https://doi.org/10.15407/ufm.23.04.629

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CURRENT TRENDS TO OBTAIN METALS AND ALLOYS WITH ULTRAFINE-GRAINED STRUCTURE

Obtaining of materials with the improved and properly balanced physical and mechanical properties remains one of the main goals of materials science. At the same time, one of the most promising ways to improve the properties of metallic materials without changing and complicating their chemical and phase compositions is to obtain ultrafine-grained states within them. Such materials are characterized by high strength and high ductility. This combination of properties is crucially important for responsible products, where the weight and size of the part is important. For example, for medical implants, which, at maintaining the strength, can be made thinner, and, if the load is exceeded, it will not be broken, damaging the surrounding tissues, but will only bend and can be subsequently replaced. Such a combination of the strength and ductility is difficult to be obtained by other methods (*e.g.*, heat treatment). However, for the bulk ultrafine-grained materials, in addition to the requirements for a grain size, there are also requirements for the isotropism and equiaxiality of grains, the misorientation boundaries of which should be predominantly high-angle. Traditional deformation technologies (such as drawing and cold rolling) are also accompanied by structure refinement. However, in general, the substructure has a cellular character with grains elongated in the direction of drawing or rolling and contains a high proportion of low-angle boundaries. This fact contributes to the anisotropy of the properties of products in the absence of a combination of properties of high strength and ductility at the same time. Over

Citation: A.B. Naizabekov, A.S. Kolesnikov, M.A. Latypova, T.D. Fedorova, and A.D. Mamitova, Current Trends to Obtain Metals and Alloys with Ultrafine-Grained Structure, *Progress in Physics of Metals*, **23**, No. 4: 629–657 (2022)

the past 2–3 decades, the technologies of severe plastic deformation (SPD) have attracted a great interest for the production of ultrafine-grained materials. However, the growth in demand is significantly limited by the high cost of manufacturing products from such materials due to the high energy and labour intensity of their production. Therefore, this article reviews and analyses contemporary technologies for production of metals and alloys with the ultrafine-grained structure, combining both high strength and ductility, by using the relatively simple and inexpensive devices, which allow spending the minimum possible amount of time in the manufacture of products. The literature overview shows the level of the process to develop technology for obtaining the ultrafine-grained structure in metals and alloys. Such the structures provide a combination of a high level of strength characteristics with high ductility that fundamentally distinguishes such the materials from the conventional ones. This is urgent for applications, where the weight, size or special exploitation properties of the part are crucially important.

Keywords: severe plastic deformation, methods, technology, ultrafine-grained structure, properties.

1. Introduction

One of the urgent tasks of materials science and mechanical engineering is to improve the physical and mechanical properties of products and semi-finished products. Traditional industrial processes are mainly aimed at shaping and manufacturability of processes. As a rule, metal products after that have a coarse-grained structure. Ultrafine-grained (UFG) materials are polycrystalline substances (usually metals and alloys) with the size of the constituent crystallites, according to the definition of most researchers, less than 1 μm [1–4]. Such a structure is transitional from the usual grain structure of most metals and alloys to a nanostructure (NS), the upper limit of which is usually denoted as 100 nm. Materials with sizes of structural units (grains) close to 1 μm are also often referred to as subultrafine-grained (SUFG) [5].

Under conditions when the size of the structural units of the material approaches the size of 100 nm, the material more and more clearly begins to exhibit unusual properties for it and combinations of properties that are unattainable in other structural states. Thus, states with grains less than one micron in size and a special state of boundaries can significantly (2–3 times) increase the strength of commercially pure metals and alloys (1.5–2 times) in combination with a sufficiently high ductility, or even the increase in ductility. Figure 1, according to R.Z. Valiev [6], shows a comparison of the mechanical properties of UFG materials (Cu and Ti) with ordinary metals as well as with metals subjected to cold rolling with various degrees of deformation (plots of Cu and Al). This figure clearly illustrates the unusual combination of high strength and ductility, which is characteristic for UFG materials and fundamentally distinguishes them from ordinary materials.

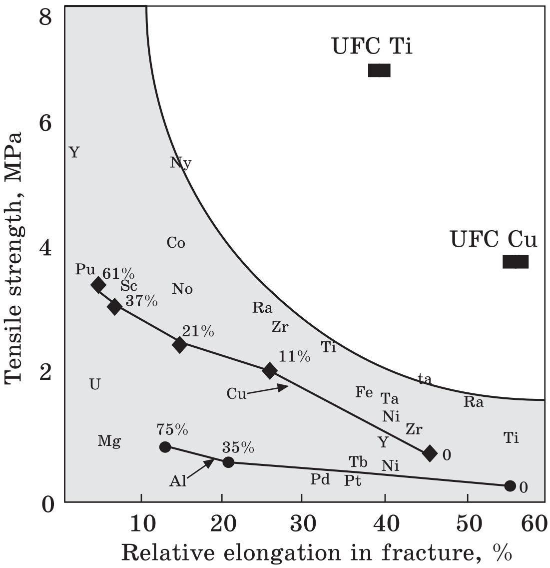


Fig. 1. Mechanical properties of conventional and UFG structural states of various metals [6]

The reason for such properties of UFG materials is due to their unusual structure, which is characterized not only by grain size, but also by a special state of grain boundaries that have a high-angle nature, a high level of internal stresses, significant distortions of the crystal lattice in the near-boundary region, and a high density of grain-boundary dislocations.

Traditional methods of thermomechanical processing using micro-alloying resources and the development of regulated thermal deformation modes of rolling, drawing also make it possible to obtain materials with a high degree of structure dispersion up to UFG. By varying the scheme, temperature, degree, and the rate of deformation, it is possible to obtain dispersed structures with small-angle grain misorientations [7]. Such structures are characterized by a high level of strength and a sufficient level of plastic characteristics immediately after production.

However, the structural instability and relatively low plasticity resource of the material during further processing have limited the scope of such materials, which has led to the need to develop technologies that can improve the plastic characteristics. Due to the small grain size, submicrocrystalline materials contain a large number of grain boundaries in their structure, which play a decisive role in the formation of their physical and mechanical properties. The concept of nonequilibrium grain boundaries is based on studies of the interaction of lattice dislocations and grain boundaries, which results in the formation of introduced grain boundary dislocations (GBDs). They form the special state of grain boundaries, which makes it possible to obtain increased physical and mechanical properties of materials [8]. This effect was studied in detail in the Refs. [9–14].

Thus, the nature and state of grain boundaries have a decisive influence on the properties of the material.

It is possible to single out the main requirements for UFG material:

- grain size less than 1 micron;
- predominantly high-angle grain boundaries;

• formation of a homogeneous structure over the entire volume of the sample;

• samples should not have destruction and mechanical damage that may occur at using of traditional methods of pressure treatment.

It is also worth noting that the fundamental difference between the hardening of metals by grinding of their grain structure from hardening by increasing the degree of work hardening (cold plastic deformation processes) lies in the higher final plasticity of UFG materials. This unique combination of properties is of great practical importance for use in mechanical engineering, since such a part will not collapse in the event of a peak load, leading to the emergency, but will only be deformed and will be changed at the next scheduled preventive repair. Therefore, obtaining the UFG state is relevant even for materials with a relatively low degree of hardening.

