Effect of aging on structure and strength of high pressure torsion processed 2024 aluminum alloy

M V Markushov, E V Avtokratova, R R Ilyasov, S V Krymskiy, A A Khazgaliieva and O Sh Saitdikov

Institute for Metals Superplasticity Problems, Russian Academy of Sciences, 39 Khalturin st., Ufa 450001, Russia.

E-mail: mvmark@imsp.ru

Abstract. Pre-quenched samples with a diameter of 20 mm and a thickness of 1 mm, cut from commercial hot-pressed rod of the 2024 aluminum alloy, were severely deformed by high-pressure torsion (HPT) with 10 revolutions under a pressure of 6 GPa at room temperature and then annealed for up to 50 hours in the temperature range of 100-190 °C. It was found that the post-deformation annealing at relatively low temperatures and/or short times can result in the alloy aging and 10-15% increased hardness. However, the conventional regimes of artificial aging of the alloy according to T6 heat treatment led to a sharp drop in its strength due to recovery and recrystallization of the matrix and simultaneous overaging. The nature of the revealed structural and property relations is discussed.

1. Introduction

Data published over the last decades have shown a high potential for enhancement the service properties of conventional metallic materials due to novel methods of their structure/phase control through nanostructuring (NS) - imparting the size less than 100 nm to the main structural components. Owing to the comparative simplicity and applicability for many metals and alloys, NS based on severe plastic deformation (SPD) to effective strains of $e>1$ [1] is of particular interest. To date, a number of SPD techniques have been developed, a wide range of nanomaterials have been processed and several industrial applications have been claimed. However, despite the huge number of studies, there are many unexplored points, starting with routes of cost affordable NS technologies. In addition, the mechanisms and factors responsible for the effective formation of nanostructures and improved service properties are still not so clear. As for wrought aluminum alloys, these points are narrowed to the effect of precipitates, which, judging by literature data, is rather ambiguous. For instance, in [2,3] it was established that the formation and/or increase in the volume fraction of disperse phases before SPD led to a greater proportion of nanograins of less size in the deformed structure. In contrast, in [4,5] it was shown that the formation of high densities of nanosized precipitates in high-strength alloys can completely suppress recrystallization of the matrix under SPD.

One of the topical issues for the age-hardenable Al alloys is the effectiveness of post-SPD aging. Along with the deformation strengthening owing to formation of new grains and subgrains, and an increase in the density of dislocations, the mechanical behavior of the SPD-ed alloy with further aging will be determined by changes in the impacts of the disperse hardening and aluminum solid solution strengthening. Though the contribution of the former to the strength of SPD materials is comparatively small, alloying elements can provide noticeable post-deformation disperse hardening of the matrix with formation of nanosized precipitates. However, their nucleation and growth in severely deformed matrix will be more heterogeneous and intense than in a non-deformed one, and may occur in a non-conventional sequence of phase transformations [6]. This behavior is commonly explained by the increased fraction of new grain boundaries and triple junctions, the high defectiveness of structure and
the level of stored energy, as well as the intensity of recovery and recrystallization [7-11]. Therefore, the development of the optimal aging regimes for SPD materials is a rather important task, which requires an understanding of the nature of structure transformations, ensuring hardening or softening. The aim of the study is to analyze the influence of post-SPD aging on the structure and strength of a medium strength age-hardenable aluminum alloy subjected to cold high pressure torsion (HPT) in a pre-quenched condition.

2. Material and procedure
A commercial hot-pressed rod of the 2024 alloy of standard chemical composition (Al-4.4Cu-1.4Mg-0.7Mn, wt.%) was used as the research material. Disk-shaped samples with a diameter of 20 mm and a thickness of 2 mm, cut out from the rod across its axis, were solution treated and quenched in water from a temperature of 505 °C. Further SPD was implemented immediately through the HPT technique with 10 revolutions under a pressure of 6 GPa at room temperature. The final artificial aging of the SPD-processed alloy was carried out in the temperature range of 100-190 °C with an exposure time up to 50 hours.

