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SEVERE PLASTIC DEFORMATION INDUCED IN Al, Al-Si, Ag AND Cu BY HYDROSTATIC EXTRUSION

ZASTOSOWANIE METODY WYCISKANIA HYDROSTATYCZNEGO DO GENEROWANIA DUŻYCH ODKSZTAŁCEŃ PLASTYCZNYCH W Al, Al-Si, Ag ORAZ Cu

The study was concerned with the effect of severe deformation induced in one pass, by hydrostatic extrusion on the properties of fine aluminum, aluminum-silicon alloy, copper and silver wires. The influence of adiabatic heating which takes place during deformation on the mechanical properties and microstructure of the wires was examined. The quality of the surface of the wires was estimated. It has been demonstrated that fine aluminum and silver wires processed by hydrostatic extrusion have very good mechanical properties and a high-quality surface.

Keywords: Hydrostatic extrusion, Grain refinement, Severe plastic deformation, Fine wires

W pracy zbadano wpływ dużych odkształceń, uzyskanych w jednym procesie wyciskania hydrostatycznego na właściwości wytworzonych produktów w postaci cienkich drutów z aluminium, stopu aluminium-krzem, miedzi oraz srebra. Uwzględniono efekty grzania adiabatycznego w trakcie procesu odkształcenia i jego wpływ na własności mechaniczne oraz mikrostrukturę produktów. Ocenie została poddana także jakość powierzchni wytwarzanych drutów. Dla aluminium oraz srebra wykazano, ze za pomocą procesu wyciskania hydrostatycznego można wytwarzać druty o wysokich własnościach mechanicznych oraz dobrej jakości powierzchni.

1. Introduction

Hydrostatic extrusion (HE) is a unique method of plastic treatment of metals and alloys. Like ECAP or HPT [1-3], it combines the ability to induce severe plastic deformations (SPD) in the material and also permits controlling the properties and geometry of the final product [4-7]. The basic difference between HE and the other methods is the three-axial compressive state of stresses which is induced in the material in both the working chamber and the deformation zone in the extrusion die. This is so thanks to the action of the pressure transmitting medium compressed to high hydrostatic pressures. The high pressure increases the plasticity of the treated material and creates favorable lubrication conditions so that friction at the tool (die)/material interface is considerably reduced. Hydrostatic extrusion therefore permits inducing severe plastic deformations during a single process pass and producing bulk products even of brittle or difficult-to-deform materials [8]. The products are characterized by a uniform distribution of mechanical properties and have a homogeneous microstructure (as observed on their cross-sections). The effect exerted by severe deformations on the microstructure and properties of metals and their alloys is commonly known. SPD refines the microstructure of the materials to a fine-grained or even nanometric level thanks to which they acquire excellent mechanical properties [1, 4-7].

In the present study the authors used hydrostatic extrusion for producing wires of aluminum, aluminum-silicon alloy (Al-1%Si), silver and copper. Such wires are commonly applied as connecting leads in electric-power installations, welding cables (Al, Cu) [9], path connections in microelectronic systems (Al-Si) [10] and high class electric leads in audio devices (Ag) [11]. A competitive method of producing wires of this type is drawing which however requires many drawing passes and often involves intermediate heating operations. For example, in the case of a copper wire shown in Fig. 1, the reduction of the diameter from 10 mm to 1 mm requires about 155 drawing passes, whereas by using HE it is possible to achieve this reduction in a one pass process.

Hydrostatic extrusion has been used thus far in practice for producing ultra-thin wires with diameters up to 25μm (0.025 mm) of pure gold (99.99%) and Al-1%Si alloy intended for thermo-compression devices [12]. In view of the very low tensile strength (between 8g and 20g depending on the state of strengthening of the material), wires with these diameters cannot be produced by drawing. They have been therefore produced by multi-pass hydrostatic extrusion using small reductions in consecutive passes.

There is no data available in the literature concerning one-pass hydrostatic extrusion with a large reduction applied to wires with diameters about 1 mm and below. The lack of literature reports is probably associated with the fact that, to

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achieve a given reduction, thin wires require extrusion pressures higher by a factor of 1.3 to 1.7 than those used in extrusion of thicker wires [13]. This is so because of the increased contribution of friction forces as a result of the increased surface area-to-volume ratio of the wire.

