Intensification of the process of equal channel angular pressing using ultrasonic vibrations

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Abstract: The work presents a new method of equal channel angular pressing (ECAP) using powerful ultrasonic vibrations (UV). The authors have developed an original device of ultrasonic ECAP, in which the waveguide with the matrix are made as a single unit, and the waveguide fastening elements are located in the nodal plane of mechanical displacements of the standing wave, the excitation of which occurs directly in the matrix and the blank during pressing. For the first time, it has been proposed to transmit ultrasonic vibrations to the zone of intersection of the matrix channels through which the blank moves, not through the punch, but by exciting vibrations in the matrix itself, i. e. the matrix is simultaneously a waveguide for longitudinal ultrasonic vibrations. This allowed increasing repeatedly the efficiency of ultrasonic action by reducing the friction forces between the surface of the blank and the surface of the matrix channels, as well as by reducing the deformation forces in the zone of intersection of ultrasonic ECAP, when the reduction in pressing force is less than 15 %, the excitation of ultrasonic vibrations directly in the waveguide – matrix allowed reducing the pressing force by 1.5–4 times. At the same time, the structure of the pressed materials also changes significantly: the grain size and their crystallographic orientations decrease, the microhardness increases. Changes in the phase composition for all samples produced by ECAP with ultrasonic vibrations, and by conventional technology are not observed.

Keywords: equal channel angular pressing; ECAP; ultrasonic vibrations; UV; bulk nanostructuring; severe plastic deformation; SPD; waveguide; matrix; deformation forces; grain structure; zinc; aluminium.

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INTRODUCTION

The production of bulk nanostructured metallic materials is a relevant and hot topic of modern materials science. Such metals and alloys are attractive for innovative applications, as they have unique properties. At the same time, the structural features of such materials (the proportion of lowand high-angle boundaries, grain size, etc.) are determined by the methods of their production. The two most widely used and studied methods of severe plastic deformation (SPD) are equal channel angular pressing (ECAP), and high-pressure torsion. ECAP, as a method of severe plastic deformation (SPD) of metallic materials, using which it became possible to produce blanks with a fine-grained structure due to bulk nanostructuring, was proposed by V.M. Segal and coworkers in the 70s of the XX century [1]. Since the early 90s, it has been used for SPD to produce submicron and nanosized metallic structures [2]. Such structures have significantly better mechanical properties optimally combining strength and ductility. In particular, the method is used to obtain submicron crystalline structures of metals such as Pd, Fe, Ni, Co, alloys based on Al, Mg, Ti, Zn, etc.

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The SPD method using ECAP involves forcing samples through intersecting at a certain angle channels of a constant cross section matrix. As a result, the samples are subjected to shear deformation in the intersection zone of the channels, which leads to a change in their structure and physical and mechanical properties¹ [3]. Thus, when carrying out the ECAP process, it is possible to accumulate an arbitrarily large shear deformation without changing the dimensions of the blank. In the process of passing through the channels, the total shear characteristics in the metal sample can be changed, due to its rotation between individual passes, i. e., structure formation during deformation directly depends on the ECAP route [4]. By repeated passes of the blank through intersecting channels, it is possible to achieve accumulation of the desired deformation degree, and as a result, the necessary structural changes. In this case, the geometric shape of the sample, with the exception of the areas near its ends, does not change. The ultrafine-grained (UFG) structure of samples with predominantly high-angle grain boundary misorientations obtained by the ECAP method, depends on many parameters: the number of passes, the route, the deformation temperature, the angle of channel intersection, the rounded radius at the intersection of the channels, the speed of the sample passage, the sample material, the type of lubricant [2; 5].

One of the ways to reduce the deformation forces during pressure metal treatment (PMT), is the use of ultrasonic vibrations (UV), for which various devices and schemes for supplying UV to the deformation zone are developed [6; 7]. In this case, the ultrasonic effect during the PMT process leads to a change in the structure, and physical and mechanical properties of materials. Depending on the frequency, UV amplitude, and the locality of the effect, it is possible to achieve both metal strengthening and its softening, plasticisation [6].

