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Research of methods of synthesis of carbidaungsten nanoparticles

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ABSTRACT: Fine-grained tungsten carbide hard alloys, when used in the manufacture of cutting tools, have higher operational properties compared to traditional hard alloys based on silicon carbide (grain size 3-8 microns). At the same time, in recent years, a whole class of low-dimensional tungsten carbide (WC) powders has appeared. These powders are classified as submicrometer (0.8 microns), ultradispersed (0.4 microns) and very fine (0.2 microns) and are already produced by the industry of leading countries and are used in hard alloys. The smaller the grain size of the tungsten carbide powder, the higher the quality of the products. At the same time, a sharp increase in the physical and mechanical properties of materials is expected only when using nanometer-sized powders (less than 100 nm). However, to date, there is still no industrial high-performance process for the production of tungsten carbide nanopowders.
This scientific study is dedicated to the development of a method for the synthesis of tungsten carbide nanoparticles. The method includes the synthesis of oxygen-containing tungsten compounds by hydrocarbons using an electric discharge plasma.

KEYWORDS: Anode, cathode, carbon, tungsten, resistance, carbide, plasma.

I. INTRODUCTION

Tungsten carbide (tungsten nanocarbide) is a chemical compound of carbon and tungsten with the formula WC. Represents the phase of implementation, which contains 6.1% C (by weight). It has a high hardness (9 on the Mohs scale) in terms of hardness, it is comparable to diamond. In addition to hardness, tungsten carbide has a high melting point and wear resistance.
Tungsten carbide is widely used in mechanical engineering for the manufacture of various metal cutting tools such as cutters, drills, milling cutters, abrasive discs and other cutting tools requiring high hardness and corrosion resistance.
Currently, for the manufacture of cutting tools, fine-grained tungsten carbide hard alloys are used, which have high operational properties. In recent years, a whole class of low-dimensional tungsten carbide (WC) powders have been increasingly used.

II. SYSTEM ANALYSIS

Tungsten carbide can be obtained in one of the following ways.
1. Direct saturation of tungsten with carbon;
2. Reduction of tungsten oxide with carbon followed by carbidization;
3. Reduction of tungsten compounds followed by carbidization;
4. Electrolysis of molten salts;
5. Monocrystals of tungsten carbide;

Tungsten carbide (WC) is an essential material in the production of high-quality, superhard, heat-resistant and acid-resistant, cutting and wear-resistant tools used in metalworking, in the petrochemical, gas and mining industries, in the construction industry and is widely used in the military-industrial complex. The global demand for tungsten carbide (of various qualities) is up to 30 thousand tons. The world turnover of tungsten carbide is about 2 billion euros. Tungsten carbide is the main material in the composition of carbide tools, up to 94%, depending on the alloy name. The world

