Studies of Highly Dispersed Titanium Carbide Powder Obtained from Scrap Tungstenless Cemented Carbide Alloys

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Abstract. The results of research of characteristics of highly dispersed titanium carbide powder obtained from carbide waste of TN-20, TN-25, TN-30 types are presented. The powder particles were studied using analytical methods including scanning microscopy, X-ray diffraction, differential thermal analysis. The obtained results confirm the formation of nanosized particles of titanium carbide of monocrystalline form.

Introduction

Titanium carbide is one of the most demanded materials widely used in industry, modern engineering and medicine [1-3]. The wide range of applications is due to the unique combination of chemical and physical properties of titanium carbide (high wear resistance, corrosion resistance, chemical inertness, high melting point, flexural modulus, etc.) [4-7]. The use of high-dispersity titanium carbide powders (micro- and nanopowders), which provide a significant improvement of the properties of materials based on titanium carbide, is becoming increasingly important [8-11]. The most studied methods of titanium carbide nanopowders production are chemical vapor deposition, self-propagating high-temperature synthesis with laser ignition, sol-gel method, self-propagating high-temperature synthesis, etc. [12-16]. All these methods are characterized by very low productivity and rather high cost of the final product. Increasing industrial use makes it necessary to reconsider the attitude to the methods of production of titanium carbide nanopowder both in terms of ensuring its high dispersity and purity and in terms of productivity, energy intensity and economy of its production technology.

In this regard, an alternative technological approach is the production of highly dispersed TiC powders from titanium-containing carbide waste by the technology [17]. This method offers ample opportunities for the production of nanopowders on an industrial scale, as it is characterized by minimal technological equipment, environmental safety, rather low operating costs and competitive prices.

Experimental part

The powder obtained from the scrap of titanium hard alloys of TN-20, TN-25, TN-30 grades was investigated. The chemical composition is presented in Table 1.

Table 1. *Chemical composition of initial raw materials for obtaining titanium carbide powder*

Alloy grade	Component content in titanium alloy scrap, mass %				
	TiC	Ni	Mo	Nb	WC
TN-20	79.0	14,0	6.0	0,05	0,95
TN-25	74.0	19,3	6.0	0.01	0.69
TN-30	70.0	22,5	7.0	-	0.50

The main component of the slab is titanium carbide TiC cemented by nickel-molybdenum bond (Ni+Mo), there are also small impurities of niobium (Nb) and tungsten carbide (WC). As a result of treatment of alloys by technology [17] specially prepared dispersing solutions, there is a destruction of the alloy matrix and particle refinement due to the improvement of the machining process.

The crystalline structure of the fillers was investigated by X-ray diffraction (ARL X'TRA, ThermoTechno) with CuKα source in the 2θ angle range from 4 to 56° in asymmetric coplanar mode with sliding angle of incidence α=3o (θ-scan). Phase identification and peak indexing were performed using the ICDD (International Centre for Diffraction Data) PDF-2 database.

The microstructure of the obtained powder was studied using a scanning electron microscope "TESCAN MIRA 3 LMU" (Poland).

For the analysis of the particle size we used the Particle Metric software of the scanning electron microscope PHENOM proX (Phenom-World B.V. (Netherlands).

Differential thermal analysis of the powder was carried out on a NETZSCH STA 449F1. Thermal analysis of the powder was carried out in a platinum crucible in an argon atmosphere in the temperature range of 25...1000 °C at heating of 10 °C per minute.

Analysis of experimental results

Fig. 1 shows a diffractogram of the powder obtained from carbide scrap.

