

# Peculiarities of Plastic Deformation of SPD Al–Li Alloy at 0.5 K

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The mechanical properties of Al–Li solid solution were studied in tensile deformation tests at extremely low temperature of 0.5 K. The purpose of this study was to investigate the flow stress and work hardening rate as well as the development of serrated (jump-like) deformation for the polycrystalline samples with different microstructure. The samples obtained by severe plastic deformation via hydroextrusion were initially tensed up to a given intermediate deformation, then unloaded, annealed to modify their microstructure, and once again deformed to fracture. The increase of the grain size and decrease of average dislocation density due to annealing were found responsible for the work hardening rate increases and the flow stress decrease in accordance with the superposition of the Hall–Petch and Taylor contributions. As opposed to the flow stress, the high ductility of the samples remains rather insensitive to the microstructure properties, apparently due to suppressed recovery processes as well as the unstable deformation mode at extremely low temperature. The high ductility makes it possible to compare the work hardening rate and the scale of jump-like plastic deformation along the stress-strain curves for samples with different microstructures. In all cases, the average amplitude of the stress jumps was observed to increase whereas the average work hardening rate decreases with deformation. The observed correlation indicates that the nature of both phenomena follows from the dislocation density evolution processes.

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## 1. Introduction

According to models [1–3], the flow stress,  $\sigma$ , work hardening coefficient,  $\theta$ , and ductility of deformed fcc metals are determined by the evolution of the dislocation substructure, in particular, by the accumulation and annihilation rates of dislocations. The grain size,  $d$ , and dislocation density,  $\rho$ , as well as the deformation temperature,  $T$ , are the main factors influencing the dislocation evolution along the stress–strain curve of a polycrystalline sample.

Preliminary severe plastic deformation (SPD) is an effective method of modifying the microstructure of samples. Usually the SPD processing leads to average grain size decrease, the dislocation substructure rearranges and the average dislocation density increases. As a result of the SPD, the strength of a polycrystalline sample increases and the ductility decreases as compared to the annealed state. The effect of the SPD on microstructure and the dislocation mechanisms of plastic deformation has been the object of many investigations in the last years.

The ratio “strength/ductility” for different microstructure states depends on the test temperature. As temperature decreases, the flow stress increases due to the thermally activated interaction of moving dislocations with

point obstacles. At the same time, the work hardening rate of fcc materials usually increases due to a suppressed dynamic recovery process, therefore, the tensile ductility also increases.

At very low temperatures, the so-called jump-like (or serrated) plastic deformation mode is observed in many crystalline materials. One of the common peculiarities found was the increase of the scale (e.g. the stress jumps amplitude) of this mode with temperature decrease. The experimental data and the nature of this phenomenon were discussed in [4, 5]. Two main hypotheses based on a so-called “local heating” and “dislocation avalanches motion” were proposed to explain some of the experimental results obtained for single- and coarse grained crystals.

In this work, the effect of annealing on the plastic deformation of SPD processed Al–Li is studied in tensile tests at extremely low temperature. The main target is to investigate the effects of microstructure properties on the average work hardening rate and the average stress jumps amplitude of jump-like deformation. These parameters of the Al–Li alloy may be evaluated with more accuracy only at extremely low temperature due to the elevated ductility.

## 2. Experimental

Cylindrical rods of Al–3.8 at.% Li solid solution were homogenized and SPD processed by a combination of the direct and equal-channel angular hydroextrusion at room temperature (for details see [6]). Plane samples

