Dynamic Synthesis of Superhardness Materials in Hypersonic Jet of Electrodischarge Plasma

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Abstract -The pre-discovery results of possible application of the pulsed coaxial magneto plasma accelerator (CMPA) with titanium electrodes for dynamic synthesis of nano-disperse crystal powders (TiO_2, TiN, TiC, $C_{0.3}N_{0.7}Ti$) are presented in the paper. Various superhardness materials are produced depending on the electro discharge plasma composition and environmental conditions. When powder-like graphite is sent into discharge plasma under normal environmental conditions, nitride TiN or titanium dioxide TiO_2 are produced, titanium carbide TiC and TiO_2 are produced under decreased atmospheric pressure (P=0,1 atm). Dynamic synthesis of TiO_2 is possible in the carbonic acid atmosphere under normal pressure. Experiments in the nitrogen atmosphere under normal pressure made it possible to produce TiN and carbon nitride $C_{0.3}N_{0.7}Ti$. The researches of the produced powders on the scanning electron microscope Philips SEM 515 showed that the particles have a spherical shape and sizes ranging from 0,05 to 0,15 μm. From 5,0 to 15,0 g of the powder like substance is produced within one work cycle.

The necessity to develop simple techniques for producing nano-disperse powder-like crystal substances on the basis of titanium with spherical shaped particles is conditioned by the availability of their application as abrasives for further hard-alloy compact elements with nano-crystal micro structure and for covering strengthened surface. In the last years electro physical methods based on the low-pressure arc discharge [1] and conductors electric explosions [2] are developing quickly.

The present paper provides information on the results of the pre-discovery researches of dynamic possibility to produce TiO_2, TiN, TiC, TiCN and nano-disperse powders production in the hypersonic electro discharge plasma jet on their basis at use CMPA [3]. CMPA refers to the erosion accelerators type. Its advantage over other types of electro magnetic and electro dynamic accelerators is that the electro erosion production of the initial material, dynamic synthesis and formation of spherical superhardness materials nano-particles from the liquid phase is carried out during one short-term work cycle (10^{-3} s) in the reactor under the pressure close to the atmospheric one.

Experiment procedure.

The principle of operation and the construction of the CMPA are described in detail in the papers [3, 4]. Accelerator barrel is made of titanium tube (BT-1-0) with the internal diameter d_{ac} = 21 mm, and wall thickness 2 mm, length of the accelerator channel l_{ac} = 230-280 mm. The central electrode is made of titanium too.

Pulsed power supply is fed by the capacitive storage with the power W_c = (100-200) kJ which was changed due to changing the charging voltage U_{ch} = 3,0-4,0 kV and capacity C = (15-48)·10^{-3} F.

The pulsed hypersonic electro discharge plasma jet was flowing into the waterproof air-filled chamber-reactor. Opening the chamber and extracting the produced material followed the full suspended fraction deposition on the walls of the chamber and natural cooling up to the room temperature.

The electrodes voltage U(t) and accelerator operating current i(t) were oscillographically tested in the experiments (fig.1).

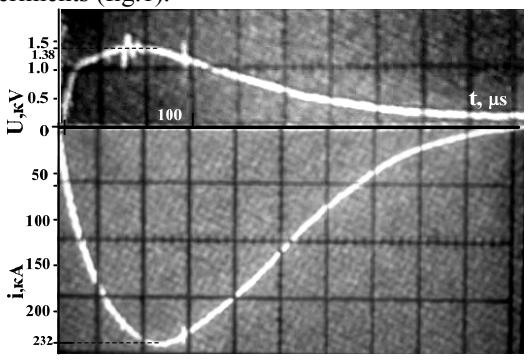

Fig.1. Typical oscillograms U(t) and i(t) of the plasma shot of the CMPA with titanium electrodes correspond to experiment №6 in the table

Dynamic characteristics of the plasma flow were studied using the high speed framing photographing on the installation ВФУ-1 (fig.2)

Analytical research of the received products was done by means of roentgen diffractometry methods on the diffractormeters DRON-4 (FeK_α – radiation) and Shimadzu XRD 6000 (CuK_α – radiation), electronic microscopy on the scanning electron microscope Philips SEM 515.