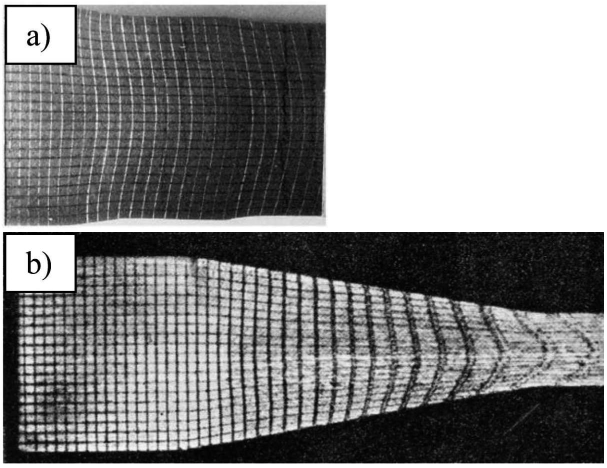

Fig. 1. (a) Drawn copper, reduction in one pass – 3% [21], and (b) HE-processed copper, reduction in one pass – 99.99% [14]

Pioneer studies on the high-reduction HE technology were reported somewhat randomly by Pugh [14] long time ago. He describes an Al wire (99.5%) with the diameter above 1 mm produced by one-pass hydrostatic extrusion from a wire above 38 mm in diameter (reduction R – about 1400). He does not however report on the properties of the final product.

The authors of the present study undertook an attempt to produce aluminum, aluminum-silicon alloy (Al-1%Si), silver and copper wires with a diameter of about 1 mm by one-pass hydrostatic extrusion using a bulk billet material. The paper describes the process parameters and discusses their influence on the properties and quality of the final product.

2. Experimental procedure

The materials examined were aluminum A1 (AA1050) with a purity of 99.5%, an aluminum alloy with 1% of silicon (Al-1%Si), silver with a purity of 99.99%, and technical M1E copper with a purity of 99.9%. In order to soften and homogenize the microstructure, the materials were annealed at a temperature of 370°C, 300°C, 600°C, and 700°C, respectively for 1h. Fig. 2 shows the microstructures of the materials in the starting state (before deformation). The grain size was the greatest in Al-1%Si, medium in Cu and Ag, and the smallest in Al (120μm, 87μm, 62μm, and 14.5μm, respectively). The Vickers microhardness was 44HV0.05 in Cu, 35HV0.05 in Ag and 28HV0.05 in Al and Al-1%Si.

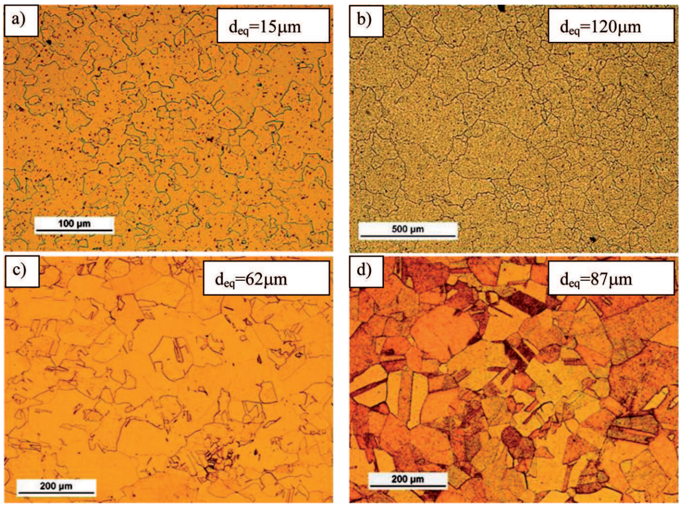

Fig. 2. Microstructures of the starting materials before HE: (a) aluminum, (b) aluminum-silicon alloy, (c) silver, (d) copper