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for tensile tests with a gauge of  $10 \times 3 \times 0.9 \text{ mm}^3$  were prepared from the rods by spark cutting and stamping. Eight of the SPD-samples were deformed in two steps at a constant rate of  $10^{-4} \text{ s}^{-1}$  using the deformation machine equipped with liquid He-3 cryostat and absorption pump. The designs of the device for deformation of materials at a record low temperature down to 0.44 K and the details of the experimental procedure were described in [7]. In the first step, each of the samples was deformed at 0.5 K to a given strain in the range of  $\varepsilon \approx 0.1\text{--}0.2$  and then unloaded. Two of the samples with the closest values of the yield stress and similar tension diagrams were selected. To modify the microstructure, sample I was annealed at 623 K for 120 min and sample II at 373 K for 20 min. In the second step the annealed samples I and II were deformed again at 0.5 K to failure. The mean parameters of stress–strain curves and the scale of the jump-like deformation observed before and after annealing were evaluated. The mean grain size and mean dislocation density changes due to SPD, annealing and the low temperature plastic deformation were controlled by standard light microscopy and using X-ray diffraction methods.

### 3. Results and discussion

The light microphotographs of Al–Li samples after SPD and annealing at two different temperatures are presented in Fig. 1. Due to processing by the combined direct and angular hydroextrusion, the mean grain size,  $d$ , decreased down to  $1\text{--}3 \mu\text{m}$  (Fig. 1a). The annealing at 373 K (sample II) leads to the slow increase of the grain size up to  $d \approx 4\text{--}6 \mu\text{m}$  (Fig. 1b), while the annealing at 623 K (sample I) leads to the increase of the grain size up to  $d \approx 30\text{--}40 \mu\text{m}$  (Fig. 1c).

The corresponding data of the mean dislocation density,  $\rho$ , for SPD processed (A), annealed (C) and tension tested (B and D) samples I and II calculated from the X-ray data for the coherent scattering domain size,  $L$ , and the microstrain,  $\langle \varepsilon^2 \rangle^{1/2}$ , are presented in the Table. As follows from calculus, the annealing at 623 K leads to a sharp decrease of the dislocation density,  $\rho$  (IC) as compared with the sample annealed at 373 K (IIC).

The  $\sigma\text{--}\varepsilon$  curves for samples I (a) and II (b) deformed in two steps at 0.5 K are presented in Fig. 2. Segments AB correspond to the SPD processed and CD to the annealed microstructure. Values of the flow stresses,  $\sigma_{\text{exp}}$ , corresponding to the points A, B, C and D of the curves I and II are listed in the Table.

In a rough approximation, the flow stress may be described assuming a simple superposition

$$\sigma_{\text{th}}(d, \rho) = \sigma_0 + \sigma_{\text{HP}}(d) + \sigma_{\text{T}}(\rho),$$

where  $\sigma_0$  is the friction stress,  $\sigma_{\text{HP}} = Kd^{-1/2}$  is the Hall–Petch stress including factor  $K$  [8, 9],  $\sigma_{\text{T}} = \alpha\mu bM\rho^{1/2}$  is the Taylor stress [1],  $\alpha$  — dislocation interaction factor,  $\mu$  — shear modulus,  $b$  — magnitude of the Burgers vector,  $M$  is the Taylor factor. The theoretical stresses,  $\sigma_{\text{th}}$ , in the Table are calculated for  $\sigma_0 = 30 \text{ MPa}$  [10];  $K = 0.16 \text{ MPa m}^{1/2}$  estimated for 4.2 K in [11];  $\alpha = 0.6$ ;