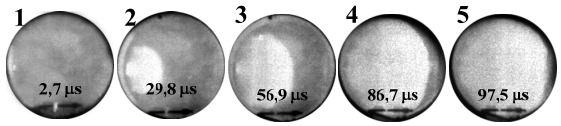

Fig.2. Typical photogram of the hypersonic plasma jet formation from the CMPA accelerating channel, it corresponds to the experiment № 8 in the table

The experiments results

The researches of the dynamic synthesis of nano-disperse crystal titanium-based materials in the hypersonic jet electro discharge plasma had a pre-discovery character.

The first experiments were carried out when plasma jet was flowing into air atmosphere under normal pressure. Structure-phase analysis of the produced powder showed the formation of crystal phases of titanium dioxide TiO_2 and titanium nitride TiN in the ratio of TiO_2:TiN = 5,7:1 (experiment 1, the table). Addition of the powder-like graphite (0,5-1,0 g) into the zone of plasma structure formation did not make it possible to produce crystal titanium carbide TiC. At the decreased pressure of air in the reactor and the energy storage of $W_c \approx 300$kJ (experience 2, table) the situation greatly changes. Two phases are also identified: tiutanium carbide TiC and titanium oxide TiO, TiC:TiO = 3:1. Thus, it is possible to assume that the nano-disperse crystal titanium carbide production is possible, when there is no oxygen, that is either in relatively low vacuum (up to 10^{-3}) or in rare gas atmosphere (not nitrogen). However, it is evident that the dynamic synthesis of titanium dioxide TiO_2 must be carried out under atmospheric conditions, which is not preferable form the safety point of view. Therefore, an alternative procedure – the possibility of dynamic TiO_2 synthesis in the carbonic acid CO_2 atmosphere under normal pressure - was considered.

Diffractometry researching of the powder-like material received under such conditions proved the expected result. Crystal TiO_2 with little dirts of Ti_3O_5 and TiC in the ratio of TiO_2:Ti_3O_5:TiC = 9:3,8:1 (experiment 3, the table) is produced.

Table. Experimental data

№	U_{ch}	C	I_m	W_c	medium	P_o
	kV	mF	kA	kJ		atm.
1	3,8	15	139	108	air	1,0
2	3,5	48	202	294	air	0,1
3	4,0	18	240	144	CO_2	1,0
4	4,0	18	203	144	nitrogen	1,0
5	4,0	18	210	144	nitrogen	1,0
6	3,0	48	232	216	nitrogen	1,0
7	4,0	18	214	144	nitrogen	1,0
8	4,0	24	172	192	nitrogen	1,0

It is evident that the production of nano-disperse crystal titanium nitrite is possible in nitrogen atmosphere under several conditions. So, for example, if the experiment is carried out under atmospheric pressure in the nitrogen, $W_c = 144$ kJ and reactor decapsulation after the plasma shot, there is partial material oxidation. Diffractograms analysis shows the production of TiN and TiO_2 in practically equal portions (experiment 4, the table). The portion of TiO_2 in the material sufficiently decreases and is about 26 %, under the same conditions, but the reactor hermiticity being kept up to the full decomposition and the cooling of the powder-like material (approximately during 1 hour), (experiment 5, the table). The presence of oxygen in this case is conditioned by the fact that before the reactor is filled with nitrogen; the air is pumped out from the reactor up to 0,1 atm. However, the concentration of crystal titanium TiO_2 under such conditions decreases below the sensitivity level of DRON-4 at the energy increase up to $W_c = 216$ kJ (experiment 6, the table).

In case of the preliminary double pumping reactor by nitrogen, TiN is practically the only dynamic synthesis phase (experiment 7, the table). It is proved by the diffractogram (fig.3) received using the Shimadzu XRD6000 roentgen diffractometer. The same result is achieved when double or triple procedure is executed in the reactor without any intermediate material selection and without nitrogen blow. The powder received is black-blue.

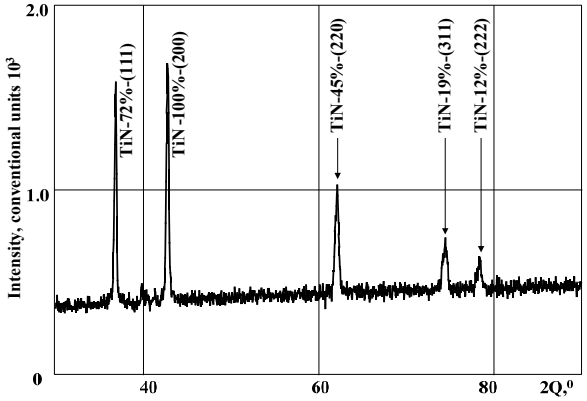

Fig.3. X-ray diffraction pattern of the nano-disperse TiN powder received in experiment №7 (the table)

The experimental researches also showed the possibility of successive execution of 10 cycles with the production of 150 g of TiN powder, which is about 75 % from initial accelerator barrel weight.

