

# Specific Features of The Structure Formation of Aluminum Alloys During Rapid Crystallization-Deformation

**Sergey Borisovich Sidelnikov**

Siberian federal university

**Ekaterina Sergeevna Lopatina**

Siberian federal university

**Denis Sergeevich Voroshilov** (✉ [d.s.voroshilov@gmail.com](mailto:d.s.voroshilov@gmail.com))

Siberian Federal University School of Non-Ferrous Metals and Material Science: Sibirskij federal'nyj universitet  
Institut cvetnyh metallov i materialovedenia <https://orcid.org/0000-0002-1406-3665>

**Nikolay Nikolaevich Dovzhenko**

Siberian federal university

**Igor Lazarevich Konstantinov**

Siberian federal university

**Yulbarskhon Nabievich Mansurov**

Tashkent State Transport University

**Vadim Mikhaylovich Bepalov**

Siberian federal university

**Aleksandr Vasilyevich Dumopyanov**

Siberian federal university

**Roman Ilurovich Galiev**

Siberian federal university

**Marina Vladimirovna Voroshilova**

Siberian federal university

**Andrey Sergeevich Bersenev**

Siberian federal university

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## Research Article

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## Abstract

The results of studies of the structure and properties of semi-finished products from aluminum and its alloys, obtained with the use of cast-free rolling-extruding are presented. It has been found that the rods obtained by the high-speed crystallization-deformation technology by the direct rolling-extrusion method have a stable ultrafine subgrain structure, which makes it possible to use them as modifiers. Experimental studies have been carried out, which confirmed the assumption that the initial structure of the modifying rod affects the melt. It was revealed that the size and density of distribution of additional crystallization centers formed in the volume of the melt based on clusters are inherited from the original subgrain structure of the modifying rod made of aluminum or its alloys. Metallographic studies have also shown that the subsequent severe plastic deformation by equal-channel angular extruding of rods obtained by direct rolling-extruding from an experimental alloy of the composition Al-0.2Zr-0.2Fe-0.4Mg makes it possible to achieve additional strengthening of the metal, since even more refines its structure, while the average grain size is 647  $\mu\text{m}$ .

## Introduction

Aluminum alloys are currently widely used in industry for the production of rolled products, extruded semi-finished products and cable and wire products [1]. To cast and deformed semi-finished products from them, increased requirements are imposed on the structure of the metal, mechanical and electrophysical properties, which is achieved, inter alia, by modifying ingots from these alloys using various ligatures [2–4].

A liquid crystallizing metal is characterized by a cluster structure that changes with decreasing temperature, and the formation of a cluster structure of a liquid upon melting of a solid metal is directly related to the initial grain and subgrain structure of the melting crystals. The subgrain structure should provide a larger number of elements of the melt structure (clusters), and hence a larger number of nuclei during crystallization. This is especially important when obtaining modifying materials in which the inherited elements of the melt structure (clusters, dispersed particles) are potential nuclei and crystallization centers [5–7].

It is possible to obtain in semi-finished products a fine grain structure and dispersed intermetallic compounds using various methods of affecting the metal: physical, physicochemical, thermal, magnetic, ultrasonic, mechanical, etc. [7].

So, for example, in works [8–10] it is proposed to use ultrasound or additives of nanopowders for this purpose.

Another such method for obtaining a fine-grained metal structure is the rapid crystallization of a metal melt, including the production of micro-ingots (granules), which can then be subjected to a certain plastic deformation [11–13].

Recently, the method of electromagnetic crystallization has become widespread [14, 15], in which the effect of electromagnetic forces on the melt makes it possible to keep liquid aluminum in the inductor. Due to the fact that electromagnetic and hydrostatic pressures are in equilibrium at the crystallization front, the height of the liquid phase remains constant during the casting process. Due to the high cooling rates affecting the crustal zone of the ingot, a uniform, fine-grained structure of the cast ingot is ensured. That is why high values of cooling rates can significantly improve the physical and mechanical properties of the cast ingot.

The authors have proposed another method [16] and a device [17], which makes it possible to obtain an ultrafine grain and stable subgranular structure of the metal due to the simultaneous high-speed crystallization of the melt

and its deformation using the operations of casting, rolling and extrusion. This method was called ingotless rolling-extrusion (IRE) [18, 19] and its essence is as follows (Fig. 1). Molten metal is poured into the mixer furnace 1. At the same time, rolls 3 and 4 with cavities for cooling 5 are brought into rotation, forming a closed caliber, which at the outlet is covered by a die 6. Crystallization of the melt begins on the surfaces of the rolls due to their cooling with the help of a coolant supplied in the cavity 5. The crystallized metal is captured and deformed by rolls in a closed caliber and through the iron-graphite insert 8 located in the die 6 is squeezed out in the form of a extruded product 10.

The technology of high-speed crystallization-deformation used in this case [20, 21] makes it possible to obtain a non-recrystallized structure, which is confirmed by the results of electron microscopic studies of thin foils (Fig. 2).

