MICROSTRUCTURE AND MECHANICAL PROPERTIES OF P/M TITANIUM MATRIX COMPOSITES REINFORCED WITH TiB

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Abstract: *The results of the estimation for the influence of titanium diboride content in the initial powder mixture on the basic mechanical properties at the tests on tension and compression are presented. It is shown that the porosity of sintered at 1250 ^{0}C preforms from TiH_2-TiB_2 powder mixture increases with increasing of titanium diboride content in the initial charge, which is due to the manifestation of the Frenkel effect at sintering. The values of tensile strength, hardness and elastic modulus, despite some porosity growth of the sintered alloy, increase with the addition of 5 % of TiB_2 powder, while increasing the content of the high modulus component in the mixture to 10 % leads to decrease in the level of these characteristics. The plasticity of sintered alloys monotonically decreases with increasing of the boride component content. At compression tests, the yield point and the compressive strength increase monotonically with increase in TiB_2 content, despite the increase in porosity of the latter, due to a significantly lower effect of porosity on the value of the resistance to deformation in compression compared with tension. The use of hot forging of sintered powder preforms leads to increase of strength properties and hardness of the composites.*

Keywords: POWDER, TITANIUM, BORIDE, HYDRIDE, COMPOSITE, STRENGTH, POROSITY, SINTERING, HOT FORGING.

1. Introduction

Titanium matrix composites (TMCs) reinforced with high strength and high stiffness particles/whiskers have generated extensive research interests due to their low cost, ease of fabrication as well as excellent mechanical properties. They have been found a wide range of applications in various fields for commercial automotive, aerospace and advanced military applications, owing to unique properties which include high specific strength, specific combination of good mechanical properties and high temperature durability and specific fatigue resistance (high cycle fatigue) [1].

A number of reinforcements have been suggested/used including Ti_5Si_3, CrB, B_4C and SiC and TiC [1–6]. The reinforcements usually need to be stiffer than the matrix, have a similar thermal expansion coefficient to the matrix and to be also chemically stable. Among the ceramic reinforcements TiB are of particular interests since they are well compatible with titanium matrix even at high temperatures and its high hardness and elastic modulus [7-9].

Technological schemes for manufacturing of the titanium matrix composites are based mainly on the use of powder metallurgy techniques [8-10].

At the same time, one of the most effective ways for production of titanium based sintered materials is the use of titanium hydride powders as raw materials instead of titanium powder, which provides a significant activation of diffusion processes during sintering due to the increased density of the crystal lattice of hydride. As well as the possibility occurs of additional purification of the interphase boundaries at the expense of atomic hydrogen released during the decomposition of titanium hydride [11-13], which leads to higher achievement of complex physical and mechanical characteristics obtained alloys. The economic efficiency of TiH_2 using is also due to its lower cost compared to pure titanium powders.

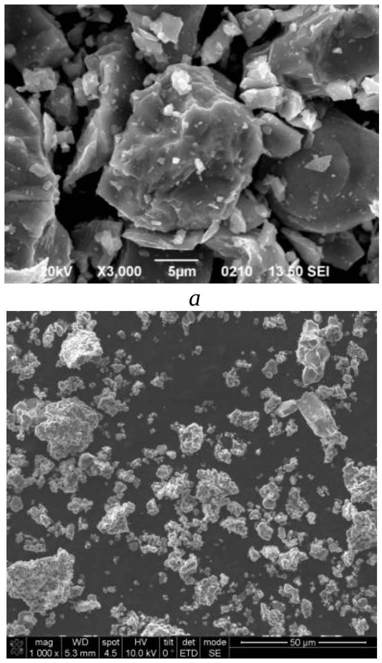
Besides that, for providing of dense (practically non-porous) material the hot forging of porous preforms is one of the most efficient processes of powder metallurgy.

The **aim** of this article was to study the regularities of the structure and phase formation at sintering and effect of hot forging on mechanical properties of titanium based composites reinforced with TiB.

2. Materials and experimental procedure

For experimental investigations of mixture composition influence the on the structure and phase composition of the pseudoallys, the mixtures of titanium hydride and titanium diboride TiB_2 powders (fig.1) were prepared.

