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Development of New Structural Materials with Improved Mechanical Properties and High Quality of Structures through New Methods Using New Type of Plasma Chemical Reactor

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Abstract Up-to-date science and technology requires further development and wide introduction of new high-performance processes to produce refractory metals. These may include plasma chemical technology of very fine powders production. Practical implementation of plasma chemical method in producing and processing of fine powders is in its initial stage. Along with this at the present time the demand for processing of structural materials with improved physical and mechanical properties is now steadily increasing. Such materials have low machinability due to high hardness and wear resistance at high temperatures which results in heavy wear of a cutting tool. To improve the efficiency when processing hard-to-cut materials it is necessary to enhance the tool's durability; this can be provided by application of new grades of hard alloys received from tungsten nanopowders. New alloy, obtained by the new developed technology, has higher degree of hardness and wear resistance compared with existing alloys and will be intended for hard materials processing.

Keywords: *tungsten oxide, mechanical properties of materials, powder materails, plasma apparatus, powders granulation*

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1. Introduction

Plasma chemical technology in comparison with the traditional one has a number of essential advantages, namely, large productivity, energy savings, environmental cleanliness and the possibility of complete mechanization and automation.

Tungsten ultrafine powders (UFP) have become common use in traditional technology production of goods and semi-finished products. Basing on them the materials have been obtained which due to their physical and mechanical properties surpasses by far serial production.

2. Method

In the Figure 1 plasma reduction apparatus (PRA-300) is a prototype specimen of serial plasma devices and operates at the workshop Nr.2 in JSC "Uzbekistan refractory and heat-resistant metals" in one - shift operating regime.

Raw material is fed by air transport into the hopper (6) from which under its own weight it continuously proceeds into four feeders (5). From feeders the raw material is moved by transporting gas through the input unit into a plasma jet into reactor 2 where the raw material's mixing with the plasma jet, heating, melting - evaporation, chemical reaction of reduction and powder condensation occur. Moving along the reactor, the powder particles collide with each other forming conglomerates that fall out of steam and gas flow and accumulate at the bottom of the settling chamber (3). The smallest particles of the powder are lifted out with the steam and gas flow to the filters (4), where the mixture clearing from the powder takes place. Purified steam gas flow is directed to a bleeder or recycle hydrogen shop collector for regeneration and further use. The apparatus provides separation of powders obtained into PRA and submicron. There are two points of load: 1) the settling chamber, and 2) the filter. Powder fraction collected in the settling chamber contains a number of sub-oxides, and therefore oxygen and water vapor content is 5 - 10% (weight). The average particle's size falls in the range of 0,8 - 1,0 mm. Tungsten anhydride in the apparatus PRA - 300 restores under the following parameters (Table 1).

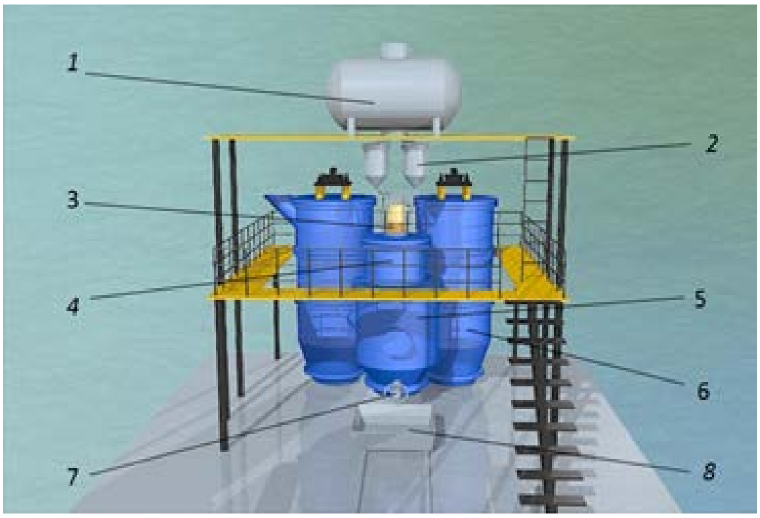

Figure 1. Plasma reduction apparatus (PRA - 300): 1 - plasma generator; 2 - reactor - 1 p; 3 - settling chamber, 4 - filters block, 5 - powder feeders; 6 - hopper for raw material; 7 and 7^1 - receiving hopper, consisting of a container and bogey; 10 - auger