Important are the prospects of using the parts made of UFG materials, because of their lower metal consumption (including the proportion of alloying elements in the metal) and weight with equal mechanical properties, which reduces not only metal consumption in production, but also energy consumption during the operation of such parts, making it possible to increase the specific power of machines that have parts made of similar materials in their design.

The use of UFG materials is relevant for such areas as aerospace industry, where weight is critical, medical implants, where size is important, general mechanical engineering, where exploitation properties are important.

2. Conditions and the Main Methods for Production of the UFG Materials

It is possible to obtain ultrafine-grained materials in macrovolumes in two fundamentally different ways: by means of compaction of the mass of already obtained isolated particles of material having the appropriate size [15–18] or by severe plastic deformation (SPD) of a macroblank with grinding the size of the structural units of the material to the required size.

The first method involves first controlled condensation of crystals from the gas phase, or grinding the material in ball mills to the state of the ultrafine powder, and, then, compacting it into a finished part. This direction was studied in the works [8, 19, 20] and others.

The method has a number of disadvantages associated with the residual porosity of the finished products, geometric restrictions of the sizes of the obtained parts, and the inevitable impurities in the powder. In addition, the technological and economic part of the issue is very important, which is expressed in the need to use successively two complex

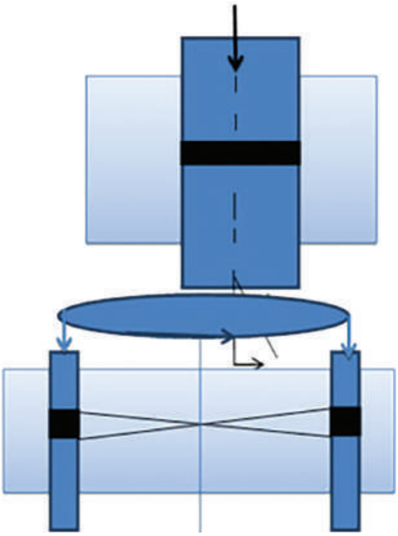


Fig. 2. High-pressure torsion scheme of the disc and ring [30]

and very energy-intensive processes on complex equipment.

The described disadvantages are absent in the methods of severe plastic deformation. Obtaining by this method equiaxed UF grains with high-angle boundaries, according to numerous studies [1, 3, 11–17, 21, 22], is possible under the following conditions [20]:

(1) achieving of high degrees of deformation for grain refinement ($e > 6$–8);

(2) formation of high hydrostatic pressure preventing the destruction of the sample and the annihilation of different types of defects [23–25] in the crystal lattice (1 GPa and higher);

(3) deformation at temperatures of about 0.4 of the melting point and below, preventing recrystallization;

(4) ensuring turbulence and nonmonotonicity of deformation, which contribute to the formation of high-angle grain boundaries.

Similar conditions can be obtained by deforming the metal by such methods as high-pressure torsion, equal-channel angular pressing, screw extrusion, all-round forging, and some others.

High pressure torsion (HPT), as an evolution of the Bridgman anvil, was one of the first methods for obtaining of bulk UFG and nanostructured samples, later developed in Refs. [1, 14, 26–28].

Samples obtained by deformation by high-pressure torsion are in the form of discs. The sample is clamped between a punch and a calliper and compressed under the applied pressure of several GPa. The calliper rotates, and the surface friction forces cause the sample to deform by shear.

The geometric shape of the sample is such that the bulk of the material is deformed under conditions of quasi-hydrostatic compression under the action of applied pressure and pressure from the outer layers of the sample. As a result, the deformable sample, despite the high degree of deformation, is not destroyed [26]. In this case, the deformation of the sample has a radial inhomogeneity, which, according to studies [26–28], can be minimized by a large number of revolutions. The high-pressure torsion method can also be used to process a workpiece in the form of a ring, according to the scheme proposed in Ref. [29]. The process diagram is shown in Fig. 2 [30].

This method has been developed and improved to increase in various ways the homogeneity of the resulting structure in the centre and on the periphery of the sample, manufacturability and expansion of the types of materials produced.

So, in the patent [31], it is proposed in the process of torsion under high pressure to carry out a cyclic change in the specific pressure by 10–20% of the current value with a frequency of 0.1–1.5 of the set rotation speed of the striker. In addition, the task is achieved by the fact that the speed of rotation of the striker in the process of deformation also changes cyclically. In addition, the task is achieved by changing the direction of rotation of the striker in the process of deformation in increments of 0.1–1.5 turns.

The technical result is achieved by the fact that the load cycling during SPD by torsion leads to a change in the concentration of vacancies in the material of the workpiece, which, in turn, affects the rate of 'crawling' of the dislocation and thereby the mechanisms of deformation and the mechanisms of formation of an ultrafine-grained structure, ensuring its uniformity. Load cycling during SPD by torsion is similar to sample rotation during equal-channel angular pressing, which leads to a change in sliding systems during processing and, due to this, provides a more uniform microstructure of the material and, consequently, an increase in physical and mechanical properties, such as tensile strength and microhardness.

Thus, the authors of Ref. [31], for further increasing the homogeneity of the sample microstructure, focus on providing condition (4), *i.e.*, nonmonotonicity and turbulence of deformation.

Patent [32] describes a method for producing products from magnetically soft amorphous alloys by high-pressure torsion with a cryogenic deformation temperature (77 K). According to the authors of Ref. [22], this approach makes it possible to improve the magnetic (hysteresis) characteristics of amorphous soft magnetic alloys by grinding the structural units of the material to 100 nm or less. Here, for ultrahigh grinding, the authors used condition (3), ensuring the lowest possible temperatures, despite the fact that the deformation of materials of this class without destruction, especially at low temperatures, is possible only under high hydrostatic pressure.

There are also a number of works aimed at technical improvement of units and parts of the installation itself [27, 33]. In addition, there are attempts to introduce elements of torsion as intensifying shear deformations into other SPD methods [1, 34].

Using the high-pressure torsion method, many researchers in many materials managed to obtain a structure with the smallest grain size (up to 20 nm), study its features, and evaluate a number of their mechanical and physical properties [26–28, 35].