Analysis of the structure of foil samples cut from A7 aluminum rods obtained by the BPP method shows that there are individual grains and groups of grains with a high intragranular dislocation density, about  $10^9 \text{ cm}^{-2}$  (Fig. 2b, c, e-g), which indicates an uneven distribution of the dislocation structure. A sufficiently high density of dislocations inside subgrains is retained during warm deformation, which is explained by a slight decrease in the deformation temperature at the stage of extruding in the technology of obtaining a rod. Measurement of all subgrains in electron microscopic images of the structure in the lobar and transverse directions showed that the average size of subgrains along the axis of the rod is  $1.3 \mu\text{m}$ , while the number of subgrains with a size of  $< 1 \mu\text{m}$  was equal to  $\approx 70\%$ , the rest were  $> 1 \mu\text{m}$ . The average subgrain size in the transverse direction was  $1.2 \mu\text{m}$ , with a predominant size of  $0.7\text{-}1.0 \mu\text{m}$ . Consequently, the technology for producing rods by the direct rolling-extruding method provides a stable subgrain structure.

Bars with such a structure can be used to modify ingots from aluminum-based alloys [21]. To study the effect of the structure of the modifying rod on the melt, experimental studies were carried out in which aluminum grade A7 was melted at a temperature of  $720^\circ\text{C}$ , and then a rod 3 mm in diameter made of aluminum grade A7, obtained by the BPP method, was introduced into the melt, preventing its complete dissolution. When the rod was introduced, the melt was not stirred. Cooling was carried out in air. For metallographic studies, the resulting ingot was cut vertically along the axis of the introduced rod. Microstructural studies (Fig. 3) revealed that, depending on the conditions of reflow and subsequent crystallization, a fundamentally different structure is observed in different volumes of the solidified metal. This difference is observed in the region of the melting rod, in thin layers of crystallizing aluminum adjacent to the surface of the rod, and in the region of liquid initial aluminum, which is not affected by the modifying rod.

In the zone, the unmelted part of the bar, a fibrous structure is found inherited from the original deformed bar (Fig. 3a). A fine-grained unrecrystallized structural state of the metal is observed in the reflow zone of the bar and in the adjacent zones of the crystallizing liquid, which perceive the direct influence of the modifying bar, both along the lateral cylindrical surfaces and under the end of the bar, which is comparable in size to the structural elements of the structure of the reflowed bar (Fig. 3b-d). Far from the boundary, between the liquid and the introduced rod, an equiaxed coarse-grained structure is observed (Fig. 3f). Consequently, the liquid formed during the reflow of the introduced rod interacts with the crystallizing melt, changing the kinetics of the crystallization of the melt upon cooling. This affects the size of the crystals formed in the zones of the liquid adjacent to the former surface between the bar and the liquid, diffusively interacting with each other within a period of several seconds after the start of solidification of the metal. In areas inaccessible to the diffusion penetration of the liquid of the melting modifier rod, the grain size remains large, unmodified.

Analysis of changes in the microstructure along the axis of the melting rod shows that after the rod is immersed in the melt, the boundaries of recrystallized grains that form before melting are the first centers of melting of the solid rod. The body volumes of these grains transform into such elements of the melt structure (clusters), which at the moment of solidification of the sample become the nuclei of new crystallizing grains. This pattern of structure formation is observed not only directly in the volumes corresponding to the bar, but also in the adjacent volumes of the main liquid, i.e. in those layers in which, in a very short period of time from the reflow of the rod to the beginning of crystallization, cluster formations of liquid formed during the dissolution of the rod can penetrate.

Thus, it was revealed that the structure of the solid melted rod material introduced into the melt can influence the development of the solidification processes of the main crystallizing liquid. Therefore, by controlling the structure of the ligature rod, it is possible to influence the refinement of the grain structure of the ingot.

The cluster structure of the liquid arising during the reflow of the modifying rod is formed by the formation of interlayers of disordered atoms not along the grain boundaries, but along the subgrain boundaries, which significantly increases the number of clusters formed during melting, affecting the subsequent crystallization of the modified ingot [21].

To confirm this assumption, experimental studies were carried out in which rods obtained by different technologies and, accordingly, having a different initial structure were introduced into the aluminum melt. The rods were obtained from A5 grade aluminum by the IRE method and by the technology of continuous casting and rolling on a casting and rolling unit (CRA), the main difference of which from the IRE method is that the crystallized billet undergoes deformation. In the zone of bar reflow (Fig. 4a, b, e, f) and in the adjacent zones of the crystallizing liquid, which perceive the influence of the modifying bar, a fine-grained state of the metal is observed. A little further from the melting zone of the rod, one can observe the presence of both small and larger grains (Fig. 4c, g). Far from the boundary, between the liquid and the introduced rod, an equiaxed coarse-grained structure is observed (Fig. 4d, h). In areas inaccessible to the diffusion penetration of the liquid of the melting modifier rod, the grain size remains large, unmodified.

Studies have shown that rods obtained by various technologies grind the grain of the ingot, but to a different extent. A bar made by the IRE method provides finer grain throughout the entire volume of the ingot than a bar produced by CRA. This confirms the assumption that the original structure of the modifying rod affects the melt. The sizes and density of distribution of additional crystallization centers formed in the volume of the melt on the basis of clusters are inherited from the original subgrain structure of the modifying rod. Therefore, in order to effectively refine the grain in the ingot, the modifying rod must have a stable ultrafine subgrain structure. Obtaining such a bar structure is possible using the high-speed solidification-deformation technology.

