

## PHASE AND STRUCTURAL TRANSFORMATIONS IN FOIL BILLET DURING ROLLING OF HELICAL ROLLERS AND LONGITUDINAL WEDGE MILL (LWM)

Received – Priljeno: 2020-08-26  
Accepted – Prihvačeno: 2020-11-05  
Preliminary Note – Prethodno priopćenje

In the article, by changing the technological modes of strip rolling in helical rolls and a longitudinal wedge mill, the phase and structural transformations in the foil blank are studied. It was found that rolling in helical rolls with twelve passes of the initial alloy billet 2017 provides its nanostructuring with the formation of a mixed structure. It is shown that rolling alloy 2017 in helical rolls leads to an increase in the density of dislocations. It has been proved that with an increase in the number of passes, the excess phases, mainly of coarse particles of the (CuFeMn)  $3\text{Si}2\text{Al}15$  phase, are significantly crushed. The volume fraction of these particles does not change.

*Keywords:* aluminum alloy 2017, rolling, sheet, structure, temperature

### INTRODUCTION

Currently, scientists in many countries pay great attention to research areas that can significantly improve the service properties of metals and alloys through the development and application of new ways to control their structural-phase state without changing the chemical composition [1]. The most effective areas of science include research on the development of methods for producing nanostructures. Of all the nanostructuring (NS) method of metals and alloys, of particular interest is the method of obtaining non-crystalline structures of severe plastic deformation (SPD). Such interest is associated with the comparative simplicity of the SPD method and its applicability to all classes of crystalline materials [2]. At present, a number of SPD methods have been developed, a wide range of NS metallic materials has been obtained and studied, and work is underway to introduce them into industry. However, despite the huge amount of research, the mechanisms and factors responsible for the efficiency of the formation of new nanoscale phases (grains) are still largely unclear [3]. The issues of the initial and intermediate structural phase state on the structure and properties of materials after final processing also belong to poorly studied issues [4]. As applied to wrought aluminum alloys, these questions are practically reduced to revealing the role of

the second phases. Moreover, in the literature there is no unequivocal opinion about their role. So, for example, it is stated in the works that the introduction of dispersed particles and an increase in their volume fraction leads to the production of SPD structures with a smaller size and a larger proportion of new grains [5]. The purpose of this work is to study the phase and structural transformations in a 2017 aluminum alloy foil billet during SPD in helical rolls (HR) and rolling in a longitudinal wedge mill (LWM).

### MATERIALS AND METHODS

The spectrum of the disorientation boundaries, the average angle of disorientation ( $\Theta_a$ ), share high-angle boundaries, shape, and grain size ( $D_g$ ) and sub grain ( $D_s$ ), the volume fraction of recrystallized grains ( $V_g$ ), was determined by the EBSD maps obtained using microscope TESCAN MIRA 3 LMH registration system OXFORD and HKL CHANNEL5 software package. The size of the study area (raster) was  $200 \times 150$  points with scanning steps of  $0,5 \mu\text{m}$  and  $70 \text{ nm}$ . When processing the data, a standard Tango filter was used. Samples for x-ray analysis (XRA) measuring  $5 \times 5 \text{ mm}$  and a thickness of  $2$  to  $0,2 \text{ mm}$  were cut from rolled sheets [1]. To eliminate the influence of the rivet introduced during the cutting of the samples and the oxidized layer that arose during heating, the samples were mechanically ground and polished on diamond pastes with an abrasive dispersion from  $14 / 7$  to  $1 / 0 \mu\text{m}$  before the XRA. X-ray diffractometer DRON-4-07 in Cu-K $\alpha$  radiation at a voltage of  $40 \text{ kV}$  and a current of  $30 \text{ mA}$  with a wavelength  $\lambda = 1,54418 \text{ \AA}$  was used for x-ray analysis (x-ray) in the rolling plane. The value of the lattice parameter  $a$ , the mean square microdeforma-