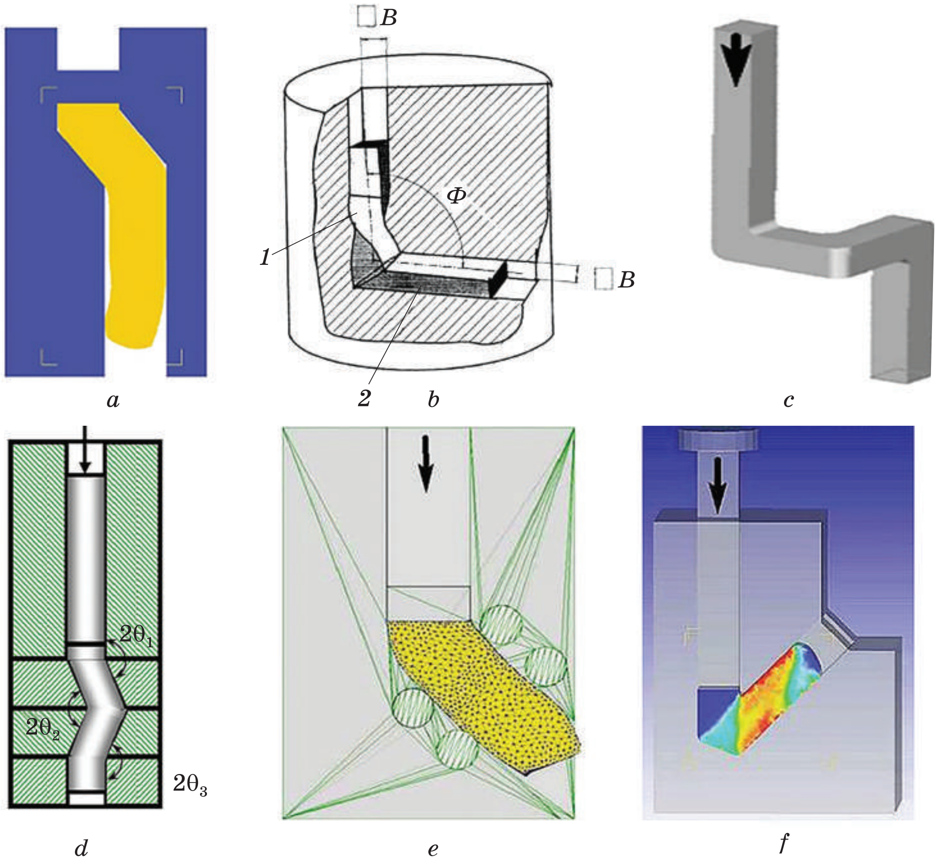


Fig. 3. Different types of equal-channel angular pressing: ECAP in a step matrix (*a*), combined method: torsion deformation and ECAP (*b*) [41], multiangle equal-channel pressing (*c, d*) [14, 45], ECAP in a roller matrix (*e*) [49], ECAP with quasi-small angles (*f*) [104]

However, the prospects for using high-pressure torsion as an industrial method have significant drawbacks, primarily due to the small dimensions of the workpieces being machined and the low tool life due to high loads on it. This fact seriously limits the practical application of this method and actually limits it to the academic environment.

The method of equal-channel angular pressing (ECAP) is devoid of many of the above disadvantages and makes it possible to obtain bulk prismatic samples with a uniform UFG structure with a grain size of 100–200 nm and does not require complex equipment. The method consists in forcing the workpiece through the angular channel of the matrix and implements a simple shift scheme.

This method was invented and patented by V. Segal in 1973 [36] for crushing the cast structure of ingots. The idea of performing a significant deformation without changing the shape of the blanks turned out to be attractive.

However, the use of ECAP for the targeted production of UFG and NS materials began only in the 1990s by R.Z. Valiev, T.G. Langdon, Z. Horita, V. Segal and others. The anatomy of ECAP and its various variations is most fully summarized and presented in Refs. [1, 9–14, 36, 37].

According to many studies (see above), to obtain a uniform UFG structure over the entire volume of the workpiece, four to eight passes are needed with an intermediate tilting of the sample [37]. This method is the most studied and is one of the most frequently mentioned in scientific articles devoted to the study of the properties of various UFG and NS materials. In addition, this method has many variations of the technical and technological performance presented in the patents of different countries. Various versions of ECAP are shown in Fig. 3.

Improvements are mainly aimed at increasing the degree of deformation and in one pass, and increasing the uniformity of the study of the structure throughout the volume. Let us dwell on the most interesting modifications.

The most attention will be paid to the stepped RCA matrix (in some sources, the CCA matrix with parallel channels) [38, 39] (Fig. 3, *a*), which makes it possible to implement two alternating deformation zones at once, provided that the input and output channels are co-directional. This scheme is also energy saving, since it allows you to realize a large degree of deformation in one pass with the same force. Due to the co-direction of the input and output channels and the relatively low pressing force, matrices of this type are most convenient for creating combined processes.

A variant of RKU-pressing with angles less than 90° has also been developed. For example, in Ref. [40], a pressing method was proposed in an equal-channel angular matrix with a channel junction angle of 45° and a special form of channel conjugation, which provides the highest degree of deformation of the workpiece per pass, with reduced force and maintaining the correct shape of the front end of the workpiece.

There is a certain tendency to combine torsion deformation with pressing and other OMD processes, which can be concluded from a review of patent sources and work [14]. It is also claimed that torsional deformation somewhat reduces the required deformation force.

In the method described in the patent [41] (Fig. 3, *b*), combined severe plastic deformation of workpieces is carried out in the following sequence: torsion deformation in a helical channel, then the workpiece passes into the matrix section, which implements equal-channel angular pressing.

When pressing the workpiece through the helical channel of the matrix, it experiences intense shear deformation mainly in the cross section, which is superimposed during the subsequent ECA pressing uniform intense volumetric shear deformation. In the process of a full cycle of pressing in the material of the blanks, the structure is refined, while the proposed sequence leads to a more isotropic structural state. This is explained by the fact that a more uniform and intense shear deformation during ECA pressing is superimposed on a less homogeneous deformation by torsion, which smoothies the resulting inhomogeneity after torsion, both in structural terms and in obtaining more uniform properties. The reverse sequence leads to the superimposition of a more inhomogeneous state on a homogeneous one, which is fixed in the workpiece after pressing, while all known methods of pressure treatment show that the values of the specified characteristics of the material in different directions of the workpiece differ significantly.

Close to the described invention, there are methods [42, 43], which combine ECA pressing and torsion deformation provided by rotation of the workpiece in a horizontal channel by means of a gear wheel.

There is also a tendency to create multi-angle equal-channel matrices (Equal channel multiangular extrusion, ECMAE) in order to implement sign-alternating deformations. The most interesting solutions are presented in the patent [44], paper [45] (Fig. 3, *c*), and in Refs. [14, 46] (Fig. 3, *d*). These methods increase the degree and uniformity of deformation in one pass, but require significantly more effort and are not suitable for all materials and temperature-speed conditions of deformation.

Pressing in them is accompanied by a change in the shear direction in the next zone, including the opposite one, from the point of view of the nature of structure formation, this is important, since it means a change in the sign of deformation. In each zone, the main deformation axes undergo rotation, and when the material passes through each subsequent zone, the direction of rotation of the axes is reversed. This contributes to the effective fragmentation of structural components and the formation of isotropy of the structure in each cycle of deformation. With each subsequent focus and cycle of deformation, because of the appearance of a large number of directions, along which an elementary shift can occur in the microvolumes of the deformable material, the microdistortions of the crystal lattice are redistributed between individual microvolumes of the plastically deformable material. This also contributes to the intensive process of fragmentation of mosaic blocks and individual crystallites, as a result of which a finely dispersed structure is observed [14]. There are also several works [47, 48] showing the combination of ECAP and cryogenic cooling, which helps to further refine the structure and obtain higher mechanical properties.

