

Formation of micro-discontinuities during thermal processing and plastic deformation of polycrystalline materials

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Influence of initial micro-defective structure and microdefects arising during thermal and mechanical treatment on mechanical characteristics of polycrystalline aluminum samples has been studied experimentally. It is shown that heat treatment of samples with strongly distorted structure results in a decreased sample density due to generation of porous structure. Formation of pores on grain boundaries hinders recrystallization and results in appearance of microcracks near the pores at plastic straining that causes the grain-boundary failure character of fine-grained samples. It is shown that pre-straining promotes decrease in porosity during high-temperature annealing that, in turn, causes coarsening of grains.

Экспериментально исследовано влияние исходной микродефектной структуры и микродефектов, возникающих в процессе термической и механической обработки, на механические характеристики поликристаллических образцов алюминия. Показано, что термическая обработка образцов с сильно искаженной структурой приводит к уменьшению их плотности вследствие образования пористой структуры. Образование пор на границах препятствует рекристаллизации и приводит к возникновению при пластическом деформировании в области пор микротрещин, что обуславливает зерно-границный характер разрушения мелкозернистых образцов. Показано, что предварительная деформация способствует уменьшению пористости в процессе высокотемпературного отжига, что, в свою очередь, приводит к укрупнению зерен.

In [1], the development of strain-induced microdefects (pores and microscopic cracks) has been studied experimentally in single- and polycrystalline samples of various metals under straining conditions varying within a rather wide range. It has been shown that the density reduction caused by the micro-defective structure arising under plastic straining is localized in the near-surface layer. Thus, when determining the density reduction value using the integral method, the maximum sensitivity will be attained for samples with the large free surface-to-volume ratio, and in this connection, the research using the foil samples will be most effective.

As the study objects, about 150 μm thick polycrystalline aluminum samples of about $100 \times 20 \text{ mm}^2$ size have been used. The density reduction was determined in initial samples after annealing at 400°C and 600°C of various duration, coarse-crystal samples with the average grain size $\bar{d} \approx 1\text{--}15 \text{ mm}$, as well as in the latter samples strained under active tension conditions at a constant straining rate $\dot{\epsilon} = 10^{-5} \text{ s}^{-1}$. For all samples, the mechanical characteristics (ultimate stress σ_t , maximum plastic strain ϵ_{max}) were determined from the stress-strain curve and the failure character was investigated *in situ* using video filming [2]. The samples with the various average grain size

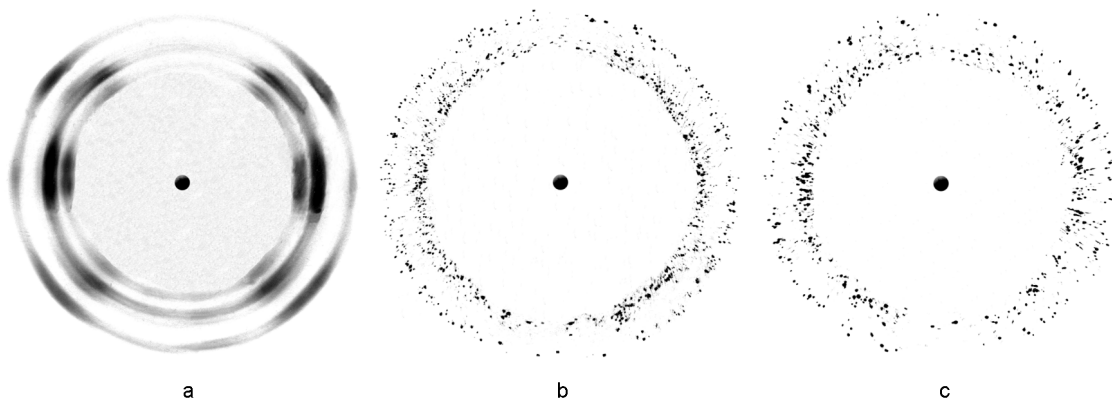


Fig. 1. X-ray diffraction patterns of polycrystalline aluminum samples: initial (a), annealed at 400°C for 2 h (b), annealed at 400°C for 2 h + 600°C for 2 h (c).