The formation of an ultrafine grain structure and high performance properties of longish semi-finished products provides the combined application of the IRE technology and severe plastic deformation (SPD) [21, 22]. The object of the study was a rod with a diameter of 9.5 mm from an experimental alloy of the composition Al-0.2Zr-0.2Fe-0.4Mg, obtained using the IRE method, and a wire with a diameter of 2.0 mm, manufactured by the method of equal channel angular extrusion on an ECAE installation - Conform (Fig. 5).

The results of studying the microstructure of deformed semi-finished products obtained by the BPP method are shown in Fig. 6, and the ultimate tensile strength  $R_m$ , elongation to failure  $A$ , microhardness HV and electrical resistance  $\rho$  of the rod and wire of various diameters are given in Table 1.

Metallographic studies of rods and wires have shown that their microstructure consists of grains of the  $\alpha$ -solid solution elongated in the direction of deformation and eutectic inclusions of phases such as Al-Fe-Si and Mg<sub>2</sub>Si.

An increase in the degree of cold deformation leads to an increase in strength characteristics up to 250 MPa, microhardness up to 63 kgf/mm<sup>2</sup> and a decrease in plasticity up to 2%. The obtained values of the specific electrical resistance are 0.0335 Ohm·mm<sup>2</sup>/m for rods and 0.0342 Ohm·mm<sup>2</sup>/m for a wire with a diameter of 2 mm.

Table 1  
– Average values of mechanical and electrophysical properties of deformed semi-finished products from a test alloy after IRE

Semi-finished product	$R_m$ , MPa	$A$ , %	HV, kgf/mm <sup>2</sup>	$\rho$ , Ohm·mm <sup>2</sup> /m
Rod: diameter 9.5 mm	145	26	42 ± 1.7	0.0335
Wire: diameter 3.9 mm, diameter 2.0 mm	206	3	–	–
	250	2	63 ± 1.2	0.0342

Microscopic analysis by transmission electron microscopy (TEM) showed that after treatment of the samples by the ECAE method, an ultrafine grained (UFG) structure of the alloy is formed (Fig. 7), and the quantitative analysis of the fine structure made it possible to estimate the average grain size after SPD, which was 647 nm (Fig. 8).

This change in structure, in turn, leads to a significant hardening of the rods. The use of SPD, which is characterized by a higher intensity of plastic deformation, makes it possible to achieve additional hardening of rods and wires due to the formation of an ultrafine structure in them.

## Summary

Thus, the use of high-speed crystallization-deformation (the IRE method) and the use of the ECAP method in the future ensures the production of new materials from aluminum and its alloys in the form of:

- rods-modifiers, having a stable fine subgrain structure of the metal, which is necessary for the effective modification of ingots from aluminum alloys;
- longish deformed semi-finished products (rods, wire rods, profiles and wire) with an ultrafine metal structure and a high level of mechanical and electrophysical properties;
- other deformed semi-finished products (strips, profiles, etc.) of small cross-section for the needs of various industries.

## Declarations

### Ethical Approval

The work contains no libelous or unlawful statements, does not infringe on the rights of others, or contain material or instructions that might cause harm or injury.

## Consent to Participate

The authors consent to participate.

## Consent to Publish

The authors consent to publish.

## Authors Contributions

The authors declare that they are all participants in the work and none of them performed only administrative functions.

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## Competing Interests

The authors declare about the absence of competing interests.

## Availability of data and materials

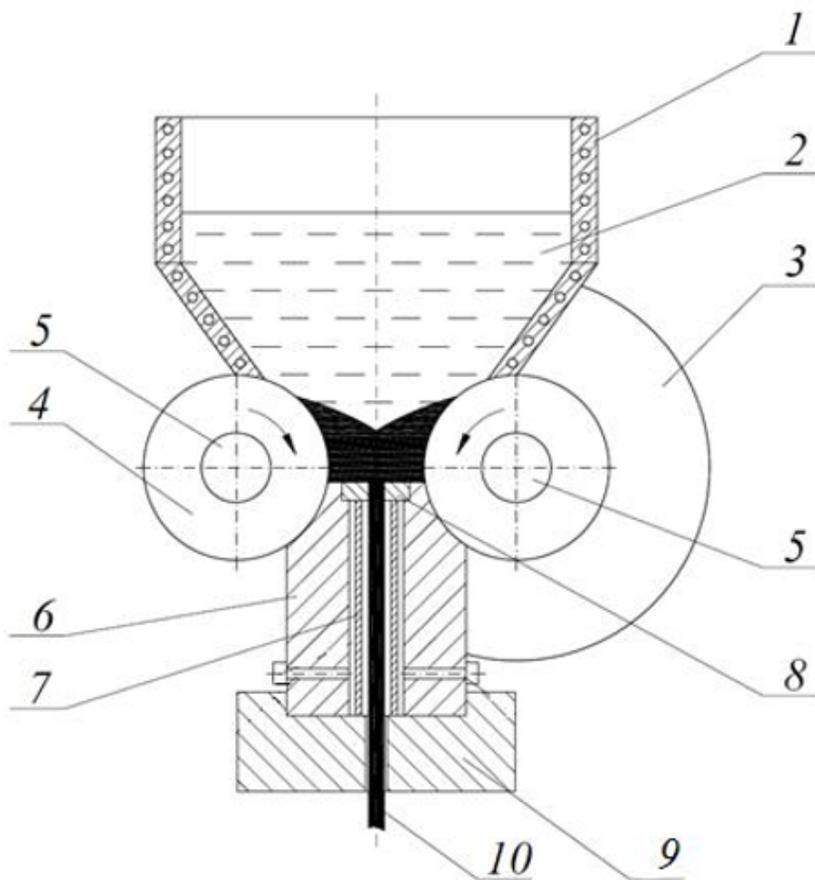
Not applicable.

