

Effect of structural-phase transformations on the microhardness of the Al-22%Si-3%Cu-1.7%Ni alloy after high-pressure torsion and annealing

G R Khalikova, G F Korznikova and V G Trifonov

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

E-mail: gulnara.r.khalikova@gmail.com

Abstract. The squeeze casting Al-22%Si-3%Cu-1.7%Ni alloy was subjected to high-pressure torsion (HPT) and subsequent annealing for investigation of their effect on the microstructure and microhardness. Specimens with a diameter of 8 mm and a thickness of 0.45 mm, cut from a squeeze cast ingot (region of ingot without primary silicon particles), were severely deformed by HPT via 5 revolutions under a pressure of 4 GPa at room temperature. The edge parts of the deformed samples were annealed at temperatures of 300, 400 and 500 °C for 5 minutes. HPT resulted in the refinement and partial dissolution of the eutectic silicon and primary intermetallic particles in an aluminum matrix with the formation of abnormal supersaturated solid solution and fragmentation of the structure that caused an increase in microhardness from 149±10 HV for the squeeze casting state to 285±14 HV for the deformed state. Subsequent annealing leads to the decomposition of the abnormal supersaturated solid solution, which occurred simultaneously with the recovery and recrystallization of the fragmented structure, which led to a decrease in the microhardness to 183±5, 108±5 and 132±6 HV at temperatures of 300, 400 and 500 °C, respectively.

1. Introduction

Severe plastic deformation techniques are actively used to form ultrafine grained and nanocrystalline structures in various aluminum alloys, which significantly change their physical and mechanical properties [1–3]. At the same time, during severe plastic deformation, a change in the phase composition can occur simultaneously with a structural change of the matrix of alloys, caused by the accelerated decomposition of the supersaturated solid solution i. e. dynamic strain aging [4,5] or dissolution of the second phases, including transition metal aluminides [6,7].

It is known that in the course of the conventional heat treatment of strain ageing aluminum alloys, along with recovery and recrystallization, a sequential decomposition process of a preliminarily supersaturated solid solution can occur [8]. However, after severe plastic deformation, the supersaturated solid solutions are metastable and at heating, accelerated decomposition undergoes, in some cases, with a change of its kinetics. As a result, the structure may differ from the structure obtained by conventional treatment [4,9–10].

The aim of the study was to evaluate the influence of high-pressure torsion and annealing on the microstructure, phase composition and microhardness of the Al-22%Si-3%Cu-1.7%Ni alloy.

2. Experimental

The commercial casting heat-strengthening Al-22%Si-3%Cu-1.7%Ni (AK21 in Russian classification) alloy was used in the present study. The initial state represents squeeze casting (SQ). The ingot was



characterized by inhomogeneity of a distribution of primary silicon crystals. In this work, parts of an ingot without primary silicon crystals were investigated.

Samples in the form of a disc with a diameter of 8 mm were mechanically cut and polished to a final thickness of about ~0.45 mm and subjected to severe plastic deformation by means of high pressure torsion (HPT) for 5 revolutions under a pressure of 4 GPa at room temperature. Then, the processed samples were subjected to annealing at 300, 400 and 500 °C for 5 minutes. The edge parts of the deformed and annealed samples were examined.

Microstructural characterization in the initial condition, after HPT and annealing was performed using a Tescan Mira 3LMH scanning electron microscope (SEM) by means of secondary electrons (SE) and back-scattered electrons (BSE) detectors. The fine structure of the HPT alloy was analyzed by a JEOL-2000 EX transmission electron microscope (TEM). The chemical composition of aluminum solid solution and secondary phases was investigated using SEM Tescan VEGA 3 SBH with an energy dispersive X-ray spectroscopy (EDS) supplement. Electron Backscatter Diffraction (EBSD) analysis of the annealed samples was performed by a Tescan Mira 3 LMH SEM.

A quantitative estimation of the average area (S) and the volume fraction (V) of the primary (Pr) and secondary (Sec) phases was performed using computer analysis techniques circling each particle around the perimeter; the average grain size was calculated by the line intercept method. The microhardness of the alloy in all conditions was evaluated by the Vickers hardness method. Statistical analysis was performed with a confidence level of 95%.

3. Results and discussion

The typical microstructure of the Al-22%Si-3%Cu-1.7%Ni alloy in the initial squeeze casted state is shown in figure 1 a. The structure of the alloy consisted of a solid solution of α -Al, double eutectic (α -Al)+Si^{Eu}, and particles of intermetallic phases (IPh^{Pr}): Al₅Cu₂Mg₈Si₆, Al₃CuNi, (NiCu)₂Al₃, (FeMnCu)₃Si₂Al₁₅ and CuSi₄Mg₅Al₄ (identified by EDS analysis).