The dispersion composition of the powders was determined using the Malvern Mastersizer 2000 laser analyzer.



a

b

Fig. 1. *The morphology of the initial TiH_2 (a) and TiB_2 (b) powders*

The mixtures of titanium hydride and 5 or 10 % (mass.) titanium diboride powders were blended in a ball mill and then consolidated at 650 MPa to obtain compacts, which were then sintered at 1250 ^{0}C for 4 hour in vacuum. As the base material for the comparative assessment of high-modulus component effect on the alloys characteristics, the compacts made from the titanium hydride were sintered without the use of reinforcing TiB_2 additives in the initial mixture.

The density and porosity of the blanks after consolidation and sintering were determined by the method of hydrostatic weighing.

Some of the sintered samples were then heated for 10 min. in argon and hot forged on the mechanic arc-type stator press in the semiclosed die (fig. 2).

Mechanical tensile and compression tests of the produced composites were carried out at room temperature using the Instron 3376 machine.

The fracture structure of the samples after the mechanical tests was studied using fractographic analysis. The study of materials microstructure was carried out using scanning electron microscope

TESCAN VEGA 3. The Vickers hardness was measured on a Wolpert hardness meter.

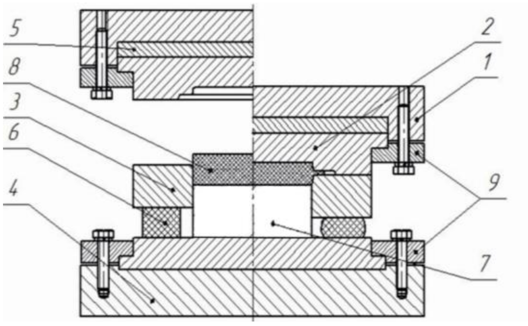


Fig. 2. *The experimental die for hot forging: 1 - the top plate; 2 - upper semimatrix; 3 - lower semimatrix; 4 - lower support plate; 5 - support plate; 6 - elastic element; 7 - lower punch; 8 - stampable blank; 9 - upper and lower fixing flanges*

Investigation of physical processes occurring during the heating of powder compacts took place on an automated dilatometric complex [14]. The X-Ray Diffraction (XRD) analysis of the samples was performed using DRON-3M diffractometer with CoKα radiation in the range of angles from 20 to 130 degrees. The sample during the analysis were rotated around its axis. The analysis of XRD data was performed by standard methods using the ASTM card index.

3. The results and discussion

The estimation of the initial powders granulometric composition showed that average size of the titanium diboride particles did not exceed 10 microns, whereas the maximum of the distribution curve for the particles of the titanium hydride powder was about 30-40 microns (Fig. 3).

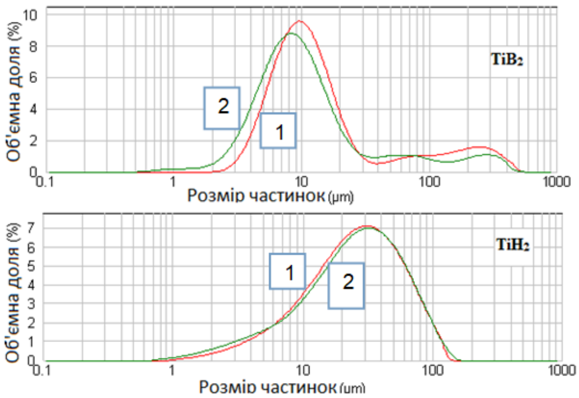


Fig. 3. *Particles size distribution of the used TiH_2 and TiB_2 powders: 1 - initial powders; 2 - powders after ultrasonic treatment*

Sintering process of titanium hydride based powder mixtures is accompanied by the emission of atomic hydrogen contained in the titanium hydride crystalline lattice, which is an effective metal cleaner from unwanted admixtures such as oxygen and chlorine. During high-temperature sintering hydrogen is almost completely removed from titanium and the hydride is converted into sintered, technically pure titanium with residual content of hydrogen in the alloy at the level of ~0.005 %.

As it is shown in [7, 13], in the process of sintering of mixtures of titanium powder with titanium diboride, TiB_2 particles at high temperatures actively interact with the titanium matrix phase by the reaction $TiB_2+Ti \rightarrow 2TiB$ with the release of titanium monoboride particles.

