2700	2700	2000	2000	2000	2000	0
Deformation							
φ, %	180	500	0	250	600	350	0
ψ, %	600	710	475	670	820	700	595

average value corresponding to the accumulated macroscopic deformation ε, Fig. 3, what contributes to the deformation delocalization over the length of the samples. Stable deviations ε_l of ε and hence deformation localization take place not long before the fracture.

The behaviour ε_l of other samples relative ε was different, Fig. 3. A concentration of the deformation ($\varepsilon_l > \varepsilon$) in some segments and a stable prevention of the deformation ($\varepsilon_l < \varepsilon$) in others were observed implying an early deformation localization.

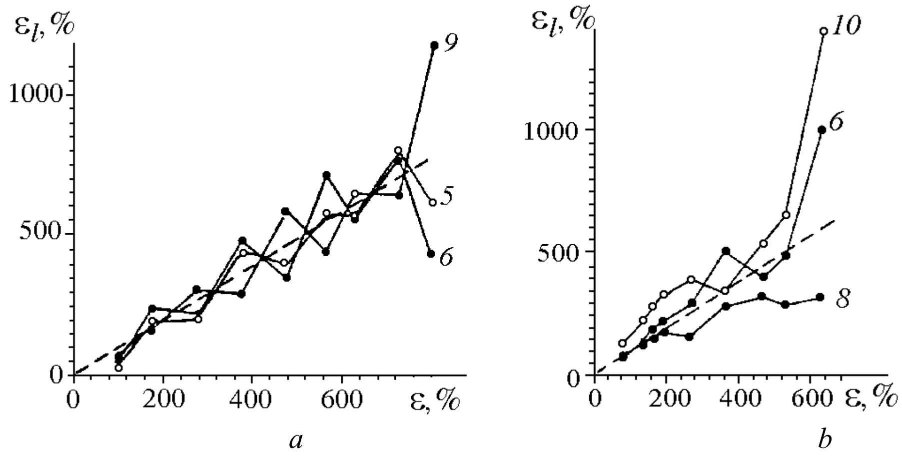

Fig. 3. Local deformations as a function of macroscopic deformation at 20°C and $\dot{\varepsilon}$ = = 0.01 s^{-1} of SP deformed (*a*) and annealed (*b*) samples. The figures stand for the number of local regions

A decisive role of the dynamic activity of the nonequilibrium grain boundaries in SP flow realization manifested itself at creep of a fine-grained alloy Zn–0.4 wt.% Al at tension at a room temperature. Its SP flow is determined by the grain boundary sliding (GBS) [11]. A nonequilibrium structure in the samples was created by rolling. By a subsequent aging at 20°C it was transformed into a more equilibrium state.

After rolling at 20 and 150°C in the samples the average size of grains was D = 0.8 and 1.2 μm, respectively, and there were spontaneously distributed small particles of the Al phase. At creep the samples showed SP, accumulating large ψ with a high rate, m up to 0.43, Fig. 4. An intensive GBS, migration of boundaries, a considerable growth of grains, absence of dislocations in grain boundaries, dislocation substructure and gliding lines in grains were typical of their creep. Dislocations moved easily in grains and were accepted by their boundaries. Grain boundary diffusion in zinc controlled SP flow. It stopped when a growth of grains stopped. However, at high stresses there were 10^9 cm^{-2} dislocations in grains. They were observed in the grain boundaries, grain growth intensity decreased.

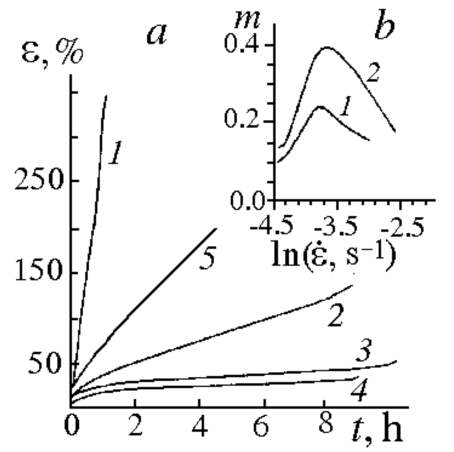

Fig. 4. *a* – the creep curves of Zn–0.4% Al sample rolled at 20°C (*1*) of those aged for 3 (*2*), 6 (*3*), 12 (*4*) months and sample rolled at 150°C (*5*); σ = 90 MPa, 17°C. *b* – changes in m with creep rate at 17 (*1*) and 28°C (*2*)

Aging within 3, 6, 12 months of the alloy rolled at 20°C resulted in the growth of grains up to D = 1.1, 1.2 and 1.4, respectively, a predominant location of particles on the grain boundaries and their junctions, a decrease of m down to 0.18, 0.12 and 0.1, respectively, a decrease of creep rate and ψ, Fig. 4, and

SP disappearance. Gliding lines and dislocation substructure in the majority of grains and high density of lattice dislocations in the grain boundaries were typical of the creep. Volume self-diffusion in zinc controlled creep rate. There was no migration of boundaries and growth of grains. GBS was expressed weakly.

