## THE OBTAINING OF CERAMIC COMPOSITE MATERIAL ON BASE TITANIUM NITRIDE FROM MIXTURE TiH<sub>2</sub>-BN BY METHOD OF A REACTIONARY ELECTRODISCHARGE SINTERING

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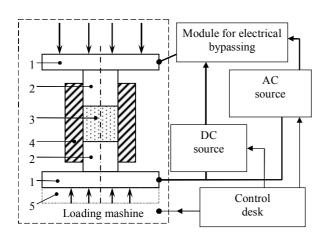
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## Introduction

The method for obtaining of ceramic composite material (CCM) on base TiN–TiB<sub>2</sub> by processing a powder mixture TiH<sub>2</sub>–BN under method of electrodischarge sintering (EDS) is offered. This technological approach has a number of advantages, which simplified technological process of obtaining CCM by a method reactionary EDS in comparison with [1, 2].

## **Consideration of problem**

The CCM on base TiN-TiB<sub>2</sub> obtaining in graphite mould under direct passing of superposition of electric currents of direct and alternating of higher frequency. The schematic diagram of equipment for obtaining CCM on base TiN-TiB<sub>2</sub> by method EDS is showed in Fig. 1.



**Fig. 1.** The schematic diagram of equipment for EDS ERAN 2/1

- 1- current-carrying plate of loading machine;
- 2- electrodes-punches (graphite MPG-6);
- 3– treating powder mixture;
- 4– matrixes of mould (graphite MPG –6);
- 5- blocks of pressing.

The duration of this process of processing powder mixture up to 3 min.

At treatment of powder mixture TiH<sub>2</sub>–BN simultaneously with direct current is influence by alternating electric current of higher frequency (5 kHz) also. The mixed effect on powder mixture

necessary because of presence in mixture of nitride of boron, as a consequence of the mixture of powders is dielectric. The total denseness of superposition electric currents during processing, how is determined from practice, should be  $(8-9)\times10^6 \text{ A/m}^2$ .

Up to a moment of an electrical breakdown of powder mixture the main part of direct electric current is passes through a graphite mould along circuit 2–4–2 (from top to bottom, see Fig. 1). Because of this body of the powder mixture, in main, is heated up at the expense of passing an alternating current. When temperature processing is increased up to 460-580 C the electrical breakdown of a powder mixture is take place and subsequent firing of selected hydrogen. In outcome of an electrical breakdown is increasing electroconductivity of a mixture and value of direct current through a mixture will increasing also. It results to more force to heating of a mixture by a passing of direct current and of a reaction of synthesis in all volume of a mixture of powders. During synthesis pass the following chemical responses:

$$3\text{TiH}_2 + 2\text{BN} = 2\text{TiN} + \text{TiB}_2 + \uparrow 3\text{H2}$$
 (1)

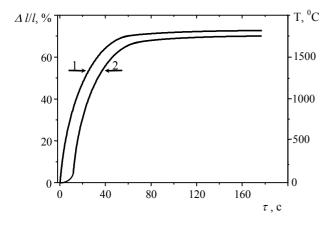
$$TiH_2=Ti+\uparrow H_2$$
 (2)

$$3Ti+2BN=2TiN+TiB_2$$
 (3)

In an offered method of obtaining CCM the hydrogen not only is deleted, but also during processing plays a role of protective gas medium of a reaction of synthesis. Also for want of expansion dehydrate of titanium (2) the formation of chemical particles of active metal titanium happens, that positively has an effect for forming of CCM.

In consequence of electroprocessing the processes of forming and sintering of CCM are accelerated and pass more completely, that promotes formation of material with homogeneous denseness. In Figure 2 is shown, as take place shrinkage of powder mixture and increasing of

temperature by processing EDS method at obtaining CCM on base TiN–TiB<sub>2</sub>.



**Fig. 2.** Dependence of shrinkage and temperature during of EDS processing of a composition TiN–TiB<sub>2</sub>: dependence of temperature on sample surface (1) and dependence shrinkage of sample (2) from time.

Relative denseness synthesized of samples CCM is measured by a hydrostatic method, hardness – Vickers method, crack-resistance – indention method [4].

The research work for possibility of obtaining fine-grained and high dense refractory composite on base of TiN by EDS method. As initial work material for obtaining of powder mixtures  $TiH_x+BN$  and  $TiH_2+BN$  for ratio  $TiH_x(TiH_2):BN$  in accordance with stoichiometry for reaction (1).

Substances  $TiH_x$  and  $TiH_2$  were obtained from iodide and magnesium of titanium, correspondingly, and have different quantity of admixtures [5]. The substances  $TiH_2$  have cubic structure for crystal lattice and  $TiH_x$  – tetragonal structure. The tetragonal structure for  $TiH_x$  is showing about more content of hydrogen in substance [5].

The initial powders was mixed and milled in planetary stirrer and following treatment by EDS method. The properties of foreign analogous and obtaining materials are shown in Table 1.

The relative denseness of samples CCM up to 97–98,5 % was amounted. The X-ray analysis shown, that synthesized material is content only the phases TiN and TiB<sub>2</sub>. Sintered CCM have decreased hardness, but increased crack-resistance – this is

phenomenon of influence hydride during of processing form of CCM structure.

For present way of obtaining CCM by EDS method ceramic material on base TiN–TiB<sub>2</sub> with high mechanical properties are obtained. This ceramic material can be using for making elements of cutting instrument.

**Table 1** – The properties of foreign analogous and obtaining materials on base TiN– TiB<sub>2</sub>.

Obtained material	Crystalline size, nm	Microhardness, HV, under loading ГПа Н		Crack- resistance, MPa/m <sup>2</sup>
36%TiB <sub>2</sub> — TiN(1%Ni) [1]	2000–3000	20.5	196	6.20
53%TiB <sub>2</sub> –TiN [3]	<100	20.6	2	6.50
$36\% TiB_2$ — $TiN$ (innit. $TiH_x$ )	500–700	17.2 19.8	2 196	<del></del> 8.00
36%TiB <sub>2</sub> –TiN (innit. TiH <sub>2</sub> )	500–700	19.1 18.6	2 196	— 7.44

## References

- 1. Zhang G., Jin Z., and Yue X.  $TiN-TiB_2$  Composites Prepared by Reactive Hot Pressing and Effect of Ni Addition. // J.Am. Ceram. Soc. 1995. 78, No 10. P. 2831-2833.
- 2. Patent № 5794113 USA, Simultaneous synthesis and densification by field–activated combustion / Munir Z., Shon I., Yamazaki K. Οπy $\overline{0}$ π. 11.07.1998. 3. Lee J.W., Munir Z.A., Shibuya M. Synthesis of Dense TiB<sub>2</sub>–TiN Nanocrystalline Composites through Mechanical and Field Activation // J. Am. Ceram. Soc.–2001. V.84, №6. P.1209–1216.
- 4. Evans A.T., Charles E.A. Fracture toughness determination by indentation // J. Am. Ceram. Soc.  $-1978. N_{\odot} 7-8. p. 371.$
- 5. Гидриды металлов // Под ред. Мюллера В., Блэкледжа Д., Либовица Дж. М.: Атомиздат, 1973. C. 283–290.