With these methods, the intensity of hardening is less than with traditional monotonic plastic deformation (*e.g.*, hydroextrusion), due to

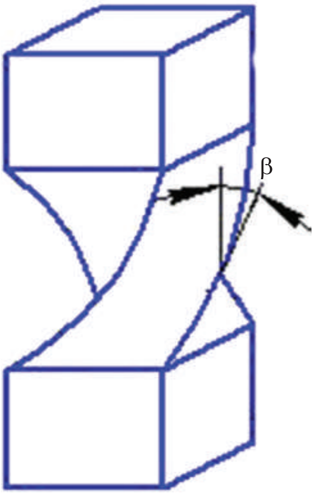


Fig. 4. Twist extrusion [51]

some relaxation of microstresses as a result of more intense fragmentation of the structure during intermittent alternating flow of the deformation process.

The above-described physical features of the deformation in multiangular ECA matrices can also be applied to the description of the processes occurring in the metal during pressing in a stepped matrix.

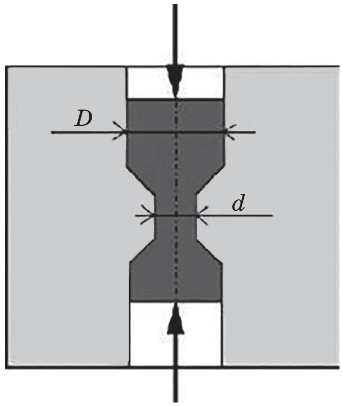
With all the described advantages of multiangle ECA pressing in the formation of a homogeneous UFG and nanostructure, it also has disadvantages associated with a large force or the need for a large number of passes, or is not suitable for all materials and temperature-speed deformation conditions.

The construction of a *T*-shaped matrix, described in [14] (Fig. 3, *d*), creates too uneven deformation over the volume of the workpiece and requires a large number of cycles to obtain an isotropic structure.

A common disadvantage of the ECAP process is the need for a large deformation force when processing massive bodies and low tool life. Elimination of these shortcomings is possible by pressing blanks in a roller matrix. The roller die known from [49] makes it possible to obtain a UFG structure with much less effort, but this requires a larger number of passes. Nevertheless, relatively small efforts make it possible to recommend such designs for the creation of combined installations.

The described methods and devices are the most effective and popular for obtaining UFG and nanostructures. However, there are other methods, which also make it possible to obtain UFG products by severe plastic deformation.

Twist extrusion (TE) is shown in Fig. 4 [50–52]. The essence of TE is that a prismatic sample is pressed through a matrix with a channel containing two prismatic sections separated by a helical section. During processing, the material undergoes intense shear, maintaining the identity of the initial and final sections of the sample. The latter circumstance makes it possible to carry out its multiple extrusion with the aim of accumulating a large deformation, which leads to a change in the structure and properties of the material, which makes this process similar to ECA. The most important distinguishing feature of TE from other SPD methods is a powerful vortex flow in the deformation zone, which provides intensive mixing of the deformed material and creates the prerequisites for the formation of unusual structures and the formation of new phases. The efficiency of processing by the TE method is currently



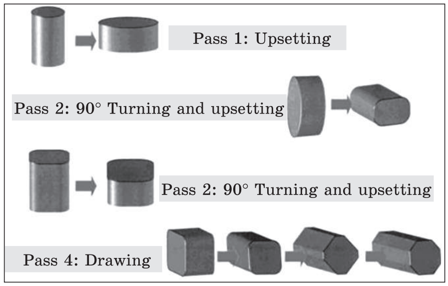


Fig. 5. Cyclic extrusion-compression [53]

◄ *Fig. 6.* Multiple isothermal forging [55]

shown on some metals and alloys: secondary Al alloys (AK9, AK5M2, AV87), Ti alloys (VT1-0, VT-6, VT-22, VT3-1), Cu–P alloys, Al–Mg–Sc alloys, and others.

Another way to deform workpieces, which makes it possible to implement significant shear deformations, is invented by M. Richert (Krakow University, Poland) [53] cyclic extrusion–compression (CEC, 'hourglass') shown in Fig. 5.

This method consists in repeated deformation of the metal by extrusion or pressing through a narrowed hole in the tooling, the longitudinal section of which has the shape of an hourglass. If you apply an extrusion force to the upset part of the workpiece on the other side, the process will be repeated. Such a scheme of deformation in a multicycle mode makes it possible to obtain an UFG structure in workpieces with a diameter of 15 mm and a length of up to 100 mm.

The method was first applied to the accumulation of large plastic deformations in pure aluminium at room temperature. Experiments show that stress saturation occurs after 4–5 extrusion cycles. To date, the method has been applied only to the deformation of a limited number of materials [53–55].

Multiple isothermal forging can also be used to form UFGs and nanostructures in bulk samples (Fig. 6). This method proposed in Ref. [55] was improved by Gosh. The process of all-round forging is usually accompanied by dynamic recrystallization. Closed forging is a modification of the all-round forging process [56].

The scheme of comprehensive forging is based on the use of repeated repetition of free forging operations: upsetting–broaching with a change in the axis of the applied deforming force. The uniformity of deformation in this technological scheme is lower compared to ECA pressing or torsion. However, this method makes it possible to obtain the UFG state in fairly brittle materials, since processing is started at elevated temperatures and small specific loads on the tool are provided. The choice of appropriate temperature-rate deformation conditions makes it possible to obtain very fine grains with a size of about 100–500 nm in pure metals and less than 100 nm in alloys. Typically, this approach is implemented at temperatures of plastic deformation in the range (0.3–0.6) T_{melting}.

In addition, to intensify the deformation of the forging process, special-shaped dies are used, after broaching in which it is possible to obtain a more uniform structure in less time than with all-round forging [57–60].

The disadvantages of forging processes are their high labour and energy intensity, with a nonuniform distribution of deformation over the volume of the workpiece.

There are attempts to achieve an intensive and uniform study of the structure by rolling, by using a special form of rolls or calibres in them. The scheme of hot rolling, the number of passes, the temperature of deformation, especially the temperature of its end, determine the course of the processes of decomposition of austenite together with the influence of the cooling rate. Traditional forms of rolling, developed and tested over a number of years, optimize the deformation process in terms of the forces of the stands, the stability of the roll, and the productivity of the mill. The control of the rolling end temperature and cooling rate, as well as the microalloying of steels, makes it possible to obtain fine grains with a large number of dislocations in the grain body, fixed by impurity atoms [14]. So, in Ukraine, E.G. Pashinska patented a scheme for hot rolling of wire rod with the implementation of intense shear [61], which makes it possible to obtain an equiaxed structure with a grain size of up to 2 μm [62].