were prepared as follows. The samples cut out from a foil were strained under active tension conditions after annealing at 400°C during 2 h. The strain value was about 1 to 7 %. The samples with above-mentioned average grain size were obtained using subsequent annealing at 600°C during 2 h. To reveal the grain boundaries, the Keller etchant [3] was used. The density of samples was determined using the hydrostatic weighing. Weighing in water and in air was carried out using a VLR-20 balance with an absolute error of about $\pm 2.5 \cdot 10^{-5}$ g and relative error of about $\pm 3 \cdot 10^{-3}$ %. The absolute and relative errors of sample density determination did not exceed $\pm 3 \cdot 10^{-4}$ g/cm³ and ± 0.01 %, respectively. The main results obtained are presented in Table.

The average density value of initial samples (about 2.6925 g/cm³) is close to X-ray aluminum density (about 2.6986 g/cm³). The insignificant scatter of sample density values within limits of about 2.6920 to 2.6930 g/cm³ is due to distinctions in the sample cutting areas (at edge or near the middle part). After annealing at 400°C during 2 h, the density of various samples and its scatter decrease appreciably. The density values of such samples are between about 2.6851 and 2.6857 g/cm³. The subsequent annealing at 600°C during 2 h results in an essential density decrease. The average value of density decrease $\Delta\rho$ as compared with initial samples amounts 0.0450 g/cm³ ($\Delta\rho/\rho = 1.7$ %).

It is known that heating of strongly distorted crystal structure causes an increased concentration of nonequilibrium vacancies (due to high activity of diffusion processes) as well as the coalescence thereof resulting in pore formation, increase in the number

and sizes of pores [4], that should no doubt reduce the sample density. Fig. 1 shows the X-ray diffraction patterns for the initial sample and for samples annealed at 400°C and 600°C. The pattern for the initial sample (the lines are continuous and diffuse) evidences a high degree of the crystal lattice distortion and the average grain size that does not exceed 10^{-5} cm [5]. The patterns for aluminum samples after annealing at 400°C and 600°C are found to be of point type. Determination of the average grain size from the number of points in the Debye ring using a known technique [6, 7] shows that it amounts 0.2–0.3 mm and is practically the same for samples annealed at different temperatures. Thus, an essential density reduction of polycrystalline aluminum samples after annealing at 600°C as compared to those annealed at 400°C cannot be explained by features of recrystallization of such samples (by various length of grain boundaries in samples depending on the recrystallization temperature).

To determine the localization of the density reduction, in samples of various type (initial samples and samples annealed at 400°C and 600°C) the density was measured after the layer-by-layer thinning of samples using chemical polishing with following etchant composition: H₃PO₄ 350 ml, HNO₃ 50 ml, H₂SO₄ 100 ml, CuH₁₀O₉S 0.5 g [8]. An about 20 μm thick layer was removed. For initial samples, an insignificant density reduction (about 0.1%) was observed in the near-surface layer of about 20 μm thickness. The density practically does not vary over the thickness in samples annealed both at 400°C and 600°C. In spite of the fact that the samples were thinned using the polishing composition [8], the etching action

