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ECAE Methods of Structure Modification of Materials

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Abstract Equal-channel angular extrusion (ECAE) is one of the most efficient methods of severe plastic deformation of materials. The possibilities of structure modification of materials of different nature (metals, polymers) by new varieties of ECAE, equal-channel angular hydroextrusion (ECAH) and equal-channel multiple-angular extrusion (ECMAE) are investigated. Devices and technique of ECAH and ECMAE are described. The use of this methods ensured strength σ_T = 686 MPa, elongation to failure δ = 2% and electrical conductivity at the level of 86.4% IACS for a copper wire of 0.5-mm in diameter, up to two times increase in critical current density and slightly influences the temperature of the transition to the superconducting state of NbTi alloy, up to several times increase in microhardness, rigidity and strength of polymers with conserved high level of plasticity.

Keywords Equal-Channel Multiple Angular Extrusion, Equal-Channel Angular Hydroextrusion, Technique, Metal, Polymer, Properties

1. Introduction

Equal-channel angular extrusion (ECAE) is one of the most efficient methods of severe plastic deformation (SPD) permitting accumulation of high plastic deformation with remained form and size of a sample. Traditional ECAE reaches this effect by extrusion of a billet through a matrix consisting of two channels with equal cross-sections intersecting usually at the angle of 90°[1-5]. Metal processing causes intensive fragmentation of the grains and formation of submicrocrystal or nanocrystal structure. In the case of high-molecular compounds, macromolecules become oriented along the deformation axis[6-8]. As a result of structure transformations, the set of parameters of the mentioned subjects can be significantly improved and states with unique combination of physical and mechanical characteristics can be realized. Determined by technical and commercial attraction of the method, active interest of researchers in ECAE contributed to its development. Side by side with the traditional ECAE scheme, S-shape extrusions, the process using a rotary-die, the side-extrusion process, ECAE-conform process have been suggested[5].

There are other potential variants of ECAE application, in particular, equal-channel multiple-angular extrusion (ECMAE), equal-channel angular hydroextrusion (ECAH), as well as combined methods of processing based on them. Unlike to the enlisted methods, they are not so well known. The aim of this paper is to make the results of our investigation visible and accessible to new audiences around the word. The present paper is focused on possibilities of the listed technological processes with respect to the materials of different nature (metals, polymers) for the sake of efficient application of promising methods of SPD.

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2. ECAH Method

A new version of ECAE, called equal-channel angular hydroextrusion, and the facility for ECAH were suggested in[9]. ECAH implies that a billet is extruded through the die channel by fluid pressure. The basic features of ECAH have been investigated using the facilities shown in Figure 1[9],[10].

The main part of the facilities is the high-pressure container with the operating pressure up to 1.6 GPa. In Figure 1(a), a conical die and an angular die are located in the lower part of the container channel. The conical die channel and the input segment of the angular die channel are aligned with the high pressure container channel. The conical die is placed before the angular die in order to provide billet centring during its insertion into the container channel. In addition, the fluid should be sealed at the interface between the conical die and the billet. In the case illustrated by Figure 1(b), the high-pressure container and the S-type angular die are pressed in the common belt (not shown).

The diameters of the conical die calibrating bore and the input segment of the angular die channel are equal. The diameter of the output segment of the angular die channel was made slightly larger than the diameter of the input segment for repetitive ECAH without any additional operations of billet thickening before each pass through the conical die.