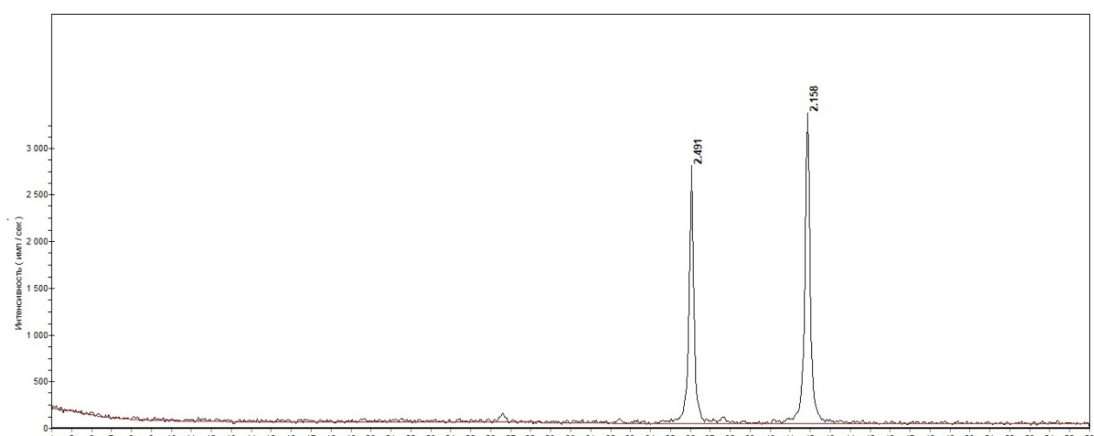

Fig. 1. *Powder diffractogram*

Two characteristic reflexes at angles 2 θ 36 and 42°, which correspond to pure titanium carbide, are present on the X-ray image (PDF card No. 31-1400).

The microstructure of the powder is shown in Fig. 2

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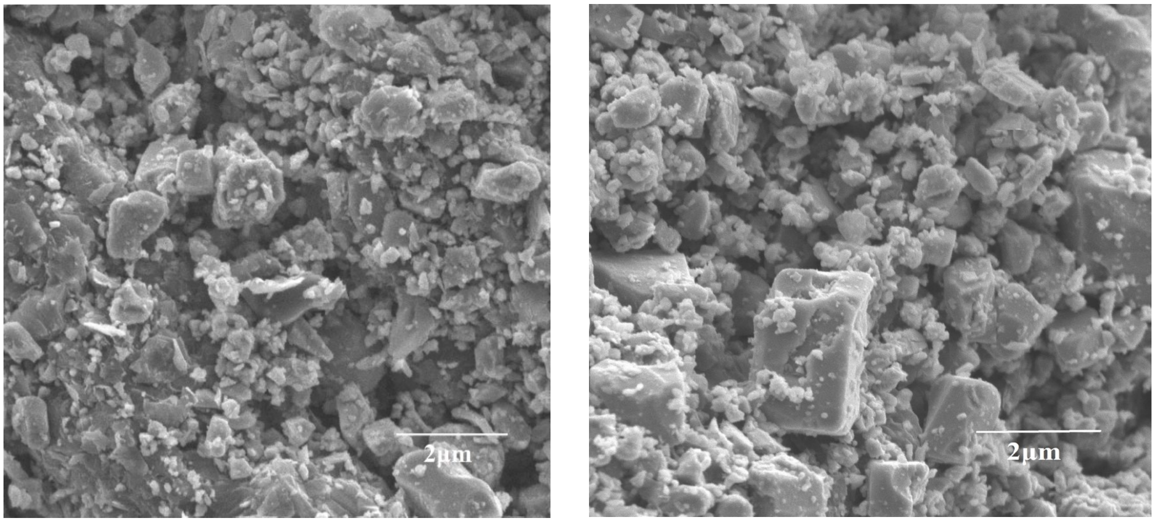

Fig. 2. *Microstructure of titanium carbide (TiC) powder*

As can be seen from the presented microphotographs (Fig. 2) the powder under study is prone to agglomeration. The microstructure of the particles is heterogeneous, the particle shape is irregular. The average particle size is 60-150 nm, the size of agglomerates is 200 nm–2.5 μm (Fig. 3).

