**THE INFLUENCE OF THE HEATING RATE IN THE PROCESS OF SPARK-PLASMA SINTERING ON THE KINETICS OF COMPACTION, STRUCTURE FORMATION AND PROPERTIES OF THE MATERIALS OF Fe – Ti – C – B SYSTEM**

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**Abstract:** Regularities of the influence of spark plasma sintering heating rate on the structure and properties of dispersion-strengthened materials based on Fe – Ti – C – (B) system are studied. Increasing heating rate of the samples from 10 °C/s to 20 °C/s induced by increasing current rise rate leads to decrease the average grain size in the material matrix of the Fe – Ti – C – B system from 2.5 to 1.5 microns. This allows to increase hardness from 48 HRC up to 70 HRC and wear resistance 7 times compared to R6M5 (full analog of HSS M2) steel during SPS consolidation in the mode with a heating rate of 20 °C/s and isothermal exposure 1100 °C for 180 s.

**Keywords:** spark plasma sintering, high voltage electric discharge, consolidation, hardness, wear resistance, powder, carbide steel

1. **Introduction**

The contemporary industry needs the structural materials with high hardness and wear resistance which could replace the costly tungsten-containing materials. This can be achieved by creating composite materials with heterogeneous structure being a plastic metal matrix with hard inclusions. Such materials include metal-matrix composites, including carbide steels, which are obtained by powder metallurgy methods [1 – 4]. The Institute of Pulse Processes and Technologies of NAS of Ukraine (IPPT NASU) developed the technology of producing carbide steels of the Fe – Ti – C – B system with levels of hardness and wear resistance similar to tungsten-containing alloys. Improved physical and mechanical characteristics were obtained by changing the dispersion and phase composition of the initial powders using the high-voltage electric discharge (HED) treatment in the hydrocarbon liquid and their subsequent spark-plasma sintering (SPS) [5 – 7].

2. **Problem discussion**

It is known that physical and mechanical and functional properties of powder compacts largely depend on the porosity and grain structure [8], and that SPS provides rapid consolidation of powder materials and in many cases restricts the grain growth compared to conventional sintering [9]. Therefore, the study of the influence of the heating rate of SPS consolidation on the structure and properties of dispersion-strengthened materials based on Fe – Ti – C – (B) system is an urgent task.

**Objective of this paper** is to establish regularities of the influence of SPS heating rate on the structure and properties of dispersion-strengthened materials based on Fe – Ti – C – (B) system.

3. **Objective and research methodologies**

The SPS was carried out using experimental complex, designed at IPPT NASU (see Fig. 1) [10]. The mixture of powders of initial mass composition of 75% Fe – 20% Ti – 5% B4C was chosen as the charge for sintering to be treated by the HED with a specific energy of 6.25 MJ/kg, which provided the content of the strengthening phase to 25% sized from 100 nm to 600 nm and an average metal-matrix particle diameter of ~ 15 µm (see Fig. 2) [6, 7].

**Fig. 2.** Electron micrograph ×1700 (a) and the phase composition (b) of the charge of Fe – Ti – C – B system

Consolidation was carried out at a heating rate of 10 °C/s at a temperature of isothermal aging of 1100 °C, holding time τ = 180 s, cooling rate 10 °C/s. The heating rate was varied in the range of 10 °C/s to 20 °C/C by increasing the current strength amplitude rise from 14 A/s to 30 A/s. The experimental analysis of changes in the structure of the consolidated compacts was carried out by means of computer metallography according to DSTU ISO 643:2009 using the Biolam-I optical microscope and ImageJ software. Phase analysis was carried out on DRON-3 diffractometer according to ref. [11].

Hardness of samples was measured according to ISO 6508-1:2013. Wear resistance was investigated by SMC-2 friction machine based on the “roller-pad” pattern. The counter-body was the 1A1 diamond grinding wheel with grain size of AC80/63. The results were compared with test results of the sample of steel R6M5 (GOST 19265-73, full analog of HSS M2 steel).

The average grain size of the matrix of the Fe – Ti – C – B system after HED treatment is ~ 0.5 µm. In the process of SPS at heating rate of 10 °C/s its average size increased to ~2.5 µm. Increasing the heating rate to 20 °C/s allowed to reduce the average grain size of the matrix after sintering to ~1.5 µm.

**Table 1.** The atmosphere

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<th>The atmosphere</th>
<th>Vacuum 10⁻³ MPa</th>
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**Table 2.** The matrix material

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<th>The matrix material</th>
<th>Graphite MPG-3</th>
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**Table 3.** Axial pressure

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<th>Axial pressure</th>
<th>60 MPa</th>
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**Table 4.** The amplitude of the superposition

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<th>The amplitude of the superposition</th>
<th>1.1 kA</th>
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**Fig. 1.** SPS complex and its parameters
The increase of the heating rate of SPS also led to increasing hardness from 48 HRC up to 70 HRC and wear resistance; the mass loss for the samples consolidated with a heating rate of 20 °C/s was 0.2 %, which is 7 times lower than that of samples of R6M5 (full analog of HSS M2) steel (hardness of 60 HRC). It is assumed that this is due to the decay of residual boron carbide and occurrence of the carbide and boride formation process during heating, which is characterized by heat release, as evidenced by the heating curves obtained during the experiment (see Fig. 4, b).

Additionally, a set of experiments on the consolidation of powder mixture of Fe – Ti – C – B system with a heating rate of 20 °C/s was conducted to achieve a temperature of 1100 °C and subsequent holding from 0 to 180 s with a step of 60 s. The analysis of experimental data allowed us to establish that with the increasing holding time to 180 s the content of the boride phase of source metals is increasing in the composition of the sample (see Fig. 5), which leads to increased hardness and wear resistance (see Fig. 6).

3. Conclusions
1. It is established by experiments that increasing heating rate of the samples from 10 °C/s to 20 °C/s induced by increasing current rise rate allows to decrease the average grain size in the material matrix of the Fe – Ti – C – B system from 2.5 to 1.5 microns.
2. It is established that increasing heating rate from 10 °C/s to 20 °C/s affects the flow of diffusion processes in the material during SPS, i.e. slowing them, and helps to increase hardness from 48 HRC up to 70 HRC and wear resistance 7 times compared to R6M5 (full analog of HSS M2) steel during SPS consolidation in the mode with a heating rate of 20 °C/s and isothermal exposure 1100 °C for 180 s, which is connected with intensification of carbide and boride formation processes.

References