Pressure metal treatment with the UV imposition began to be widely used after the discovery of the acoustoplastic effect [8]. The effect consisted in a sharp decrease in the stress of plastic flow of the metal under ultrasonic action. The degree of reduction depends on many factors, primarily on the power of ultrasonic action and the technological parameters of metal forming processes. In particular, the vibrational speed of the ultrasonic tool should be much higher than the deformation rate of the metal [6; 7]. From this point of view, ECAP is an ideal process for intensification using UV, since the pressing speeds are low (except for explosive ECAP), and the friction forces are high, i. e. UV, by reducing friction forces, should affect both the force conditions of the ECAP process, and the properties of the resulting blanks [6; 7]. However, despite the obvious effectiveness of ultrasonic action on ECAP, ultrasonic vibrations have not been used in this process until recently, due to the complexity of introducing them into the deformation

zone. The authors of studied the reduction in the sliding friction force of metals using longitudinal or transverse UV^2 [9]. The results of their study show that vibrations in the longitudinal or transverse direction can be used to reduce significantly the sliding friction forces between the interacting surfaces. In the works [10; 11] the effect of applying ultrasonic vibrations to a punch, during the ECAP process, was numerically investigated. Calculations showed that a reduction in the deformation force should occur, which depends on the amplitude of vibrations and the speed of movement of the punch.

In fact, the results of experimental studies of ECAP of aluminium alloys with ultrasonic action on the deformed metal through a punch confirmed that the use of ultrasonic vibrations reduces the pressing force by 10 % [11]. At the same time, the amplitude and frequency of ultrasonic vibrations have a significant impact on reducing the pressing force [11–13]. The use of USV in ECAP leads to an increase in the yield strength, tensile strength and hardness of metallic materials [12; 14]. The disadvantage of the proposed method of ultrasonic action on ECAP is its low efficiency [10; 12; 15]. This is associated with the impossibility of introducing significant ultrasonic energy into the deformation zone through the punch, which is an element of the acoustic system – a waveguide for longitudinal ultrasonic vibrations.

The Institute of Technical Acoustics of the National Academy of Sciences of Belarus has developed an original device for ultrasonic ECAP, in which the waveguide with the matrix are made as a single unit with a total length equal to

$$l=n\frac{\lambda}{2},$$

where λ is the length of the longitudinal ultrasonic wave in the matrix-waveguide material;

n is an integer.

The waveguide fastening elements are located in the nodal plane of mechanical displacements of the standing wave, the excitation of which occurs directly in the matrix and the blank during pressing [16].

The purpose of the work is to intensify the equal channel angular pressing process using ultrasonic vibrations, as well as to study their effect on the force characteristics of ECAP of metal materials, and the properties of the produced samples when ultrasonic vibrations are excited in the matrix, i. e. directly in the deformation zone.

METHODS

Zinc with a purity of 99.9 wt. % and A7 aluminium alloy of standard chemical composition (Table 1) were selected as the material.

The initial samples of zinc and A7 aluminium alloy had a length of 20 mm and a diameter of 5 mm. The source of

¹ Shivashankara B.S., Gopi K.R., Pradeep S., Raghavendra Rao R. Investigation of mechanical properties of ECAP processed AL7068 aluminium alloy. IOP Conference Series: Materials Science and Engineering, 2021, vol. 1189, article number 012027. DOI: <u>10.1088/1757-899X/1189/1/012027</u>.

² Gudimetla K., Kumar S.R., Ravisankar B., Prathipati R.P., Kumaran S. Consolidation of commercial pure aluminium particles by hot ECAP. IOP Conference Series: Materials Science and Engineering, 2018, vol. 330, article number 012031. DOI: <u>10.1088/1757-899X/330/1/012031</u>.

