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Regularities of the Effect of Process Parameters of SHS-Extrusion on the Structure and Properties of Long-Dimensional Rods from TiB / 30 wt. % Ti Materials

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Abstract

In this paper, the effect of the process parameters of the SHS-extrusion method (the delay time before applying pressure, the speed of movement of the press plunger) on the length and quality of extruded cylindrical rods with a diameter of 3 mm, obtained from TiB – 30 wt. % Ti materials, was studied. Their optimal values were determined: the delay time interval was 3–3.5 s, the press plunger speed interval was 40–50 mm/s. Using the results of SEM and XRD, the structural features of the obtained materials were studied, and their texture was determined. The physical and mechanical characteristics of the obtained materials were measured: porosity, hardness, microhardness.

Keywords

SHS; SHS-extrusion; ceramic material; titanium borides; titanium alloy.

Introduction

Titanium alloys and composite materials have found wide application in aviation technology, rocketry, automotive industry, in medicine as biological implants [1, 2]. Promising materials include titanium borides, which have a high hardness up to 25–35 GPa and a high melting point of 3225 °C, which makes it possible for parts made on their basis to work in corrosive environments. The presence of titanium borides significantly increases the tribological characteristics of titanium alloys [3–5].

Over the past five years, researchers have implemented many different approaches and methods to obtain composite materials based on titanium borides. These methods include: selective laser melting [6], hot forging [7], equal channel angular pressing [8], spark plasma sintering [9], hot extrusion [10], etc. Each method has its own advantages and disadvantages, but all they involve a large number of technological steps and require a large amount of energy and time. SHS extrusion is a promising method for obtaining materials based on titanium borides [11, 12]. The objective of this method is to give the synthesized combustion products a certain shape and size by extruding them through a forming die. In this case, the structure formation of the material occurs under conditions of high-temperature shear deformation. A significant advantage of SHS extrusion over most of the known methods is the ability to obtain a finished product in tens of seconds, while the synthesis of the material and the formation of a product of a given size and shape occurs in one technological operation.

The present paper is aimed at obtaining long cylindrical rods with a diameter of 3 mm and a length of up to 350 mm from a cermet material based on TiB – 30 wt. % Ti by the SHS extrusion. The effect of process parameters on the length and quality of extruded rods is studied, the microstructure is studied, and the porosity, hardness and microhardness of the materials obtained are measured.

Experimental

A composite material based on titanium monoboride with the presence of excess titanium was chosen as the object of research. The choice of the titanium binder is due to the fact that it imparts plasticity to the synthesized material during high-

Table 1

Characteristics of raw materials

Powder	Powder grade	GOST, TU	Basic substance content, at least, %	The particle size of the main fraction, less than, μm	Bulk density, g/cm^3
Ti	PTOM	TU 14-22-57-92	99.0	80	1.36
B (бор черный)	B-99A	6-02-585-75	99.5	10	2.34

temperature shear deformation, and also has a coefficient of thermal expansion close to titanium monoboride, which makes it possible to obtain a material with minimal levels of thermal stress. As it was established earlier, materials based on titanium monoboride, containing 20–40 wt. % of titanium binder, have the best formability [13]. In this paper, the composition TiB – 30 wt. % Ti was chosen. Table 1 shows the characteristics of the original powders.

Long cylindrical rods were obtained by SHS-extrusion, combining combustion processes in the mode of self-propagating high-temperature synthesis (**SHS**) and high-temperature shear deformation [12]. The main structural and technological parameters for SHS extrusion are: delay time, speed of movement of the press plunger, mass and relative density of the original workpiece, pressing pressure, degree of deformation and geometry of the matrix.

In a ball mill, a charge mixture was mixed from the initial components of 87.1 wt. % Titanium and 12.9 wt. % Boron. Cylindrical workpieces weighing 40 g, 25 mm in diameter, 40 mm in height, and a relative density of 0.43 were obtained by cold uniaxial pressing. In the experiments on the production of rods by the SHS-extrusion method, a forming die with an outlet section diameter of 3 mm, a taper angle of 120 °C, and a 30 mm length of the forming band was used. As a result, cylindrical rods 3 mm in diameter, up to 350 mm long with a porosity of no more than 3 % were obtained (Fig. 1).

