Production of tungsten pseudo-alloy powders in DC Arc Thermal Plasma Reactor for ISPC25 21-26May 2023, Kyoto, Japan

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Abstract: The presentation presents the results of the DC arc plasma jet processes researches that led to the creation of physical and chemical bases of the thermal plasma processes, providing production of powders of tungsten pseudoalloys with proper chemical and phase composition, including nanopowders and spherical particles in the micron size range.

Keywords: tungsten pseudoalloys, thermal plasma, nano-sized and micron powders.

General

Currently, Baikov Institute of Metallurgy and Materials Science of RAS (IMET RAS) is one of the leading organizations in the Russian Federation, where active research and development of constructive and technological schemes of plasma processes, as well as equipment for the production of nanosized, spherical powders of various materials in thermal plasma flows, is carried out [1].

In particular, an original multifunctional plasma facility has been developed for research and development of plasma processes for the synthesis and production of experimental batches of nano- and micron-sized powders. The main structural elements of this facility provide the necessary scaling for the industrial production of a wide range of nanopowders and spherical particles in the micron size range.

One of the most versatile and effective methods for obtaining nanopowders of elements, their inorganic compounds and compositions is plasma-chemical synthesis in a DC arc plasma jet [2, 3]. Due to its technical and economical features, the plasma method maintains competitive position amongst other methods used for production of broad variety of nanopowders [2, 3].

A possible application of the method of plasmachemical synthesis of nanopowders is the preparation of nanocrystalline compounds of tungsten pseudo-alloys of the W-Ni-Fe system with increased requirements for the design of products and structures for special purposes [4– 6].

Plasma-chemical synthesis of nanosized composite powders W (90 wt.%)–Ni (7 wt.%)–Fe (3 wt.%) was carried out at the IMET RAS plasma facility during the reduction of a mixture of metal oxide powders $(WO_3+Ni_2O_3+Fe_2O_3)$ in a flow nitrogen-hydrogen plasma in a plasma reactor with limited jet flow.

The formation of composite nanoparticles is carried out as a result of successive condensation of metal vapors (W, Ni, Fe) obtained as a result of physicochemical transformations of particles of the initial metal compounds. During the evolution of nanoparticles from the moment of their formation in the plasma flow to deposition on the water-cooled walls of the reactor, their dispersed and chemical compositions change as a result of particle collisions, as well as interaction with the reaction gas medium. The control of these processes is ensured by changing various process parameters - the composition, enthalpy and velocity of the plasma jet, the concentration of reagents, and the size of the reactor.

According to the results of electron microscopy (TEM, SEM), the final product consists of particles with a size ranging from 10 to 300 nm (Fig. 1). The specific surface of nanopowders is in the range from 4 to 8 m²/g. The shape of the particles is predominantly spherical with a "core-shell" structure, where the core is tungsten, and the shell (2 to 5 nm thick) is an alloy of nickel and iron. The content of total oxygen in the obtained nanopowders is at the level of 1.5 wt.%. An additional stage of thermochemical treatment in a hydrogen environment makes it possible to reduce the oxygen content to 0.009 wt.%.



Fig. 1. Micrographs of the nanopowder of the W–Ni–Fe system: a — scanning electron microscopy (SEM), b characteristic composite nanoparticles, transmission electron microscopy (TEM).

To obtain spheroidized powders of the W-Ni-Fe system in the micron size range, a plasma spheroidization of metal powders was used, developed at IMET RAS.

The spheroidization of powders of the W-Ni-Fe system is based on the intensive heating of the initial particles fed into the plasma flow based on the argon-hydrogen mixture by the transporting gas argon, their melting and the acquisition of spherical shape by the melt drops due to surface tension forces. As a precursor for plasma spheroidization, dense spherical nanopowder microgranules were used (Fig. 2 a, b), obtained as a result of granulation of a nanopowder of the system W (90 wt.%)-Ni (7 wt.%)-Fe (3 wt.%) (Fig. 2 a, b) with an organic binder (sucrose) on a Buchi Mini Spray Dryer B-290. The characteristic size of the obtained nanopowder microgranules is in the range from 25 to 63 μ m. Microgranules are characterized by a homogeneous microstructure that does not contain internal cavities (Fig. 2 c). The value of their bulk density is 2.5 g/cm³, the fluidity is 37 s/50 g. The content of total oxygen in the obtained microgranules is at the level of 2.5 wt. %.



Fig. 2. SEM images of nanopowder microgranules (a, b) and the internal structure of a characteristic microgranule after layer-by-layer etching with gallium ions (c).

As a result of the process of plasma spheroidization of nanopowder microgranules of the W - Ni - Fe system, spherical microparticles were obtained in the size range from 10 to 50 μ m (Fig. 3 a, b) and a submicron internal structure characterized by a grain size in the range from 0.5 to 2 μ m (Fig. 3c, d). A decrease in oxygen impurities in the spheroidized product to 0.7 wt.% was established. The value of the bulk density of the product is 9.4 g/cm³, the fluidity is 10 s/50 g.



Fig. 3. SEM images of spheroidized micro-powder of the W – Ni – Fe system (a, b), microparticles after their mechanical grinding (c) and the internal structure of a characteristic microparticle (d).

Conclusions

The expected results of experimental studies have the fundamental possibility of using the process of plasmachemical synthesis to obtain nanosized composite powders of the W – Ni – Fe system within sizes from 10 to 300 nm with a "core-shell" structural particle, as well as the process of plasma spheroidization when nanopowder microgranules are detected for obtaining spherical microparticles in sizes from 10 to 50 microns with an internal submicron structure characterized by a grain size ranging from 0.5 to 2 microns.

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