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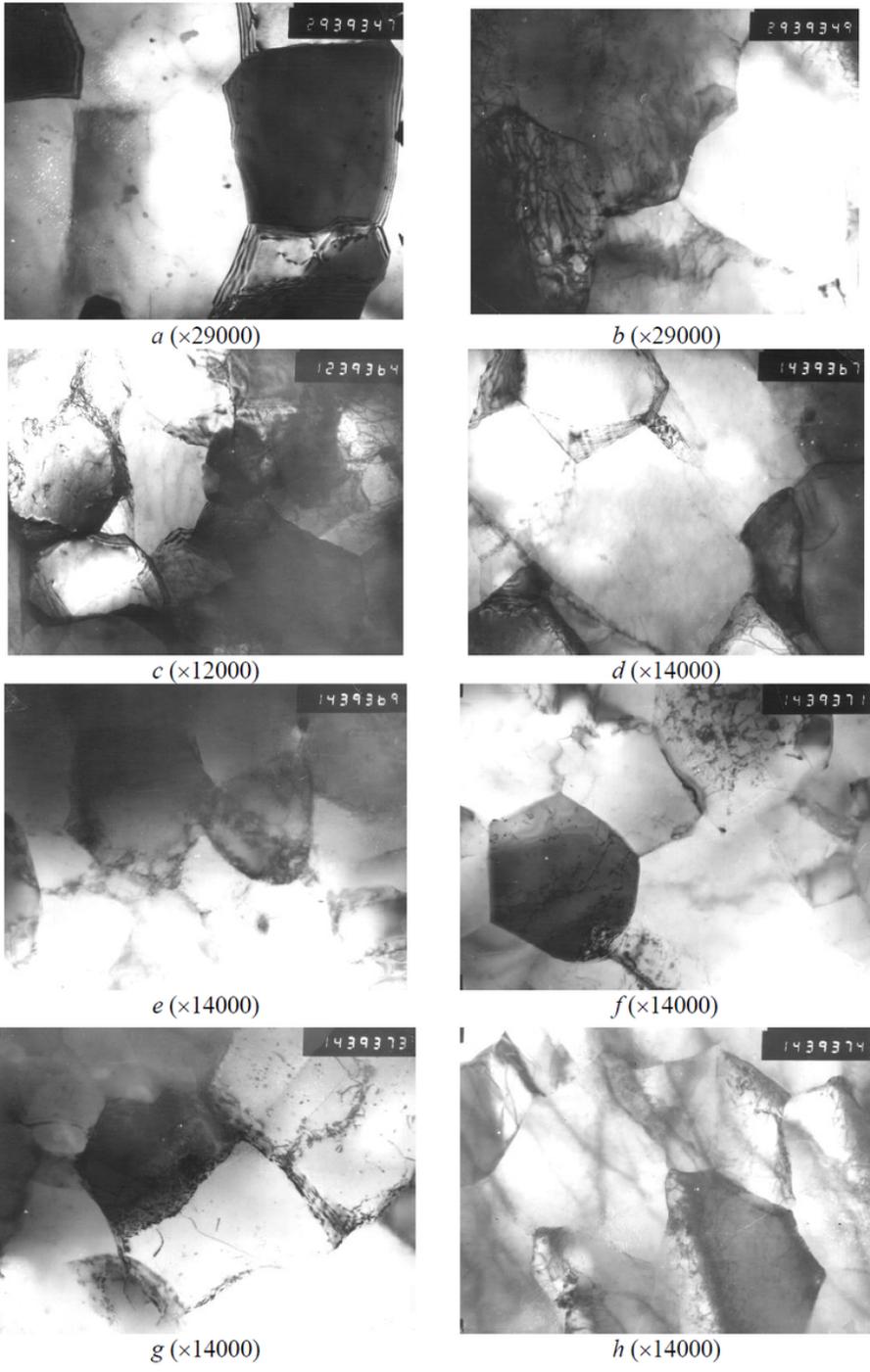
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## Figures



