

# A new technology for making advanced materials

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## ABSTRACT

A new approach is proposed to control the processes of billets plastic deformation during metal treatment by the methods of severe plastic deformation (SPD). High strength and plasticity have been attained for the processed copper billets after multiple repetitions of equal channel angular hydroextrusion (ECAH) and direct hydroextrusion (HE) techniques and with ECAH and HE implementation in the fractional mode. The combined SPD treatment including ECAH, HE and drawing (D) provided for fine refined tough pitch copper (Cu-F RTP) the ultimate tensile strength  $\sigma=686$  MPa, the elongation to failure  $\delta=2\%$  and the electrical conductivity (EC) at a level of 86.4% IACS and for oxidant free copper (Cu-OF)  $\sigma=576$  MPa,  $\delta=1.9\%$ , EC=96.7% IACS in the 0.5 mm diameter wire. Such treatment is efficient due to the alternative schemes of deformation, the fractional mode and the optimum degrees of plastic deformation and periodic creation of favorable conditions for relaxation and dynamic recrystallization processes in the material.

**Keywords:** equal channel angular hydroextrusion, direct hydroextrusion, drawing, copper, 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 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 to 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 treatment of lengthy billets by ECAE is difficult due to high operating pressures potentially resulting in breaking the machine-tool attachments, the punch first of all. A new version of ECAE, called the equal channel angular hydroextrusion (ECAH), has been proposed by Spuskanyuk *et al.* [3, 4] for the treatment of lengthy billets. Under ECAH a billet is extruded by high-pressure fluid through the angular die channel and the relative length of die-billet contact surface is considerably smaller as compared to ECAE. Therefore, longer billets

( $l \geq 10d$ ) can be deformed by ECAH at acceptable pressure levels making this method attractive for commercial use.

In this paper, the effect of combined treatment including the ECAH, HE and D techniques for making copper wire is investigated. Apparently, a longer wire can be produced by using ECAH of longer billets as compared to ECAE case. The main purpose of our investigation is to show that the combining of ECAH, HE and D techniques provides higher properties of wire as compared to those obtained without ECAH. For the first time, 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.

## 2 MATERIALS AND INVESTIGATION PROCEDURE

Commercial Cu-F RTP (99.95%) and Cu-OF (99.98%) hot-pressed rods of 60 mm diameter were used. 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 as illustrated in Fig.1.

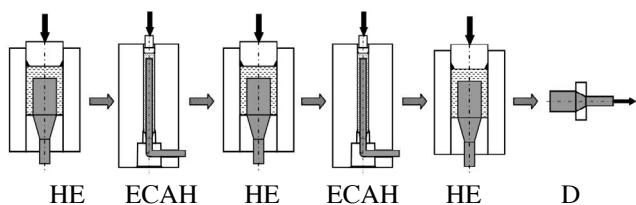


Figure 1: The multiple interchanging of HE and ECAH techniques in combined treatment including the HE, ECAH and D techniques for making wire.

The sequence of treatment steps and equivalent strain of the samples accumulated during the treatment are summarized in Table 1.

Copper	HE	ECAH	HE	ECAH	HE	D	Total
Cu-F RTP	2.3	-	0.8	-	1.9	4.6	9.6
	2.3	1.2	0.8	4.6	1.9	4.6	15.4
Cu-OF	2.0	3.5	0.8	4.6	2.4	4.6	17.9

Table 1: The equivalent strain of the samples accumulated during treatment.

The 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 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.

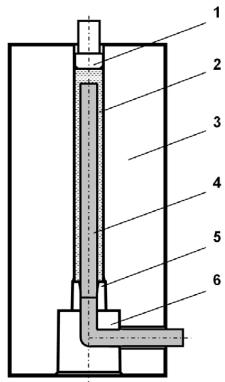


Figure 2: A schematic of the ECAH facility: 1 - plunger, 2 - fluid, 3 – high-pressure unit, 4 - billet, 5 – conical die, 6 – angular die.

Prior to plastic deformation, the billet surface was coated with a soap-based solution. The engine oil SAE 40 was used as the hydrostatic fluid. Multiple ECAH was implemented by the “billet after billet” technology. All the rods were deformed under the room-temperature conditions, the plunger traveled at a rate of 2 mm/s. HE of billets and D of wire were done by well-known methods.

Vickers hardness measurements were made 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 traveled 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 (293K) and the liquid nitrogen boiling temperature (77.3K). The resistivity was measured with a relative error under 0.5%.

### 3 RESULTS AND DISCUSSION

By the combined treatment 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

Vickers hardness (HV) of copper (Cu-F RTP) was equal to 1450 MPa (Fig.3 a), the ultimate tensile strength  $\sigma=546$  MPa (Fig.3 b). For the rods produced without ECAH, the HV of copper was equal to 1320 MPa (Fig.3 a), the ultimate tensile strength  $\sigma=473$  MPa (Fig.3 b).

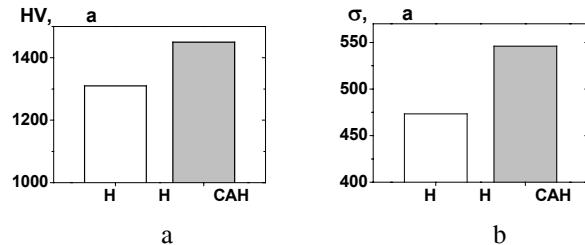


Figure 3: Vickers hardness (a) and ultimate tensile strength (b) of copper (Cu-F RTP) rods of 7 mm-diameter after different treatment variants.

