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V.Z. Spuskanyuk[1], O.A. Davydenko[1], O.M. Gangalo[1], L.F. Sennikova[1],
M.A. Tikhonovsky[2], V. Spiridonov[3]

ATTAINING A RECORD LEVEL OF COPPER-WIRE PROPERTIES BY USING SPD METHODS

[1]Donetsk Institute for Physics and Engineering, National Academy of Sciences of Ukraine
72 R. Luxemburg St., Donetsk, 83114, Ukraine

[2]National Science Center «Kharkov Institute of Physics and Technology»,
National Academy of Sciences of Ukraine
1 Akademicheskaya St., Kharkov, 61108, Ukraine

[3]OJSC Artyomovsk non-ferrous metals processing plant
42 Kirov St., Artyomovsk, 84500, Ukraine

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A new approach is proposed to control the processes of plastic deformation of materials by the methods of severe plastic deformation (SPD). High strength and plasticity have been attained for the processed copper billets after multiple repetitions of angular hydroextrusion (ECAH) and hydroextrusion (HE) methods and with ECAH and HE implementation in the fractional mode. The combined severe plastic deformation technology including ECAH, HE and drawing (D) provided the ultimate tensile strength $\sigma = 686$ *MPa, the elongation to failure* $\delta = 2\%$ *and the electrical conductivity at a level of 86.4% IACS in the 0.5 mm diameter copper wire. Such a processing is efficient due to the alternating schemes of deformation and periodic creation of favorable conditions for relaxation and dynamic recrystallization processes in the material. An important condition for the attaining of the maximum processing effect is the fractional mode and the optimum degree of deformation by the both methods.*

Keywords: equal-channel angular hydroextrusion, direct hydroextrusion, drawing, copper, physical and mechanical properties, nanocrystalline structure

1. Introduction

As demonstrated by Segal *et al.* [1], the equal-channel angular extrusion (ECAE) is a promising method for the formation of ultrafine grained (UFG) microstructures in metallic materials. Kulczyk *et al.* [2] have recently shown by the examples of copper and nickel that the combined ECAE + HE deformation greatly increases the homogeneity of the microstructure and considerably improves their mechanical properties as compared with a single technique (either HE or ECAE). In copper subjected to a combined ECAE + HE treatment at a true strain of 22.3, the ultimate tensile strength was 550 MPa.

However, the cold processing of lengthy billets by ECAE is difficult because of high operating pressures, potentially resulting in breaking the machine-tool attachments, the punch first of all. Usually the ECAE is used for billets having l/d = 4–6, where l – length, d – diameter. A new version of ECAE, called the equal-channel angular hydroextrusion (ECAH), has been proposed by Spuskanyuk *et al.* [3,4] for the processing of lengthy billets. Under ECAH a billet is extruded by high-pressure fluid through the angular die channel. The relative length of die-billet contact surface is considerably smaller than with ECAE. Therefore, longer billets can be deformed by ECAH at acceptable pressure levels making this method attractive for commercial use.

In this paper, the effect of combined technology including the ECAH, HE and D techniques (Fig. 1) for making wire is investigated. Apparently, a longer wire can be produced by using ECAH of longer billets ($l/d \geq 10$) as compared to ECAE case. The main purpose of our investigation is to show that the combined technology including ECAH, HE and D techniques significantly improves wire properties. For the first time, it has been demonstrated by the copper samples that combining the ECAH, HE and D techniques provides higher properties of wire as compared to properties obtained without ECAH. It has been also demonstrated that the repetitive HE and ECAH (HE + ECAH + HE + ECAH + HE) results in a higher level of copper wire strength as compared to the result obtained with the single ECAH technique. For the first time, the ECAH of a rod through conical and angular dies is described.