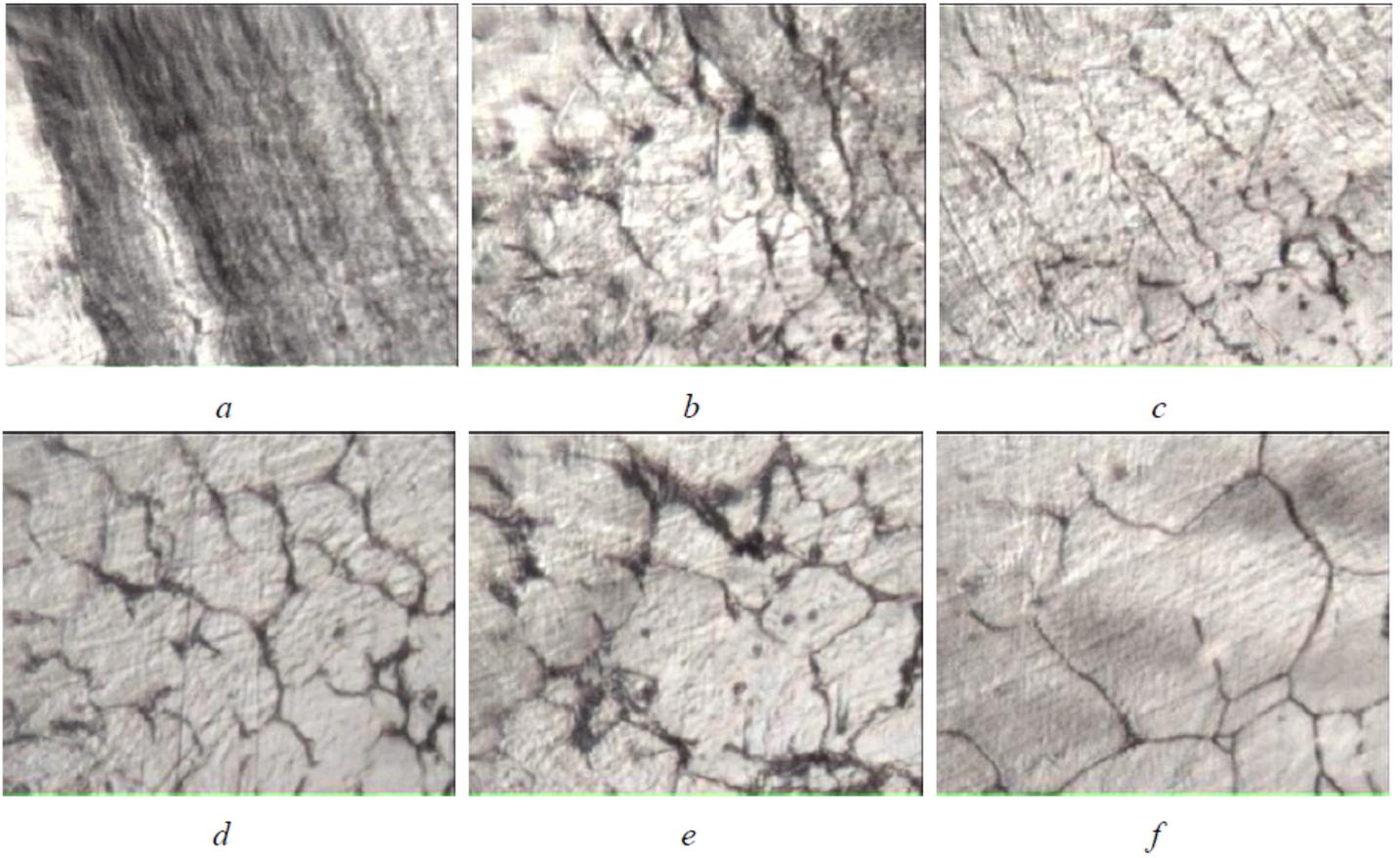
**Figure 1**

Device for ingotless rolling-extrusion [18, 19]: 1 – mixer furnace; 2 – melt, 3 – groove roll; 4 – roll with protrusion, 5 – cooled cavities in rolls, 6 – water-cooled die; 7 – pipes for supplying refrigerant; 8 – iron-graphite insert; 9 – die holder; 10 – extruded product