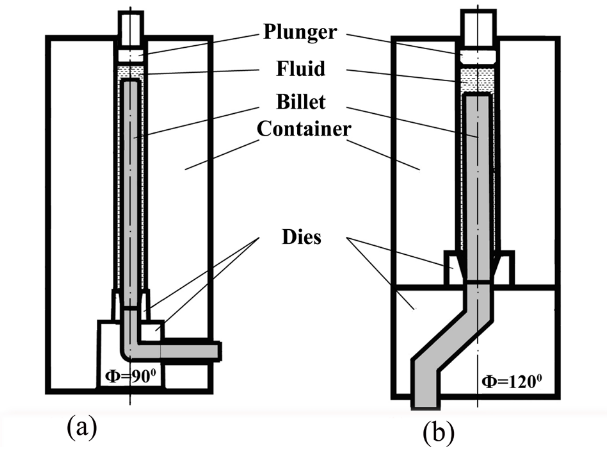

Figure 1. The scheme of the ECAH facilities with (a) one and (b) two deformation zones

The billet is fitted into an entry of the conical die to seal the fluid. Then the high-pressure unit is filled with the hydrostatic fluid and the plunger is inserted and forced into the bore by means of the press. Fluid pressure is raised until it initiated extrusion of the billet into the angular die channel input segment. Further increase of the fluid pressure forces the billet through the angular die channel and the product emerges from the output channel. Multiple ECAH is implemented by the "billet after billet" technology.

In the case of the traditional ECAE, the pressure of deformation significantly increases with a rise in the relative extent of the billet–die contact surface, at that the relative extent of this surface cannot be less of the relative length of the billet l/d. It is necessary to underline that in the case of ECAH, the relative extent of the billet–die contact surface is dependent on both the billet length l and the channel length L. Experimental values of ECAH pressure remained practically constant for $l \geq 3.5\ d$, i.e. the relative extent of the billet-die contact surface was constant as the total length of the channel segments was $L = 3.5\ d$ (Figure 2)[9]. This fact was explained by the absence of friction inside the high-pressure container because the billet was immersed in high-pressure fluid. Thereby, it has been established that ECAH makes possible effective processing of even long rods.

The relative extent of the billet-die contact surface cannot be arbitrary reduced because of required maintaining of shear deformation and straightforwardness of the extruded rod. In the case presented in Figure 1(a), the relative length of the billet part contacting with the conical die and the angular die should not usually exceed five diameters. In the case of S-type die (Figure 1(b)), a small relative extent of the billet-die contact surface was provided by using the short input and output segments of the channel and the rational minimum offset distance between them. This parameter is sensible to the reduction of the channel length but there are some design limits. Preceding analysis of strain distribution in the sample during the ECAE showed that it depends on geometrical features of the die. In particular, the minimum offset distance between the input and output channel segments should be 1.5 U (where 1 U equals the channel diameter) in order to maintain the character of the process (shearing, not bending)[11].

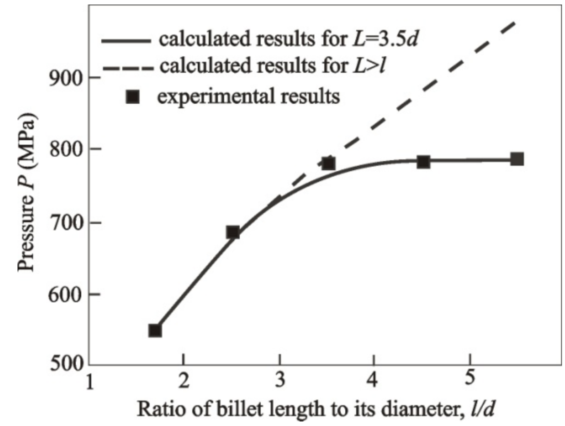

Figure 2. Calculated and experimental ECAH pressures of copper billets versus the ratio of the billet length l to its diameter d

In devices presented in Figure 1, the maximum length of the billet depends on the sizes of the high-pressure container and the plunger. The plunger length is limited by the condition of buckling failure. That is why the use of containers with channels of constant or graduated cross-section implies the billet length not exceeding l=10…15 d. Longer rod billets (l=100…150 d) can be deformed with ECAH in a device of vertical-horizontal type presented in Figure 3.