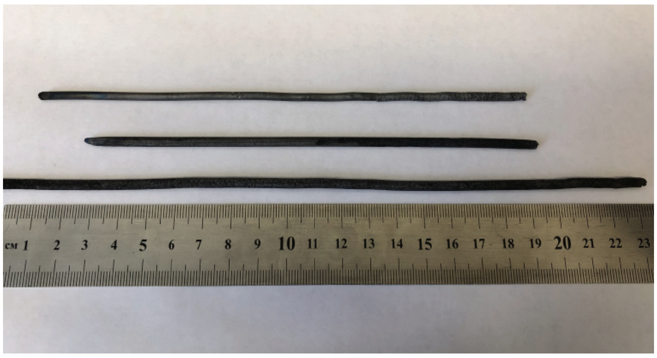


Fig. 1. TiB – 30 wt. % Ti rods obtained by SHS-extrusion

The equipment of the ISMAN Distributed Shared Use Center was used to carry out materials science studies of the obtained rods: a field emission scanning electron microscope of ultra-high resolution Carl Zeiss Ultraplus (made in Germany), an ARL X'TRA powder X-ray diffractometer, and other certified methods and techniques.

Results and discussion

In this paper, the influence of the delay time from the beginning of the initiation of the combustion process to the application of the pressure of the press plunger on the length of the extruded rods was studied, while the speed of the press plunger and the value of the applied pressure remained unchanged (U = 70 mm/s, P = 50 MPa). The main criterion for determining the optimal delay time was the maximum length of the extruded rod and its quality. To generate statistical data for each value of the delay time, five experiments were carried out, the average value of the length of the rods without macrodefects was calculated, after which a graph of the dependence of the length of the extruded rod on the delay time was plotted (Fig. 2). It can be seen from the presented graph that the optimal range of values of the delay time is $3 < t_d \leq 3.5$, which corresponds to the maximum length of the extruded rod 240–260 mm. At smaller values of the delay time $t_d < 3$ s, and as a consequence, earlier application of pressure, the material is squeezed out in the form of separate parts, without having time to compact. An increase in the delay time before the application of pressure leads to an increase in the time interval in which the material has time to consolidate and extrude in the form of a cylindrical rod, but at the same time not to lose its plastic properties. With a later application of pressure at $t_d > 3.5$ s, the synthesized material cools down, it loses its ability to high-temperature shear deformation, and the length of the extruded rods decreases. Subsequently, the area of the forming band is blocked and the extrusion is stopped.

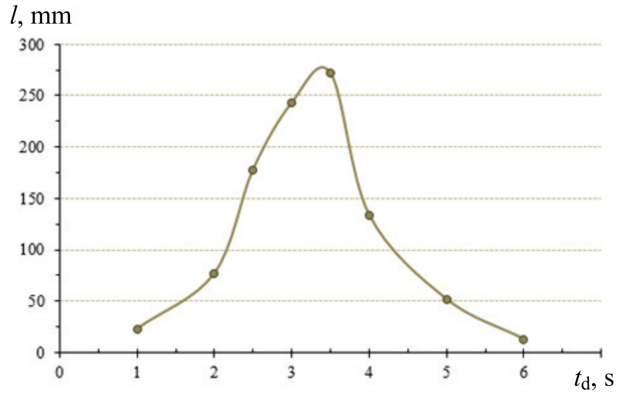


Fig. 2. Dependence of the length of the extruded rod on the delay time

At the next stage of the experiments, the speed of the press plunger was varied in the range $U = 10–90$ mm/s, at constant pressure and delay time ($t_d = 3.5$ s, $P = 50$ MPa). It was found that the optimal range of the press plunger speed is $40 \leq U < 50$ mm/s (Fig. 3), which corresponds to a stable mode of extrusion of samples with a smooth defect-free surface. In the region of high velocities $U > 50$ mm/s, an unstable extrusion mode with many cracks and high material porosity is observed. At speeds below 40 mm/s, a more rapid blockage of the matrix occurs due to increasing heat losses, a defective surface is formed and the rod may disintegrate into several parts. In the region of high velocities ($V > 50$ mm/s), the material does not have enough time for forming and consolidation, as a result, it is squeezed out in the form of separate parts. The porosity of the rods obtained was measured by the hydrostatic method. It was found that the smallest value of porosity in the materials obtained was 3 %, which was achieved during extrusion at a speed of $U = 40$ mm/s.

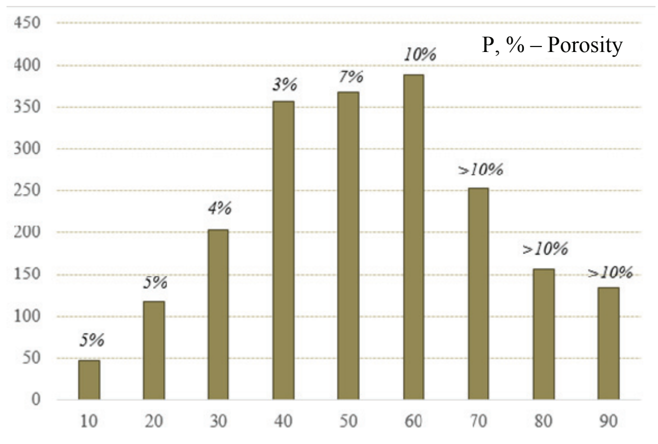
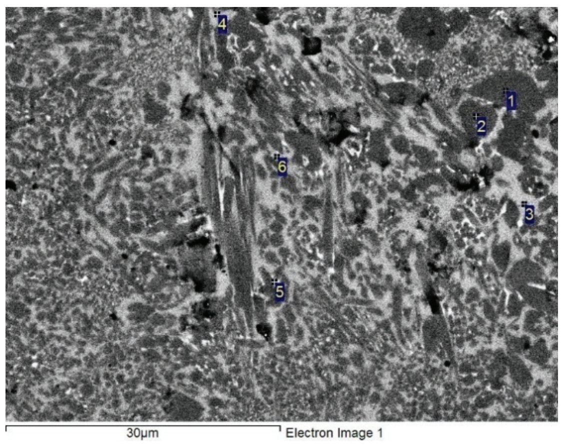