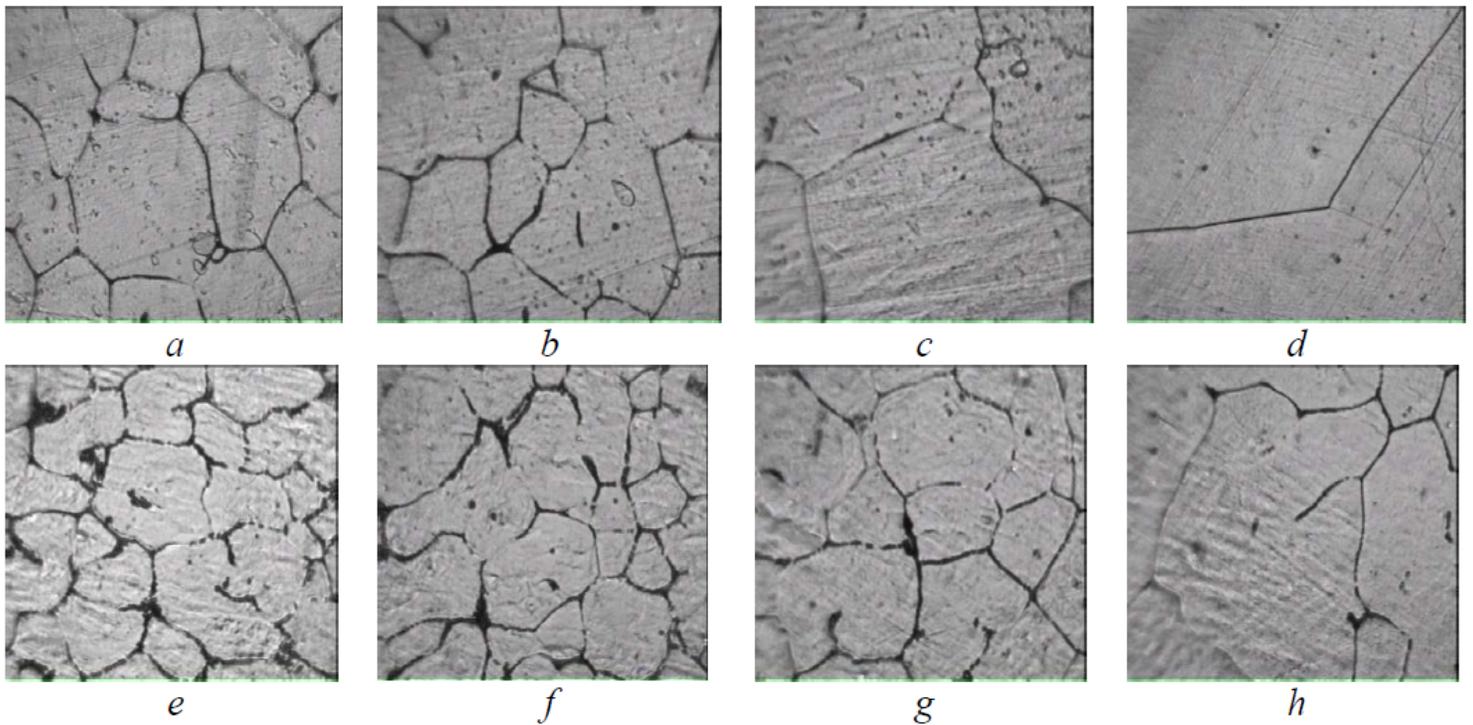
**Figure 2**

Fine structure of a rod with a diameter of 7 mm from A7 alloy, obtained by the IRE method: a-d – longitudinal section; e-h – transverse section



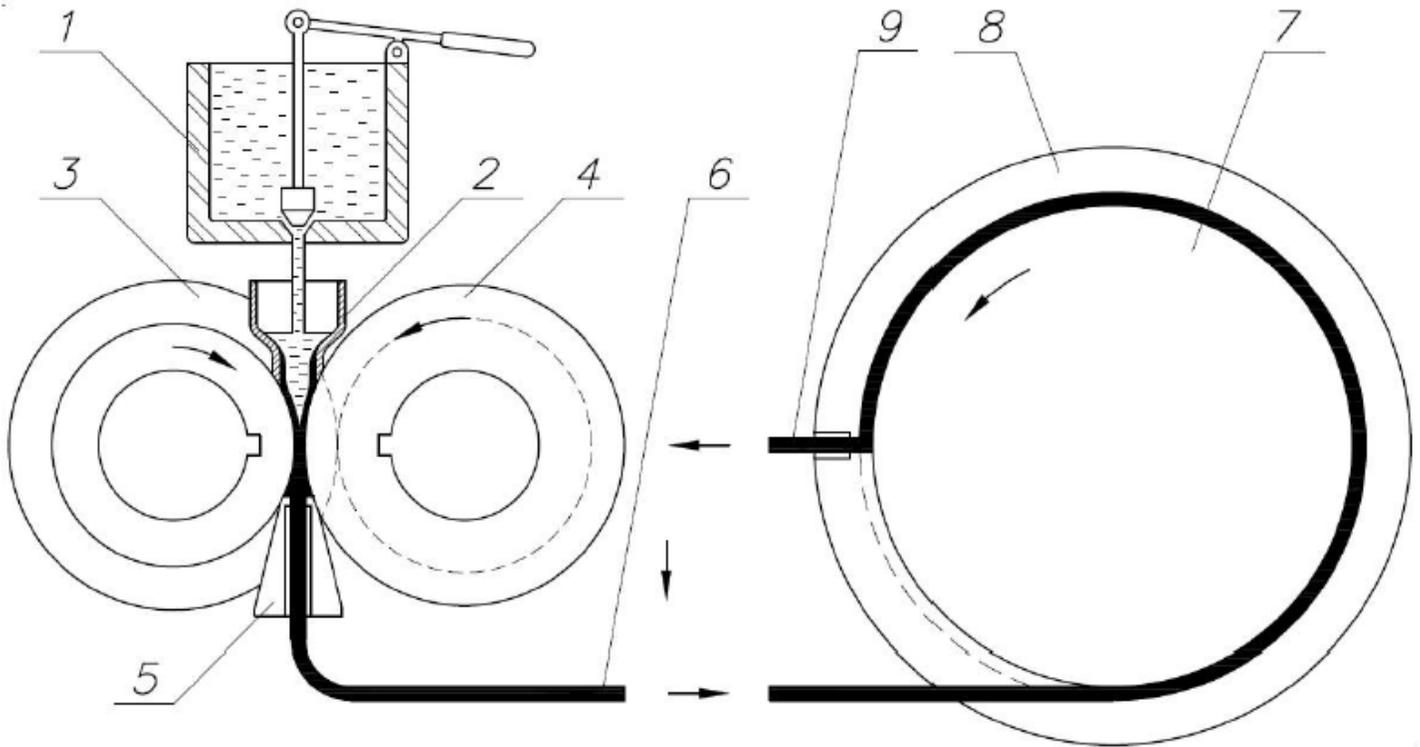
**Figure 3**

The microstructure of an A7 ingot ( $\times 640$ ) modified with a bar obtained by the IRE method: a – a bar; b – the dissolution of the bar; c – melting the bar; d, e – structure under the bar (small and large grains)