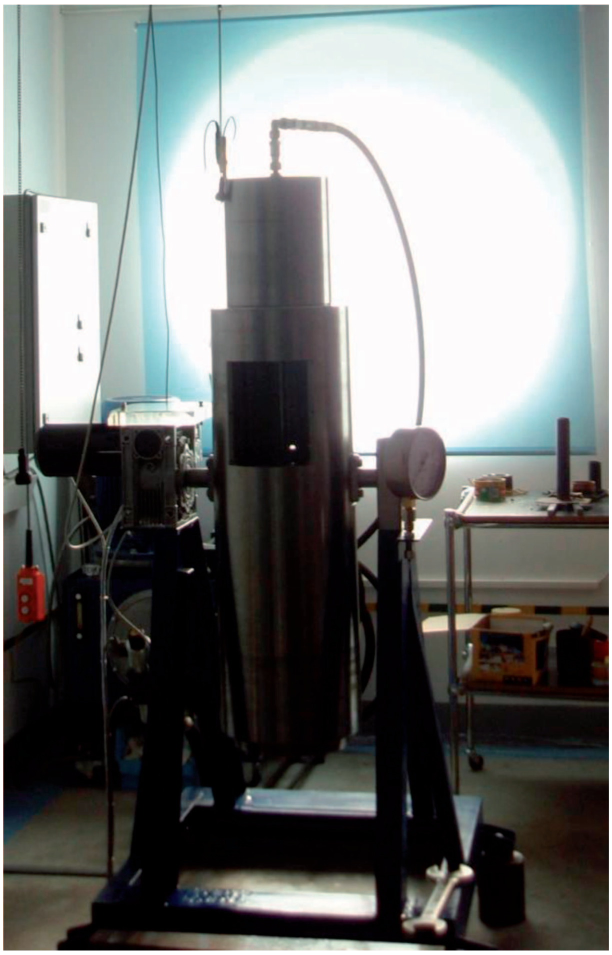

Fig. 3. Press for cold hydrostatic extrusion under a pressure up to 2.5GPa (designed and constructed at Unipress)

The hydrostatic extrusion was performed in a cold state at pressures up to 2.5 GPa in a press designed and constructed at the Institute of High Pressures Physics UNIPRESS, Polish Academy of Sciences (Fig. 3). Starting from a distance of about 15 cm from the die exit the outgoing deformed product was intensively cooled using running water and water mist, then the entire product fell down to a cooling bath filled with cold water. Aluminum and the aluminum alloy were lubricated with wax, whereas copper and silver – with a Molipas 60N. All the HE processes were conducted in one pass to the final diameter of the product to be about 1 mm, within the range of the maximum possible true strain ε which was $\varepsilon \sim 6$ in Al and Al-1%Si, and $\varepsilon \sim 5$ in Ag and Cu (Fig. 4). In HE the true strain

ε is equal to lnR where R is the reduction degree achieved during the extrusion, calculated as the ratio of the cross-section area of the starting billet to the cross-section area of the final product. The length of the HE-treated products ranged from 20 m to 60 m.

The examinations to which the deformed materials were subjected included: measurements of microhardness HV0.05, static tensile test (ultimate tensile strength UTS, yield stress YS, strain to fracture ε_f) as well as structural examinations performed in a transmission electron microscope (TEM) and in an optical microscope (LM). The surface finish of the products was examined using an optical profile-meter and measuring the arithmetic means of the deviation of the profile from the average line Ra positioned in the plane perpendicular to the wire axis. The surface topography was scanned along a measurement line of 5 mm oriented along the wire axis. Examinations of the mechanical properties of the HE-processed wires, such as HV and Ra, were performed at the randomly selected points positioned along the wires.

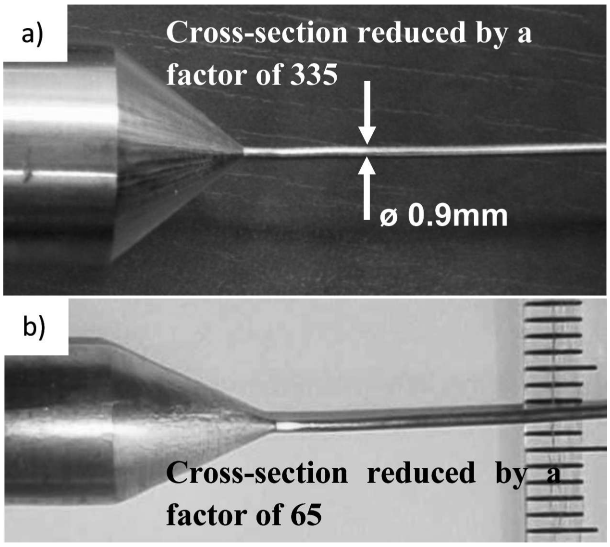

Fig. 4. Severe plastic deformation of wires produced by HE: (a) aluminum ε ~6 (b) copper ε ~5

3. Results and discussion

Figure 5 shows the pressure characteristics of the HE process, i.e. extrusion pressure vs. true strain ε induced in the extruded material, up to the value ε =6. It follows from these curves that, for the same true strain, the extrusion pressure increases from Al through Al-1%Si, Ag to Cu in which it is the highest. The coefficient of deformation strengthening during HE is the highest in copper, whereas in Ag, Al-1%Si, and Al these coefficients are almost similar. The pressure characteristics strongly depend on the effect of adiabatic heating which takes place in the deformation zone of the die.