For larger profiles, to use the 'rhombus–square' gauge system with an off-diagonal location of the rhombic gauge relative to the longitudinal axis of the rolls in such a way that two opposite sides of the rhombus are parallel to the axis of the rolls. The other two sides of the rhombus are at an angle to the axis of the rolls. As a result, a workpiece of square or rectangular cross section, which specified in such a rhombic calibre, receives, along with high-altitude reduction, a transverse shear, which ensures intensive plastic processing of the structure [63].

There are also some other methods of intensifying sectional and even sheet rolling, but none of these methods is able to provide a structure in the metal that is close in quality to the structure obtained by ECAP.

The only exception is the type of cross-helical rolling (Cross-rolling or, more rarely, Helical rolling, or, even more rarely, Screw rolling), singled out by its authors as a separate method called 'Radial shear rolling' (RSP) and patented [64]. The difference from conventional helical rolling, which used, *e.g.*, in the pipe piercing [65], is that a solid bar rolled according to a three-roll scheme with large feed angles. The scheme of the process and its features is shown in Fig. 7.

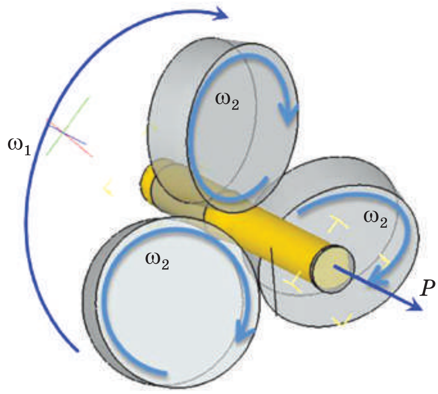


Fig. 7. Radial shear rolling [105]

The deformation zone formed by three drive rolls turned at an angle to the workpiece axis and at an angle to each other. In contrast to traditional pipe screw rolling mills, conditions are created in RSP mills not for loosening the central zone, but, on the contrary, for compaction and intensive deformation processing of metal in the entire volume of rolled products [67].

During helical rolling, in the deformation zone, a stress-state scheme is realized, which is close to all-round compression with large shear deformations. The most intense shear deformations localized in the zone of intersection of the metal slip lines, namely, the annular cross-sectional zone characteristic of the three-roll scheme. In other words, conditions created in the deformation zone that satisfy those listed at the beginning of the subsection and are optimal for the formation of UFG structures.

The main feature of helical rolling is nonmonotonicity and turbulence of deformation, as well as differences in plastic flow and elaboration of the structure of different zones of the workpiece, due to the trajectory-velocity features of the process.

In the helical rolling method [64], the reduction is carried out with slowing down the movement of the metal, reducing the length of the helical trajectories in the outer layer of the workpiece, while accelerating the movement of the metal, increasing the length of the trajectories in the inner layer of the workpiece. The thickness of the outer layer in the rolled workpiece was of 0.3–0.7 of its radius, and the ratio of linear compression strains along helical trajectories on the workpiece surface and tension along its axis was of 0.1–0.5 [66].

In the outer layer, each small trajectory-oriented element is subjected to compressive strain along the radius of the workpiece, compressive strain in the outflow direction (along the helical trajectory) and, accordingly, tensile strain across the helical trajectory. At the same time, it is important to accentuate that there is a constant gradient of velocities and flow directions along the radius, which still adds additional shear elements to the overall complex picture of the stress–strain state. Elements of the structure of the metal subjected to an expanding flow with two-way settlement (along the trajectory and along the radius) take the form of isotropic isolated particles of high dispersion (in detail, see, for example, reference [64]).

The speed of particles in the axial fibre and its length, as in the case of longitudinal rolling, increase in proportion to the elongation coefficient. The cross section of the central current tubes decreases. The elaboration of the metal structure acts like longitudinal rolling in calibres with multilateral reduction or pressing. Elements of the structural structure stretched and thinned with the formation of a characteristic structural banding.

In general, within the bulk of the workpiece, the helicoidal outflow of metal with deceleration of the surface layers and acceleration of the central ones creates the effect of volumetric macroshear, which also contributes to the deepening of the metal structure.

By screw rolling with the above features, it is possible to roll a wide range of materials up to complexly alloyed hard-to-deform special alloys of ferrous and nonferrous metals [64, 67–69]. In Ref. [67], by combining section rolling with rolling on a mill described in the patent [70], it was possible to obtain a homogeneous globular ultrafine-grained structure with an average grain size of 150 nm from commercial grade titanium with grain sizes from 50 nm to 500 nm.

Improvement of the same method allowed the authors of Ref. [71] to obtain long bars of UFG titanium 8 mm in diameter with an average grain size of 90 nm; in this case, grains from 30 nm to 300 nm are recorded, and the fraction of grains with a size of less than 100 nm was 64%.

Having become acquainted with the main methods for obtaining UFG materials with nonequilibrium high-angle grain boundaries, we can conclude that the high-pressure torsion method satisfies all the conditions that provide the required structure most completely. However, it is the least suitable for any practical application, since it allows processing samples of only small sizes (discs of the order of 10 mm in diameter and 0.5 mm thick), which is not enough even for laboratory studies. The method is very energy-intensive and unsuitable for modernization.

Equal-channel angular pressing in its various forms is the most compromise method for obtaining bulk products with a high quality, homogeneous UFG structure, but also has its own basic limitations. They are attributed to the inability to produce long products, the need to perform a large number of deformation cycles, strong tool wear, insufficient manufacturability, and, at still high energy costs for industrial production, which significantly reduces the productivity of the process and its commercial attractiveness. In its current form, the industrial implementation of ECAP is suitable at best only for small-scale subsidized production of small products with properties, which cannot obtained in other ways (medical implants, *etc*.).

Comprehensive forging and forging in special dies that implement SPD to some extent allows you to bypass the restrictions on the size of manufactured products, but at the cost of high energy costs compared to ECAP, and in addition, the production of UFG products by forging is not very suitable for creating high-performance, cost-effective production. The production niche of forging is the production of massive piece UMP products that cannot obtained in other ways.

Either different types of shear rolling do not provide a sufficient level of isotropy of the structure, or the resulting structure does not fully comply with the requirements set forth at the beginning of the subsection, as a rule, the special state of the grain boundaries shown in Fig. 1 is not provided throughout the entire volume. In particular, helical rolling, forming an UFG structure at the periphery, does not provide sufficient elaboration of the axial zone of the workpiece.

Having critically studied the materials on the listed methods for producing UFG products, we can make an unambiguous conclusion that in the current state, none of the known methods, due to high-energy costs, is ready for economically justified widespread industrial use. The problem can be solved only by ensuring the continuity of the process.

The solution to this problem, apparently, lies in the field of combining processes.

3. Combined SPD Processes

It is possible to reduce radically the energy consumption by building a combined process that allows you to obtain long-length workpieces in a continuous way.

As a basis for creating combined SPD methods, a special place is occupied by the Conform continuous pressing method, as the most studied in scientific works and already having some industrial application, and it seems to be especially promising in nonferrous metallurgy [72–74]. The scheme of the combined process ECAP–Conform is shown in Fig. 8.