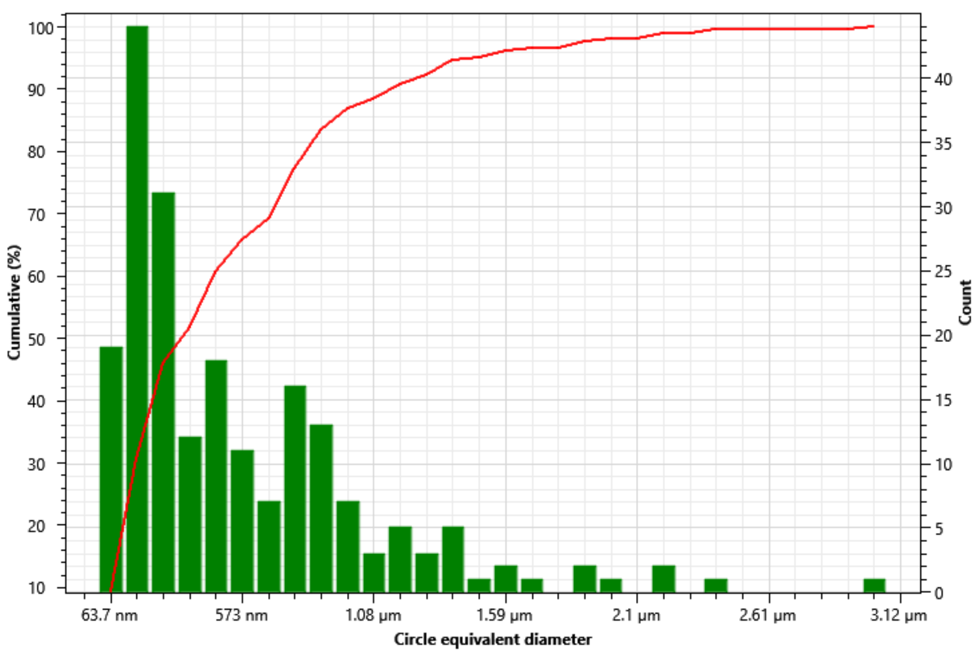

Fig. 3. *Titanium carbide powder particle size distribution.*

Elemental analysis of the powder (Fig. 4) showed the presence of impurities of molybdenum and tungsten, which is associated with the initial composition of raw materials used for the synthesis of titanium carbide powder.

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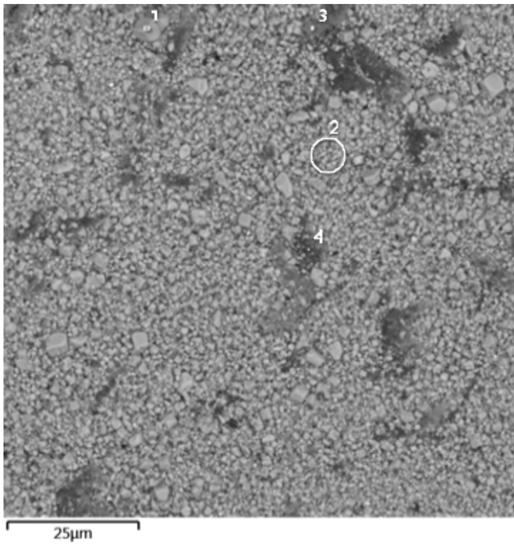

Spectrum name	Elemental composition, wt.%				
	C	O	Ti	Mo	W
Spectrum 1	21.63	4.53	60.03	6.15	7.66
Spectrum 2	18.10	7.07	64.92	6.33	3.58
Spectrum3	55.58	8.30	30.94	3.35	1.82
Spectrum 4	58.29	6.79	29.49	3.55	1.88

Fig. 4. *Results of spectral analysis of titanium carbide powder*

Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) of the powder under study are shown in Figure 5.