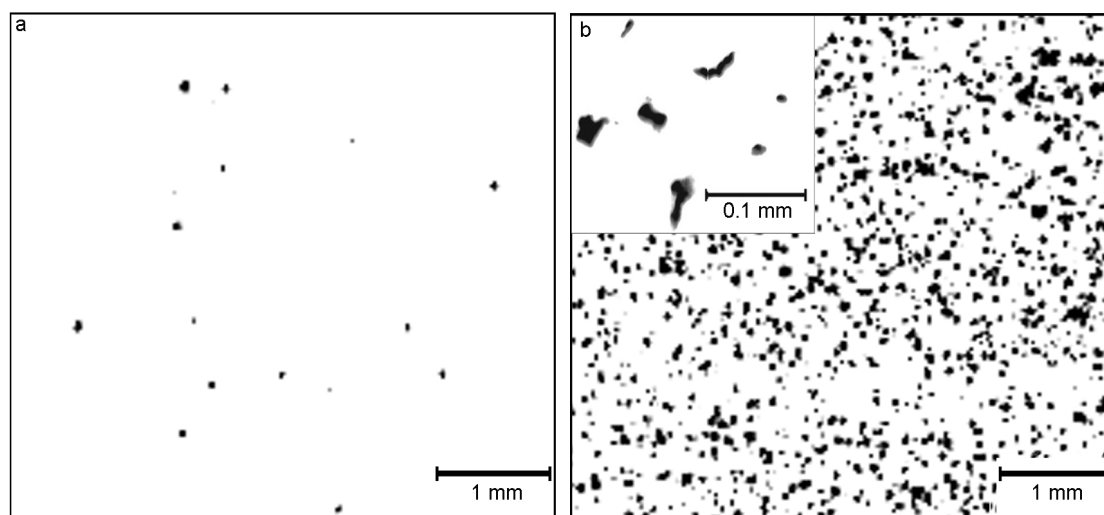


Fig. 2. Through pores in heat treated aluminum samples thinned by chemical polishing down to 100 μm thickness: annealed at 400°C for 2 h (a); annealed at 400°C for 2 h + 600°C for 2 h (b).

was appeared to be selective. In samples annealed at different temperatures and thinned down to thickness of about 100 μm , through pores were found (Fig. 2), and the pore density in samples annealed at 400°C (2 h) + 600°C (2 h) is two orders of magnitude higher than in those annealed at 400°C. In microphotos of the sample surfaces shown in Fig. 2, the pores are obviously formed by fusion of neighboring pores both at the sample surface and over its thickness.

Consideration of the enlarged ($\times 10$) microphoto fragment of a sample with through pores (Fig. 2b) makes it possible to assume, taking into account the grain size value, that the through pores correspond to the etched parts of grain boundaries. The metallographic researches of the samples porous structure confirm it (Fig. 3). In samples annealed at 400°C, pores have been observed in parts of separate grain boundaries (Fig. 3a). After annealing at 600°C, the pores appear practically in all boundaries of all grains (Fig. 3b).

Initial samples and samples after the thermal and thermal-mechanical treatment were strained under uniaxial tension conditions. The obtained mechanical characteristics for such samples and data on the failure character are presented in Table. The highest ultimate strength σ_t and the lowest strain before failure ε_{max} have been observed in initial samples. The ultimate strength σ_t amounts ≈ 600 MPa, and ε_{max} does not exceed 0.5 %. The failure character of such samples is always quasi-brittle.

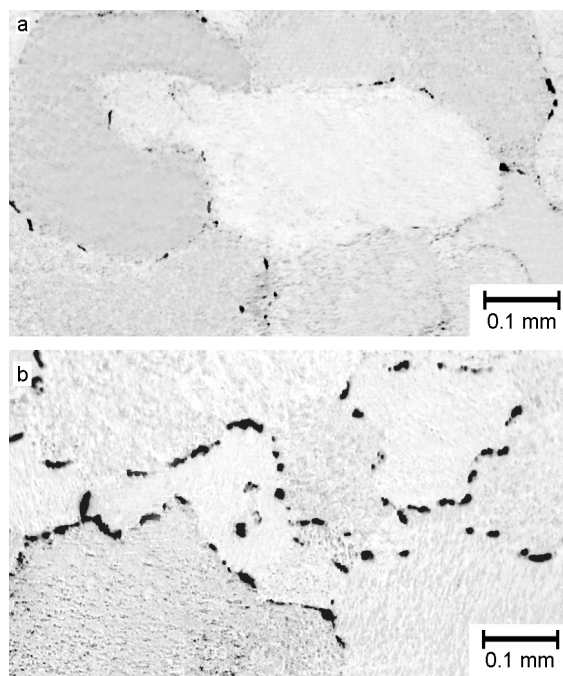


Fig. 3. Pores on grain boundaries visualized by chemical etching in aluminum samples: annealed at 400°C for 2 h (a); annealed at 400°C for 2 h + 600°C for 2 h (b).