The essence of the proposed method of deformation is as follows. The workpiece preheated to the temperature of the beginning of deformation is fed to the rolling rolls, which, due to the forces of contact friction, capture it into the throat of the rolls, and at the exit from it, they push it through the channels of an equal-channel stepped die. After the billet has completely left the gap of the rolls, the next billet is fed to them, which, having passed through the rolls and getting into the matrix, pushes the previously deformed billet out of the matrix. That is, in this case, the process of pressing workpieces in an equal-channel stepped die realized through the use contact-friction forces, which occur on the contact surface of the metal with rotating rolls. The disadvantage of this process is that the workpiece is in a heated state, which complicates the technology and requires constant temperature control that is not quite suitable for mass-producing.

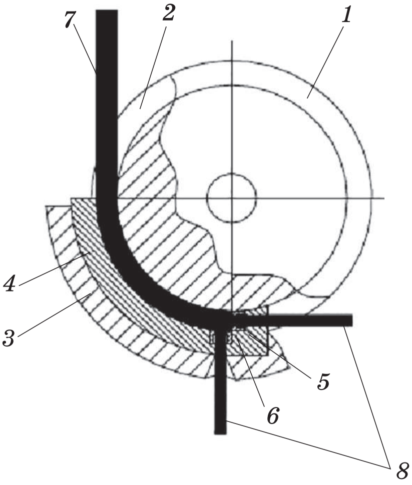


Fig. 8. Scheme of Conform continuous pressing: *1* — driving wheel; *2* — groove; *3* — ring groove; *4* — shoe; *5* — matrices; *6* — ring groove; *7* — billet; *8* — product [72]

The deforming force formed due to the active friction forces acting on the working surface of the rotor. In this case, the working channel of pressing formed by the rotor and the working surfaces of the clamp and stop.

Using this method allows you to remove the limitation on the length of the pressed billet. However, to obtain a uniform UFG structure, it is required to carry out several pressing cycles, and therefore, new problems arise related to maintaining the temperature regime for a long workpiece.

The successful application of the combined ECAP–Conform process for obtaining an UFG structure in a metal was made by G. Raab [72], and, since the autumn of 2014, a combined the Conform–ECAP unit, previously created on the basis of the Conform 315i serial machine [75]. The facility produces UMP titanium for medical implants and some other applications of Ti-based alloys [76, 77]. This is a real field of application of UFG materials, as evidenced, for example, by a US patent [78] received back in 2002.

Similar devices described in articles and patents by scientists of the Siberian Federal University under the guidance of Prof. S. Sidelnikova [79–84]. There are a number of combined processes developed and patented by them based on Conform, such as 'rolling–pressing' [81], 'casting–rolling–pressing' of solid [82, 83] and hollow profiles [84]. In addition, from the patent of O. Lekhov [85] and work [92], the combined process of 'casting–rolling of a bimetallic bar' is known. The general essence of the methods is that the melt is poured directly into the mould rolls, crystallized in the form of a rectangular billet, which deformed using the same rolls, and then squeezed out through the calibrating hole of the matrix.

For the first time, such a method of manufacturing press products patented in England under the name Castex. According to this method, the leading companies in this direction, Babcock Wire Equipment and Holton Machinery LTD, manufacture and replicate lines for continuous casting and pressing of nonferrous metals based on Conform installations.

Almost all of the listed methods (except for ECAP–Conform), despite their advantages and innovative potential, are not aimed at obtaining an UFG structure and their use is limited only to nonferrous metals.

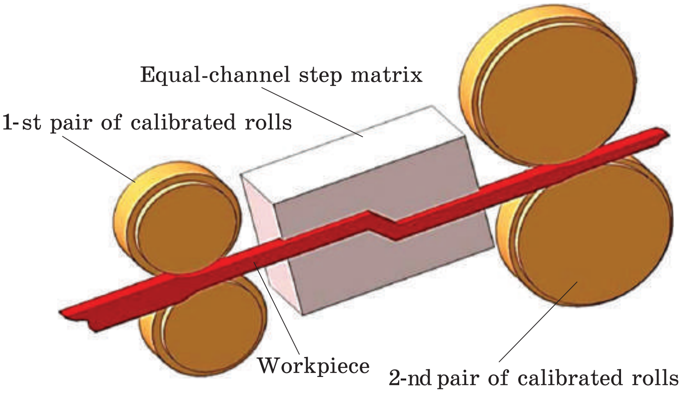


Fig. 9. The combined method of rolling and ECA pressing [57]

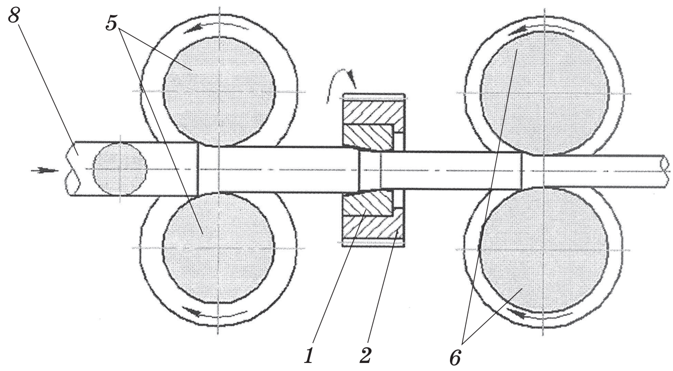


Fig. 10. Combined process 'rolling–torsion–rolling': *1* — matrix; *2* — gear wheel; *3* — calibrating rolls; *4* — blank; *5* — feed rolls [90]

For the purposeful formation of the UFG structure of a wide range of materials, a continuous combined method of 'rolling–pressing' invented using a stepped RCA matrix [87–89] (method is illustrated in Fig. 9).

Theoretical study and substantiation of the first process was carried out in Ref. [89], an experimental study of the rolling–pressing of aluminium in Ref. [87]. Already after three cycles of continuous pressing, it was possible to achieve grain refinement from 180 microns to 3.5 microns. The method is aimed at energy-saving obtaining of the UFG structure; however, it does not allow develop the required level of the degree of deformation in one pass, and the shape of the resulting product is limited to a rectangular section.

It is possible to increase the intensity and nonmonotonicity of deformation by including torsion elements in the combination scheme, as a device claimed in the patent [90] and is shown in Fig. 10.