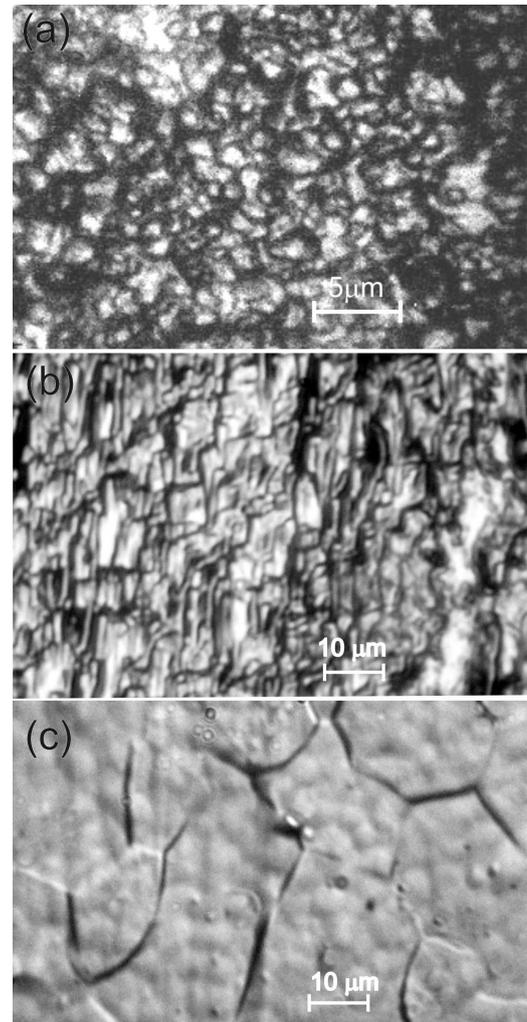


Fig. 1. Light micrographs of Al–Li samples after SPD (a) and annealing at 327 K (b) and 623 K (c).

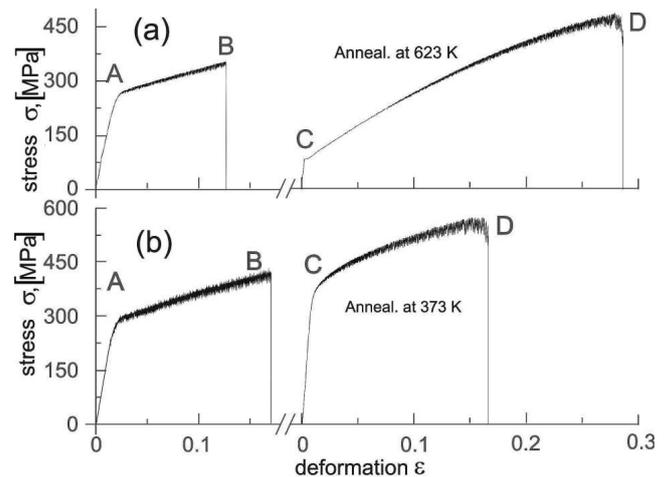


Fig. 2. The stress–strain curves  $\sigma\text{--}\varepsilon$  at 0.52 K obtained for samples I and II of Al–Li before (segments AB) and after (segments CD) annealing at (a) 623 K and (b) 373 K.

$\mu = 32$  GPa taken at 0 K from [12],  $b = 0.286$  nm and  $M = 3.05$ . The values of  $\sigma_{th}$  and  $\sigma_{exp}$  are in good agreement in the points A, B and C of the curves for both samples. This indicates that the annealing influences the flow stress level but it does not affect the hardening mechanism based on the predominant role of the grain boundary and dislocation interactions. In other words, the grain size and dislocation density range in our case (see the Table) seems to be insufficient to modify the hardening mechanism of the Al–Li alloy at low

temperature. The deviations of  $\sigma_{th}$  and  $\sigma_{exp}$  at high strains (points D) may be attributed to some accommodation of dislocations not described by the simple stress superposition at high dislocation density. Another possible reason is an error in the dislocation density evaluation following from the X-ray data obtained at room temperature. No controlled relaxation of the low temperature dislocation substructure may be responsible for a considerable error at high strains.

TABLE  
Microstructure and mechanical parameters of Al–Li.