Table 1. Chemical composition of the A7 material (GOST 11069-2001), % **Таблица 1.** Химический состав материала А7 (ГОСТ 11069-2001), %

Fe	Si	Mn	Ti	Al	Cu	Mg	Zn	Ga	Impurities
≤0.16	≤0.15	≤0.03	≤0.01	min 99.7	≤0.01	≤0.02	≤0.04	≤0.03	Others, 0.02 each

ultrasonic vibrations was an ultrasonic generator with a power of 4.0 kW with a PMS-15A-18 magnetostrictive transducer (Russia). The amplitude of vibrations at the end of the waveguide-matrix was up to 30 μ m, the vibration frequency was ~18 kHz. The ECAP process was carried out at a temperature of 20–22 °C using a PGPR hydraulic hand press with a punch travel speed of 4÷10 mm/s.

The structure of the samples was examined using a MICRO-200 optical microscope (Republic of Belarus), as well as a LEO1455VP scanning electron microscope (SEM) (Germany). The obtained raster electron microscopic images were adapted to the NEXSYS ImageExpert Pro 3 software environment. The grain structure was studied by the electron backscattered diffraction (EBSD) method, implemented using the HKL EBSD Premium System Channel 5 (UK) phase analysis diffraction attachment to the SEM. To determine the orientation of the grain structure, longitudinal (in the direction of deformation) sections of samples were prepared both in zinc and in the aluminium alloy.

Using a PMT-3M microhardness tester (Russia), the microhardness of zinc and aluminium alloy samples was determined after conventional ECAP and ECAP with ultrasonic vibrations. The microhardness value was determined using the Vickers method by measuring the lengths of the indentation diagonals, taking into account the load value of 0.196 N. To calculate the microhardness value, the average value of four measurements in each area under consideration was taken.

Graphite lubricant was used when pressing the samples. Zinc and A7 aluminium alloy blanks were forced through a matrix with two channels of equal cross-section with a diameter of 5 mm intersecting at an angle of 90° (Fig. 1).

RESULTS

It was found that the application of ultrasonic vibrations during ECAP of zinc and A7 aluminium alloy leads to a decrease in the pressing force by 1.5 times or more (Fig. 2). Changing the pressing speed within $4\div10$ mm/s has virtually no effect on the pressing force, and the dynamics of its change is determined by the value of the punch displacement. After one pass of ECAP with the application of USV (Fig. 3 a), the microstructure of zinc differs from the microstructure of zinc obtained with conventional ECAP (Fig. 3 b), and from the microstructure of the initial sample (Fig. 3 c). Grain refinement occurs, and the grains take a more equiaxial shape. The grain refinement process does not depend on changing the pressing speed in the studied speed range.



Fig. 1. Schematic (a) and external appearance (b) of the acoustic unit of the ultrasonic assisted ECAP device Рис. 1. Схема (a) и внешний вид (b) акустического узла устройства ультразвукового РКУП



Fig. 2. Dependence of the pressing force of zinc on the displacement of the punch (a) and A7 aluminium alloy (b) *Puc. 2.* Зависимость усилия прессования цинка от перемещения пуансона (a) и алюминиевого сплава A7 (b)



Fig. 3. Microstructure of zinc: a – initial; b – after 1 pass of ECAP without UV; c – after 1 pass of ECAP with UV **Puc. 3.** Микроструктура цинка: a – исходного; b – после 1 прохода РКУП без УЗК; c – после 1 прохода РКУП с УЗК

The results of the study of the analysis of the grain structure of zinc are shown in Figs. 4–7. In the initial alloy, the average grain size is 10.6 μ m. However, according to the data of the grain size distribution by size groups (Fig. 6 a), there is a large scatter of grain sizes. Comparison of the SEM images of the microstructure and the EBSD results allows stating that the volume fraction of large grains with a size exceeding 20 μ m is high. At the same time, a high density of low-angle grain boundaries is observed in large grains, as shown in the grain misorientation histogram (Fig. 5 a).

After deformation during the ECAP process, the average grain size decreases to 3.6 μ m (Fig. 6 b). Analysis of the grain structure shows that small grains are formed at the boundaries of large grains (Fig. 4 b). The proportion of low-angle boundaries in the grain decreases by 2 times (Fig. 5 b). This allows assuming that during one ECAP pass, deformation is carried out due to the movement of low-angle boundaries to the grain boundary. Since zinc has a pronounced anisotropy of the crystal lattice and, accordingly, mechanical properties, such a mechanism can occur for grains with a favourable orientation.

