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TECHNOLOGIES OF AFFINAGE FOR REFRACTORY RARE METALS V GROUP

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There are considered technologies of affinage for refractory rare metals of V group for the periodic system of elements: vanadium, niobium and tantalum. The methods of cleaning of vanadium and tantalum from impurities are expounded briefly with references in works, where these methods are considered with sufficient plenitude. Technologies of affinage for niobium are analyzed in detail with pointing of technological parameters for separate operations and estimation of efficiency of technology on the whole, the methods of electrolytic and iodide affinages, cathode-ray melting, floating-zone refining with the receipt of monocrystals and combined methods, allowing to get niobium of maximally high degree of cleanness for the special aims, are here considered.

Keywords: vanadium, niobium, tantalum, electrolytic and iodide affinage, cathode-ray and zone melting, electrotransfer, sprayed targets.

Vanadium. All methods of cleaning of vanadium from impurities divide into previous methods and methods of the deep cleaning. Electrolytic affinage in molten salts and cathode-ray melting take to the previous methods. An electrolytic affinage is applied for cleaning, both draft vanadium and wastes of vanadic alloys. The cathode-ray melting is usually preceded to other methods which use for the receipt of high-clean vanadium, to which a vacuum distillation, zone melting and electrotransference.

The zone melting provides moving away of impurities, both after the mechanism of zone пере-кристалізації and by evaporation from the surface of molten zone. As initial materials it follows to use materials after them previous affinage be methodes, which execute cleaning from impurities, which difficult remove at the zone melting. For the receipt of cleanness high degree vanadium, niobium and tantalum it is recommended after the zone melting to execute the finish cleaning from gas and gaseous impurities: for vanadium - by electrotransfer, for niobium and tantalum - high temperature ultrahigh-vacuum ($1 \cdot 10^{-8}$ Pa) annealing. The use of such complex technology of affinage allows to get vanadium, niobium and tantalum of record degree of cleanness.

Niobium. Metallic niobium of technical cleanness is got by the methods of metal- or carbothermic reduction its pentaoxide. A cleanness of niobium is a critical factor in progress of production of high-frequency superconductivity resonators for accelerant. In high-clean niobium content of impurities which retire most difficult, - tantalum, tungsten, carbon, oxygen, nitrogen, hydrogen - it must fold about $1 \cdot 10^{-5}$ %. For the receipt of high-clean niobium the complex of methods, which foresees electrolytic and iodide affinage, vacuum distillation, cathode-ray and zone melting,

and also high-vacuum annealing is used. Niobium of ultrahigh cleanliness can be the got by melting in the stream of hydrogen plasma or method of hardphase electrolysis (electrotransfer).

At the receipt of refractory rare metals of high-purity practically an electrolytic affinage in molten salts is an obligatory process. Niobium with the enough high-purity (99,5 %) was got by means of electrodeoxidation of pentoxide niobium in molten salts (variety of process of FFC Cambridge Process). For the receipt of high-clean niobium use also iodide affinage.

Tantalum. For desoxydating of tantalum and tantalum scrap a carbon reparer can be applied, thus a quantity of carbon, that enter to the charge, must be more than 90 % from the stoichiometrical calculating on making monooxide of carbon (CO). Also for the deep cleaning of metallic tantalum from the impurities of oxygen and carbon use a plasma meltback in helium-hydrogen mixture of $He + (3-6 \%) H_2$. Decline of content of oxygen, and also sodium, potassium and fluorine in tantalum condenser powders, must be the attained by their heat treatments at presence of metals-deoxidants, which are characterized by high pressure of pair, for example to magnesium.

An electrolytic affinage of tantalum in molten salts is most effective at cleaning of it from the impurities of tungsten and molybdenum. At the same time the deep cleaning from the impurities of introduction, which it is most difficult to withdraw from refractory rare metals originates.

The affinage of tantalum by the method of a vacuum cathode-ray melting on one-two orders reduces content of most metallic impurities, here content of impurities of introduction goes down notably.