Here the fluid pressed by plunger in vertical container flows from the vertical channel to the horizontal one and extrudes the billet through the angular die. The sizes of the channels are calculated so that the volume of the fluid under high pressure was sufficient to extrude a billet of the required length. It is well known that this type of plants is used for the direct hydroextrusion of long length rods. ECAH of long length billets enhances labour productivity and the use factor of metal compared to the traditional ECAE.

It has been established that SPD by ECAH provides substantial transformation of the structure and properties of materials[9],[10]. For instance, when commercial copper (99.7%) cold deformed rods were used, the initial grain size was ranging from 6 to 40 μm with the average size of 16 μm. After three passes through the S-type die (Φ = 120), the final dominant grain size of copper was not in excess of ≈1 μm; most of the grains were of 200–600 nm in size while larger ones were also fragmented with the average size of fragments about 100–200 nm.

Figure 4 demonstrates the results of compression tests of the samples made of commercial copper and obtained with the use of ECAH. As in the case of ECAE, the increase in the degree of accumulated deformation of the billets above $\varepsilon \approx 2.3$ does not practically affect the copper yield point. Hardness of copper after 6 ECAH passes ($\varepsilon \approx 6.8$) through the die with Φ = 90° (Figure 1(a)) approached 1340 MPa.

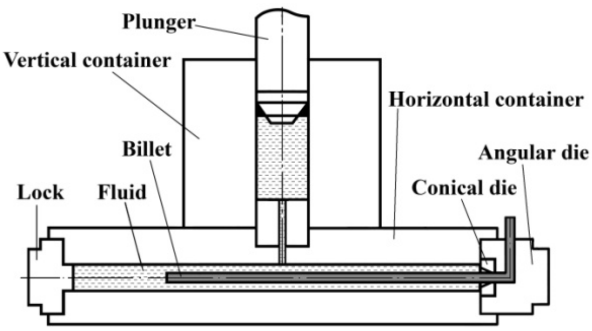

Figure 3. The scheme of the ECAH facility with connected vertical and horizontal containers

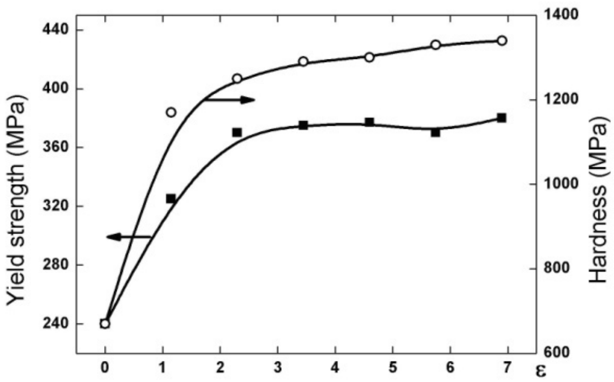

Figure 4. Effect of ECAH ($\Phi = 90°$) on copper properties

3. ECMAE Method

ECMAE is a sort of ECAE realized by pressing of a billet through the channel where several zones of shear deformation are organized by intersection of channel segments at ordered angles[3],[11-13]. ECMAE enhances labour productivity of treatment in comparison with multi-pass ECAE of billets in devices with one shear zone. At the same total strain of billets, ECMAE and multi-pass ECAE give the similar resulting changes of microstructure and hardness[3]. A significant drawback of ECMAE is the increase of the surface of contact friction of billets with the instrument and determined higher pressure of deformation.

Experiments have shown that the development of homogeneous microstructure with the grain boundaries having high angles of misorientation requires intense plastic strain during ECAE and therefore, it is necessary that the angle Φ should be equal to, or very close to 90°[14]. Therefore, Nakashima et al.[3] suppose that the construction of any multi-pass die requires that each separate plane is associated with an angle Φ close to 90°.

We have developed ECMAE device where large angles (Φ=110…160°) are used along with Φ=90°, so technological abilities of the method become expanded. The deforming block has the form of a set of thick-walled deforming disks with intersecting channels of the same diameter (Figure 5)[13]. Channels of the container and the lower deforming disk are vertically coaxial. Pair of disks with inclined channels form so-called bend. When using several pairs of deforming disks, the position of the bend can be changed by turning disks about the vertical axis.