The ultimate strength decreases and the maximal plastic strain increases at annealing. So, after annealing at 400°C and 600°C, ε_{max} amounts ≈ 5 %. The grain-boundary failure character of such samples has been observed. The microcracks are originated from pores on grain boundaries (Fig. 4).

In this work, it is shown experimentally that the probability of grain-boundary fail-

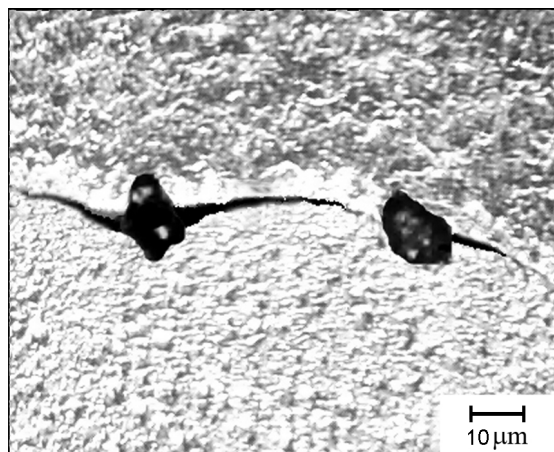


Fig. 4. Microcracks in pores on grain boundaries in polycrystalline aluminum sample ($d = 1$ mm, $\varepsilon = 10$ %).

ure increases with reduction of the average grain size (Fig. 5), that corresponds, as has been shown before [9, 10], to increasing fraction of general type grain boundaries when passing from coarse-grained samples to fine-grained ones. The source of the grain-boundary failure is not the grain boundary type, but the presence of pores at the boundaries which are formed, as a rule, on general type grain boundaries. During primary recrystallization under annealing at 400°C , in samples with strongly distorted structure and high concentration of excess vacancies, pores are formed due to coalescence of nonequilibrium vacancies on grain boundaries simultaneously with recrystallization.

Fig. 6 presents the average grain size and density of aluminum samples as functions of the annealing duration at 400°C . These results show convincingly that the recrystallization process essentially stops as the certain density of pores (the sample density) is achieved. The pore density estimated for average grain size $\bar{d} \approx 0.2$ mm (Fig. 6) shows that about 10 pores of about $5\text{--}10$ μm size are sufficient to block a grain boundary migration. Formation of defects such as pores on grain boundaries, first, hinders not only primary recrystallization but apparently collective one; second, it obstructs generation of the special type grain boundary or of a similar one; third, it results in occurrence of microcracks near the pores under plastic straining.

Thus, the results obtained, first, show once again that the presence of pores on grain boundaries causes grain-boundary failure; second, testify that pre-straining prior to high-temperature annealing initi-

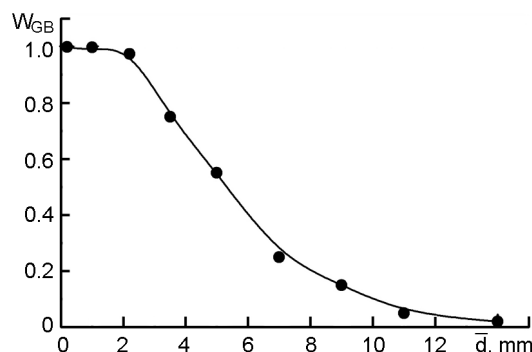


Fig. 5. The grain-boundary failure probability W_{GB} as a function of average grain size d .

