Effect of preliminary heterogenization on the structure and hardness of cryorolled aluminum alloy D16

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Abstract. The effect of preliminary heat treatment on transformations of the grain structure of the aluminum alloy D16 under isothermal rolling at the temperature of liquid nitrogen and subsequent annealing was investigated. It was found that heterogenization by intensifying the nucleation of new grains facilitates the nanostructuring of the alloy during cryorolling, as well as the formation of a fine-grained structure upon static recrystallization at post-deformation annealing.

1. Introduction
In recent years, aluminum alloys processed by severe plastic deformation at low (cryogenic) temperatures have become of the increasing scientific and commercial interest. This is reasoned by the fact that such processing results in nanostructuring of the alloy and a noticeable increase in its strength [1-3]. Along with the deformation conditions, their intensity is determined by a number of structural factors, including the degree of heterogeneity of the initial structure of the alloy. However, many aspects relating to this effect on the structure and the related properties, developed upon cryo-straining, still do not have a systematic description and remain unexplored.

The aim of the work is to study the effect of the main strengthening phases on the structural and microhardness changes in the aluminum alloy D16 during cryogenic rolling and post-deformation annealing in a wide temperature range.

2. Material and procedure
A hot-pressed rod of the aluminum alloy D16 of standard chemical composition (Al-4.4Cu-1.4Mg-0.7Mn, wt.%) with a diameter of 60 mm possessing a fibrous non-recrystallized structure (fiber thickness 100-200 µm, subgrain size 2 µm) was cut into plates with thickness of about 5 mm. After water quenching from a temperature of 505 °C, some of the plates were heterogenized for 5 hours at 400 °C. Subsequent isothermal rolling up to a total strain of \( \varepsilon \approx 2 \) was performed at the temperature of liquid nitrogen on a laboratory six-roller mill. Therewith, the long axis of the plates coincided with the rolling direction. Subsequent annealing was carried out in air in the temperature range of 190-500 °C with an exposure time of 0.5 hours.

The microstructure of the alloy was studied using a Nikon L150 optical microscope after etching of mechanically grinded and polished templates in a standard Keller's reagent. The volume fraction of secondary particles was estimated on the images obtained using a TESCAN MIRA 3 LMH scanning electron microscope (SEM). Objects for electron backscatter diffraction (EBSD) analysis and transmission electron microscopy (TEM) were prepared using a TenuPol-5 twin-jet polisher at -28 °C in a 20% solution of nitric acid in methanol. The EBSD analysis was carried out using an Oxford Instruments HKL Channel 5 system. On the EBSD maps, low-angle boundaries (with misorientation...
from 2 to 15°) were highlighted by thin white-, and high-angle boundaries (more than 15°) - by thick black lines. The dislocation structure was analyzed using a JEOL 2000EX transmission electron microscope (TEM) at an accelerating voltage of 160 kV.

Vickers microhardness (HV) was measured by a standard procedure at a load of 1 N at room temperature on an Axiovert – 100 microscope equipped with a MHT-10 microhardometer.

3. Results and discussions
The TEM analysis showed that the matrix of the pre-quenched alloy, supersaturated with the main alloying elements, contained only the compact T-phase (Al_{20}Cu_{2}Mn_{3}) precipitates with a length of about 340 nm and a thickness of about 70 nm (figure 1 a). In addition, platelet particles of the 0 and S phases precipitated and coagulated during subsequent heterogenization annealing (figure 1 e) were formed owing to the decomposition of the aluminum solid solution. The average size of such precipitates located along the grain boundaries reached about 1000 x 250 nm, while in the grain interiors they were much finer, not exceeding 380 x 70 nm. The volume fraction of these phases was about 8%.

Cryorolling of a pre-quenched alloy formed an inhomogeneous deformation structure consisting of arrays of deformation bands with predominantly weakly misoriented cellular structures (figure 1 b, c). Dislocation pile-ups were mainly noticed near the T-phases (figure 1 d), even though individual crystallites of a nanometric size with a TEM contrast typical for highly deformed nanograins were also present. In the pre-heterogenized state, on the contrary, a more homogeneous structure was formed with a more uniform distribution of dislocations, cells and dislocation boundaries, as well as a larger fraction of nano-sized crystallites, preferably located near particles (figure 1 f-h).