The mechanical work of the plastic deformation is transformed into thermal energy, an effect which is about proportional to the extrusion pressure and inversely proportional to the density of the material and to its specific heat [7]. This was confirmed and measured many times during various plastic deformation processes including HE [15-18].

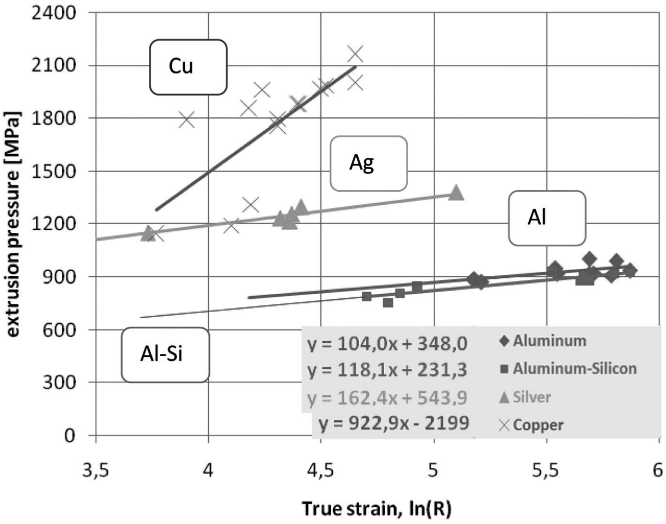

Fig. 5. Pressure characteristics of HE (p=ln(R)) obtained for aluminum, aluminum-silicon alloy, silver, and copper within the range of severe deformations

In the wires with a diameter of about 1 mm extruded in our experiments, in which the plastic strain in one pass was high and, thus, such were the extrusion pressure and the relative share of friction, the adiabatic heating effects were important (Table 1). Table 1 gives the minimum and maximum homologous temperature (defined as the ratio of the melting temperature to the adiabatic heating temperature, both in K) to which the materials were heated during the HE, their melting temperatures, and the values of their stacking fault energy (SFE). The highest homologous temperature was observed in aluminum and the Al-1%Si alloy which are characterized by high values of the stacking energy. Because of the high values of these two parameters, the susceptibility of the materials to plastic deformation increases due to the thermally activated dynamic recovery effect which weakens the strengthening of the material through the annihilation of defects and their regrouping into low-energy systems. The lowest homologous temperatures were observed in copper.

TABLE 1

Stacking fault energy, homologous temperature and melting temperature of the materials examined in the present experiments

Material	Stacking fault energy (SFE) [mJ/m^2]	Melting temperature [°C]	Homologus temperature$^{(1)}$ for HE
Aluminum [14]	250	660,32	0,6-0,7
Aluminum silicon [13,14]	<250	600	0,65-0,75
Silver [15]	22	961,78	0,55-0,65
Copper [15]	55	1084,45	0,3-0,35

$^{(1)}$ Homologous temperature = temperature of adiabatic heating (K)/ melting temperature (K)

Figure 6 shows the microstructures of the materials examined after subjecting them to cold HE, and Table 2 gives their average grain size. HE-processed aluminum had a microstructure composed of equiaxial grains with the average grain size d_{eq} =1μm (Fig. 6a). The aluminum-silicon alloy had a bimodal microstructure where greater grains of 4-5μm are accompanied by agglomerates of finer grains with sizes of about 1.3μm (Fig. 6b). In silver, apart from equiaxial grains of about 2μm we can observe local deformations in the form of thin bands of about 500nm (Fig. 6c). The microstructure of copper was composed of large recrystallized grains with an average grain size of about 11μm (Fig. 6d).