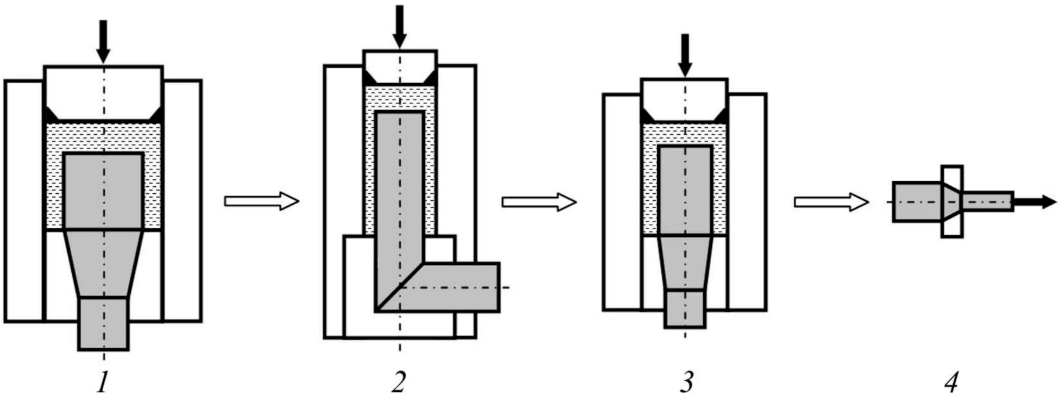


Fig. 1. A new combined technology including the HE (1, 3), ECAH (2) and D (4) techniques for making wire

2. Materials and investigation procedure

Commercial Cu-FRTP (fire refined tough pitch copper) hot-pressed rods of 60 mm diameter were used. The chemical composition of this material is given in Table 1. Impurity analysis of copper was done by the atom-emission method using an optical-emission spectrometer ARL4460 Metals Analyzer.

Rods were processed by HE, ECAH and D followed by annealing of copper in some cases.

Table 1

The chemical composition of Cu-FRTP, %

Pb	Fe	Sn	Si	Sb	As	Mn	Al	Co	Zn
0.0060	0.0019	0.0062	0.0002	0.0012	0.0004	< 0.0002	< 0.0001	< 0.0001	0.0039
Bi	**P**	**S**	**Ag**	**Cr**	**Ni**	**Se**	**Te**	**Cd**	**Cu**
0.0002	0.0116	0.0039	0.0051	0.0006	0.0101	< 0.0001	< 0.0001	< 0.0001	99.948

The experimental conditions, such as processing modes, sequence of processing steps, equivalent strain ε_{HE} of the samples by the hydroextrusion, temperature of annealing (for 1 h), the number of ECAH cycles *n*, ECAH route, total equivalent strain $\varepsilon_{\text{ECAH}}$ accumulated during the ECAH, total equivalent strain ε_{D} accumulated during the drawing are summarized in Table 2. For the 7-th variant of processing modes the billets were machined after the first ECAH in order to provide the same diameter as that of billets after the second HE.

Table 2

Experimental conditions

Processing mode	HE	A	ECAH			HE	ECAH			HE	D
	ε_{HE}	*t*, °C	*n*	route	$\varepsilon_{\text{ECAH}}$	ε_{HE}	*n*	route	$\varepsilon_{\text{ECAH}}$	ε_{HE}	ε_{D}
1	2.3	–	–	–	–	0.8	–	–	–	1.9	4.6
2	2.3	350	2	C	2.3	0.8	–	–	–	1.9	4.6
3	2.3	350	2	C	2.3	0.8	1	–	1.2	1.9	4.6
4	2.3	350	2	C	2.3	0.8	2	C	2.3	1.9	4.6
5	2.3	350	2	C	2.3	0.8	3	C	3.5	1.9	4.6
6	2.3	–	1	–	1.2	0.8	–	–	–	1.9	4.6
7	2.3	–	1	–	1.2	–	3	C	3.5	1.9	4.6
8	2.3	–	1	–	1.2	0.8	4	C	4.6	1.9	4.6

The original ECAH technology was applied with the purpose of refining structure of lengthy billets. The rods ($l = 10d$) were extruded by ECAH through an angular die with $2\Phi = 90^\circ$ using a hydraulic press of 1 MN force. The original ECAH facility is schematically shown in Fig. 2.