The high strength of 0.5mm-diameter copper wire was achieved by drawing the strengthened rods. The combining of HE, ECAH and D techniques (Fig.1.) have resulted in a higher level of copper wire strength as compared to the result obtained without ECAH technique. In the case of combined treatment the ultimate tensile strength  $\sigma=686$  MPa and the elongation to failure  $\delta=2\%$  have been reached for Cu-F RTP and  $\sigma=576$  MPa,  $\delta=1.9\%$  for Cu-OF (Fig.4). In the case of Cu-F RTP treatment without ECAH the ultimate tensile strength  $\sigma=556$  MPa, the elongation to failure  $\delta=1.4\%$ .

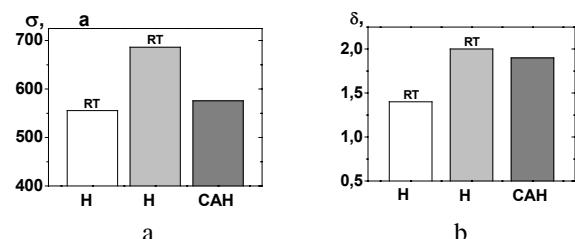


Figure 4: The ultimate tensile strength (a) and elongation to failure (b) of 0.5 mm-diameter copper wire

The electrical resistance of the most strengthened copper wire produced using ECAH and strengthened wire produced without ECAH differ insignificantly (Table 2).

Copper	Processing mode	$\rho_{293}$ , $\mu\text{Ohm}\times\text{cm}$	$\rho_{77}$ , $\mu\text{Ohm}\times\text{cm}$	$\rho_{293}/\rho_{77}$
Cu-F RTP	HE	1.972	0.512	3.852
	HE&ECAH	1.995	0.537	3.715
Cu-OF	HE&ECAH	1.782	0.314	5.675

Table 2: The electrical resistance of 0.5 mm-diameter copper wire samples.

Thus, electrical conductivity is low-dependent on processing mode and for high-strength Cu-F RTP wire it is

equal to 86.4 % IACS. Conductivity of high-strength Cu-OF wire is equal to 96.7 % IACS.

The high-strength copper (Cu-F RTP) wire produced by combining HE, ECAH and D techniques preserves the highest values of ultimate tensile strength to the annealing temperature  $T \leq 100^\circ\text{C}$ , and the plasticity increases insignificantly (Fig. 5).

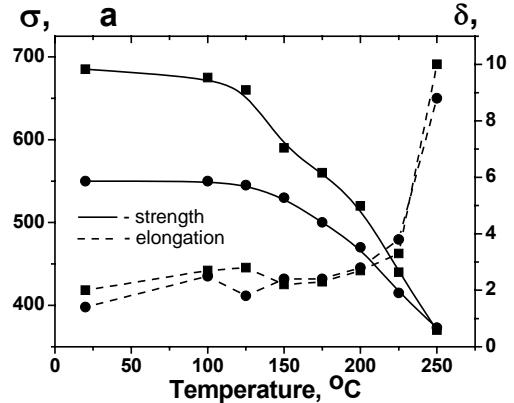


Figure 5: Mechanical property of copper (Cu-F RTP) wire versus the annealing temperature during 1h:  
—■— using ECAH, —●— without ECAH.

Figure 6 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

It is seen that, in the general case, the electrical conductivity of copper alloys drops abruptly with the growth of strength.

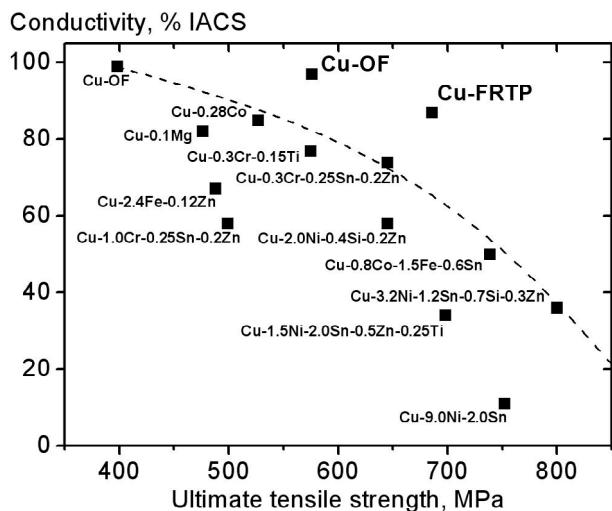


Figure 6: Physical and mechanical properties of copper and copper alloys after cold deformation.

The properties of Cu-F RTP samples produced by us using the combination of HE, ECAH and D techniques are

above the curve. The repetitive application of HE and ECAH techniques in technological chain of copper treatment in fractional mode and with the optimal degrees of deformation resulted 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. Similar result was achieved for Cu-OF samples: the high level of ultimate tensile strength  $\sigma=576$  MPa and electrical conductivity at a level of 96.7% IACS.

The proposed combined treatment is efficient due to the alternative 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 that the repetitive application of HE and ECAH techniques in technological chain of copper treatment in fractional mode and with the optimal degrees of deformation resulted in the formation of a unique complex of physical and mechanical properties.

The high ultimate tensile strength  $\sigma=576$  MPa, the elongation to failure  $\delta=1.9\%$  and the electrical conductivity at a level of 96.7% IACS have been reached for 0.5 mm-diameter copper (Cu-OF) wire. For Cu-F RTP samples  $\sigma=686$  MPa,  $\delta=2\%$  and the electrical conductivity at a level of 86.4% IACS have been reached. Such complex of strength and electrical conductivity is the record for the copper and copper alloys.

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