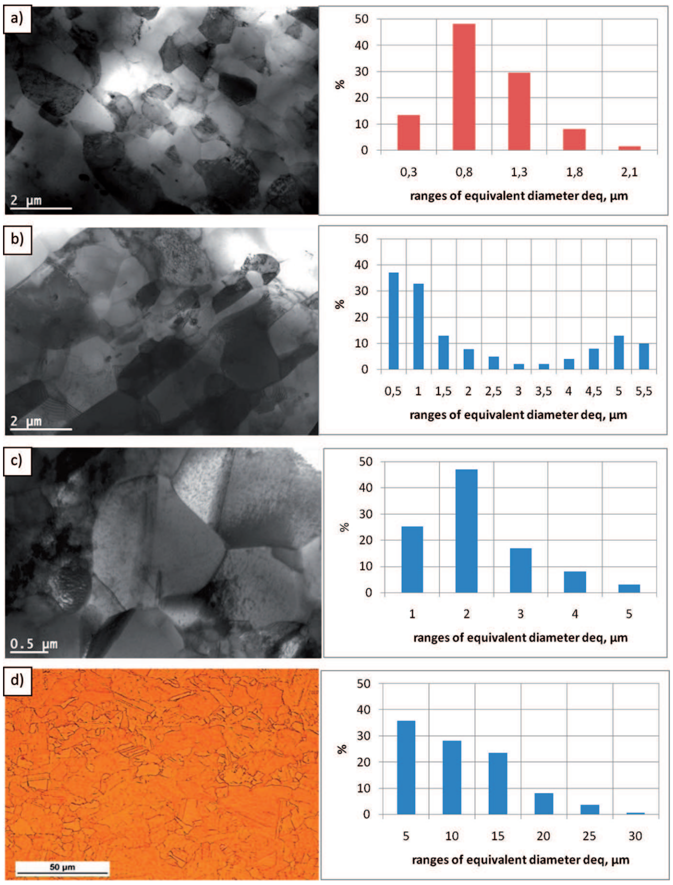

Fig. 6. Microstructures and grain sizes distribution of the wires after HE conducted with severe deformation in one pass: (a) aluminum ε =5.7, (b) Al-1%Si alloy ε =5.7, (c) silver ε =4.4, and (d) copper ε =4.4

TABLE 2

Grain size characteristic of the materials examined before and after cold hydrostatic extrusion conducted with severe deformation

Material	Initial		After HE		
	d_{eq} [μm]	CV (d_{eq})	d_{eq} [μm]	CV (d_{eq})	True strain ε
Al	14.5	0.31	1	0,41	5,7
Al-1%Si	120	0,44	1,3	0,52	5.7
Ag	62	0,39	2,2	0,43	4.4
Cu	87	0,30	11	0,52	4.4

(1) d_{eq} – Mean equivalent grain diameter = $(2S/\pi)^{-1/2}$, where S=equivalent surface area of a circle

(2) Cv (d_{eq}) – Coefficient of variation = standard deviation/mean value

The stacking fault energy of copper is lower than that of aluminum and the aluminum-silicon alloy. This favors dynamic recrystallization which results in the deformed grains being recrystallized and their high-angle boundaries migrating. In view of the considerably higher homologous heating temperatures in silver, its structure after recovery and recrystallization is more inhomogeneous, which enhances the strengthening effect (the slope of the p=ln(R) curve is not so sharp) compared to that occurring in copper.

Figure 7 shows the strength properties of the extruded wires compared with those of commercial wires produced by drawing [19]. In aluminum and silver, the yield strength is considerably higher, namely by above 140% and 26%, respectively, than that in commercial wires. In HE-processed copper, the yield stress is lower because of the susceptibility of copper to recrystallization enhanced by the adiabatic heating effect. Despite intensive cooling, the thermally-activated softening processes which proceed in Cu within the deformation zone are not effectively hindered. Higher strength in commercial Al-1%Si is probably due to the more effective strengthening during the drawing process.

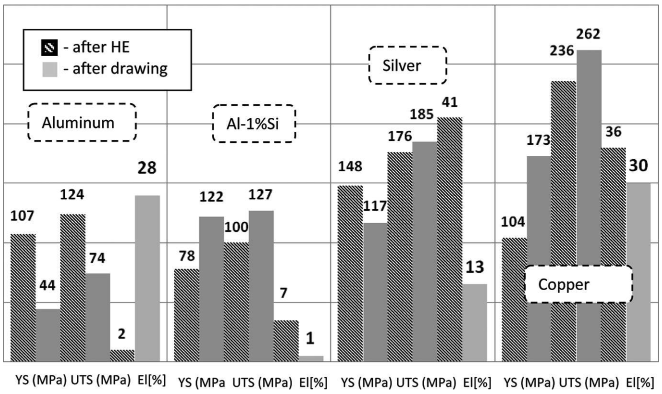

Fig. 7. Yield stress, ultimate tensile strength, and strain to fracture of the HE-processed aluminum, silicon-aluminum alloy, silver, and copper wires compared with the properties of drawn commercial wires