The structure of the alloy was analyzed using optical metallography (OM) and transmission electron microscopy (TEM). The objects for analysis were prepared by standard procedures with electropolishing at -28°C in 20% solution of HNO3 in CH3OH. Microhardness was determined at a 15-second load of 1N. The hardness and structure of the alloy were examined in the middle of the radius of the SPD-ed disk.

![Figure 1. Structure of the pre-quenched 2024 alloy rod. a – OM, b and c – TEM.](image)

3. Results and discussions
The initial pre-quenched alloy had a predominantly coarse-fibered structure with a fiber thickness of ~100-200 µm (figure 1). The grain and subgrain boundaries were decorated with coarse particles of excess and impurity phases (figure 1a), and their interiors contained elongated in the pressing direction precipitates of secondary T-phase (Al12Cu2Mn3) with the sizes of 330×70 nm and densities about 3.4×10^2 µm^-3 (figures 1b, c). With the subsequent HPT the structure of the matrix – aluminum solid solution supersaturated with Cu and Mg, transformed into a non-equilibrium nano-fragmented one with a fragment / (sub)grain size of about 75 nm (figures 2a, e). Judging by the TEM analysis, the deformation structure of the alloy was not so homogeneous: in particular, it was easy to find out the band components, indicating the development of a microshear banding under HPT. Such heterogeneity was virtually caused by the non-uniform distribution of primary and secondary phases, involving T-phase precipitates, and by their interaction with dislocations. Thus, frequent observations of these precipitates at non-equilibrium boundaries of fragments mean their active participation in the dynamic recrystallization of the matrix [7].

As a result of HPT, the alloy was considerably strengthened and its hardness increased from ~130 up to 270 Hv. Providing the almost maximum hardness, recorded for HPT aluminum alloys [9], was caused by deformation strengthening, resulting from nanostructural hardening and maintaining the solid solution strengthening. Subsequent annealing at 100°C led to a further increase in the hardness of
the alloy, and its gain proceeded up to 50 hours (figure 3). This fact testified that the additional post-deformation hardening of the alloy could be caused by the effect of disperse strengthening (artificial aging) only. The signs of the latter in the form of lamellar precipitates of the main strengthening phase(s) (S and/or Θ) were hardly distinguished and found only in separate fragments (figure 2f) after transformation of their structure into a more equilibrium one due to recovery. The morphology of these phases had no obvious differences from those commonly observed, except for their extremely small length, limited by the size of a fragment. Thus, it is possible to claim that during aging of the alloy at 100 °C, along with the decomposition of aluminum solid solution and the predominant formation of Guinier-Preston-Bagaryatsky zones, the structure of the fragments underwent static recovery. Therewith, losses in the strength of the alloy due to elimination of crystal defects, as well as from a decrease in the level of alloying of aluminum solid solution were compensated and overcame by disperse strengthening with an even more than 10% increase in alloy. First of all, two maxima on the dependence of hardness on annealing time have been observed (figure 3). The first one was probably conditioned by hardening at the above mentioned stage of zone aging, and the second one – at the stage of phase aging. It should be noted that the latter was found at much lower temperatures and shorter times than those under conventional T6 route (artificial aging to maximum strength is usually realized in the pre-quenched alloy under annealing at T = 185-195 °C during 11-13 hours). This finding was not surprising, since the decomposition of an aluminum solid solution should occur more rapidly in a severely deformed alloy, which suggested a shift of the maximum on the kinetic hardness curves to lower temperatures and times. By the way, a similar result was obtained for highly work hardened conventional sheets, showing the maximum of hardness after 7-9 hours of aging at conventional temperatures.