In the samples subjected to ECAP with the imposition of ultrasonic vibrations, a decrease in the average grain size to 2.9 μ m was revealed (Fig. 7). At the same time, a narrowing of the grain size distribution by size groups and a low concentration of low-angle grain boundaries are observed, as shown in Fig. 4 c and 5 c, respectively. The obtained result allows asserting that during deformation under the influence of ultrasound, the movement of dislocations is activated both in favourably oriented grains, and in the entire volume of the sample. From the analysis of the grain misorientation histograms, it is evident that in the sample of zinc subjected to one ECAP pass without ultrasonic vibrations, and with ultrasonic vibrations, the share of low-angle boundaries has an advantage (Fig. 5).

The zinc texture was studied by the EBSD method after ECAP without UV (Fig. 8 a) and ECAP with UV (Fig. 8 b). Fig. 8 shows the direct pole figures of the projections of the planes {0001}, {10-1-2}, {10-11} onto the *OX-OY* plane of the sample. The projection of the normal to the surface is located in the centre of the circle (Fig. 8). The maximum density of projections located near the centre of the circle in both cases falls on the reflection from the plane (0001).

The average microhardness value for zinc samples obtained by ECAP with UV was \sim 30.6 HV, for samples without UV – \sim 26.9 HV. At the same time, for all zinc samples,



Fig. 4. Grain structure of zinc: a – initial; b – after 1 pass of ECAP without UV; c – after 1 pass of ECAP with UV; d – colour code. Bold black lines highlight grain boundaries with a misorientation greater than 10°, thin black lines highlight low-angle boundaries with a misorientation greater than 2° and less than 10° Puc. 4. Зеренная структура цинка: a – исходного; b – после 1 прохода РКУП без УЗК; c – после 1 прохода РКУП с УЗК; d – цветовой код. Жирными черными линиями выделены границы зерен с разориентацией больше 10°,

тонкими черными линиями – малоугловые границы с разориентацией больше 2 и меньше 10°

a slight increase in microhardness is observed from the centre to the edges in the cross section.

As for the A7 aluminium alloy, the average grain size in the initial sample was ~16 μ m (Fig. 9 a, 10 a). The application of ultrasonic vibrations during ECAP changed both the grain size and shape (Fig. 9 b, 9 c). From the analysis of the grain size distribution histograms, it follows that ECAP results in grain fragmentation. The average grain size in the A7 aluminium alloy sample, after one ECAP pass without ultrasonic vibrations was 6.0 μ m (Fig. 10 b), while with ultrasonic vibrations it was 3.5 μ m (Fig. 11).

In the A7 aluminium alloy samples subjected to one ECAP pass without ultrasonic vibrations, an increase in the proportion of high-angle boundaries, and a two-fold decrease in the proportion of low-angle boundaries was found. The application of ultrasound resulted in a four-fold decrease in the proportion of low-angle boundaries compared to the initial alloy (Fig. 12).

Fig. 13 shows the results of the study of the texture of the aluminium samples. It was found that the initial sample had a pronounced (101) texture. After one ECAP pass without ultrasound, the proportion of grains oriented by the (101) plane decreases by 24 times. After exposure to ultrasound, the preferred orientation of the grains disappears.

The average microhardness value for A7 samples ob tained by ECAP with ultrasonic vibrations was ~ 23.4 HV, for samples without ultrasonic vibrations – ~ 19.1 HV. A slight increase in microhardness from the centre to the edges in the cross section is also observed.

DISCUSSION

The results obtained in this work showed that the use of ultrasonic vibrations in the ECAP process reduces the friction forces between the sample and the matrix, and therefore the pressing force of metal materials, changes the structure and physical and mechanical properties of the deformed metal.

