ABSTRACT

Currently there is an increased interest in ultrafine-grained (UFG) materials produced by severe plastic deformation (SPD). This communication examines the features of structural and mechanical behavior of aluminum alloy under high plastic deformation with intense cooling at low temperatures. Aluminum alloy 2024 is studied. It is subjected to ECAP in an equal-channel step matrix of an angle of the channels junction of 125° and a step matrix of the same angle of the junction channels, but with the use of nitrogen. The average grain size is determined by transmission electron microscopy, while the strength limit, the conditional yield strength and the elongation to failure of the samples are determined using the method of mechanical tensile tests. The study reveals that the microstructure of aluminum after pressing with cryogenic cooling is more fine-grained and provides better mechanical properties. It is determined that the new UFG grains of the pre-tempered alloy 2024 obtained in the course of equal-channel angular pressing (ECAP) in a matrix of parallel channels are formed through the development of continuous dynamic recrystallization. This work involves a deeper understanding of the theoretical concepts and the practical aspects of the methods of SPD with structure formation.

Keywords: microstructure, ECAP, aluminum, cryogenic cooling, microhardness.

INTRODUCTION

One of the topical tasks of materials science and machine building refers to the increase of the physical and mechanical properties of the products and the semi-finished products. The solution of such problems lies in the field of creating highly efficient technologies using modern and advanced processing methods. In recent years, the creation of new products is limited by the high demands in respect to their technological and operational properties. The use of the traditional materials cannot provide the proper meeting of these requirements, while the constructive methods of strength increase lead to an increase of the mass of the structures and the complication of the technology for their manufacturing [1 - 3]. The possibilities of alloying have largely been exhausted by now, and the development of completely new alloys requires large material costs for the creation of new compositions, their certification and implementation. One of the solutions to this problem is the development of new materials with an ultrafine-grained (UFG) structure and high mechanical properties [4 - 7]. Therefore, a new direction in material science and material forming is intensively developing in the course of the recent decades. It refers to the formation of ultrafine-grained and nanostructures in metals and alloys. On the basis of this direction, it is possible to create a fundamentally new complex of physico-chemical and mechanical properties in conventional structural materials [8 - 15].

The production of a metal structure of crystallite sizes of 1 μm - 10 μm is achieved quite simply by using various thermomechanical treatments [15 - 17]. However, to obtain a submicroscopic and a nanostructure, new methods have been developed, commonly called “severe plastic deformation” (SPD). ECAP is the most common form [18 - 22]. However, from a practical point of view, this method
is labor and resource-consuming (high material costs and a complex tooling construction). It is also inefficient when scaling the process to obtain long-length workpieces. In this regard, work is underway to improve it. Also, the average grain size after ECAP, especially in pure metals, is within 300 nm - 500 nm [23 - 25]. The limitation of the grain refinement effect with the increase of the level of deformation by the traditional methods of SPD is explained by the dynamic recrystallization (recovery) occurring when certain deformation degrees are reached [26]. That is why the development of other methods for the production of UFG materials, devoid of the indicated shortcomings of traditional SPD methods, presents a great interest. Recently, there has been an increased attention to using the possibilities of intensive deformation at the temperature of liquid nitrogen or close to it [27 - 31]. This treatment is not accompanied by a dynamic recovery or recrystallization, which allows more efficient grinding of the grain structure. Another advantage of this deformation refers to the significant increase of the strength at a less deformation effort when compared to that of traditional methods of SPD.

Thus, the relevance of this work is related to a deeper understanding of the theoretical concepts and the practical aspects of SPD methods application with cryogenic cooling to nanostructuring processes and the characteristics of the metallic materials strength and plasticity. The possibility of significantly expanding the field of application of industrial metals and alloys by creating advanced technological processes for the production of ultrafine-grained semi-finished products and products of a qualitatively new level of physical and mechanical properties is also envisaged.

EXPERIMENTAL

The combination of SPD at a room temperature with ECAP method with cooling of the deformed workpiece by liquid nitrogen immediately after leaving the die was currently investigated. The theoretical basis of this method of processing referred to the representation of the process of plastic deformation of all crystalline bodies as a physico-chemical transformation, accompanied by recrystallization [32] in the process of deformation.