Sample in points (see Fig. 2)	I A	II A	I B	II B	I C	II C	I D	II D
$d$ [ $\mu\text{m}$ ]	1–3		–	–	30–40	4–6	–	–
$L$ [nm]	$110 \pm 10$		$60 \pm 10$	$50 \pm 10$	$170 \pm 20$	$80 \pm 10$	$60 \pm 10$	$50 \pm 10$
$(\varepsilon^2)^{1/2}$ [ $10^{-4}$ ]	$2.0 \pm 0.4$		$4.0 \pm 1.0$	$6.0 \pm 1.0$	$< 0.1$	$4.5 \pm 1.0$	$8.0 \pm 1.0$	$8.5 \pm 1.0$
$\rho$ [ $10^{14} \text{ m}^{-2}$ ]	$0.4 \pm 0.2$		$1.5 \pm 0.2$	$2.7 \pm 0.4$	$< 0.01$	$1.3 \pm 0.2$	$3.0 \pm 0.4$	$3.8 \pm 0.4$
$\varepsilon$	0.02		0.12	0.17	0.02		0.28	0.17
$\sigma_{exp}$ [MPa]	$242 \pm 5$   $251 \pm 5$		$341 \pm 5$	$388 \pm 5$	$69 \pm 5$   $329 \pm 5$	$468 \pm 5$	$579 \pm 5$	
$\sigma_{th}$ [MPa]	$248 \pm 30$		$347 \pm 30$	$417 \pm 30$	$67 \pm 10$   $290 \pm 30$	$348 \pm 30$	$430 \pm 30$	

The data of dislocation densities evaluated at the points A, B (see the Table), and the positive slopes of the A–B segments of the stress–strain curves in Fig. 2 testify the high capacity of SPD samples to the work hardening corresponding to the dislocation accumulation during plastic deformation at low temperature. The accumulation capacity indicates that at 0.5 K, the dislocation substructure of the sample previously processed by SPD at room temperature becomes unsaturated. As a result, the ductility of the SPD sample tensed at low temperature increases as compared with elevated temperatures.

Annealing at 623 or 373 K leads to the work hardening rate increase (segments C–D in Fig. 2) more pronounced for sample I treated at high temperature. In terms of the work hardening model [3] and assuming no changes of the average grain size with the strain at extremely low temperature, the observed increase of the work hardening coefficient,  $\theta$ , after annealing may be attributed to the decrease of the dislocation density and corresponding increase of the dislocation accumulation rate. Data of the sharp increase of the average dislocation density due to the low temperature tensile deformation are listed in the Table (see points C and D).

The pronounced jump-like deformation mode in the form of successive stress jumps (serrations) in the  $\sigma$ – $\varepsilon$  curves is observed for samples I and II in Fig. 2. Due to the high ductility at 0.5 K, more than a hundred stress jumps may be registered in the  $\sigma$ – $\varepsilon$  curves for SPD (AB) and annealed (CD) samples. As a result, the average amplitude of the stress jumps,  $\Delta\sigma$ , at a given stress,  $\sigma$ , be-

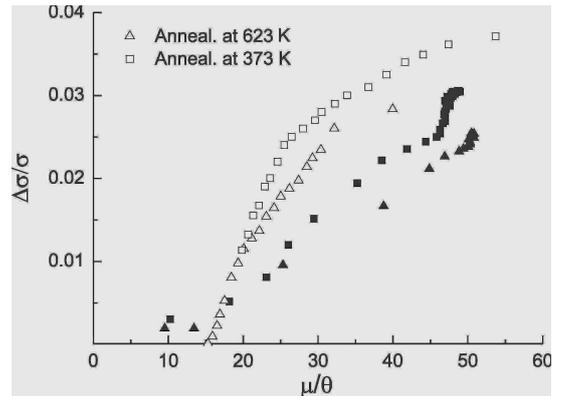


Fig. 3. Normalized stress jump amplitude,  $\Delta\sigma/\sigma$ , vs. inverse work hardening coefficient,  $\mu/\theta$ , of Al–Li samples I and II. Data are calculated at the same points of  $\sigma$ – $\varepsilon$  curves. Triangles — after SPD (dark) and annealing at 623 K (clear); squares — after SPD (dark) and annealing at 373 K (clear).

comes the representative scale characteristic of the jump-like mode. As follows from Fig. 2, the average stress jumps amplitude,  $\Delta\sigma$ , for samples I and II increases with strain but decreases after annealing. These variations of the jump-like deformation scale correlate with the behaviors of the work hardening due to the strain and annealing. To illustrate this correlation, the plot of the normalized stress jumps amplitude,  $\Delta\sigma/\sigma$ , vs. the inverse work hardening coefficient,  $\mu/\theta$ , is presented in Fig. 3.