Table 1. Tungsten anhydride recovery parameters

№	Parameter Name	Rate
1	Arc current, kA	from 0.45 to 0.55
2	Arc voltage, V	from 380 to 410
3	Consumption of hydrogen through the plasma jet, m^3/ h	from 60 to 70
4	Consumption of hydrogen for tungsten anhydride transportation, m^3/h	from 2 to 4
5	Water rate for the plasma jet, m^3/ h	from 2.15 to 3.6
6	Water pressure in the high pressure header, Pa kgc/cm^2	$8.73 x 10^5$ - $11.77 x 10^5$ 9-12
7	The temperature difference of softened water at the inlet and outlet of the plasma jet, °C, not more	20
8	Gas pressure in the unit, Pa, not more than kgc/c^2, not more	$0.98 x 10^4$ 0.1
9	Softened water pressure at the inlet to the water distribution comb, Pa, kgc/cm^2	$78.48 x 10^4$ 8
10	Industrial water pressure in the water pipeline of low pressure, Pa kgc/cm	$16.62 x 10^4$ – $39.24 x 10^4$ 2-4
11	Hydrogen pressure in the gas pipeline at the inlet to the gas distributing comb, Pa., no less kgc/cm^2, not less	$3.9 x 10^4$ 0.4
12	Softened water pressure at the outlet from plasma jet, Pa, not less, kgc/cm^2, no less	$(-1.76) x 10^4$ (-1.18)

The powders obtained have found application in the following types of products:

• powders brand 1.9 - 2.2 for ceramic metallization;

• tungsten powder brands PVV, PVO, PV1, PVPV and others for the production of technical tungsten powders.

The new type of plasma chemical reactor for hydrogen reduction of tungsten and molybdenum oxides has a distinctive feature of energy supply to the reaction zone. The energy is introduced not only in the form of plasma jet, but also as an additional stream, heated to a high temperature gas entering the reaction zone through the porous, permeable wall, heated by an electrical air heater.

The new type of plasma chemical reactor has been created, based on the following considerations. The process of plasma chemical reduction in the standard reactor, where d/D is equal to 1/10, is divided during no more than 0.03 seconds, and the plasma flow at free discharge in a large volume quickly loses thermal energy store, therefore some part of the powder that gets into the peripheral area of the jet remains underreduced. Thus, it is necessary to extend stay time of tungsten oxides particles in the hot area.

The constructive scheme of plasma chemical reactor is shown in Figure 2. Plasma jet with diameter of 30-40 mm is crimped by porous molybdenum tube with inner diameter of 40-50 mm, which is located coaxially in the porous tube made of stainless steel. Between the porous tubes there is a molybdenum heater for heating the flowing gas.

The above described construction is placed into a sealed case, where additional hydrogen is blasted, when heated between the porous tubes, crimps the plasma jet.

Technological researches of the new reactor were conducted on the strengthened plasma laboratory apparatus. Plasma capacity was kept within the range of 45-55 W, calorifer capacity was 16-18 kW, hydrogen consumption through the plasma generator was 20 m^3/h, through the heater was from 40 to 60 m^3/h. Consumption of tungsten oxide was 6-10 kg/h. Hydrogen temperature heated by the calorifer was 1500 -1600 C^0. Thus, the total capacity is not more than 75 kW, and the total hydrogen consumption is up to 80 m^3/h, while plasma jet capacity under the traditional scheme is 100 kW at a flow rate of hydrogen 75 m^3/h.