Aluminum alloy 2024 was the material studied, as it was easy to adjust the parameters of the secondary phases, including their modality, morphology and size distribution, to the change of the structure of the die from coarse-grained to recrystallized, including UFG state. The experimental sample referred to a hot-pressed rod of a diameter of 30 mm of industrial deformable heat-strengthened alloy 2024 of the standard chemical composition (Al, 4.4 mass % of Cu, 1.4 mass % of Mg, 0.7 mass % of Mn). The workpieces of a square section of 15 mm x 15 mm x 70 mm cut along the axis of the rod were heated to 500°C and, after a half hour holding, were quenched in water to fix the supersaturated aluminum solid solution. The samples obtained were further subjected to ECAP in a usual die of parallel channels of an intersectional angle of 125°. ECAP was carried out along the route Be with a rotation of the workpiece by 90° around the longitudinal axis. The friction between the tool and the workpiece was decreased by applying palm oil with graphite as a lubricant. The deformation was carried out at a room temperature.

In the first series of the experiments, each workpiece was deformed by the ECAP method. In the second series of experiments after each conventional passage of the workpiece through the die, it entered a container with liquid nitrogen (Fig. 1). In both cases, the number of passes through the die of parallel channels was equal to 4.

The preparation of the samples for the metallographic analysis was carried out on the electrolytic sample preparation unit Struers.

Fig. 1. The scheme ECAP with cooling with liquid nitrogen.
The middle plane of the sample was examined in all cases to avoid the influence of the peripheral areas. The transverse and the longitudinal sections of the samples obtained were considered. The structure and the phase composition of the alloy were analyzed by optical and transmission electron microscopy. The qualitative and the quantitative analysis of the microstructure of the groundmass and the primary phases was carried out using an optical microscope LEICA equipped with an attachment for determining the microhardness of individual phases as well as with software for determining the grain score and the number of the phases on the mechanically polished and etched by Keller’s reagent thin sections.

The fine structure was examined on a transmission electron microscope (TEM) JEM2100 in the magnification range from 1000 to 50000 times. The objects for TEM were prepared by polishing with a Tenupol-3 device at a temperature of -28°C and a voltage of 20 V in a 20% solution of nitric acid in methyl alcohol.

The misorientation was calculated between neighboring (adjacent) scan points. The dimensions of the scan step were previously determined from the measurement areas and the expected grain or subgrains sizes. The scanning was carried out on sections of 50 μm × 50 μm in 0.2 μm increments. Various misorientations between the grains were established using the minimum resolution of misorientation 2°. In view of the experimental error of the EBSD method, all small-angle boundaries of a misorientation of less than 2° were excluded from consideration. All scanned reference points of a confidence index of ~ 0.1 were excluded from the sets aiming to improve the overall accuracy of images.

The metallographic analysis of the structure of the alloy after ECAP in a die of parallel channels at a room temperature and with cooling in liquid nitrogen shows that highly deformed grains/subgrains are formed in both cases. However, in experiments using nitrogen, the structure is more dispersed with a smaller grain size (Fig. 2c), because the cryogenic treatment suppresses the recovery process characteristic of aluminum at a room temperature. The movement of dislocations is limited in the course of this treatment and which is why the dynamic recovery is inhibited. It leads to an increase of the dislocation density up to ρ = 6×10^{14} m^{-2}. Therefore,
the microstructure obtained after ECAP with using nitrogen is characterized by diffuse, nonequilibrium and weakly expressed grain boundaries. This is also verified in ref. [26]. Dislocation cells and dense dislocation walls are also visible. After ECAP at a room temperature, the microstructure is characterized by a lower dislocation density $\rho = 4 \times 10^{14} \text{ m}^{-2}$ and more precise grain boundaries.

EBSD analysis is performed as it is not possible to divide quantitatively the crystallites into grains and subgrains by their mutual misorientation and volume fraction analyzing the TEM images. The results obtained are presented in Fig. 3.