At the same time, these data testify to a low thermal stability of the obtained nanostructure and a lowered interval of working temperatures for HPT-processed nanostructured alloy. Judging by the TEM data (figures 2c, g), static recovery and continuous static recrystallization were the main reasons for the softening behavior of the alloy during annealing at 140 °C. Recrystallization occurred through in-situ mechanism and was followed by intense migration of fragment/grain boundaries. The latter resulted in simultaneous changes in the morphology of the secondary phases, giving them a compact

Figure 2. TEM structures of the HPT processed (a, e) and post-HPT aged for 48 hours at 100 (b, f), 140 (c, g) and 190 °C (d, h) alloy.
shape (figure 2g) due to the early violation of their coherency by continuous changes in the lattice of the surrounding matrix.

The overaging stage and intensive decrease in the hardness of the alloy due to the loss of disperse strengthening were observed at the annealing times of beyond 10 hours).

During annealing at 190 °C the zone stage of aging was not fixed and a short phase hardening was followed by a strong hardness drop due to the activation of both discontinuous and continuous static recrystallization and strengthening phase coagulation (figure 2). As a result, a bimodal recrystallized structure was formed with spherical precipitates rather evenly distributed (figure 3). Though a considerably decreased strength was observed, the alloy did not lose its nanostructured nature, since the sizes of all phase components, including statically recrystallized grains, still retained in the nano range.

Besides, it should be noted that the detected behavior of the alloy was virtually caused by processes at the level of interaction of mobile dislocations with clusters containing atoms of Cu and Mg – fragments of zones and metastable phases formed during processing. Such a point of view was formulated and proved in [12], and the results of this study act in its support.

![Figure 3. HPT alloy hardness vs time of aging at 100 (▲), 140 (♦) and 190 °C (■).](image-url)

4. Conclusions
Few (nano)structured states of the 2024 aluminum alloy, having a hardness in the range of 150-300 Hv, were processed by thermomechanical treatment using HPT and artificial aging. It is shown that the ambient temperature strength of the nanostructured alloy is determined not only by grain boundary strengthening, as is traditionally believed, but also strongly depends on the dislocation density, nature, morphology and distribution of the second phases. The data obtained testify to low thermal stability of the HPT-processed nanostructured alloy and to decreased interval of its working temperatures. The main reasons for this behavior of the alloy are intensive recovery and recrystallization of the matrix with the simultaneous effect of overaging during the post-SPD annealing.

Acknowledgments
The work was performed under the grant No. 16-19-10152 of Russian Science Foundation on the basis of Collective center of IPSM RAS "Structural and physics-mechanical analyses of materials".

References
[1] Valiev R Z, Estrin Y, Horita Z, Langdon T G, Zehetbauer M J and Zhu Y 2016 JOM 68 1216
[2] Rabinovich M Kh, Markushev M V and Murashkin M Yu 1997 Mater. Sci. Forum 243 591
[3] Barlow C Y, Hansen N and Liu Y L 2002 Acta Mater. 50 171
[4] Krymskiy S V, Shtidkov O Sh and Markushev M V 2016 AIP Conf. Proc. 1785 040030
[5] Markushev M V, Burdastykh Yu L, Krymskiy S V and Shtidkov O Sh 2017 Lett. Mater. 7 101
[6] Sha G, Wang Y B, Liao X Z, Duan Z C, Ringer S P and Langdon T G 2009 Acta Mater. 57 3123
[7] Humphreys F J, Hatherly M 2004 Recrystallization and Related Annealing Phenomena (Oxford: Elsevier) p 658
[8] Krymskiy S V, Sitdikov O Sh, Avtokratova E V, Murashkin M Yu and Markushev M V 2012 Rev. Adv. Mater. Sci. 31 145
[9] Markushev M V, Avtokratova E V and Sitdikov O Sh 2017 Lett. Mater. 7 465
[10] Markushev M V 2009 Phys. Metals Metallogr. 108 161
[11] Markushev M V, Avtokratova E V, Krymskiy S V and Sitdikov O Sh 2018 J. Alloys Comp. 743 773
[12] Chen Y, Gao N, Sha G, Ringer S P and Starink M J 2015 Mater. Sci. Eng. A 627 10