The main part of a facility was a high-pressure vessel with the operating pressure to 1.6 GPa. A conical die and an angular die are located in the lower part of the vessel channel. Conical die channel and input segment of the angular die channel were aligned with the high-pressure vessel channel. The diameters of the conical die calibrating bore and the input segment of the angular die channel were equal. The diameter of output segment of the angular die channel was made slightly larger than that of input segment for a repetitive ECAH without any additional operations of billet thickening before each pass through the conical die.

Prior to plastic deformation, the billet surface was coated with a soap-based solution. The initial billet with a small nose machined on one end was fitted into an entry of the conical die to seal the fluid. The high-pressure unit was then filled with the hydrostatic fluid (engine oil SAE 40) and the plunger was inserted and forced into the

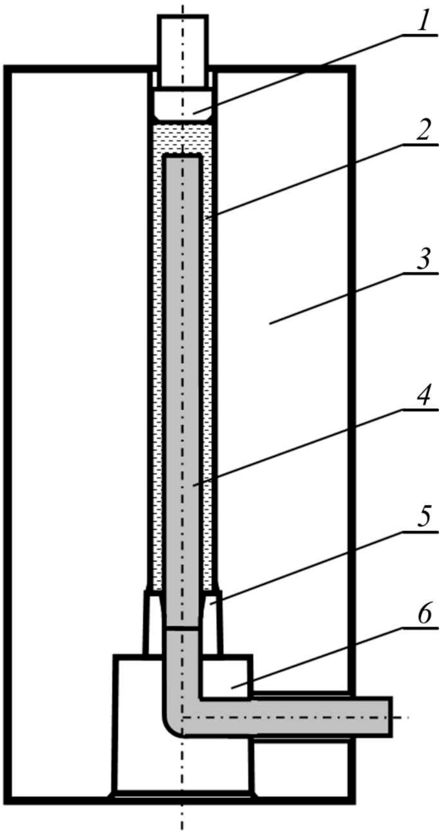


Fig. 2. A schematic of the ECAH facility: *1* – plunger, *2* – fluid, *3* – high-pressure unit, *4* – billet, *5* – conical die, *6* – angular die

bore by means of the press. The appropriate fluid pressure initiated extrusion of the billet into the input segment of the angular die channel. Subsequently, when the pressure was increased, the fluid forced the billet through the angular die channel and the product emerged from the output channel segment. Multiple ECAH was implemented by the «billet after billet» technology. All the rods were deformed under the room-temperature conditions, the plunger travelled at a rate of 2 mm/s. HE of billets and D of wire were done by well-known methods. For instance, HE and D techniques were described by Blazynski [5].

Microstructure of copper was studied by methods of optical and electron microscopy. Vickers hardness measurements were done using a HV-5 Low V.H. Tester manufactured by L.H. Testing Instruments Co, Ltd. Mechanical tensile tests for samples and wires were done using machines of 2167 R-50 type manufactured by «Tochpribor» Co and ZM 20 174.21 type manufactured by «Fritz Heckert» Co, respectively. Diameter of the working part of a tensile-tested sample was equal to 3 mm, the length of the base was 15 mm; wire diameter was equal to 0.5 mm, the length of the base was 100 mm. Tensile tests were done at the room temperature, the cross-piece travelled at a rate of 10^{-4} m/s. Electrical resistance of copper wire was measured by the standard four-probe method under the room temperature (293 K) and the liquid nitrogen boiling temperature (77.3 K). The resistivity was measured with a relative error under 0.5%.

3. Results and discussion

By the combined processing of original billets by HE and ECAH techniques the high-strength rods for wire drawing have been produced. For the rods of 7 mm-diameter (the 8-th variant of processing mode of Table 2) the HV of copper was equal to 1450 MPa (Fig. 3,*a*), the ultimate tensile strength σ = 546 MPa (Fig. 3,*b*). For the rods produced without ECAH (the 1-st variant of processing mode of Table 2), the HV of copper was equal to 1320 MPa (Fig. 3,*a*), the ultimate tensile strength σ = 473 MPa (Fig. 3,*b*).