market for tools is estimated at 17 billion euros, of which carbide tools account for 4 billion euros.
The quality of tungsten carbide products is mainly determined by the fineness, i.e. grain size, original powder. The smaller the grain size of the tungsten carbide powder, the higher the quality of the products. Products made of nanosized (grain size: 50-200nm), ultradispersed (size: 250-850nm) and submicron (size: 800-1500nm) powders have especially high characteristics. To date, there is still no industrial high-performance process for the production of tungsten carbide nanopowders. In such conditions, the deficit in the country is compensated by importers from the USA, Germany, Japan and China. The problem of improving the quality of hard alloys all over the world is solved by nanostructuring them.
To solve these scientific problems, the following specific research objectives are formulated:
- design development and manufacture of a plant for carrying out plasma-chemical processes;
- study of the possibility of using various binders for the manufacture of briquettes from tungsten oxide powders;
- development of a method for making briquettes of a given shape and stoichiometry from a charge consisting of powders of tungsten oxide, carbon black and fresh material;
- development and implementation of a method for the synthesis of tungsten carbide nanopowders in a plasma-chemical process;
- study and assessment of the phase composition of tungsten carbide nanopowders;
- optimization of synthesis conditions and possible subsequent processing of tungsten carbide nanopowders to increase the selectivity of the target carbide phase;
- production of prototypes of carbide cutting tools based on tungsten carbide nanopowders;
- testing of prototypes of carbide cutting tools based on tungsten carbide nanopowders in industrial conditions.
Compactness and high productivity of production, simplicity of the technological process and environmental friendliness, low cost and high profitability, high quality and properties of tungsten carbide powder will provide the product with high competitiveness in the domestic and world tungsten markets.
At present, all over the world, most of the proposed synthesis methods provide for the production of tungsten nanopowders with subsequent carbidization of metallic tungsten nanoparticles. This path obviously leads to an increase in the cost of the resulting product, requiring additional huge material and energy costs. Thus, it is necessary to solve scientific problems associated, on the one hand, with an increase in the productivity of the synthesis method and a decrease in the cost of nanopowders, on the other hand, with an increase in the selectivity of the resulting material in terms of phase composition.
The aim of this scientific work is to develop a technology for producing tungsten carbide nanopowders by plasma-chemical reduction synthesis directly from tungsten oxide to create nanostructured hard alloys.
The scientific novelty of the research lies in the fact that a technology for producing tungsten carbide nanopowders with particle sizes less than 100 nm will be developed, the use of which is expected to increase wear resistance, impact resistance and service life of cutting tools. - a device for carrying out plasma-chemical processes has been developed;
- the possibility of using various binders for the manufacture of briquettes from tungsten oxide powders has been studied;
- development of a method for making briquettes of a given shape and stoichiometry from a charge consisting of powders of tungsten oxide, carbon black and fresh material;
- development and implementation of a method for the synthesis of tungsten carbide nanopowders in a plasma-chemical process;
- study and assessment of the phase composition of tungsten carbide nanopowders;
- optimization of synthesis conditions and possible subsequent processing of tungsten carbide nanopowders to increase the selectivity of the target carbide phase;
- production of prototypes of carbide cutting tools based on tungsten carbide nanopowders;
- testing of prototypes of carbide cutting tools based on tungsten carbide nanopowders in industrial conditions.
The scientific work relates to a method for the synthesis of tungsten carbide nanoparticles.
The method includes the synthesis of oxygen-containing tungsten compounds by hydrocarbons using an electric discharge plasma. In this case, the morphology of the synthesized nanoparticles is controlled by evacuating the vacuum chamber to a pressure of 1.33 Pa and filling it with an inert gas, the pressure of which is 666.61-66661 Pa, before igniting the arc, and igniting an electric arc having a discharge current equal to 80-200 A, and a discharge voltage of 20-30 V between the graphite electrode and the composite electrode. The composite electrode is a graphite rod with a drilled hole in the center and is filled with an oxygen-containing tungsten compound selected from the group containing $WO3$, $W(CO)6$, and the mass content of graphite and tungsten compound is selected in a ratio of 1:0.5 to 1:2. The arc burning conditions are maintained by varying the interelectrode, spraying the composite electrode in an

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electric arc, and depositing the resulting nanoparticles on a cooled screen. The technical result consists in creating a method for the synthesis of tungsten carbide nanoparticles, which makes it possible to control the morphology and composition of the resulting nanoparticles.
A large number of studies are devoted to the processes of formation of especially fine-grained tungsten carbide hard alloys, which should have significantly higher operational properties.
The scientific work is based on the task of creating a method for the synthesis of tungsten carbide nanoparticles in a direct current electric arc, which makes it possible to control the morphology of synthesized nanoparticles by varying the determining synthesis parameters.
The problem is solved by using new operations in the sequence of operations of the known plasma-arc method in order to obtain a new result - nanoparticles of compounds of refractory metals such as tungsten carbide with a given morphology.
The proposed method for the synthesis of tungsten carbide nanoparticles includes: pumping out the vacuum chamber to a pressure (1.33 Pa), filling with an inert gas selected from the range: He, Ne, Ar, Kr, Xe, igniting an electric arc between the graphite and composite electrodes, maintaining combustion conditions arc by varying the interelectrode distance, sputtering in an electric arc of a composite electrode, which is a graphite rod with a drilled cavity in the center, filled with a tungsten compound, deposition of a nanostructured material on a cooled screen.
The morphology of the synthesized nanoparticles is controlled by varying the defining parameters of the synthesis: the composition of the electrodes, the composition and pressure of the inert gas, and the characteristics of the electric discharge. The connection for filling the graphite electrode is selected from the group: WO3, W(CO)6, MoO2, MoO3. The mass content of graphite and tungsten compounds is selected in a ratio from 1:0.5 to 1:2. During the synthesis of tungsten carbide nanoparticles, the inert gas pressure varies within 666.61-66661 Pa, the discharge characteristics vary within the following range: discharge current 80-200 A, discharge voltage 20-30 V.
The method is carried out in a plasma-arc reactor (Fig.-1), which is designed and assembled at the Scientific Institute of Ion-Plasma and Laser Technologies named after U.A. Arifova which includes a sealed vacuum chamber made of stainless steel, with a fixed consumable composite electrode and a movable graphite electrode, the design of which allows you to vary the interelectrode distance to maintain the arc burning conditions, vacuum pumping system, DC power supply, water cooling system, gas supply and discharge system, measuring systems for pressure control and electrical parameters of the discharge, as well as measuring systems for monitoring the temperature, flow rate of the gas phase and its composition.