Analysis of the thermogravimetric curve analysis (Fig. 5) showed that when increasing the temperature to 1000 °C the sample practically does not lose mass (-0.32%), indicating its relatively stable behavior in this temperature range. The endothermic process peaks at 133.1 °C, associated with the removal of adsorbed water. Differential thermal analysis (DTA) results indicate the presence of two exothermic effects with peaks at 441.9 °C and 476.4 °C. The first peak is associated with the oxidation of free carbon, with a loss of mass, the second peak indicates the oxidation of titanium carbide. The increase in mass during oxidation of the sample begins at 476.4 °C and is 1.26 % up to 900 °C, which indicates partial oxidation.

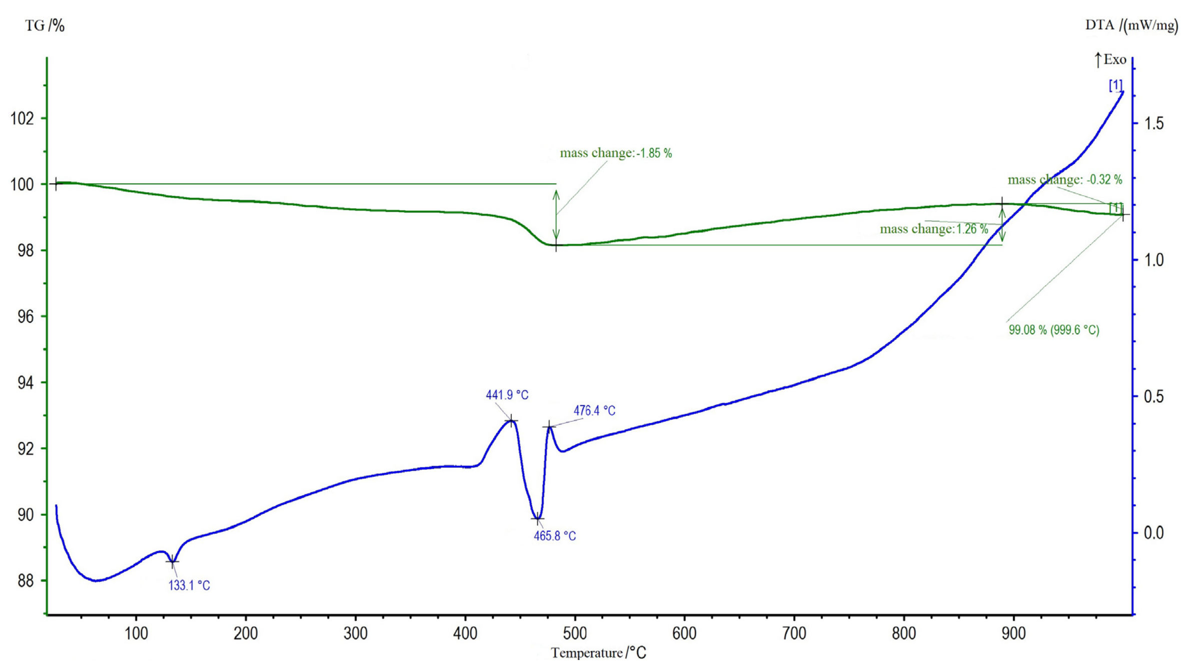

Fig. 5. *TGA and DTA curves of titanium carbide (TiC) powder*

Conclusions

The powder obtained from TN 20, TN-25, TN-30 carbide wastes contains single-phase titanium carbide and impurities of molybdenum and tungsten. The carbide particles have the size of 60...150 nm and are mainly agglomerated. The particle size of agglomerates is 200 nm...2.5 µm. It is relatively stable in the high temperature range.

Thus, titanium carbide powder obtained from carbide wastes can be used as an alternative to those available on the market as a component in the production of heat-resistant hard alloys, reinforced alloys, abrasive materials, coatings for cutting tools, etc., but this requires further research.

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