A similar result was previously obtained for AA-1050 industrial aluminium³. However, in the device described

³ Donič T., Martikán M., Hadzima B. New unique ECAP system with ultrasound and backpressure. IOP Conference Series: Materials Science and Engineering, 2014, vol. 63, article number 012047. DOI: <u>10.1088/1757-899X/63/1/012047</u>.







Fig. 6. Histograms of grain sizes of zinc: **a** – initial; **b** – after 1 pass of ECAP without UV **Puc. 6.** Гистограммы размеров зерен цинка: **a** – исходного; **b** – после 1 прохода РКУП без УЗК



Fig. 7. *Histograms of grain sizes of zinc after 1 pass of ECAP with UV Puc.* 7. *Гистограммы размеров зерен цинка после 1 прохода РКУП с УЗК*



Fig. 8. Direct pole figures of zinc: a – after 1 pass of ECAP without UV; b – after 1 pass of ECAP with UV Puc. 8. Прямые полюсные фигуры цинка: a – после 1 прохода РКУП без УЗК; b – после 1 прохода РКУП с УЗК

in [12; 14; 17], the UV energy was used inefficiently. When the UV source, which was a magnetostrictive transducer with a vibration amplitude of 12 μ m, and a frequency of 20 kHz, makes contact with the moving part of the deformation unit (punch), the mass and geometric size of the vibrating system of the device change. As a result, the vibrating system goes out of resonance, the amplitude of the punch vibrations decreases sharply. As a result, the achieved reduction in the deformation force was less than 15 %.

The results of the studies described in [11] also confirmed that the use of UV reduces the pressing force, but only by 10%: 162.5 kN and 147.7 kN for conventional ECAP, and ECAP using UV, respectively. The main reason for this decrease is associated with a decrease in the contact friction force between the sample and the matrix.

In our studies, the UV effect on the sample was carried out not through the movable part of the deformation unit (punch), but through the matrix [16], i. e., the excitation of a standing ultrasonic wave occurred directly in the matrix,



Fig. 9. Grain structure of the A7 aluminium alloy: a – initial; b – after 1 pass of ECAP without UV; c – after 1 pass of ECAP with UV Puc. 9. Зеренная структура алюминиевого сплава A7: a – исходного; b – после 1 прохода РКУП без УЗК; c – после 1 прохода РКУП с УЗК



Fig. 10. Histograms of grain sizes of the A7 aluminium alloy: \mathbf{a} – initial; \mathbf{b} – after 1 pass of ECAP without UV Puc. 10. Гистограммы размеров зерен алюминиевого сплава A7: \mathbf{a} – исходного; \mathbf{b} – после 1 прохода РКУП без УЗК



Fig. 11. Histograms of grain sizes of the A7 aluminium alloy after 1 pass of ECAP with UV Puc. 11. Гистограммы размеров зерен алюминиевого сплава A7 после 1 прохода РКУП с УЗК



Fig. 12. Histograms of grain misorientation of the A7 aluminium alloy: a – initial; b – after 1 pass of ECAP without UV; c – after 1 pass of ECAP with UV Puc. 12. Гистограммы разориентации зерен алюминиевого сплава A7: a – исходного; b – после 1 прохода РКУП без УЗК; c – после 1 прохода РКУП с УЗК



Fig. 13. Direct pole figures of the A7 aluminium alloy: a – initial; b – after 1 pass of ECAP without UV; c – after 1 pass of ECAP with UV Puc. 13. Прямые полюсные фигуры алюминиевого сплава A7: a – исходного; b – после 1 прохода РКУП без УЗК; c – после 1 прохода РКУП с УЗК

and the blank during ECAP. The use of a waveguide with a matrix made as a single whole (waveguide-matrix) ensured the exclusion of the transition region between them as such. The UV energy is supplied directly to the processing zone without losses at the waveguide-matrix interface. The absence of this boundary also provides a significant increase in the service life of the device as a whole. At the same time, the deformation force is significantly reduced both by reducing the friction forces between the punch surface, and the waveguide-matrix channels, and by reducing the forces of metal deformation shear in the channel intersection zone. The degree of refinement of the blank structure increases, and the sinks of dislocations of the blank are facilitated. As for the grain misorientation of both zinc and A7 aluminium alloy, the proportion of low-angle boundaries has an advantage (Fig. 5 and 12). This is associated with the increase in the deformation degree. It is difficult to achieve a homogeneous equiaxial structure with a high content of high-angle grain boundaries in one ECAP pass, with and without ultrasonic vibrations. In the future, it is necessary to carry out ECAP with a large number of passes both with and without ultrasonic vibrations.