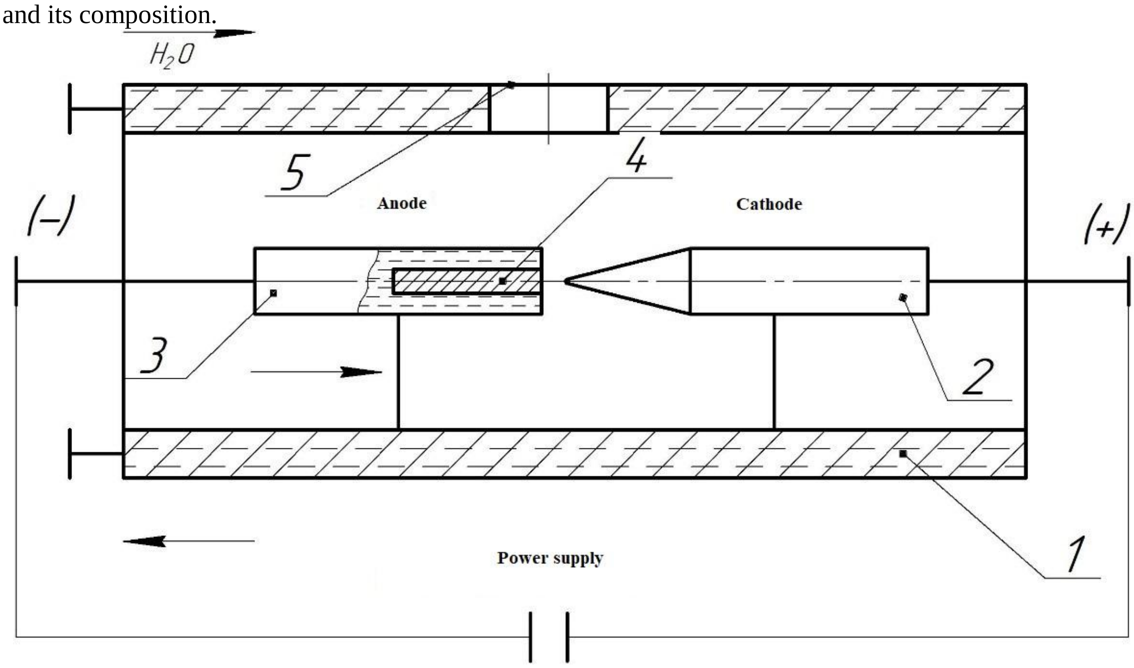

Fig-1. Plasma-arc reactor diagram.
1-cooled shield, 2-graphite electrode, 3-composite electrode, 4-tungsten rod, piercing window.

The method is carried out by performing a series of sequential operations. The vacuum chamber of the reactor is evacuated to a pressure of 1.33 Pa and then filled with an inert gas, which is selected from the series: He, Ne, Ar, Kr, Xe, to the required pressure. Then, in a vacuum chamber between two electrodes, in an inert gas atmosphere, an electric arc is ignited. The fixed consumable electrode is a graphite rod with a drilled cavity in the center filled with a tungsten compound, the movable electrode is a graphite pellet.

III. RESULTS

Maintaining the arc burning conditions is carried out by varying the interelectrode distance, moving the electrode using the bellows unit for the transmission of reciprocating motion. When heated to temperatures realized in the arc, the composite electrode is sprayed. Sputtering a composite electrode in an electric arc leads to the formation of atomic components of tungsten, carbon and oxygen. Diffusion of these components in an inert gas leads to a whole spectrum of possible reactions, including the formation of tungsten carbides: WC and W2C. Subsequently, the synthesized products, which are nanostructured material, are deposited on a cooled screen. The kinetics of chemical reactions and subsequent condensation of synthesis products in an inert gas medium determines the morphology and composition of the synthesized material. The morphology of the synthesized nanoparticles of tungsten carbides is controlled by varying the determining parameters of synthesis: the composition of the electrodes, the composition and pressure of the inert gas, and the electrical characteristics of the discharge. The connection for filling the graphite electrode is selected from the group: WO3, W(CO)6. The mass content of graphite and compounds in the composite electrode is set in a ratio from 1:0.5 to 1:2. The inert gas pressure varies from 666.61 to 66661 Pa, the electrical characteristics of the discharge vary within the following limits: discharge current from 80 to 200 A, discharge voltage from 20 to 30 V.

Thus, in the proposed method for the synthesis of nanoparticles from refractory metals in an electric arc, not metals (W), but their compounds (WO3, W(CO)6) are subjected to pyrolysis to atomic components. Varying the governing parameters of the synthesis allows the synthesis of tungsten carbide nanoparticles with a given morphology and properties.

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