S. A. Mashekov, (mashekov.1957@mail.ru), Satbayev University, Almaty, Kazakhstan

G. A. Smailova, Kazakh National Agrarian University, Almaty, Kazakhstan

B. A. Nurlybayev, S.A. Orynbayev, Taraz State University named after M. Kh. Dulaty, Taraz, Kazakhstan

Zh. S. Abdimuratov, A. Zhauyt, Almaty University of Power Engineering and Telecommunications, Almaty, Kazakhstan

tion of the crystal lattice  $\langle \varepsilon^2 \rangle^{1/2}$  and the size of the coherent scattering regions of the  $D_{\text{csr}}$  were calculated by automatic full-profile analysis of diffraction patterns in the software complex "MAUD" (Materials Analysis Using Diffraction). The measurement error of  $a$ ,  $\langle \varepsilon^2 \rangle^{1/2}$  and  $D_{\text{csr}}$  did not exceed  $\pm 0,0001 \text{ \AA}$ ,  $0,001 \%$  and  $5 \text{ nm}$ , respectively. Based on the experimentally obtained values  $a$ ,  $\langle \varepsilon^2 \rangle^{1/2}$  and  $D_{\text{csr}}$ , the dislocation density  $\rho$  in the material after various treatments was calculated by the formula used in:

$$\rho = 2\sqrt{3} \langle \varepsilon^2 \rangle_{\text{csr}}^{1/2} / D_{\text{CSR}} \cdot b$$

where  $b$  – Burgers vector dislocation (for aluminum  $b = 0,286 \text{ nm}$ ).

## RESULTS AND DISCUSSION

At the same time, the crystal lattice of the matrix also had a low level of microstresses ( $\langle \varepsilon^2 \rangle^{1/2}$ ) –  $0,12 \%$ , and relatively large size  $D_{\text{csr}} \sim 145 \text{ nm}$ , characterizing the size of the regions of the crystal lattice, misoriented at angles of the order tens of angular minutes. On the x-ray of the initial sample, reflexes of high and low intensity are observed. The former were indexed as aluminum solid solution. The second ones belonged to the  $\theta$  ( $\text{CuAl}_2$ ) and  $S$  ( $\text{Al}_2\text{CuMg}$ ) phases and indicated that the metal of the initial sample had a completely undissolved phase. Reflexes from other phases, including  $T$  ( $\text{Al}_2\text{Cu}_2\text{Mn}_3$ ) phases, could not be fixed because of their small volume fraction, which is beyond the sensitivity of the method X-ray analysis ( $< 5 \%$ ) [12].

By optical metallography (OM) and scanning electron microscopy (SEM) in the initial alloy, particles of two excess phases were observed, which differed in morphology: large complex (skeletal) forms and smaller compact particles. After etching the first became black, and the second dark brown. Comparison data of etch ability and chemical analysis of particles allowed to refer them to  $(\text{CuFeMn})_3\text{Si}_2\text{Al}_{15}$  and  $S(\text{Al}_2\text{CuMg})$  phase, respectively. The total volume fraction of the particles of both phases was  $(11,9 \pm 1,5) \%$ .

Thus, it can be concluded that in 2017 aluminum alloy, as a result of its rolling into HR with twelve passes, a mixed nano (sub) grain structure is formed, with crystallite size  $\sim 120 - 360 \text{ nm}$  and coherent scattering areas  $\sim 70 - 80 \text{ nm}$ . At the same time, the dislocation density did not change and the grain and sub grain boundaries on the TEM images became even clearer and thinner [2]. Judging by the Energy backscattered diffraction (EBSD) maps, these areas alternated and were arranged form of stripes stretched across the rolling direction. The share of high angle borders (HAB) in such a structure reached  $83 \%$ , and the average misorientation angle of the boundaries is  $\sim 36^\circ$ . At the same time, the distribution spectrum of boundary misorientations became close to the theoretical random distribution with a maximum of about  $48^\circ$ . An indirect confirmation of this is the nature of the change in the lattice parameter of the aluminum matrix during

rolling in the HR (see below). To study the changes of morphology of the excess phase after rolling in the HR has allowed establishing that during the rolling phase was milled. Moreover, particles were predominantly ground  $(\text{CuFeMn})_3\text{Si}_2\text{Al}_{15}$  - phase, while the size of the initially smaller particles of the  $S$ -phase decreased slightly.