Both orientation maps shown in Fig. 3 demonstrate a fairly uniform microstructure. The average grain size after ECAP at a room temperature is $\approx 1 \text{ µm}$, while it is $\approx 0.5 \text{ µm}$ after cryogenic treatment. The distribution of the boundaries along the misorientation angles in both states is close. The fraction of the small-angle boundaries is 15% for treatment with nitrogen and 19% for ECAP. The share of the special boundaries detected

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Fig. 2. Microstructure of aluminum 2024 the initial state (a), after 4 passes of ECAP (b); after 4 ECAP passes with cooling with liquid nitrogen (c).

Fig. 3. Orientation microstructure maps aluminum 2024 after 4 passes of ECAP (a), after 4 ECAP passes with cooling with liquid nitrogen (b).
by EBSD analysis is minimal – 1 %. The total share of the large-angle boundaries in both cases is higher than 80 %, which provides to assume the formation of a UFG structure with predominating large-angle boundaries.

Based on the studies of TEM and EBSD analysis, it can be seen that two types of a structure are obtained by one deformation mechanism – dynamic recrystallization.

The results of the mechanical tests are shown in Table 1. The strength characteristics of aluminum are represented by the values of the conditional yield stress \( \sigma_{0.2} \) and the tensile strength \( \sigma_v \); the plastic characteristics are expressed through the values of the relative constriction and elongation of the samples prior to destruction.

The results of the microhardness study of alloy 2024 in both cases indicate that ECAP provides a fairly homogeneous microhardness over the entire section. The microhardness increases approximately 2 times after four ECAP passes when compared to that of the initial state, i.e. it increases from 1210 MPa in the initial state to 2450 MPa in the treated state. The microhardness increases from 1210 MPa to 2710 MPa with the use of nitrogen under ECAP.

The analysis of the mechanical properties of the alloy is carried out by means of a tensile test at a room temperature. Stress jumps are observed on the “stress-strain” diagrams in the process of stretching. This can be associated with cyclic hardening-softening under the action of motion and dislocations fixing on the barriers.

The tensile tests of the samples show that the strength of aluminum increases after 4 passes of ECAP without intensive cooling by 48 % - 50 %. The tensile strength \( \sigma_v \) increases from 400 MPa in the initial state to 596 MPa after four passes, that is, it increases almost 1.5 times. The conditional yield stress \( \sigma_{0.2} \) increases from 320 MPa in the initial state to 525 MPa after four passes, that is, it increases almost 1.6 times. The relative elongation during the tensile test shows that the level of plastic properties of aluminum decreases by 6 % after 4 passes of ECAP. The tensile tests of the samples after 4 passes of ECAP with intensive nitrogen cooling show that the strength of aluminum is increased by 75 % - 80 %. The strength limit and the conventional yield strength increase from 400 MPa to 740 MPa (the absolute increase is 340 MPa) and from 320 MPa to 700 MPa (the absolute increase is 380 MPa), respectively. Thus, the strength indicators increase almost 2 times. The level of the alloy plastic properties with the use of intensive cooling between each pass of ECAP is decreased by 4 %.

According to the above data, it can be concluded that the microstructure of aluminum after pressing with intensive cooling is more fine-grained and has higher microhardness values by suppressing the post-dynamic recrystallization of the alloy during its intensive cooling between the deformation cycles. The preservation of the nonequilibrium deformed state at the beginning of each ECAP cycle in case of nitrogen cooling explains the higher efficiency of grinding the structure according to the proposed technology.

**CONCLUSIONS**

A new technology is developed. It includes accelerated cooling of alloy 2024, previously subjected to cold plastic deformation by ECAP method in an initially quenched condition in order to reduce the number of passes and increase the productivity. It is revealed that the proposed technology provides a higher efficiency of grinding the structure when compared to that obtained by ECAP - is possible to obtain the characteristics after six ECAP passes in case of only 4 passes with intensive nitrogen cooling.

It is established that intensive nitrogen cooling following each ECAP pass leads to a significant hardening of alloy 2024 due to the suppression of post-dynamic recrystallization.
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