Fig. 3. Dependence of the total length of the extruded rod of the press plunger speed

The microstructure of the material (Fig. 4) is presented in the form of titanium monoboride whiskers (dark gray areas) with a cross-sectional size of 0.5–2 μm, located in a titanium matrix (light gray areas). Also, the material contains a small proportion of larger inclusions of titanium monoboride with a diameter of 3–4 microns and a length of up to 10–12 microns. The presence of elements such as Al, Cr, Fe, Ni, and W in the material is explained by the insufficient purity of the starting powders. Carbon is present on the surface of the section from polishing pastes. All impurity elements contain less than 5 %, which is confirmed by the results of X-ray phase analysis (Fig. 5). The X-ray diffraction pattern also revealed a shift of the main peaks relative to the standard position, which indicates texturing in the material during SHS extrusion. It can also be concluded that not all boron has reacted with titanium to form titanium monoboride, but is also present in the form of a solid solution of boron in titanium – α–Ti [B].

The microhardness of the obtained materials was studied, which was, respectively, at a load of 100 g: 537 and 978 kg/mm^2 along and across the rod. The scatter of values was at the level of measurement error, which indicates a uniform distribution of structural components in the volume of the extruded rod.



Spectrum	B	C	Al	Ti	Cr	Fe	Ni	W
1	27.20	9.02	0.25	63.54				
2	26.42	7.97	0.24	65.37				
3		4.33	0.58	95.09				
4		12.16	0.57	87.28				
5		5.92	0.76	86.31	3.26	0.95	0.84	1.95
6		4.58	0.66	87.93	2.93	0.67	1.01	2.22

Fig. 4. Microstructure of a TiB rod – 30 wt. % Ti in a cross section

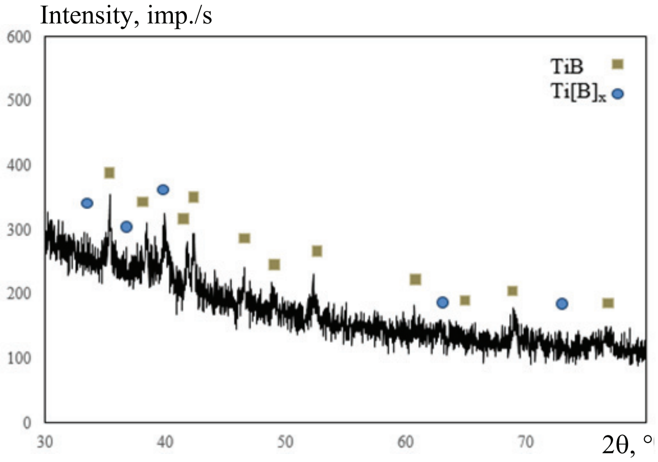


Fig. 5. XRF of a thin section TiB – 30 wt. % Ti

The difference in the obtained values can be explained by the presence of texturing of the material, and we also find ourselves in the area enriched and depleted in titanium binder. The macrohardness of the materials obtained was 69–70 HRC.

Conclusion

For the composition TiB – 30 wt. % Ti, the technological modes of SHS extrusion (the delay time and the speed of movement of the press plunger) were studied and optimized. It has been established that the dependence of the length of the extruded rods on the indicated parameters is extreme. The optimal range of values for the delay time before the application of pressure is $3 < t_d \leq 3.5$ s, and the optimal range of the press plunger speed is $40 \leq U < 50$ mm/s. Subject to optimal technological conditions, cylindrical rods with a diameter of 3 mm, a length of up to 350 mm with a porosity of no more than 3 % were obtained.

The microstructure of the rod is presented in the form of titanium monoboride whiskers with a cross-sectional size of 0.5–2 µm, located in a titanium matrix. Also, the material contains a small proportion of larger inclusions of titanium monoboride with a diameter of 3–4 microns and a length of up to 10–12 microns. It has been established that during high-temperature shear deformation under SHS-extrusion conditions, texture is formed in the material.

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