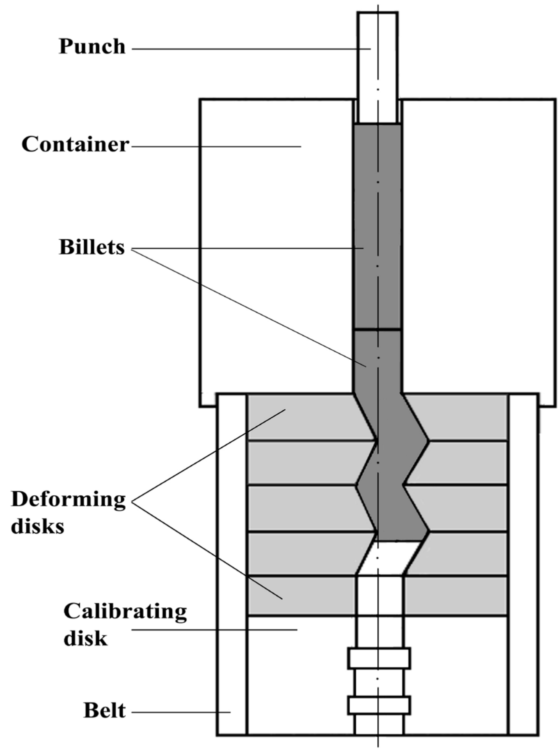

Figure 5. The scheme of the ECMAE facility

The main benefits of the developed device are as follows:

– Deformation of a billet can be realized by simple shear with varied space orientation of shear planes.

– It is sufficient to modulate deformation intensity in shear zones by means of replacement of a pair of disks. At the joint of vertical channels and inclined ones, the values of intersection angles are forcedly accepted to be $\Phi \geq 135$, in order to secure approximate coincidence of cross-section configurations of connected channels. At other deformation zones the values of intersection angles are $135 \geq \Phi \geq 90$.

– Commercial application of ECMAE ensures acceptable stability of the deforming disks due to use of well-forged billets or rolled billets of small diameter made of tool steel in the course of disks production.

Experiments showed that ECMAE at angles of Φ=110…160 can be successfully applied to processing of bimetal or multimetal billets with formation of submicrocrystal structure in all components[15]. Bimetal billets cut from a hot-pressed rod of the NbTi alloy in copper shell were extruded twelve times through the channel with three shear zones. The degree of deformation accumulated per one pass was $\varepsilon = 0.82$ and the extrusion pressure was about ≈ 800MPa. Consisting from a core made of an aluminium alloy and a copper shell, composite billets were processed in analogous way. Quality of the main part of obtained product was an evidence of homogeneous character of flow of the billet components. We have established possibility of multiple ECMAE according to "billet-after-billet" scheme without any intermediate processing of the samples between the passes.

We used a multimetal billet consisting of a set of wire elements wrapped by common external copper shell. Each wire element was the NbTi alloy core covered by a copper layer. We carried out multiple ECMAE of such billets through the channel with three shear zones at the degree of accumulated deformation per one pass ε=0.82. The formation of submicrocrystal structure of composite fibres and strengthening of the alloy were obtained.

Polymers are promising subjects of ECMAE. Solid-phase extrusion of these materials without heating of the billet does not result in noticeable improvement of their properties because of mechanical destruction of polymer chains prevailing over their orientation. The increase of the ECAE temperature of a polymer enhances the mobility of the chains and reduces probability of the destruction resulting in favorable modification of physical and mechanical characteristics. The effect of ECAE and ECMAE of semicrystalline polymers realized at the optimum temperature revealed the main advantage of the ECMAE method[16]. It is associated with the possibility of significantly higher accumulation of deformation per one pass, compared to ECAE. As a result, microhardness, rigidity and strength were increased several times with conserved high level of plasticity[16–18]. Multiple ECAE does not allow achievement of the same result because of the processes of thermally stimulated relaxation of deformation during the cooling and the succeeding heating of a polymer billet[17].