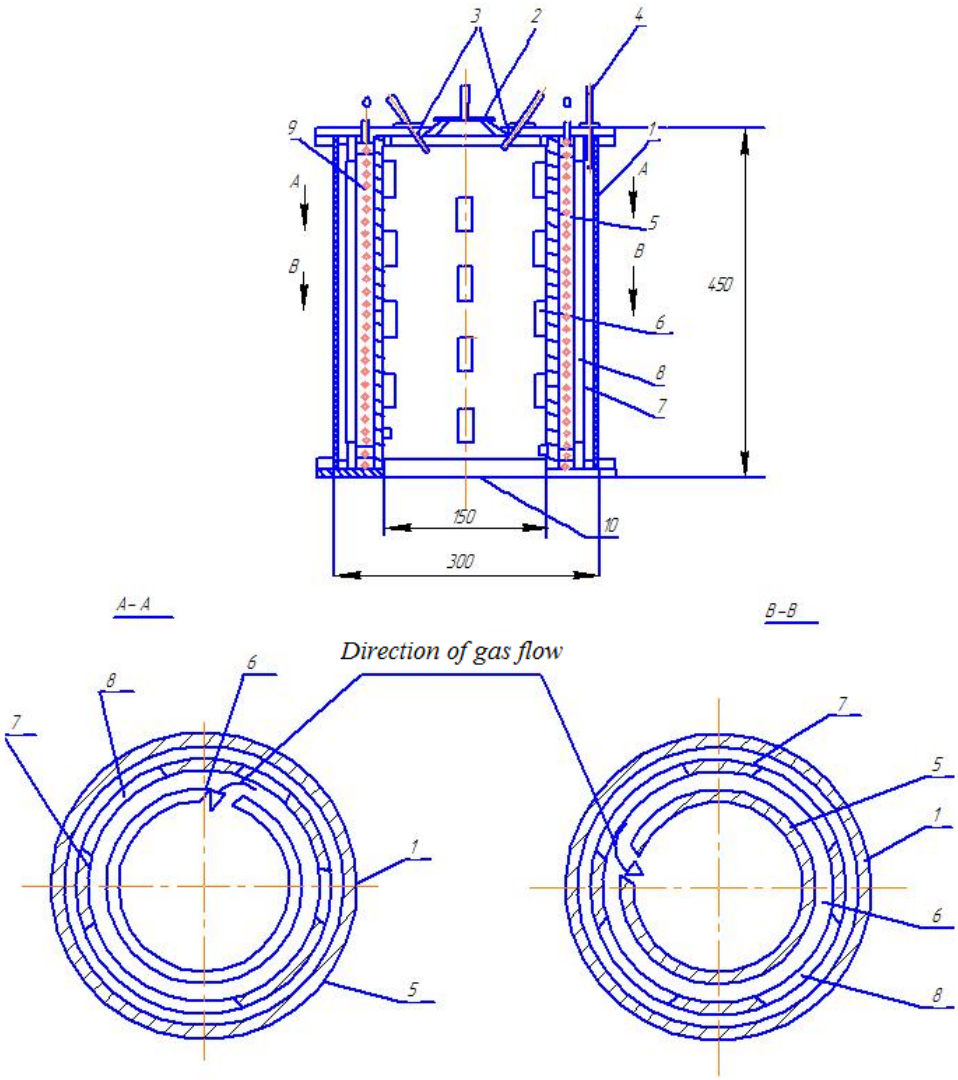

Figure 2. Plasma chemical reactor with a high degree of raw material processing**:** 1 - porous molybdenum cylinder, 2 – porous stainless steel cylinder; 3 - plasmatron A-26 (PG-2, I); 4 - case.

3. Results and Discussions

The study of the obtained powders showed that the average grain size is 0.07 - 0.09 microns with the content of oxygen and moisture vapor 1.5% wt (Table 2).

Table 2. The grain size and the mass fraction of oxygen in powders obtained in plasma-chemical reactor.

№/№	Extraction point	The grain size due to Fischer, micron	Mass fraction of oxygen and moisture vapor,%
1	W plasma from the filter	0.08	1.5
2	W plasma from the filter	0.09	1.5
3	W plasma from the settling chamber	0.09	1.5
4	W plasma from the filter	0.07	1.4

X-ray phase analysis showed the presence of β - tungsten up to 50%, I - Tungsten up to 35%, and the rest is tungsten oxide without amorphous phases.

In the process of powders production in the new reactor in the settling chamber under the reactor practically no powder was found; it testifies that the entire obtained tungsten oxide was reduced and UFP tungsten entered the filters.

Thus, proceeding from the analysis of technological tests of the new plasma chemical reactor one can come to the following conclusions:

• raw material processing degree increases up to 95%;

• significantly smaller and more active UFP has been received;

• the actual number of amorphous tungsten in the powders produced has been revealed;

• process control is increasing, including the dispersion of the powder;

• uniformity of grain size is increasing;

• coefficient of reduction process activity is increasing.

The process of tungsten anhydride reduction in the plasma chemical reactor occurs in the plasma jet of hydrogen due to the reactions:

$WO_3 + H_2 = WO_{2.90} + H_2O,$

$WO_{2.90} + H_2 = WO_{2.72} + H_2O,$

$WO_{2.72}+H_2=WO_2 + H_2O$,

$WO_2+H_2=W+H_2O$,

The apparatus has two points of powder discharge (Figure 1): settling chamber; filter with the powder accumulation in the settling chamber and filter it is discharged into the intake reservoirs. Depending on the variety of raw material and places of discharge one can obtain:

- plasma reduction powder - unannealed (from the filter);
- plasma reduction powder - unannealed powder (from the settling chamber);

Unannealed plasma reduction powder from the filter of black colour.

Unannealed plasma reduction powder from the settling chamber of gray-black colour.

The powder should not have cakes and balls.

Dispersity of the powder is characterized by an average particle size and must be not more than 0,4 microns. The powder mass loss at hydrogen calcinations is not more than 3%.

Before-reduced plasma powder has the following characteristics:

- oxygen content is 0.5%;
- average grain size is 0,2-0,4 micron.

Tungsten plasma powders are designed for the production of tungsten powders of various grades to improve the quality of products, to intensify the sintering process, fine-grained carbides and hard alloys.

The application of tungsten UFP obtained as an alloying component of many high-temperature and heat-resistant materials and the products received from them allowed to improve the quality of these products, to reduce the sintering temperature and lower energy consumption during the operation "Welding."