The workpiece 4 with the help of feed rolls 5 enters the horizontal channel of the matrix 1. By means of the gear wheel 2, the workpiece deformed by torsion in the centre located between the feed rolls and the torsion unit. The degree of deformation depends on the speed of rotation of the gear wheel and the speed of rotation of the feed 5 and calibrating

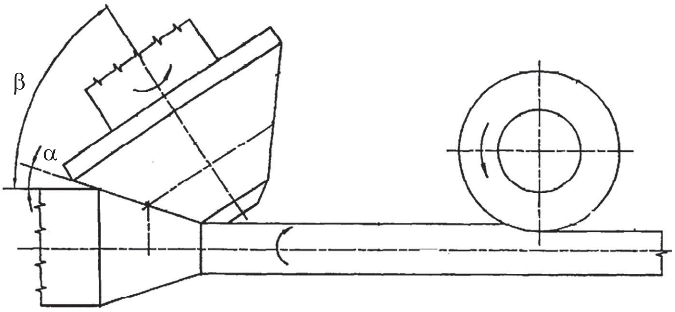


Fig. 11. Combined processes with continuous ECAP: rolling–pressing [92]

rolls 3. The presence of comb-like protrusions in the conical part of the horizontal channel eliminates the possibility of turning the workpiece inside the matrix. Next, the workpiece falls on the calibrating rolls 3 for the final fine-tuning of the shape and size to the desired. In addition, when processing with gauge rolls, the surface layers of the workpiece are hardened (rolling deformation) and the surface roughness reduced. By changing the speed of rotation of the calibrating rolls, the pressure (backpressure) required to achieve higher degrees of deformation can changed smoothly without stopping the machining process. Torsional deformation and rolling according to the present invention provide severe plastic deformation in the material of the workpiece, which leads to an improvement in mechanical properties and an increase in the quality of the workpieces machined.

However, to create a uniform UFG structure, such a deformation may not be enough; therefore, methods have been developed for combining helical rolling and section rolling in various ways with the implementation of intense twisting of the workpiece [91–93]. Thus, in addition to the intense deformation that occurs during helical rolling, torsion deformation and a slight reduction in the bar stand added.

In the patent [91], the 'screw rolling–section rolling' method, which, according to the authors, makes it possible to obtain a more uniform finely dispersed structure by twisting a billet heavily deformed after helical rolling in front of the bar stand calibre.

The proposed method was developed and tested at VNIIMetMash on a pilot plant consisting of an induction furnace, a screw rolling stand, two longitudinal rolling stands and shears. The method carried out as follows. The heated billet fed to the helical rolling stand, captured by the work rolls and reduced to the required size. Then, the front end of the roll captured by the rolls of the longitudinal rolling stand and torsion deformation occurs in the inter-stand gap.

Then, in order to stabilize the process and more accurately control the extra-cage shear deformations, the method improved by the authors and claimed in a patent [92]. The improvement consisted in the fact that

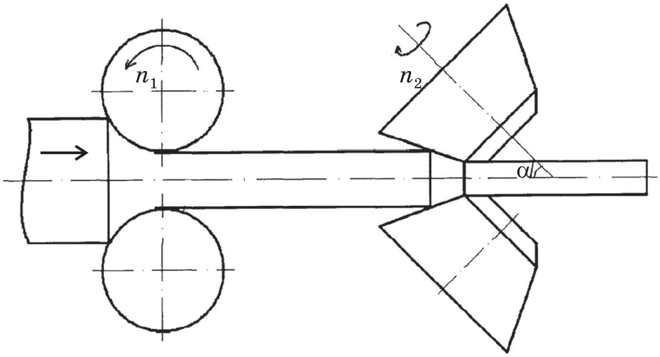


Fig. 12. Combined processes with continuous ECAP: casting–rolling–ECAP [93]

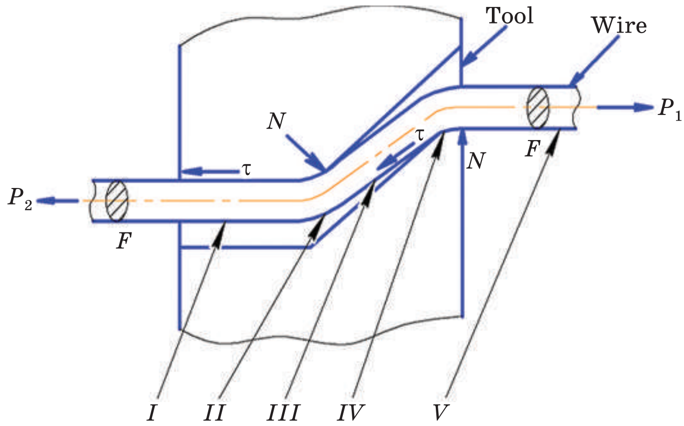


Fig. 13. ECA-broaching scheme: F — the cross-sectional area of the processed billet; P_1 — broach force; P_2 — antitension force; N — force of normal pressure; τ — friction force [94]

the out-of-roll deformation of the billet by twisting is carried out in the helical rolling stand, stopping the rotation of the billet at the exit from the rolls by roller guidance, and then the bar is captured by the rolls of the bar stand. This method is shown in Fig. 11. There is also a variant of the reverse combination of longitudinal rolling and helical rolling, declared in the patent [93]. The method is shown in Fig. 12.

Like the previous one, this method includes out-of-roll deformation of the workpiece by twisting in the gap between adjacent stands of helical and longitudinal rolling. The deformation, accompanied by a change in the physical and mechanical properties of the metal and the formation of a fine-grained structure, is ensured due the processing started by longitudinal rolling. After that, the cross-helical rolling is carried out with circular reduction and rotation of the workpiece; acting on the workpiece with compressive stress due to the backwater force achieved due to the difference in metal outflow velocities in the process of longitudinal and helical rolling.

The value of the support force depends on the mechanical properties of the material and the degree of shear deformation and selected em-

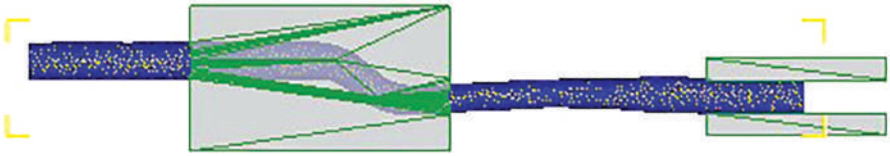


Fig. 14. Combined processes with continuous ECAP: pressing–drawing [96]

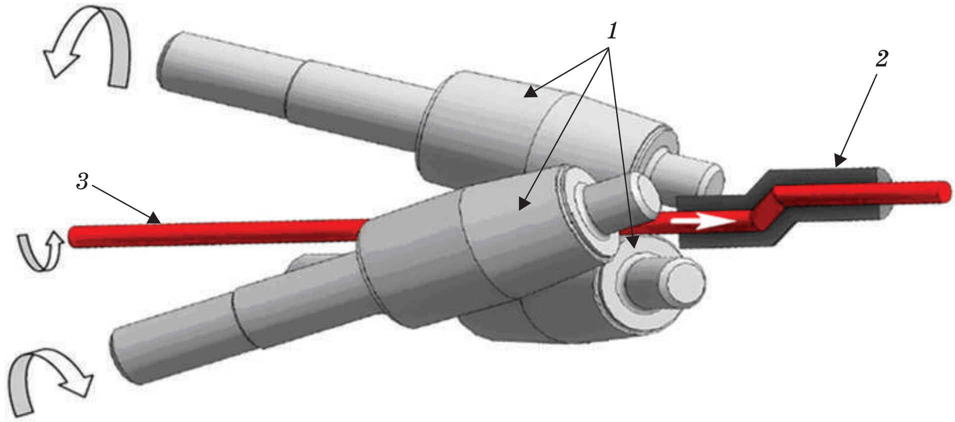


Fig. 15. The combined process 'screw rolling–pressing': *1* — billet; *2* — roll node; *3* — equal-channel step matrix [100]

pirically. The backwater force, according to the authors of the invention, can be achieved up to 10 GPa [93].