Figure 4 illustrates the plastic properties of copper in the rods. The highest level of plastic characteristics was achieved for the 2-nd variant of processing modes. Note that after different modes of processing, the difference in plastic characteristic is not so significant as in the strength properties of copper.

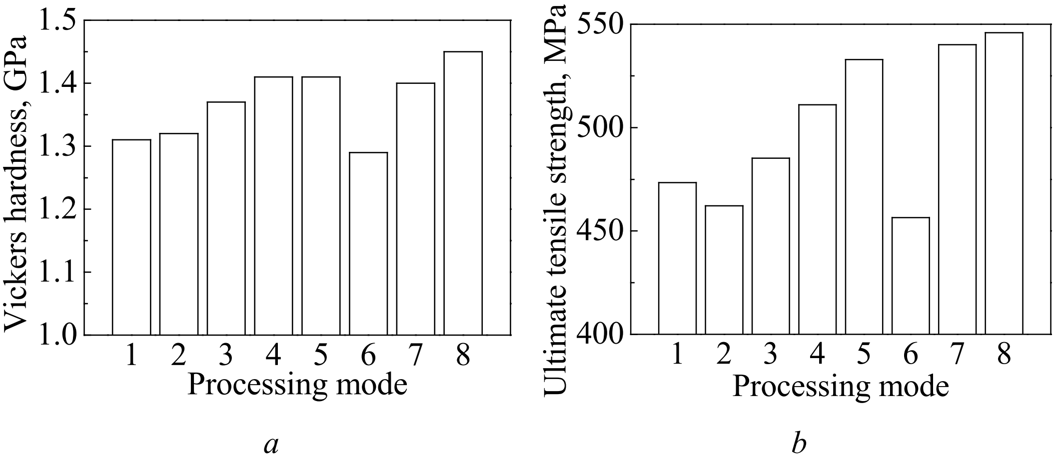


Fig. 3. HV (*a*) and ultimate tensile strength (*b*) of copper (Cu-FRTP) rods of 7 mm-diameter after different processing modes

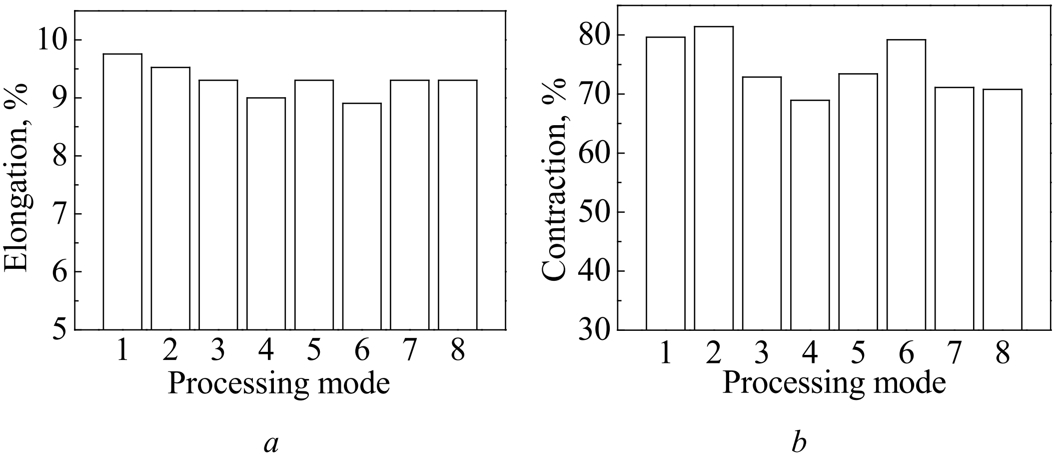


Fig.4. The elongation to failure (*a*) and contraction at tension (*b*) for copper specimens

The high strength of 0.5 mm-diameter copper wire was achieved by drawing the strengthening rods. The multiple interchanging of HE and ECAH techniques (Fig. 5.), processing without annealing or using a low temperature of annealing have resulted in a higher level of copper wire strength (Fig. 6).