The use of a device providing change of space orientation of shear planes in the course of ECMAE (Figure 5) creates additional possibilities to control the structure and the properties of polymers. For instance, Table 1 presents the characteristics of the initial samples of high-density polyethylene (HD1000, GUADRANT) and extrudates processed with ECAE and ECMAE including varied routes of deformation: A, when the direction of simple shear did not change (ECAE, Φ=90°); C, when shear direction was changed in each deformation zone through 180° within the same plane (ECMAE); E, when the shear directions were changed through 180° and ±90° with respect to the deformation zone and being located at planes turned through ±45° around the extrusion axis and the normal to the axis (ECMAE). The temperature of extrusion was 110°C. Here ε is the degree of accumulated deformation; ρ is the density of a polymer; $\overline{H}$ is the average value of microhardness measured at cross-section; E, σ_T, ε_T are modulus of elasticity, ultimate tensile strength and deformation of destruction measured at tension.

It follows from Table 1 that in the case of polymers, not only the temperature and the value of accumulated deformation but also the routes of the process are of significant importance for the improvement of the set of the properties. E route is the most preferred among the considered ones because of provided higher level of physical and mechanical characteristics.

Table 1. Influence of ECAE and ECMAE on Properties of High Density Polyethylene

Method of deformation	ε	ρ g/cm^3)	$\overline{H}$ (MPa)	E (MPa)	σ_T (MPa)	ε_T (%)
No deformation	–	0.962	30	220	22	520
ECAE, route A	1.2	0.966	74	370	35	465
ECMAE, route C	9.1	0.970	105	690	65	448
ECMAE, route E	9.1	0.980	137	950	95	505

4. Combined Processing of Materials

4.1. Production of High-Strength and High-Conductivity Copper Wire with Using of ECAH Method

In[10], for the first time, it was demonstrated by the example of copper that combination of the ECAH, hydroextrusion (HE) and drawing (D) techniques provides higher properties of wire as compared to properties obtained without ECAH. Commercial Cu-FRTP (fire refined tough pitch copper) hot-pressed rods of 50 and 60 mm in diameter were used. When rods of 50-mm diameter were used, ECAH applied (scheme of HE+ECAH+HE+D) to the production of 0.5-mm diameter copper wire resulted in a higher level of the ultimate tensile strength (σ_T=546 MPa) and the elongation to failure (δ = 2.4%), as compared to σ_T = 474 MPa and δ=1.4% obtained without ECAH. It was also ascertained that these properties of copper wire were practically preserved for 18 months. When rods of 60-mm in diameter were used, the repetitive HE and ECAH techniques (scheme of HE+ECAH+HE+ECAH+HE+D) resulted in a higher level of copper wire strength as compared to the result obtained with single ECAH technique. In this case, σ_T = 686 MPa and elongation to failure δ = 2% was achieved for a copper wire of 0.5-mm in diameter.

The ECAH does not result in a considerable growth of resistivity. The electrical properties of the highest strength copper wire produced with ECAH and hardened wire produced without ECAH differ negligibly. The repetitive application of HE and ECAH techniques in technological chain of copper treatment in fractional mode and with the optimal degrees of deformation formed a unique complex of physical and mechanical properties, such as σ_T = 686 MPa and electrical conductivity at the level of 86.4% IACS, which is the record for the copper and copper alloys. Such strength is almost 1.5 times as much as that of copper subjected to monotonous deformation[10, 19].

The proposed combined treatment is efficient due to alternative schemes of deformation and periodic creation of favourable conditions for relaxation and dynamic recrystallization processes in the material. High plastic deformation by the simple shear scheme results in saturation of grain refinement and metal hardening. But with further processing of the billets, the method of HE provides a higher degree of material hardening, whereas ECAH implies conditions for stress relaxation, healing of micro-discontinuities and smaller exhaustion of plastic resource.