This work aimed at improving the ECAP technology as one of the SPD methods is relevant. The use of ultrasonic vibrations in ECAP fundamentally changes the properties of metals and alloys when forming UFG structures in them, which will make it possible to implement largely a combination of high strength and ductility. Research into the unusual combination of strength and ductility of nanostructured materials is of great fundamental and practical importance. From a fundamental point of view, these studies are of interest for investigating new deformation mechanisms. From a practical point of view, the creation of nanomaterials with high strength and ductility can sharply increase their fatigue strength, impact toughness, and reduce the brittle-viscous transition temperature, which will increase the service life, and consequently, the scope of application of many promising materials.

CONCLUSIONS

An original ECAP device, where the waveguide with the matrix are made as a single unit, has been developed.

For the first time, the ECAP method has been applied to metal materials such as zinc and A7 aluminium alloy, with longitudinal ultrasonic vibrations applied directly to the deformation zone by exciting them in the matrix-waveguide.

It has been found that the application of ultrasonic vibrations during ECAP of zinc and A7 aluminium alloy, leads to a decrease in the pressing force by 1.5 times or more due to the excitation of vibrations in the matrix itself, which served as a waveguide for longitudinal ultrasonic vibrations. Changing the pressing speed within $4\div10$ mm/s has virtually no effect on the pressing force.

The effect of ultrasonic vibrations on the ECAP process is also an effective way to change the structure of metal materials. Thus, after one pass, the microstructure of the material obtained by pressing with the application of ultrasonic vibrations differs significantly from the microstructure of samples obtained without ultrasonic vibrations: a decrease in the grain size, and a change in their crystallographic orientation, an increase in the mechanical properties of the deformed metal, and an increase in microhardness are observed.

Therefore, one can argue that ultrasonic exposure in the ECAP process allows changing significantly the force characteristics of the process, and the properties of metallic materials.

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Интенсификация процесса равноканального углового прессования с помощью ультразвуковых колебаний

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Аннотация: Представлен новый способ равноканального углового прессования (РКУП) с использованием мощных ультразвуковых колебаний (УЗК). Разработано оригинальное устройство ультразвукового РКУП, в котором волновод с матрицей выполнены как единое целое, а элементы крепления волновода расположены в узловой плоскости механических смещений стоячей волны, возбуждение которой происходит непосредственно в матрице

и заготовке в процессе прессования. Впервые предложено передавать УЗК в зону пересечения каналов матрицы, через которые перемещается заготовка, не через пуансон, а посредством возбуждения колебаний в самой матрице, т. е. матрица одновременно является волноводом продольных УЗК. Это позволило многократно повысить эффективность ультразвукового воздействия за счет снижения сил трения между поверхностью заготовки и поверхностью каналов матрицы, а также за счет снижения деформационных усилий в зоне пересечения каналов матрицы, где происходит простой сдвиг деформируемого металла. В результате по сравнению с известными способами ультразвукового РКУП, в которых снижение усилия прессования составляет менее 15 %, возбуждение УЗК непосредственно в волноводе-матрице позволило снизить усилие прессования в 1,5–4 раза. При этом существенно меняется и структура прессуемых материалов: уменьшается размер зерен и их кристаллографические ориентировки, увеличивается микротвердость. Изменения фазового состава для всех образцов, полученных РКУП с УЗК и по обычной технологии, не наблюдается.

Ключевые слова: равноканальное угловое прессование; РКУП; ультразвуковые колебания; УЗК; объемное наноструктурирование; интенсивная пластическая деформация; ИПД; волновод; матрица; деформационные усилия; зеренная структура; цинк; алюминий.

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