From the point of view of commercial applications of fine wires, an important factor is the quality of their surface, which, as follows from the literature reports, is often even the critical parameter of their application. Drawing processes generate defects in fine wires such as e.g. pores or protuberances [20]. Fig. 8 shows scans of surface topography and the roughness profiles of HE-produced Al, Al-1%Si, and Cu wires compared with the profile obtained for a commercial aluminum wire produced by drawing [19]. All the values of the parameter Ra measured for HE-treated wires fall within the range from 0.33 to 0.36, which is near the requirements of the 6th accuracy class of the surface finish obtained by grinding. In the drawn aluminum alloy Ra is twice as high, the surface is porous, and the cross-section is not perfectly circular (Fig. 8b). Similar defects were observed in the other commercial wires. Our results therefore demonstrate that the HE process ensures better conditions of lubricating and shaping the wires than the drawing process.

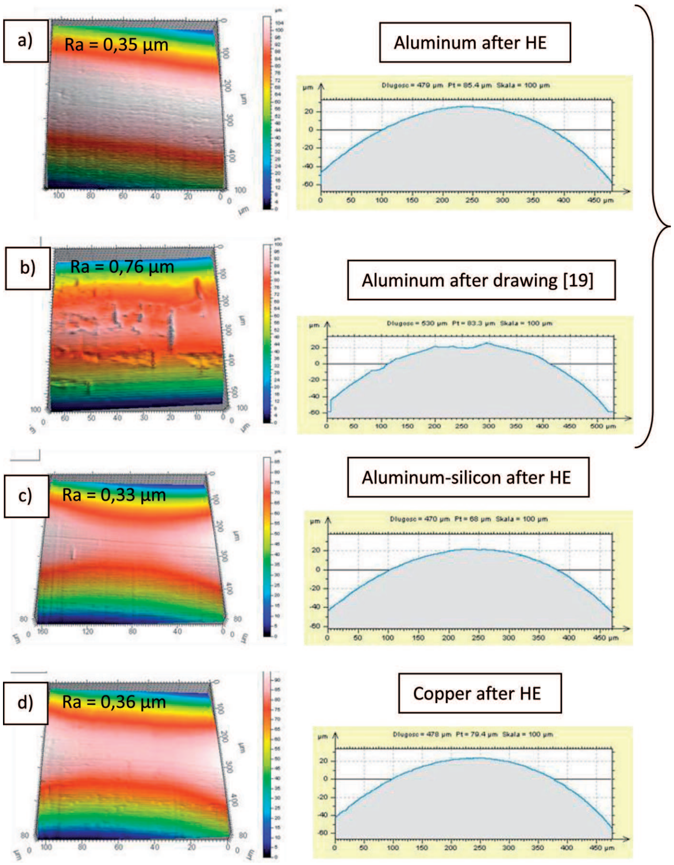

Fig. 8. Images of surface topography and roughness profiles obtained for the wires: (a) HE-processed aluminum, (b) drawn commercial aluminum, (c) HE-processed aluminum-silicon alloy, (d) HE-processed copper

4. Conclusions

Cold hydrostatic extrusion, which is a severe plastic deformation (SPD) method, was used for producing aluminum, aluminum-silicon alloy, copper, and silver wires with diameters of about 1 mm. The true strains applied in one-pass operation were 4 and 6. During the extrusion, a strong adiabatic heating effect took place, ranging from 0.36 to 0.7 of the homologous temperature.

In all the HE-treated materials examined the microstructure was refined, with the refinement degree being the smallest in copper because of its susceptibility to recrystallization.

Compared with the yield stress of the drawn commercial wire, the yield stress of the HE-treated aluminum was higher by more than 140% and that of the HE-treated silver – by 25%. In copper and in the aluminum-silicon alloy a converse effect was observed: the strength of the drawn commercial wires was higher than that of HE-extruded wires. In copper, this can be attributed to its susceptibility to recrystallization associated with the low stacking fault energy, whereas in the aluminum-silicon alloy – to the more effective deformation strengthening during the drawing operation.

The advantageous conditions of lubrication prevailing during the hydrostatic extrusion and the possibility of shape control ensured that the wires thus produced had a smooth surface and very good circularity. We can conclude that for certain applications that require specific wire parameters, the commercial wires with a diameter of about 1 mm can be successfully produced by hydrostatic extrusion.

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