It should also be noted the method of equal-channel angular free broaching (ECA-Broach), which leads to the formation of an UFG structure in long billets of round cross section (Fig. 13). This process consists in repeatedly pulling the wire through special dies, the design of which provides for two channels intersecting at an angle [94]. In the work [95], the influence of heat treatment and the design of matrices on the mechanical properties of the obtained samples studied, it also shown that in order to obtain an UFG structure, it is necessary to carry out from 4 to 10 processing cycles, which is the main disadvantage of this process. It should be noted that during ECA broaching, an inhomogeneous UFG structure is observed in the processed workpieces even after eight passes.

Scientists of the Karaganda State Industrial University have developed a new scheme of the combined process 'pressing–drawing' using an equal-channel stepped matrix and a sizing tool (Fig. 14) [96, 97]. The difference between this scheme and drawing is that, before drawing, the workpiece passes through an equal-channel stepped die. As shown in Ref. [97], to obtain an UFG structure in Al and Cu samples, it is necessary to carry out at least three processing cycles, which increases the complexity of the process. This method being a direct prototype of the ECAP will inherit all its shortcomings.

At the same university, another method of billet deformation was proposed, namely, the combined helical rolling–pressing process using an equal-channel stepped die (Fig. 15) [98, 99], which makes it possible to obtain round cross-section billets, the metal of which will have an ultrafine-grained structure.

The essence of the proposed method of deformation is as follows. The workpiece preheated to the temperature of the beginning of deformation fed to the rolling rolls, which, due to the forces of contact friction, capture it into the rolls, and at the exit from them, they pushed through the channels of an equal-channel stepped die. After the billet is completely out of the rolls, the next billet is fed to them, which, having passed through the rolls and getting into the matrix, pushes the previously deformed billet out of the matrix. In this case, the process of pressing workpieces in an equal-channel stepped die, as well as in the previously given combined process, is realized through the use of contact friction forces arising on the contact surface of the metal with rotating rolls [98].

However, this method also has disadvantages, which consist in limiting the length of the deformable workpiece, which cannot exceed 5–7 m.

Concluding analysis of the combined methods, we have to mention other methods that are not included in the detailed review. There are known combined methods of continuous pressing by the method of Extrolling [100], Linex [101], and some others [102, 103], which are either too highly specialized or do not provide the formation of a structure of the desired quality.

Thus, the analysis of existing methods for producing aluminium wire rod showed that at present, there are promising methods for processing materials, but each method has both advantages and disadvantages. Therefore, the combination of the advantages of traditional methods with SPD methods is a promising idea for the development of technology for obtaining materials with an ultrafine-grained structure with improved mechanical and operational properties.

4. Conclusions

Of all the methods of obtaining ultrafine-grained materials considered in this article, the most promising is intense plastic deformation, due to the possibility of obtaining isotropic products of a larger volume, without internal discontinuities. However, for successful structure refinement by SPD methods, special conditions are required, such as the creation of large, at the same time, nonmonotonic deformations under high hydrostatic pressure and low temperatures.

Many SPD methods either do not provide the formation of an isotropic structure over the entire cross section, or have significant limitations on the size of the resulting product, which significantly limits the scope. In addition, a significant drawback of all known SPD methods, which limits their industrial applications, is their high energy and labour intensity. To circumvent and solve these problems, attempts are periodically observed to modify existing processes (*e.g.*, the introduction of torsion elements into the processes of pressing or rolling).

However, having critically studied the materials on the listed methods for producing UFG products, we can make an unambiguous conclusion that in the current state, none of the known methods, due to high-energy costs, is ready for economically justified widespread industrial use. The problem can be solved only by ensuring the continuity of the process, *i.e.*, by building a combined SPD process that allows you to obtain long workpieces in a continuous way. The use of the few known combined processes, as a rule, is limited to nonferrous metals and not aimed at obtaining an UFG structure, since it requires a large number of passes, which leads to a decrease in plastic properties. Therefore, further development and research of such methods of SPD is very important.

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Received 02.06.2022;
in final version, 13.10.2022

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СУЧАСНІ ТЕНДЕНЦІЇ ОДЕРЖАННЯ МЕТАЛІВ І СТОПІВ З УЛЬТРАДРІБНОЗЕРНИСТОЮ СТРУКТУРОЮ

Одержання матеріалів з підвищеними та правильно збалансованими фізико-механічними властивостями залишається однією з головних цілей матеріалознавства. Одним з найбільш перспективних способів поліпшити властивості металевих матеріалів, не вдаючись до зміни та ускладнення їхнього хімічного та фазового складів, є одержання в них ультрадрібнозернистих станів. Для таких матеріалів характерною є висока міцність за високої пластичности. Таке поєднання властивостей критично важливе для відповідальних виробів, де значущими є вага та розмір деталі. Наприклад, для медичних імплантатів, які, за збереження міцности, можна зробити тоншими, і в разі перевищення навантаження імплантат не зруйнується, ушкоджуючи навколишні тканини, а лише погнеться та може бути згодом замінений. Подібне поєднання міцности та пластичности складно одержати іншими способами (наприклад термообробленням). Проте для об'ємних ультрадрібнозернистих матеріалів, окрім вимог до розміру зерен, ще пред'являються вимоги до ізотропности та рівновісности зерен, межі розорієнтування яких мають бути переважно висококутові. Традиційні технології деформування (такі як волочіння та холодне вальцювання) також супроводжуються подрібненням структури. Одначе в основному субструктура має пористий характер із зернами, подовженими у напрямку волочіння чи вальцювання, та також містить високу частку малокутових меж. Цей факт сприяє анізотропії властивостей виробів за відсутности поєднання властивостей високої міцности та пластичности одночасно. За останніх 2–3 десятиліття великий інтерес для виробництва ультрадрібнозернистих матеріалів привертають технології інтенсивної пластичної деформації (ІПД). Втім зростання попиту істотно обмежується високою вартістю виробництва виробів із таких матеріалів, зумовленою високою енерго- та трудомісткістю їхнього виробництва. Тому в даній статті оглянуто та проаналізовано сучасні технології одержання металів і стопів з ультрадрібнозернистою структурою, що поєднують одночасно високу міцність і пластичність, в умовах використання відносно простих і недорогих пристроїв, які уможливлюють витрату мінімального часу для виробництва. Літературний огляд показує, на якому рівні знаходиться процес розроблення технологій для одержання ультрадрібнозернистої структури в металах і стопах. Такі структури забезпечують поєднання високого рівня характеристик міцности з високою пластичністю, що принципово відрізняє такі матеріали від звичайних. Це актуально для сфер застосування, де критично важливі вага та розмір або особливими є експлуатаційні властивості деталі.

Ключові слова: інтенсивна пластична деформація, методи, технологія, ультрадрібнозерниста структура, властивості.