The study used plasma powders of tungsten, selected from the filter of the industrial apparatus PRA-300. Plasma tungsten powders specification is given in Table 3.

Table 3. Plasma tungsten powders specification

Powder	Oxygen content in %	Bulk density, g/cm^3	Specific surface area, m^2/g,
Tungsten 1	2.0	0.70	4.0
2	2.4	0.82	5.8
3	3.0	0.90	8.0

The oxygen content was calculated using the weight method, annealing powder in hydrogen stream at 900 °C.

Weight by volume was determined using volumeter and the specific surface was determined by the argon thermal desorption method.

Powders granulation was done in the system of “drunken barrel”. Pressing of industrial samples was done on the presses of P474A, P807 brands. Testing regimes of extraction, sintering, welding was carried out on the industrial equipment of shop 2 (PRA-300, CEP-214, CTN-1, 6).

Powders were used in the experiment that included the gases under mass- spectrometry data: O_2, H^2, W_2, H_2O, CO, CO_2.

Compact half-finished products preparing. The experiment was carried out using initial tungsten fine powders with introduction of a plasticizer (alcohol, glycerol, 1:1) into their composition. Mixing of powders with a plasticizer and at the same time their granulation was done out on vibromixer.

Formation of finely dispersed powders is associated with sharp increase in resistance to the punch, due to friction along the walls. Therefore, to obtain rods without compacting crack at pressing is practically impossible. Using methods of impact compaction does not give positive results too. The value of bulk density is shown in Table 4.

Table 4.

Powder	Ultrafine			Standard
	initial	Sifted	granulated	sifted
Bulk density W, g/cm^3	0.7-0.9	1.4-1.8	2.0-2.2	3.4-3.65

Quality rods are obtained from granular tungsten powders, the compaction process of which can be represented as follows: in the initial period of pressing the compaction occurs due to the movement of granules associated with the destruction of bridges and arches formed at the free fill of powder. This results in an increase in stress at the particles contact places, which in turn causes irreversible elastic deformation and brittle fracture of grains. Further compaction occurs under the usual scheme by filling the voids by the particles formed by the destruction of the granules.

Compacts with uniform density are obtained by hydrostatic pressing of powders in elastic shells due to the development of plastic deformation by brittle solids under all-round compression.

Specific features of formation and sintering of ultrafine tungsten powders. Recrystallized mechanism of sintering, based on the formation of the nonequilibrium vacancies excess concentration at recrystallization, can be resulted in considerable activation of self-diffusive processes. Shrinkage in the initial period of sintering occurs under two mechanisms of mass transfer: particles grain boundary sliding and diffuse - viscous flow (liquid-like coalescence).

Shrinkage in this case will depend on the initial porosity of compacts; at that the largest one will not necessarily be observed in the compacts with minimal porosity. Obtained data show that the most favorable values of pressing efforts for ultrafine tungsten powders are in the range 100-200 MPa.

Production of rods from ultrafine tungsten powders. 5% of plasticizer was added into superfine tungsten powder; powder granulation was carried out for an hour. Granulated powder was pressed into rods by section 12x12x500 at effort of 200 MPa. The density of compacts was 10.2 g/cm^3. The rods have been sintered in a furnace TSEP214 at 1373 ° K for two hours. Rods’ welding was carried out under the existing technology at various welding currents. Standard density can already be obtained at a welding current equal to 2.6 kA. The results of the welding rods from ultrafine tungsten powders are shown in Table 5.

Table 5.

Wn/n	Welding current, kA	Rods density, g/cm^3
1	2.2	17.0
2	2.4	17.8
3	2.6	17.9
4	2.8	18.0
5	3.0	18.2

Currently, the basic requirements for durable cutting materials include high hardness and uniform finely dispersed structure.

To equip the cutting tool the industry produces alloys of the type "M" (VKZ-M VK6-M, VK10-M) with a grain size of WC-phase 1.8 mm without alloy additions.

The developed new alloy has higher degree of hardness and wear resistance compared with existing alloys and will be intended for hard materials processing.

The most efficient means to create such an alloy are the use of tungsten nano-powders and WC-intensive grinding phase. The paper shows that the researches done revealed that the developed alloy is different from the standard fine alloys by its ultra-finely grained structure. Thus, the volume of WC-phase grain up to 1 micron is 80-85%, and in alloys VK10-M - 65-75%. The average grain size of WC-phase is 1.1 microns, and in the alloy VK10-M - 1, 3-1, 5 microns.

4. Conclusions

Based on the provided work it was identified that the new type of plasma chemical reactor for hydrogen reduction has significant advantage than the previous analogs, as there energy introduced not only in the form of plasma jet, but also as an additional stream. In the case of ne reactor the total capacity reduced to 75 kW, which was 100 kW previously and the total hydrogen consumption have been increased up to 80 m^3/h from 75 m^3/h.

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