The volume fraction of particles in aluminum alloy 2017 when rolling HR with twelve passes ( $13,3 \pm 0,4$ ) % was almost identical to that in the undeformed state ( $12,3 \pm 0,8$ ) %. The obtained data meant that in the process of rolling in the HR dissolution of excess phases did not occur, but only their grinding took place. The latter, apparently, was the main reason for some decrease in the average value of the volume fraction of the phases, as part of the particles became less than  $1 \mu\text{m}$  in diameter and accordingly, was not recorded. In this paper, there are dependences of the change in the lattice parameter on the degree of deformation obtained on the basis of full-profile analysis of diffraction patterns. From these materials it can be seen that these dependencies are nonmonotonic. First, with an increase in the number of passes to four lattice parameter of the alloy decreases sharply. With further deformation with the number of passes up to eight, the value of the lattice parameter varies only slightly and with a subsequent increase in the number of passes up to twelve, it increases rapidly [3]. The non-monotonous type of dependence indicates a simultaneous and unequal influence (contribution) of several factors that determined the absolute value and trends of the lattice parameter during the rolling process in alloy 2017. In our opinion, in the early stages of deformation, the decrease in the lattice parameter was caused by a significant increase in the defect structure of the material. Note that a similar effect was observed in SPD of pure metals for example, copper and nickel as well as nickel obtained by ball grinding. The author of suggested that a decrease in their lattice parameter is caused by the appearance of strong fields of elastic compressive stresses due to a significant increase in the fraction of grain boundaries due to the formation of a nanocrystalline structure. In this case, in the 2017 alloy only the formation of a developed cellular structure was observed. Apparently, the cell boundaries could also be a sufficient source of compressive elastic stresses, which influenced the lattice parameter value of the alloy. In this paper, the study of the quantitative characteristics of the particles was carried out at two structural levels – at the meso level by SEM and at the micro level by TEM. In the initial sample, large particles up to  $7 \mu\text{m}$  in size are observed at the meso level, with the most likely size of large particles being  $3 \mu\text{m}$ , the average distance between the particles being  $21,0 \pm 0,3 \mu\text{m}$ . At the micro level, the most probable size of small particles lay in the range of  $2 - 12 \text{ nm}$ , the average distance between the particles is  $132 \pm 7 \text{ nm}$ . Thus, the initial billet had a coarse-grained state with a certain concentration of solid solution (SS) with large (up to  $7 \mu\text{m}$ ) and small ( $2 - 12 \text{ nm}$ ) particles.

The results of the study size and distribution of particles at the mesa-level studied on SEM, after rolling into HR with a different number of passages, showed that with increasing accumulated strain, the average distance between particles decreases from 21 to 14  $\mu\text{m}$ , which corresponds to an increase in particle density by about 2 times. This can be explained by one of the processes determining the evolution of particles, the mechanical fragmentation of large particles. In our opinion, in alloy 2017 there is at least one coinciding slip plane between the particles and the aluminum matrix, through which the mechanical break of the particles can pass. The results of the study size and distribution of particles observed Transmission electron microscopy (TEM) after different numbers of rolling passes in HR showed that with increasing accumulated strain, the average distance between particles decreases twice from 132 to 70 nm, which corresponds to an increase in the density of particles of about 10 nm in size by about 4 times.

The study of the evolution of an ensemble particles of the second phases showed that after complex processing, including rolling HR with twelve passes and rolling LWM, the average particle size was  $53 \pm 7$  nm, the most probable value falls on 30 - 40 nm - particles of this size constitute more than 75 % of the total. The average distance between particles increased to  $730 \pm 20$  nm that is the density of particles decreased by 2 times [4].