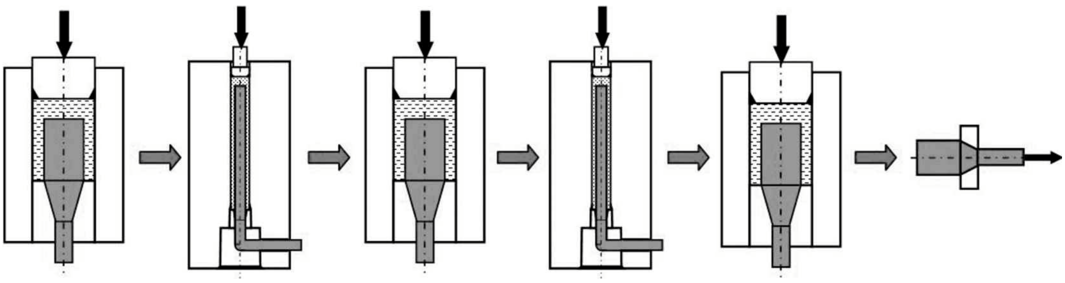
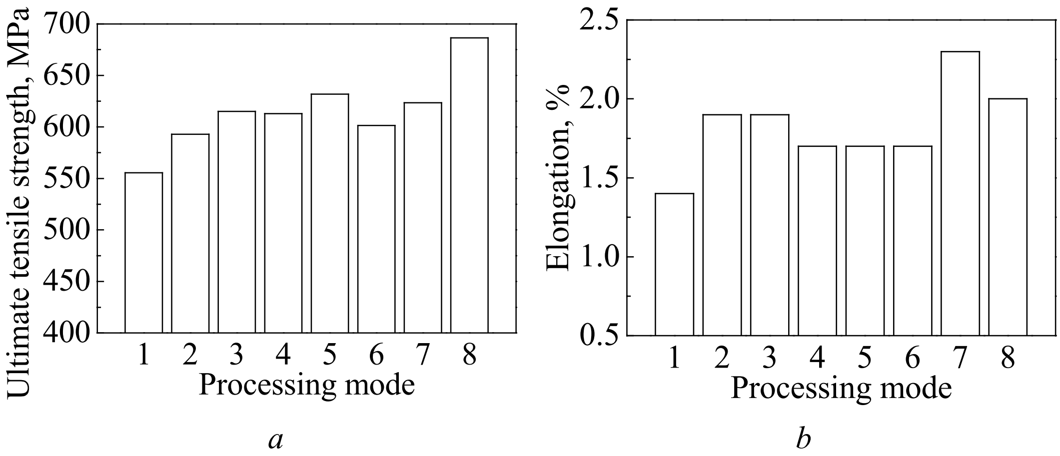


Fig. 5. The multiple interchanging of HE and ECAH techniques in combined technology including the HE, ECAH and D techniques for making wire

The best result (Fig. 6) was achieved for the 8-th variant of processing mode of Table. 2: the ultimate tensile strength σ = 686 MPa, the elongation to failure δ = = 2%. In the case of the processing mode without ECAH (the 1-st variant) the ultimate tensile strength σ = 556 MPa, the elongation to failure δ = 1.4%.

The high-strength copper wire produced by combining HE, ECAH and D techniques preserves the highest strength characteristics to the annealing temperature $T \leq 100$°C, and the plasticity increases insignificantly (Fig. 7).



a *b*

Fig. 6. The ultimate tensile strength and elongation to failure of 0.5 mm-diameter copper wire (column numbers correspond to variants of processing modes of Table 2)

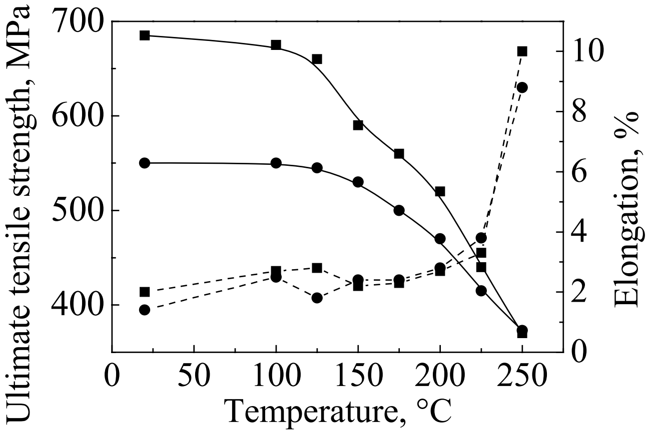


Fig. 7. Dependence of ultimate tensile strength (■—■—■ – using ECAH, ●—●—● – without ECAH) and elongation to failure (■---■---■ – using ECAH, ●---●---● – without ECAH) of 0.5 mm-diameter copper wire on annealing temperature (1 h)

