

PRODUCTION OF SUPER LARGE TUNGSTEN SINGLE CRYSTALS

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Tungsten is characterized by extremely high and stable mechanical and physical properties over a wide range of temperatures. In the single crystal state, it exhibits anisotropy of properties that significantly increase the performance properties of tungsten products, taking into account the orientation of the structure in the individual elements.

Traditionally, tungsten single crystals are grown as rods with a diameter of 25-30 mm. Increasing the linear size of single crystals presents two problems: the retention of a large liquid volume metal and high thermo-mechanical stresses in the body of a single crystal. Temperature gradients create thermal stresses in the crystal that in critical cases can destroy the crystal [1]. High thermo-mechanical stresses contribute to the generation of an additional number of dislocations (the dislocation density can reach $10^7 - 10^8 \text{ cm}^{-2}$) and significant disorientation of the sub-grains, which significantly degrades the quality of the structure of single crystals.

The idea of simultaneous use of two different in nature and energy concentration of electric heating sources - plasma and induction was first proposed by specialists of the Institute of Electric Welding, EO Paton NAS of Ukraine (Fig. 1) [2].

The additional heating of the crystal significantly reduces the radial and axial temperature gradients, which helps to reduce the density of dislocations and internal stresses and helps to

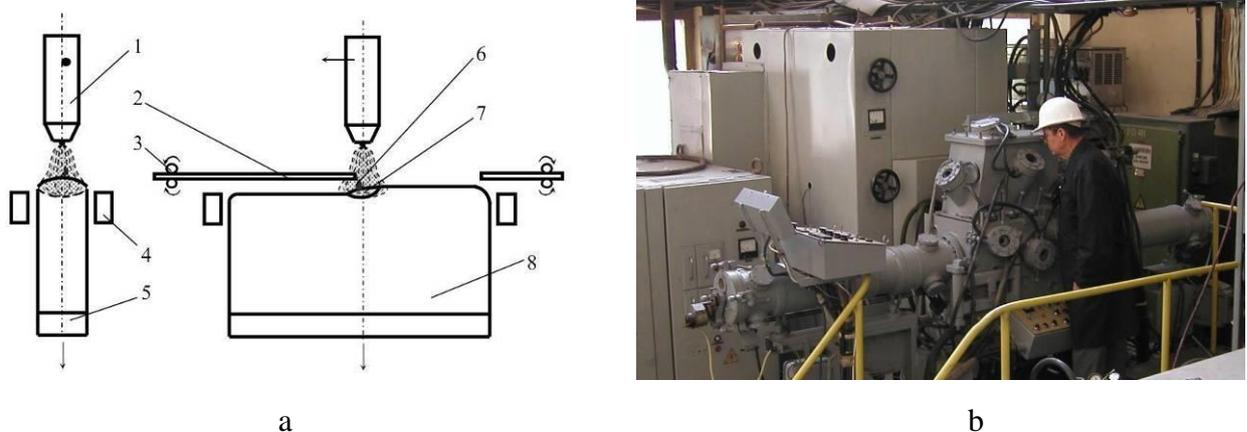


Fig. 1. Scheme of installation (a) and the equipment (b) for the additive growing of single crystals of refractory metals using the plasma-induction method: 1 - plasmatron; 2 - remelted rod; 3 - the mechanism of feeding rods; 4 - inductor; 5 - seed crystal; 6 - plasma arc; 7 - local bath melt ; 8 - single crystal

form a more perfect structure.

The essence of the method is that the plasmatron, in the reciprocating motion, moves a metal bath, which, receiving the metal from the melting rods, forms a crystal layer by layer, reminiscent of the nature of the arc surfacing. After each passage of the plasmatron, the single crystal goes down to the height of the deposited layer, thus providing stable conditions for the growing process.

Using this method, equipment and technology for growing profiled single crystals in the form of plates were created (Fig. 2).

The crystal is formed under conditions of heating by the high-frequency field of the inductor to the temperatures characteristic of the hot deformation range. It is known that at these

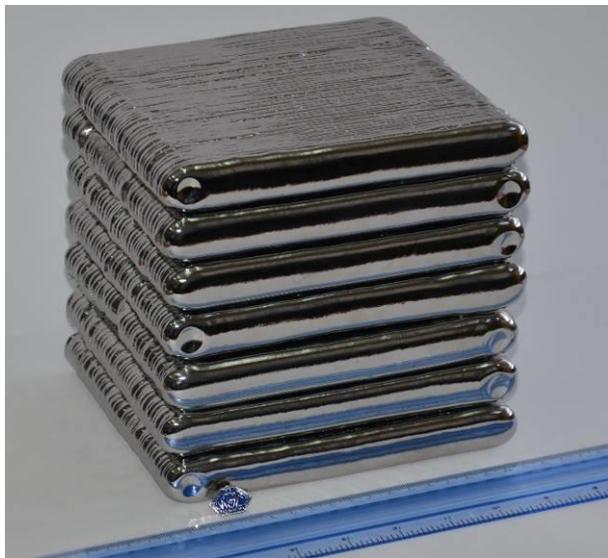
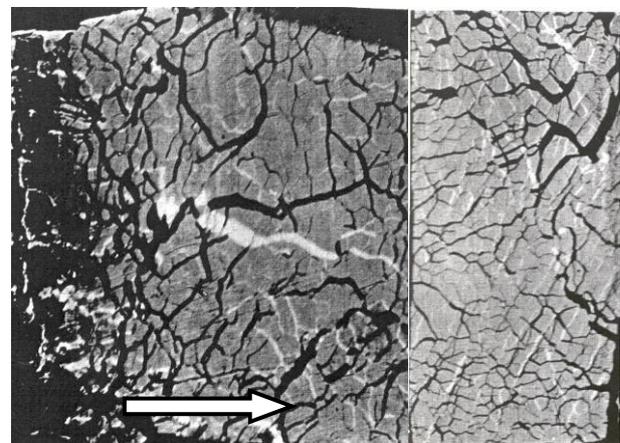