As can be seen, fine grain is not enough for SP. SP is caused by nonequilibrium and dynamic activity of grain boundaries, which are higher immediately after rolling.

SP flow is determined by GBS. However, the latter is strongly hindered in aged samples. Really, if after rolling boundaries are essentially nonequilibrium, then, during their migration in the course of aging their nonequilibrium decreases markedly. However, sliding takes place over equilibrium boundaries more slowly than over nonequilibrium ones [12]. Equilibrium boundaries are bad sewers for lattice dislocations and dislocations entering them hinder GBS [13]. Moreover, migrating boundaries meet inclusions and are enriched with them. At aging there occurs Al atom segregation from solid solution on the grain boundaries. These both factors hinder GBS.

Due to the above said, in grains there occurs dislocation structure, intensive gliding of dislocations, lines and bands of gliding are formed, the number of lattice dislocations in grain boundaries grows. Deformation is connected with the dislocation motion in grains, and GBS plays the role of accommodation process. It is scarcely developed and ψ is not high.

At SP flow GBS was realized under optimum deformation rates, given by the optimum stresses, and determined high values of ψ and m. At small stresses their decrease correlates with the reduction of grain boundary role in deformation with respect to intensity increase of grain growth in the process of creep. Their decrease under high stresses can be related to the growth of dislocation density with the stress increase [11]. Then there grows the number of dislocations, approaching the boundaries. However, the boundaries are not in time to assimilate them. Lattice dislocations are being accumulated in the boundaries and GBS is hindered [13].

The importance of the dynamic activity of the nonequilibrium grain boundaries to realize SP manifested itself markedly at creep of the polycrystal molybdenum (with the grain size of 15 μm), stimulated by the excitation of grain boundaries with a diffusional flow of nickel atoms over them. The samples were subject to tension in the vacuum at 1100°C [9]. Diffusant-nickel was deposited on the sample surface electrolytically. The values of the structure parameters, creep and SP, degree of GBS and its contribution to the deformation were determined by standard methods.

Fig. 5 presents the results. It can be seen that under some conditions of steady-state creep a flow with all SP features corresponds to the samples covered with nickel in contrast to the ones without covering. Really, the sygmaidal dependence $\log\sigma - \log\dot{\varepsilon}$, $m > 0.3$, a considerable increase of ψ and degree of deformation homogeneity (ψ/δ, where δ is a narrowing in the neck) at a simultaneous decrease of deformation resistance, an essential growth of contribution GBS, h, to deformation correspond to some intervals of deformation rate. Correlation of the above parameters with the migration velocity of grain boundaries V is also seen.

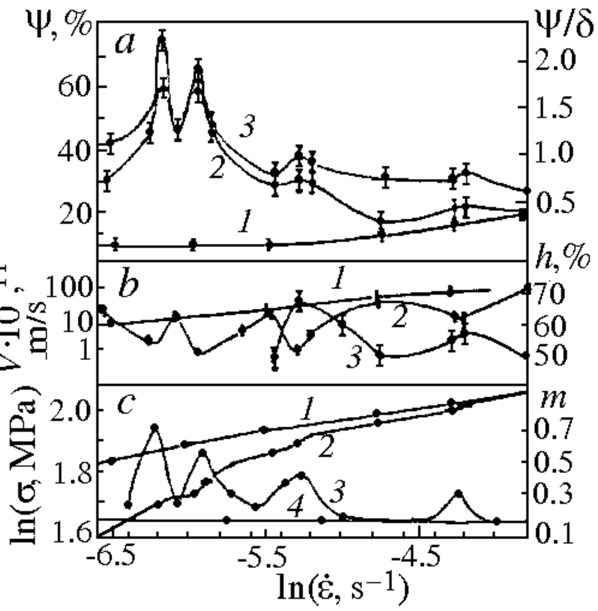

Fig. 5. Dependence on creep rate: *a* – ductility ψ for Mo (*1*), Mo–Ni (*2*) and deformation homogeneity degree ψ/δ for Mo–Ni (*3*); *b* – grain boundary migration velocity *V* for Mo (*1*), Mo–Ni (*2*) and GBS contribution *h* for Mo–Ni (*3*); *c* – flow stress for Mo (*1*), Mo–Ni (*2*) and the parameter *m* for Mo–Ni (*3*), Mo (*4*)

The experiments have also shown a correspondence of the creep activation energy of samples with covering under SP conditions to the activation energy of the molybdenum grain boundary self-diffusion. SP manifested itself only when a non-compensated diffusional flow of nickel atoms appeared on the migrating grain boundaries and the difference between concentrations of the latter on the boundaries and in the volume was maximum. That fact can be the reason of the appearance of stresses, sufficient for dislocation generation in the boundaries and, as a result, resistance decrease for GBS.