The dynamic synthesis of one of most perspective crystal nano-disperse powders - titanium carbonitride C0.3N0.7Ti was carried out by the methods described above in nitrogen atmosphere at pressure atmospheric with introduction of carbon in high current discharge plasma (experience 8, table) (fig. 4).

The powders received by the method under consideration were investigated on the scanning electron microscope Philips SEM 515. It is seen form the given microphotos (fig. 5) that the product of the dynamic

synthesis looks like accumulations (fig. 5a) with the sizes up to several tens of μm and less. As shown in the photo with the large increase (fig. 5б) agglomerates completely consist of particles of the spherical form by a diameter from (0,5 - 0,06) μm and less. The particles of awkward shape are missing.

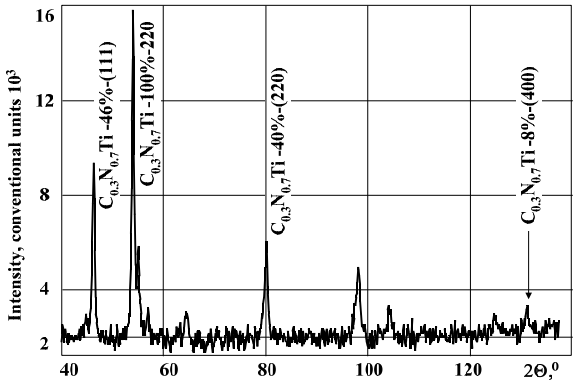

Fig.4. X-ray diffraction pattern of the nano-disperse $C_{0.3}N_{0.7}Ti$ powder received in experiment 8 (the table)

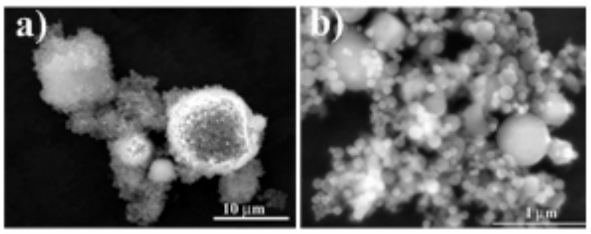

Fig.5. Microelectronic photos of nano-disperse powder TiN in different enlargements

From the law curve of particles distribution according to their sizes (fig.6) it is seen that about 90% of the powder particles are (0,05 - 0,15) μm. Besides, there are more little fractions in the powder, but now it is not possible to determine their sizes.

The researches on optimization of electroerosive acceleration channel surface wear showed that the production of the barrel can achieve 90%.

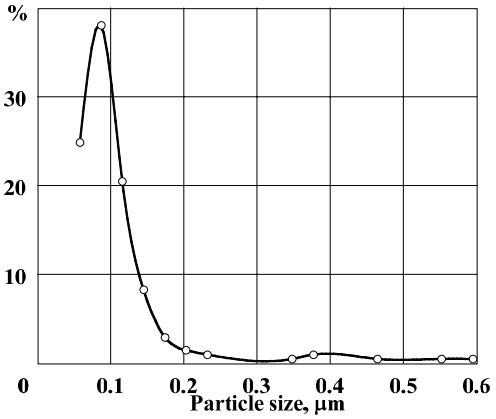

Fig.6. Distribution of the particles by sizes in the percentage to the total particles number, this number being 600

Conclusion

Therefore the results of the pre-discovery experimental researches showed the possibility to use the pulsed high current coaxial magneto plasma accelerator for producing nano-disperse crystal powders including TiN, TiC, $C_{0.3}N_{0.7}Ti$.

About (5,0-15,0) g of the powder-like material is produced during one work cycle with the power of (100-200) kJ. The method is rather simple and ecological, its application does not need preliminary preparation of the initial material and its dozed supply, it is applied in the reactor chamber under conditions close to natural and at room temperature.

References

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