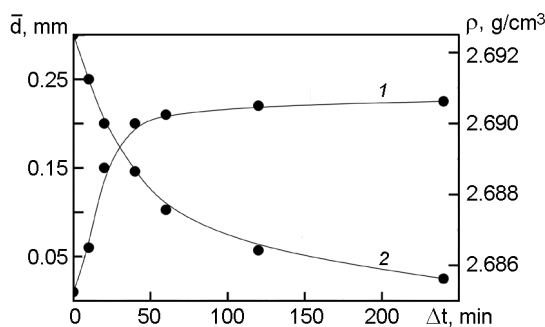


Fig. 6. Variations of the average grain size d (1) and density (2) in aluminum samples as functions of the annealing duration at 400°C .

ates diffusion processes which cause dissolution of pores and activation of recrystallization processes. Finally, Table presents the data on density reduction $\Delta\rho_\varepsilon$ (caused by formation and accumulation of micro-discontinuities during plastic deformation) in connection with the average grain size. The $\Delta\rho_\varepsilon$ was determined from the sample density values prior to straining and after failure. These results in themselves testify to a slight increase of the density reduction with diminution of the average grain size (with increasing length of grain boundaries). In [1], it has been shown that $\Delta\rho_\varepsilon$ increases linearly with the sample strain under active tension conditions. In this work, it is shown that the sample strain to the failure moment (ε_{max}) decreases by a factor exceeding 10 as of the average grain size d is reduced from 10 mm to 0.1 mm. Thus, the values $\Delta\rho_\varepsilon$ presented in Table are obtained for samples after various strain ε_{max} , and therefore, the above conclusion about slight influence of grain size on the density reduction is at least incorrect. In the next to last column of Table, presented are the density

Table. Structural state characteristics (\bar{d} , ρ — before mechanical test; $\Delta\rho_\varepsilon$, $\Delta\rho_\varepsilon/\varepsilon_{max}$ — after failure), mechanical data (σ_t , ε_{max}) and failure character of initial samples and samples after the certain thermal or thermomechanical treatment

Processing technique of sample	\bar{d} , cm	ρ , g/cm ³	σ_t , MPa	ε_{max} , %	$\Delta\rho_\varepsilon \cdot 10^3$, g/cm ³	$\Delta\rho_\varepsilon/\varepsilon_{max} \cdot 10^3$, g/cm ³	Failure character
initial	$<10^{-5}$	2.6925	620	<0.5	—	—	GB
400°C (2 h)	0.02	2.6854	250	5	4.1	82	GB
400°C (2 h) + 600°C (2 h)	0.03	2.6475	250	5	4.0	80	GB
400°C (2 h) + $\varepsilon = 1\%$ + 600°C (2 h)	0.1	2.6870	420	12	3.4	28	(GB-TC)
400°C (2 h) + $\varepsilon = 7\%$ + 600°C (2 h)	0.4	2.6895	360	20	2.6	13	(GB-TC)
400°C (2 h) + $\varepsilon = 4\%$ + 600°C (2 h)	0.7	2.6900	310	22	1.5	6.8	(GB-TC)
400°C (2 h) + $\varepsilon = 2.5\%$ + 600°C (2 h)	1.4	2.6920	230	40	1.1	2.8	TC

GB — grain-boundary failure; TC — trans-crystalline failure.

reduction values corresponding to the unit relative strain. It is obvious that the density reduction $\Delta\rho_\varepsilon/\varepsilon_{max}$ increases essentially with diminishing average grain size \bar{d} . These results show convincingly that the main part of density reduction at plastic straining is connected with grain boundaries.

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Утворення мікронесуцільностей в процесі термічної обробки та пластичної деформації полікристалічних матеріалів

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Експериментально досліджено вплив вихідної мікронефектної структури та мікронефектів, що виникають у процесі термічної та механічної обробки, на механічні характеристики полікристалічних зразків алюмінію. Показано, що термічна обробка зразків із сильно викривленою структурою приводить до зменшення їхньої густини внаслідок утворення пористої структури. Утворення пор на межах зерен перешкоджає рекристалізації та приводить до виникнення при пластичному деформуванні в області пор мікротріщин, що обумовлює зерномежевий характер руйнування дрібнозернистих зразків. Показано, що попередня деформація сприяє зменшенню пористості у ході високотемпературного відпалу, що, в свою чергу, приводить до укрупнення зерен.