4.2. Effects of Combined Processing of the NbTi Alloy with Use of ECMAE

Bimetal hot-pressed rods of the Nb–60 at.% Ti alloy wrapped by copper shell were used as initial billets. The alloy was of two-phase composition, being β-solid solution of bcc lattice with small volume fraction (~1%) of hexagonal α-phase. Test wire samples were prepared by two schemes, i.e. the traditional one (hydroextrusion + drawing) and with the additional use of ECMAE[20].

It was established that ECMAE included to the traditional scheme of processing of a superconductor results in formation of highly homogeneous nanostructure with dispersed texture and high density of grain boundaries that facilitates nucleation of α-phase and contributes to activation of β-matrix decomposition with precipitation of the secondary hexagonal α-phase at the rate of increase in nucleation place and reduction in diffusion ways of the Ti atoms[20, 21].

As a result of combined deformation with ECMAE use, significant strengthening of the alloy (up to 30%) takes place and percent elongation of bimetal superconducting wire produced on this basis is increased by 25%[22].

Included to the technological scheme of the NbTi superconductor processing, ECMAE results in significant (up to two times) increase in critical current density and slightly influences the temperature of the transition to the superconducting state. At that, the density of the critical current of the samples obtained with using ECMAE compared to that of the samples without such processing is higher practically within the whole tested range of magnetic fields irrespective of the duration of thermal treatment[20, 21, 23].

4.3. Effects of Combined Processing of Polymers with the Use of ECMAE

In[24],[25], by the example of polyamide-6 (PA-6), potentialities of combined schemes of solid-phase extrusion were considered including extrusion through a conic die (ED) and ECMAE at varied succession. Table 2 contains data of microhardness measurements and extension tests selected from the mentioned papers. Here, ε_{ECMAE}, $\varepsilon_{ЕД}$ are deformation degrees at ECMAE and ED, correspondingly; $\overline{H}^{\perp}$, $\overline{H}^{\parallel}$ are the average values of microhardness measured at longitudinal and transversal sections of the samples; $\Delta H = 1 - \overline{H}^{\perp} / \overline{H}^{\parallel}$ is the value characterizing anisotropy of microhardness.

The data of Table 2 allowed us to make a conclusion that the use of ED+ECMAE scheme realizes the state with the highest values of microhardness and the minimal anisotropy, at the same time. At that, high values of modulus of elasticity and ultimate tensile strength are achieved with conserved high level of plasticity.

Table 2. Influence of Solid-Phase Extrusion on Properties of PA-6

Parameter	Initial	ED	ECMAE	ECMAE +ED	ED +ECMAE
ε_{ECMAE}	-	-	4.0	4.0	4.0
ε_{ED}	-	0.7	-	0.7	0.7
$\overline{H}^{\perp}$ (MPa)	80	82	137	92	150
$\overline{H}^{\parallel}$ (MPa)	82	128	164	166	178
ΔH	0.02	0.36	0.16	0.45	0.16
E (MPa)	900	1095	1345	1370	1412
σ_T (MPa)	69	92	111	115	120
ε_T (%)	148	116	126	128	132

Extrudates of PA-6 demonstrated enhanced density and crystallinity degree in comparison with the undeformed polymer. The highest value is achieved when using ED+ECMAE scheme.

5. Conclusions

The results enlisted in the paper give evidences that ECAH and ECMAE substantially advance facilities of ECAE, so they are recommended first of all for processing of polymers, hardly-deformed metal materials, bimetallic and multimetallic billets (ECMAE), long metal rods (ECAH), when ECAE is not effective. Applied to metals, polymers and composites, combination of the mentioned methods of pressure treatment with the traditional ones (hydroextrusion, drawing) allows realization of structure states with the maximum level of physical and mechanical properties.

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