The electrical resistance of the highest strength wire produced using ECAH (the 8-th variant of processing modes) and hardened wire produced without the ECAH the (1-st variant of processing modes) differ insignificantly (Table 3). Thus, electrical conductivity is low-dependent on processing mode and for high-strength copper wire it is equal to 86.4% IACS.

Figure 8 combines the characteristics of strength and electrical conductivity for different copper alloys. On the diagram, the region under the curve illustrates the properties of copper alloys highly strengthened by traditional methods of cold plastic deformation [6]. It is seen that, in the general case, the electrical conductivity of copper alloys drops abruptly with the growth of strength. The properties

Table 3

Electrical resistance of 0.5 mm-diameter high-strength copper wires

Processing mode	ρ_{293}/ρ_{77}	ρ_{77}, μΩ·cm	ρ_{293}, μΩ·cm
1	3.785	0.512	1.972
8	3.714	0.537	1.995

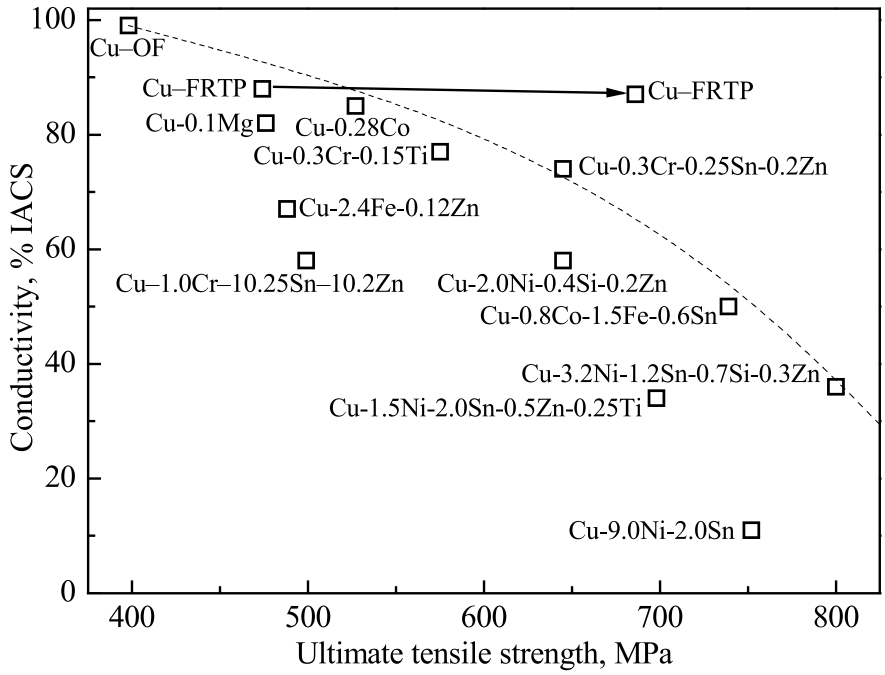


Fig. 8. Physical and mechanical properties of copper and copper alloys after cold deformation

of commercially pure copper (Cu-FRTP) produced by us using the combination of HE, ECAH and D methods are above the curve. The repetitive application of HE and ECAH methods in technological chain of processing the materials in fractional modes and with the optimal degrees of deformation results in the formation of a unique complex of physical and mechanical properties, such as strength $\sigma = = 686$ MPa and electrical conductivity at a 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. Such a processing is efficient due to the alternating schemes of deformation and periodic creation of favorable conditions for relaxation and dynamic recrystallization processes in the material. A 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 – conditions for stress relaxation, healing of microdiscontinuities and smaller exhaustion of plastic resource.