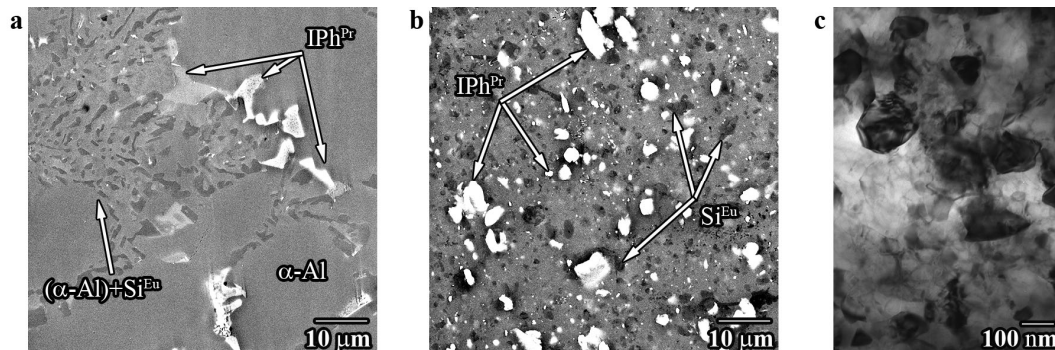


Figure 1. Typical structure of the Al-22%Si-3%Cu-1.7%Ni alloy in the initial squeeze casted state (a) and after HPT (b, c): a, b – SEM (SE imaging mode), c – TEM bright field.

High-pressure torsion resulted in fragmentation of excess phases of the alloy and a decrease in their volume fraction (figure 1 b). The quantitative characteristics of the structural constituents of the alloy are given in the table 1. The fine structure of the deformed alloy consisted mainly of individual fragments ranging in size from 20 to 200 nm, surrounded by nonequilibrium low and high angle boundaries (figure 1 c).

Since the Al-22%Si-3%Cu-1.7%Ni alloy was obtained by squeeze casting, during which the saturation of the solid solution by alloying elements did not occur due to the high cooling rate of the melt, deformation aging at HPT was suppressed. However, according to the results of the EDS analysis, HPT at room temperature resulted in an increase in the content of dopant and impurity elements in α -Al, contributing to the additional supersaturation of the solid solution (figure 2).

During the subsequent annealing, accelerated decomposition of the anomalously supersaturated solid solution occurred with the precipitation of particles of secondary phases (figure 3). Such rapid processes of nucleation and growth of the secondary phases during annealing of the deformed alloy

were related to the nonequilibrium state of the alloy after HPT, which is confirmed by the results of fine structure analysis, and an increased concentration of deformation-induced defects in similar materials [11].

Table 1. Average area (S) and volume fraction (V) of silicon particles (Si), primary (Pr) and secondary (Sec) intermetallic phases (IPh) of the Al-22%Si-3%Cu-1.7%Ni alloy after various treatments.

Phase	Parameters	SQ	HPT	Annealing for 5 minutes at the temperature, °C		
				300	400	500
Si	S, μm^2	2.9 $\pm$ 0.1	1.2 $\pm$ 0.1	0.45 $\pm$ 0.01	0.53 $\pm$ 0.02	1.08 $\pm$ 0.05
	V, %	14 $\pm$ 1	8 $\pm$ 1	11 $\pm$ 1	13 $\pm$ 1	13 $\pm$ 1
IPh	S ^{PR} , μm^2	5.6 $\pm$ 0.5	0.49 $\pm$ 0.05	0.46 $\pm$ 0.04	0.53 $\pm$ 0.04	0.50 $\pm$ 0.04
	S ^{SEC} , $10^{-3} \mu\text{m}^2$			20.1 $\pm$ 1.2	34.4 $\pm$ 2.2	42.9 $\pm$ 3.2
	V, %	11 $\pm$ 1	6 $\pm$ 1	9 $\pm$ 1	10 $\pm$ 1	14 $\pm$ 1

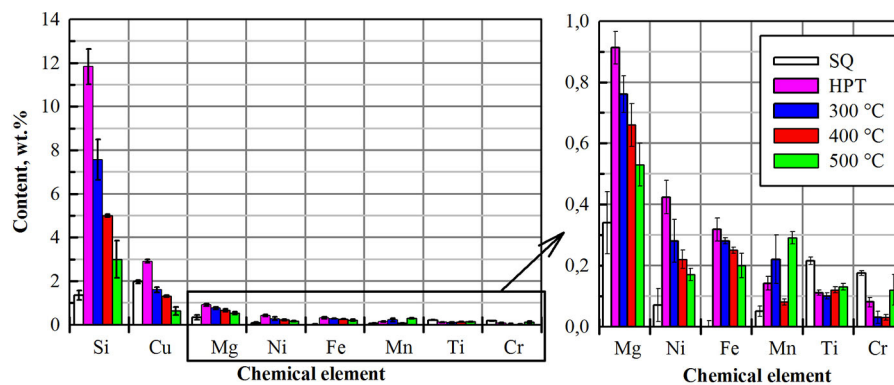


Figure 2. Distribution weight content of alloying and impurity elements in solid solution of Al-22%Si-3%Cu-1.7%Ni alloy in the initial squeeze casted (SQ) state, after the HPT and subsequent annealing at 300, 400, and 500 °C.