Fig. 2 Appearance of flat tungsten single crystals 170x160x20 mm



growth direction

a

b

Fig. 3. X-ray topograms by the Berg-Barrett method; longitudinal (a) and transverse (b) cross section of the crystal

temperatures the movement of dislocations occurs under the action of both external stresses and temperature. Dislocations are not rigidly tied to "their" slip plane and can move from one plane to another, choosing the easiest path. This is seen as an additional degree of freedom of dislocations. With such unregulated movement of dislocations, the probability of their meetings increases, and therefore, on the one hand, the number of cases of their annihilation increases (the density of dislocations decreases), and on the other - the tendency to form regular dislocation structures, characterized by the unification of dislocations within low-angle boundaries. The conditions under which the formation of the single crystal occurs provide a higher quality of the single crystal structure than in the ways in which no additional heating (electron beam and plasma arc) is used.

Crystals grown using the above method have a less smooth lateral surface, but this does not prevent their use without additional machining as billets for widescreen rolling.

Interesting are the results of the study of the structure of crystals using optical and x-ray methods. In Fig. 3 shows the results of the X-ray study of single crystals.

Comparative studies have shown that the structure of such crystals is more perfect than plasma-arc, and there is practically no macromosaic.

The developed additive technology for the cultivation of large single crystals of refractory metals of technical purity is based on long-lasting thorough researches that allowed to establish and study:

- distribution of thermal fields of single crystals using mathematical models and experimental data [3];
- working ranges of changing technological parameters of the crystal growth process;
- structural characteristics and patterns of structure formation of grown single crystals [4].

Further study of the process allowed to create on the same basis the newest installation for growing super-large single crystals of tungsten in the form of bodies of rotation (Fig. 4), where the arrows indicate the rod movement direction into the melting zone of the plasma arc and the direction of rotation of the single crystal.



Fig. 4 The newest equipment with computer-controlled unit (a) for the production of single crystals of refractory metals of rotation bodies (b)

The equipment created is a qualitatively new generation of equipment, featuring a fully computerized control system for actuators, motion sensors and single crystal growth control. The design provides for the possibility of growing single crystals in the form of rotation bodies (cylinder or hollow cylinder) with an outer diameter of up to 100 mm (4 inches). Now a thermal module has been created and the technology of growing tungsten single crystals diameter 85 mm is being developed (Fig. 5).



Fig. 5 The appearance of a tungsten crystal with a diameter of 85 mm

Fundamentally the technology of growing cylindrical single crystals is based on the technology of growing flat single crystals, but in the new installation, the crystal is continuously rotated around the vertical axis. The crystal seed used is a cylindrical workpiece made of a flat single crystal of predetermined orientation.

Single crystals of refractory metals (tungsten, molybdenum) can find an alternative application, both in the form of wide-format rolled metal (sheets, thermal screens), and in the form of articles made of ingots. The most promising applications may be: X-ray technology (electrodes), electronics (sputtering targets, crucibles),

electrical engineering (contacts), laser technology (mirrors for optical and X-ray lasers), nuclear power (thermionic power converters of space power plants, parts of structures reactors), aerospace engineering (nozzles).

References

1. G. Marinelli, F. Martinaa, S. Gangulya, S. Williams. Development of Wire + Arc Additive Manufacturing for the production of large-scale unalloyed tungsten components // *International Journal of Refractory Metals and Hard Materials*, Volume 82, August 2019, Pages 329-335.
2. V.A. Shapovalov, V.V. Yakusha, A.N. Gnizdylo and Yu.A. Nikitenko. Application of additive technologies for growing large profiled single crystals of tungsten and molybdenum // *The Paton Welding Journal* № 5-6, 2016 p.134-136.
3. V.A. Shapovalov, V.V. Yakusha, Yu.A. Nikitenko, V.V. Dolinenko, A.N. Gnizdylo, V.V. Zholud. Studying the temperature field of profiled tungsten single-crystals produced by plasma-induction process // *Advances in Electrometallurgy*, Translated from *Sovremennaya Elektrometallurgiya* 2014, №3. pp. 31–35.
4. G.M. Grigorenko, L.I. Markashova, E.N. Berdnikova, V.A. Shapovalov, E.V. Polovetskii, V.V. Yakusha, Yu.A. Nikitenko and A.N. Gnizdylo. Structure and properties of profiled tungsten single-crystals produced by method of plasma-induction growing // 6-th International Samsonov conference “Materials science of refractory compounds”. May 22-24, 2018. Kyiv, Ukraine. P.34.