4. Summary

For the first time, it has been demonstrated by the example of copper that the combining of ECAH, HE and D techniques provides higher properties of wire as compared to properties obtained without ECAH. It has been also demonstrated that the repetitive application of HE and ECAH methods in technological chain of copper processing in fractional modes and with the optimal degrees of deformation results in the formation of a unique complex of physical and mechanical properties.

The high ultimate tensile strength $\sigma = 686$ MPa, the elongation to failure $\delta = 2\%$ and the electrical conductivity at a level of 86.4% IACS have been reached for 0.5 mm-diameter copper (Cu-FRTP) wire. Such complex of strength and electrical conductivity is the record for the copper and copper alloys.

Such a processing is efficient due to the alternating schemes of deformation and periodic creation of favorable conditions for relaxation and dynamic recrystallization processes in the material. A 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 – conditions for stress relaxation, healing of microdiscontinuities and smaller exhaustion of plastic resource.

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В.З. Спусканюк, О.А. Давиденко, О.М. Гангало, Л.Ф. Сеннікова, М.А. Тіхоновський, Д.В. Спірідонов

ДОСЯГНЕННЯ РЕКОРДНОГО РІВНЯ ВЛАСТИВОСТЕЙ МІДНОГО ДРОТУ МЕТОДАМИ ІПД

Запропоновано новий підхід до керування процесами пластичної деформації матеріалів при обробці заготовок методами інтенсивної пластичної деформації (ІПД). Високий комплекс міцності й пластичності оброблених мідних заготовок отриманий після багаторазового чергування методів кутової гідроекструзії (КГЕ) і прямої гідроекструзії (ГЕ) з реалізацією КГЕ й ГЕ в дробовому режимі. Комбінована ІПД-технологія, що включає КГЕ, ГЕ й волочіння (В), забезпечує в мідному дроті діаметром 0.5 mm межу міцності $\sigma = 686$ MPa, відносне подовження $\delta = 2\%$ і електричну провідність на рівні 86.4% IACS. Ефект такої обробки обумовлений чергуванням схем деформацій і періодичним забезпеченням сприятливих умов для протікання в матеріалі процесів релаксації й динамічної рекристалізації. Важливою умовою досягнення максимального ефекту обробки є дробовий режим і оптимальні ступені деформації обома методами.

Ключові слова: кутова гідроекструзія, пряма гідроекструзія, волочіння, мідь, фізико-механічні властивості, нанокристалічна структура

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В.З. Спусканюк, А.А. Давиденко, А.Н. Гангало, Л.Ф. Сенникова, М.А. Тихоновский, Д.В. Спиридонов

ДОСТИЖЕНИЕ РЕКОРДНОГО УРОВНЯ СВОЙСТВ МЕДНОЙ ПРОВОЛОКИ МЕТОДАМИ ИПД

Предложен новый подход к управлению процессами пластической деформации материалов при обработке заготовок методами интенсивной пластической деформации (ИПД). Высокий комплекс прочности и пластичности обработанных медных заготовок получен после многократного чередования методов угловой гидроэкструзии (УГЭ) и прямой гидроэкструзии (ГЭ) с реализацией УГЭ и ГЭ в дробном режиме. Комбинированная ИПД-технология, включающая УГЭ, ГЭ и волочение (В), обеспечивает в медной проволоке диаметром 0.5 mm предел прочности $\sigma = 686$ MPa, относительное удлинение $\delta = 2\%$ и электрическую проводимость на уровне 86.4% IACS. Эффект такой обработки обусловлен чередованием схем деформаций и периодическим обеспечением благоприятных условий для протекания в материале процессов релаксации и динамической рекристаллизации. Важным условием достижения максимального эффекта обработки являются дробный режим и оптимальные степени деформации обоими методами.

Ключевые слова: угловая гидроэкструзия, прямая гидроэкструзия, волочение, медь, физико-механические свойства, нанокристаллическая структура