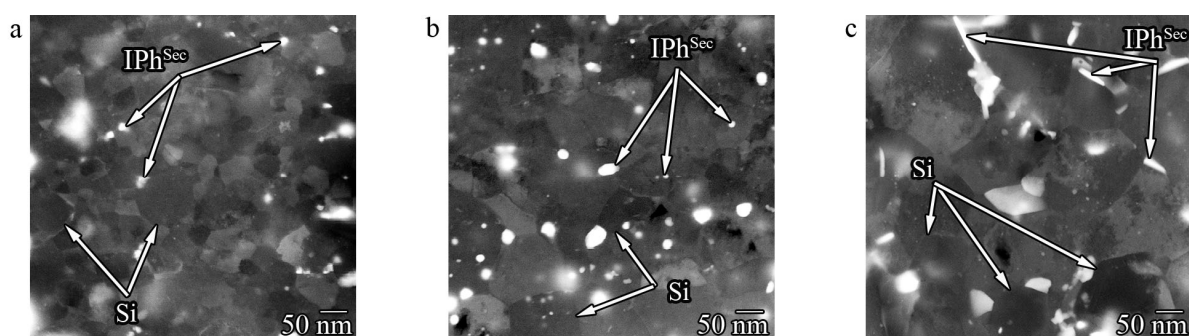


Figure 3. Typical structure of HPT Al-22%Si-3%Cu-1.7%Ni alloy after annealing for 5 minutes at 300 °C (a), 400 °C (b) and 500 °C (c) (SEM, BSE imaging mode). General view of strengthening secondary phases and (sub)grains.

The results of quantitative estimations showed that an increase of the annealing temperature resulted in an increase in the average area and volume fraction of silicon particles and intermetallic phases (table 1). This is due to the fact that the nucleation and growth of the dispersed secondary phases took place during the decomposition of an anomalously supersaturated solid solution.

The results of the quantitative estimation are in a good agreement with the results of the EDS analysis of the solid solution. After annealing of the deformed samples, the content of dissolved

elements in the solid solution decreased (figure 2), and the higher the annealing temperature, the more intensive the decomposition of the solid solution.

During the post-deformation annealing of the alloy, the recovery and recrystallization of the fragmented nanostructure took place simultaneously with the decomposition of the anomalously supersaturated solid solution. This follows from the predominance of high-angle boundaries on the EBSD-maps of the alloy, for example, annealed at 400 °C (figure 4). With increasing annealing temperature, the average grain size increased up to 0.19 ± 0.06 , 0.43 ± 0.05 and 0.67 ± 0.07 μm for 300, 400 and 500 °C, respectively.

Structural changes of the alloy after HPT and subsequent annealing are in good agreement with the results of microhardness measurements. Thus, HPT led to an increase in microhardness from 149 ± 10 HV (for SQ state) to 285 ± 14 HV. Subsequent annealing of the HPT samples at 300, 400 and 500 °C reduced the microhardness down to 183 ± 5 , 108 ± 5 and 132 ± 6 HV, respectively.

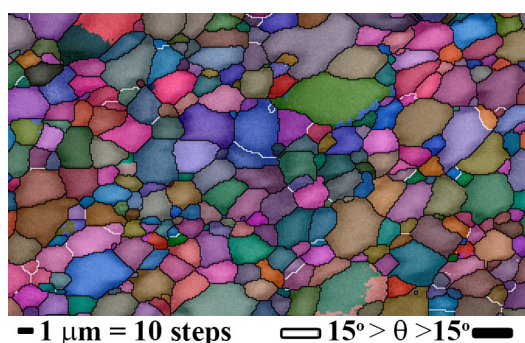


Figure 4. EBSD-map of HPT Al-22%Si-3%Cu-1.7%Ni alloy after annealing at 400 °C for 5 minutes.

4. Summary

During high pressure torsion of the Al-22%Si-3%Cu-1.7%Ni alloy, fragmentation of excess phases and their partial dissolution with the formation of an anomalously supersaturated aluminum solid solution, as well as the formation of a fragmented nanostructure with a fragment size from 20 to 200 nm took place. Subsequent annealing in the temperature range 300...500 °C led to intensive decomposition of anomalously supersaturated aluminum solid solution with the formation of secondary phase particles and simultaneous recovery and recrystallization of the fragmented nanostructure. With an increase in the annealing temperature, the average grain size raised from 0.19 ± 0.06 to 0.67 ± 0.07 μm . Such structural changes led to an increase in microhardness from 149 ± 10 HV (for SQ state) to 285 ± 14 HV (for HPT state), and then, upon subsequent annealing, – to a decrease to 183 ± 5 , 108 ± 5 and 132 ± 6 HV at 300, 400 and 500 °C, respectively.

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