The observed effect is, apparently, originated from the interaction of lattice dislocations with secondary phases. It is generally accepted that the precipitates act as obstacles for the dislocation motion and provide their accumulation in adjacent matrix regions, followed by their transformations in the nuclei of new grains. Hence, the acceleration of dynamic recrystallization/deformation nanostructuring in a heterogenized alloy was related to the fact that the main strengthening phases "stimulated" the heterogeneous nucleation and grain refinement [4].

**Figure 1.** The structure of the alloy D16 after: quenching (a), heterogenization annealing at 400°C, 5 hrs (e), and subsequent cryorolling in the pre-quenched (b-d) and heterogenized (f-h) states. TEM (a,c,d,e,g,h), SEM-EBSD (b,f).
The evaluation of microhardness showed that after quenching the hardness of the alloy was about 115 HV, while heterogenization annealing led to a drop in hardness up to 75 HV; caused by a decrease in the alloying of a aluminum solid solution due to its decomposition with the formation and coarsening of secondary phases (i.e., decreasing the effects of solid-solution hardening and dispersion hardening). After cryogenic rolling, the increase in the microhardness of the alloy was almost the same in both states (about 65 HV) due to the developed deformation structure with signs of nanostructure (figure 1). As a result, the quenched alloy showed higher hardness (about 180 HV), whereas the hardness in heterogenized state was fixed at a level of 150 HV (figure 2).

The subsequent half-hour annealing at 190 °C further increased the hardness of the pre-quenched alloy up to 200 HV owing to the decomposition of the aluminum solid solution with the formation of dispersed products (zones) in a heavily deformed matrix. In contrast, such a response to aging during low-temperature annealing of pre-heterogenized alloy was completely lost due to the low degree of supersaturation of its solid solution upon preliminary heat treatment. As a result, the hardness of the deformed alloy decreased due to the occurrence of static recovery and recrystallization, which also took place more readily in the pre-heterogenized alloy (figure 3). With increasing the annealing temperature to 400 °C, the same restoration processes in the matrix, as well as an increase in the precipitate sizes, led to an intensive decrease in the alloy hardness in both states up to the level of the initial hot-pressed rod (about 80 HV) (figure 2). At annealing temperatures of beyond 400 °C, i.e. above the solvus point for the main strengthening phases [3], the alloy matrix was normally saturated by main alloying elements in accordance with the phase diagram and, hence, water-cooling after annealing led again to the formation of a supersaturated solid solution, accompanied by the repeat solution hardening of the alloy at room temperature in both structural states (figure 2).

As mentioned above, increasing the annealing temperature promoted the occurrence of static recrystallization in the present alloy and, hence, an entirely uniform fine-grained structure with an average grain size of 10 µm was evolved in the pre-quenched alloy at 500 °C (figure 4 a, c). Preliminary heterogenization promoted, in turn, the earlier (at lower temperatures) formation of more homogeneous recrystallized structures with a finer grain size (figure 4 b, d). Thus, a more uniform structure with a finer grain size of about 8 µm was processed in a heterogenized alloy (figure 4 d). This suggests that precipitates of the main strengthening phases also served as very effective structure control agents of, which stimulated the grain refinement upon static recrystallization in a heavily deformed alloy.

**Figure 2.** Microhardness of the cryorolled alloy D16 vs temperature of 0.5 hr post-deformation annealing.

**Figure 3.** TEM structure of the cryorolled and annealed at 250 °C alloy D16 in the pre-quenched (a) and heterogenized (b) states.
Figure 4. Microstructure of the cryorolled and annealed at 400 (a, b) and 500 °C (c, d) alloy D16 in the pre-quenched (a, c) and heterogenized (b, d) states.

4. Conclusions
1) The analysis showed that preliminary heterogenization of the age-hardenable aluminum alloy D16 facilitates the formation of a nanocrystalline structure during cryogenic rolling by stimulating dynamic recrystallization, as well as the development of a fine-grained structure during subsequent annealing by stimulating static recrystallization.
2) The highest hardness of about 200 HV was achieved in the pre-quenched alloy D16 after cryorolling and the subsequent half-hour annealing at 190 °C. At the same time, cryorolling of the preliminary heterogenization alloy led to a hardness of only 150 HV, which then monotonically decreased upon the subsequent annealing below the solvus point. Post-deformation annealing at higher temperatures led to recovery and recrystallization in both structural states, which reduced the hardness level up to the value of about 80 HV, corresponding to the initial hot-pressed rod.

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