The data of  $\Delta\sigma/\sigma$  and  $\mu/\theta$  for samples I and II are plotted at the same flow stress. In all cases, the amplitude,  $\Delta\sigma/\sigma$ , increases with strain whereas the coefficient  $\theta$  decreases. The annealing, on the contrary, leads to a sharp decrease of  $\Delta\sigma/\sigma$ , while the value of  $\theta$  increases.

It was mentioned that the phenomenon of jump-like (serrated) plastic deformation is typical for metals and alloys deformed with a constant strain rate at low temperatures. Generally, this mode of plastic deformation was observed below 30 K and attributed to a set of dislocation avalanches and thermal processes or combinations thereof leading to a local shear of the lattice occurring in a short time (for details see Refs. [4, 5]). As a result, a sharp decrease of the deformation stress (single jump) may be registered in macroscopic tests at a constant strain rate. On the other hand, the dislocation-dynamic nature of plastic deformation assumes that the grain size and dislocation density are the key parameters limiting the mean free path of dislocations and therefore determining the mean work hardening rate of the sample [3]. The observed behaviors of the scale characteristic,  $\Delta\sigma/\sigma$ , confirm that the low-temperature jump-like deformation, to a great extent, is determined by the same microstructure parameters, in particular, by the current dislocation density corresponding to a given strain of the SPD or the annealed sample, respectively. A critical value of dislocation density reached during the low temperature strain may be assumed responsible for the macroscopic stress jumps registered in our tests. The critical strain necessary to accumulate this critical density of dislocations is determined by the initial microstructure; therefore, the strain is different for the SPD and the annealed samples of the Al–Li alloy. The amplitude of the stress jump as a random event may be related in this case to the scale of the random dislocation avalanche (or avalanches) leading to the local shear of the lattice.

## References

- [1] G.I. Taylor, *Proc. R. Soc. Lond. Ser. A* **145**, 362 (1934).
- [2] A. Seeger, *Dislocations and Mechanical Properties of Crystals*, Wiley, New York 1957.
- [3] U.F. Kocks, H. Mecking, *Prog. Mater. Sci.* **48**, 171 (2003).
- [4] V.V. Pustovalov, *Low Temp. Phys.* **34**, 683 (2008).
- [5] B. Skoczen, J. Bielski, S. Sgobba, D. Marcinek, *Int. J. Plasticity* **26**, 1659 (2010).
- [6] N.V. Isaev, P.A. Zabrodin, V.Z. Spuskanyuk, A.A. Davidenko, V.V. Pustovalov, V.S. Fomenko, I.S. Braude, *Low Temp. Phys.* **38**, 80 (2012).
- [7] I.N. Kuzmenko, V.V. Pustovalov, *Cryogenics* **25**, 346 (1985).
- [8] E.O. Hall, *Proc. Phys. Soc. Lond. Sect. B* **64**, 747 (1951).
- [9] N.J. Petch, *J. Iron Steel Inst.* **174**, 25 (1953).
- [10] V.S. Fomenko, N.V. Isaev, V.V. Pustovalov, *Low Temp. Phys.* **19**, 301 (1993).
- [11] Yu.Z. Estrin, N.V. Isaev, T.V. Grigorova, V.V. Pustovalov, V.S. Fomenko, S.E. Shumilin, I.S. Braude, S.V. Malykhin, M.V. Reshetnyak, M. Janecek, *Low Temp. Phys.* **34**, 665 (2008).
- [12] P.C. Noble, S.J. Harris, K. Dinsdale, *J. Mater. Sci.* **17**, 461 (1982).