A complex character or change of SP characteristics with the deformation rate, Fig. 5, including SP disappearance, seems to be caused by the corresponding changes of grain boundary migration velocity, resulting in the change of character of nickel diffusion within boundaries. Then in order to realize SP, grain boundaries are necessary, on the one hand, and observation of a kinetic correspondence between migration processes of grain boundaries and impurity diffusion at the boundaries, on the other, which, in their turn, depend on deformation rate. Grain size does not have any effect. However, such complex behaviour $V(\varepsilon)$ needs explanation.

Structural state nonequilibrium at tensile deformation with $\dot{\varepsilon} = 1.7\cdot10^{-2}$ s^{-1} of the amorphous alloy $Co_{57}Fe_5Ni_{10}Si_{11}B_{17}$ determined its SP flow at 563°C, Fig. 6, being characterized by a uniform deformation and high ductility ($\psi \approx \approx 180\%$), low deforming stress, $m \approx 0.4$, a strong dependence of ductility yield stress on strain rate. In the experiments [10] the samples were made of ribbon with the thickness of 30–40 μm obtained with spinning method. A structural state was controlled by electron microscopy and X-raying. Heating up to test temperature was carried out with the rate 50 K·min^{-1}. Deformation was performed when the required temperature was reached. Starting crystallization temperature was 563°C.

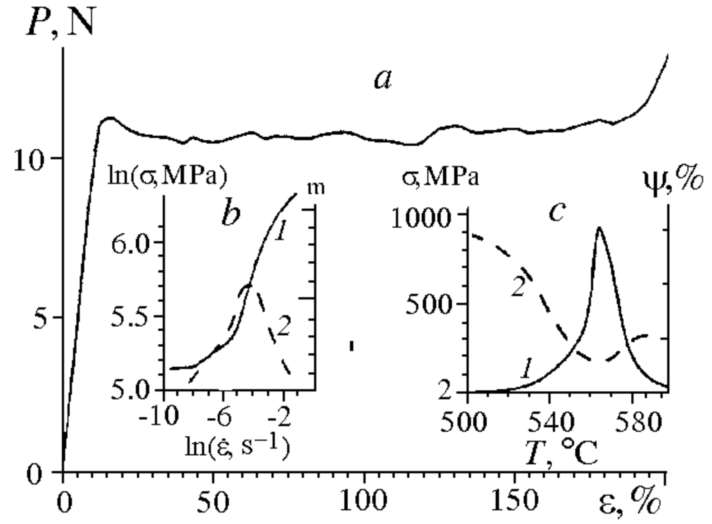

Fig. 6. Tension diagram at 563°C and $\dot{\varepsilon} = 1.7 \cdot 10^{-2}$ s^{-1} (*a*). The dependence of flow stress (*1*) and parameter *m* (*2*) on strain rate at 563°C and $\dot{\varepsilon} = 1.7 \cdot 10^{-2}$ s^{-1} (*b*). Temperature dependence of ductility (*1*) and flow stress (*2*) at $\dot{\varepsilon} = 1.7 \cdot 10^{-2}$ s^{-1} (*c*). $Co_{57}Fe_5Ni_{10}Si_{11}B_{17}$ alloy

In the process of SP flow of samples their amorphous state was preserved up to $\varepsilon \approx 60\%$. Then their crystallization started and was being developed. Before fracture the samples were practically crystalline. At $T > 630$°C crystallization intensification took place, the samples were brittly fractured ($\psi = 0$). At $T < 530$°C the samples were fractured in the amorphous state, a strong deformation localization manifested itself. In both cases SP vanished.

It follows from the above data that in order to realize SP it is necessary to observe a kinetic correspondence between the processes resulting in deformation and the ones of the structure reconstruction, contributing to relaxation of stresses, as well as a dynamic activity of structure elements responsible for crystallization in the field of mechanic stresses.

Conclusion

Finally, let us emphasize that a realization of a structural-kinetic principle is in the basis of the above considered kinds of SP, in other words, the necessity to create a sufficiently dynamic structure, on the one hand, and kinetic conditions for the maximum manifestation of its dynamics, on the other. Detailed aspects of principle might differ in different cases, the same are SP forms. A realization of the dynamic cooperative-regeneration processes of structure behaviour can ensure practically an unlimited plastic flow.

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