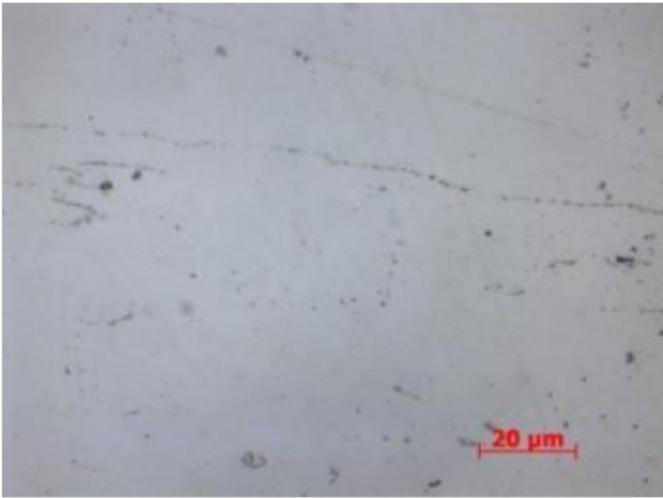
**Figure 4**

Microstructure of a high purity aluminum ingot, modified with an A5 bar, obtained by different technologies: a-d – on CRA; e-h – IRE, ( $\times 100$ )

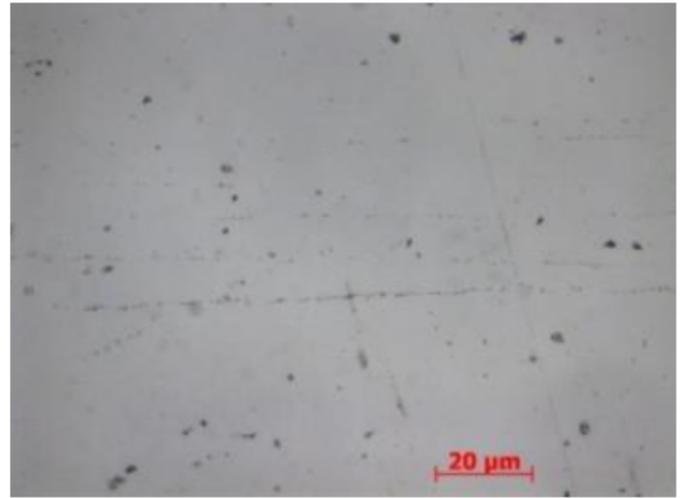


**Figure 5**

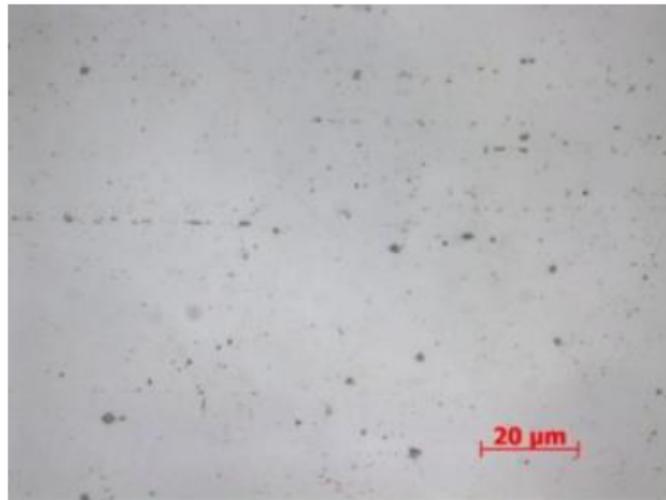
A device for experimental research using the IRE and ECAE-Conform methods: 1 – mixer furnace; 2 – funnel; 3 – roll with a groove; 4 – roll with protrusion; 5 – die with a longitudinal channel; 6 – billet; 7 – wheel of the ECAE-Conform installation; 8 – fixed block of the ECAE-Conform installation; 9 – rod