At the same time, the average size groups of particles combined by composition and morphological characteristics vary in different ways. Complex deformation treatment leads to an increase in the average size of rod-like particles up to  $74 \pm 3$  nm and round ones up to  $55 \pm 6$  nm. Large hexagonal particles appear with a significant average size of  $123 \pm 3$  nm. The size square particle does not change and the triangular and oval particles are reduced in size to values of  $42 \pm 3$  and  $31 \pm 8$  nm, respectively.

The disintegration of the SS led to the release of six-sided aluminum-honey particles, the enlargement of rod-like and round particles. As a result of the deformation-induced dissolution of particles of the second phases, the average size of triangular and oval particles decreases to 41 nm. Taking into account the fact that after complex deformation processing, the density of particles decreases twice as compared with the initial state, it can be assumed that the deformation-induced dissolution particles second phase is a process that dominates the deformation-stimulated decomposition of SS.

Particles are also observed in the grain boundaries. And here again the question arises about the nature interaction particles and dislocations. On the one hand, at the SPD stage a dislocation wall could first form, which a small-angle boundary was formed and then particles of the second phases were separated in this boundary as a result of deformation-stimulated decomposition of the SS. On the other hand, the movement of dislocations could be stopped by a wall of densely located particles

of the second phases the implementation of the hardening mechanism according [5].

The fragments of the structure after the complex SPD including cold rolling on the LWM, are so strongly elongated along the thin strips that it was not possible to estimate their longitudinal size, the transverse size was about 70 - 120 nm. EBSD analysis showed a large number of misalignment angles over five degrees. The predominant number of large-angle boundaries indicates the formation of a fragmented nanocrystalline structure in the material.

## CONCLUSIONS

1) Hot rolling in the HR with twelve passes of the initial billet from the alloy 2017 provides its nanostructuring with the formation of a mixed structure, with the size of recrystallized grains (~240 nm) with a characteristic electron microscopic contrast for the SPD materials.

2) Hot rolling alloy of 2017 into HR, without changing the volume fraction of particles excess phases, leads to their significant grinding, with predominantly coarse particles of phase  $(\text{CuFeMn})_3\text{Si}_2\text{Al}_{15}$ . The dispersed particles of the T-phase gain preferential orientation and line up in the direction of rolling with the formation of lines.

3) Rolling aluminum alloy in HR 2017 leads to an increase in the dislocation density in the alloy matrix by 8 - 9 times to a level of  $\sim 8 \times 10^{14} \text{ m}^{-2}$ , at the same time, the lattice parameter of the matrix changes non-monotonically: first, it decreases sharply and then increases, approaching the values in the unreformed alloy aged to maximum strength.

## REFERENCES

- [1] Z. Pater, J. Tomczak, J. Bartnicki, M. R. Lovell, P. L. Menezes, Experimental and numerical analysis of helical wedge rolling process for producing steel balls, *Int. J. Mach. Tools Manuf.* 67 (2013), 1-7.
- [2] S. A. Mashekov, G. A. Smailova, A. M. Alshynova, A. Zhauyt, N. S. Sembaev, M. R. Maulenova, Investigation of the formation evolution of aluminum alloy 1050 structure during rolling in the spiral rollers and the longitudinal wedge mill, *Metalurgija* 59 (2020) 2, 203-206.
- [3] H. Yang, L. Zhang, Z. Hu, The analysis of the stress and strain in skew rolling, *Adv. Mater. Res.* 538 (2012), 1650-1653.
- [4] Q. Li, M. R. Lovell, W. Slaughter, K. Tagavi, Investigation of the morphology of internal defects in cross wedge rolling, *J. Mater. Process. Technol.* 125 (2002), 248-257.
- [5] M. F. Novella, A. Ghiotti, S. Bruschi, P. F. Bariani, Ductile damage modeling at elevated temperature applied to the cross wedge rolling of AA6082-T6 bars, *J. Mater. Process. Technol.* 222 (2015), 259-267.

**Note:** The responsible for England language is Aigerim Naurzybayeva, Almaty, Kazakhstan