*a*



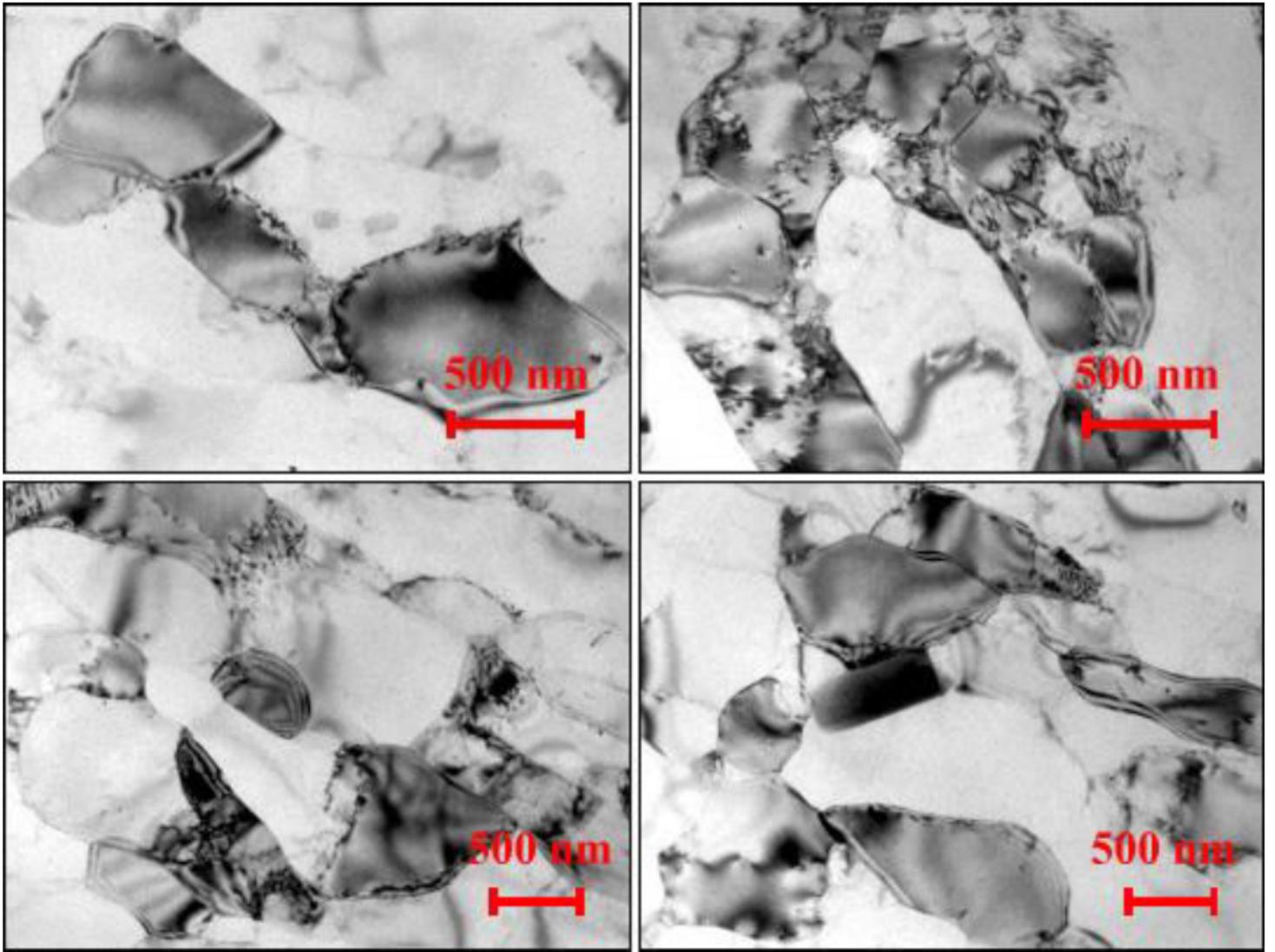
*b*



*c*

**Figure 6**

Microstructure of rods (a), obtained by the IRE method, and wires (b, c) from experimental alloy 9: a – diameter 9.5 mm; b – diameter 3.9 mm; c – diameter 2 mm



**Figure 7**

Microstructure of the wire obtained from the test alloy

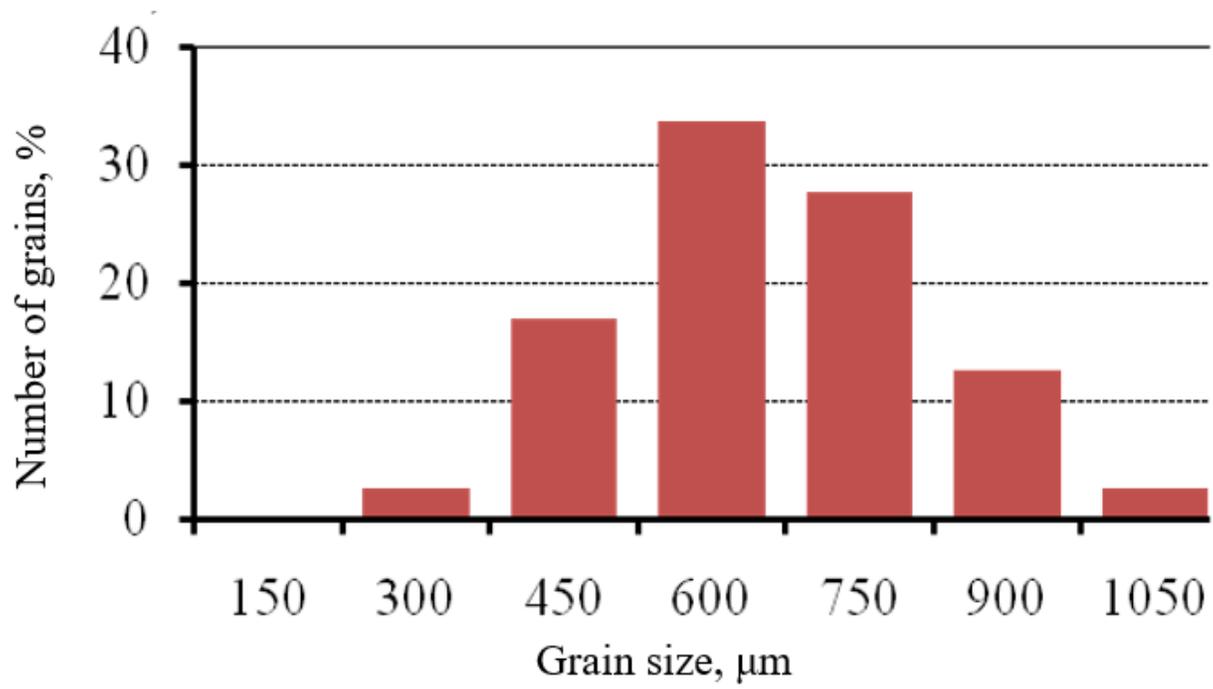


Figure 8

Grain size distribution in samples after ECAE treatment