The the X-ray diffraction analysis results of the sintered samples from the TiH_2+TiB_2 mixture (fig. 3) indicate that in the alloy, in addition to the main titanium matrix phase, the TiB phase with orthorhombic lattice and the presence of traces of titanium compounds with boron of another concentration (Ti_3B_4 and Ti_2B_5), are present in the alloy. The initial TiB_2 phase on the radiographs of the sintered alloys are not identified.

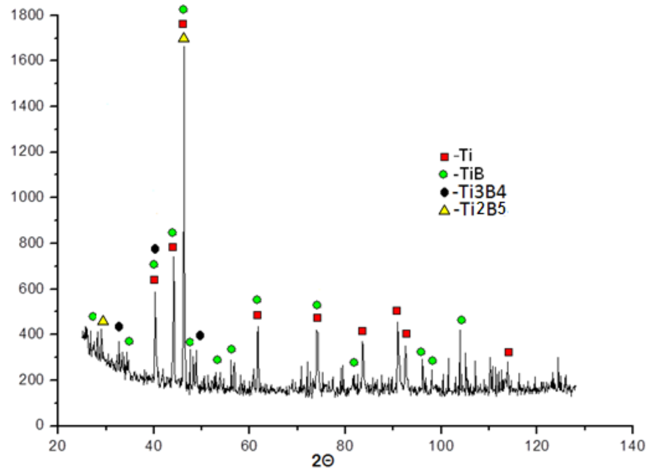


Fig. 3. *X-ray pattern of the alloy, sintered from the mixture of TiH_2+TiB_2 powders*

The estimation of component composition of the initial powder mixture influence on the amount of the sintered specimens porosity under the same sintering conditions showed that material porosity increases with increasing of titanium diboride content. While the porosity of titanium made from TiH_2 powder without reinforcing additives does not exceed 1,5 %, the specimens sintered from mixture with 10% TiB_2 have already a significant porosity of about 7,5 % (fig. 4, a).

Increasing of the sintered samples porosity with incorporation of the titanium diboride into the mixture composition can be explained by the effect of secondary pore formation. Since the Ti diffusion coefficient in boron is negligible compared to boron in titanium diffusion coefficient [15], in the process of sintering practically one-sided diffusion of boron atoms into titanium matrix occurs with the formation of needle-like TiB particles, while the flow of vacancies diffuses in the opposite direction, which leads to the formation of secondary porosity (the Frenkel effect) [16].

The presence of residual porosity makes its effect on the level of the main mechanical characteristics of the alloy. Despite the generally hardening nature of the TiB reinforcing phase in the metal matrix composite, when the content of the latter is increased in the initial powder mixture, competition occurs between the effects of reinforcing of the matrix phase (which leads to an increase in the hardness and strength of the sintered alloy), and reducing of these characteristics values due to the presence of residual porosity.

Thus, from fig. 4, b it can be seen that sintered samples obtained from titanium hydride without the use of the reinforcing phase have the tensile strength value of about 650 MPa with the elongation of ~10 %. These values are comparable to the standards for technically pure titanium (Grade 4). Incorporation of 5 % TiB_2 powder in the mixture, despite some porosity growth of the sintered alloy in comparison with pure titanium, leads to increase in the composite strength up to 750÷800 MPa (fig. 4, b), but it is accompanied by a decrease in plasticity to 2÷3 % (fig. 4, c).

With an increase of the high-modulus component content in the mixture to 10 %, the negative effect of increasing porosity prevails over the reinforcing effect and the value of ultimate stress limit of the latter decreases to 620 MPa. Furthermore, such increase of TiB_2 content in the mixture and, accordingly, TiB phase content in the sintered material, also leads to a catastrophic reduction of its plastic characteristics, which does not exceed 0,5 %.

Similar regularity concerning the effect of titanium diboride content in the mixture on composite strength appears as well for the parameters of the elastic modulus (fig. 4,e) and hardness (fig. 4,d).

Typical tensile curves for technically pure titanium and composites with different contents of TiB reinforcing phase are presented at fig. 5. Comparison of «σ-ε» diagrams type for materials with different contents of high modulus component showed their significant difference due to their significantly different plasticity. For deformation curve of titanium sintered without borides additives, the presence of a large-scale zone of plastic deformation where the deformation grows practically without increasing stresses, is characteristic. In the composite obtained from mixture with 5 % TiB_2, the length of this area significantly decreases, and with the increase of titanium diboride content up to 10 %, the view of "σ-ε" diagram corresponds to those of fragile destruction.

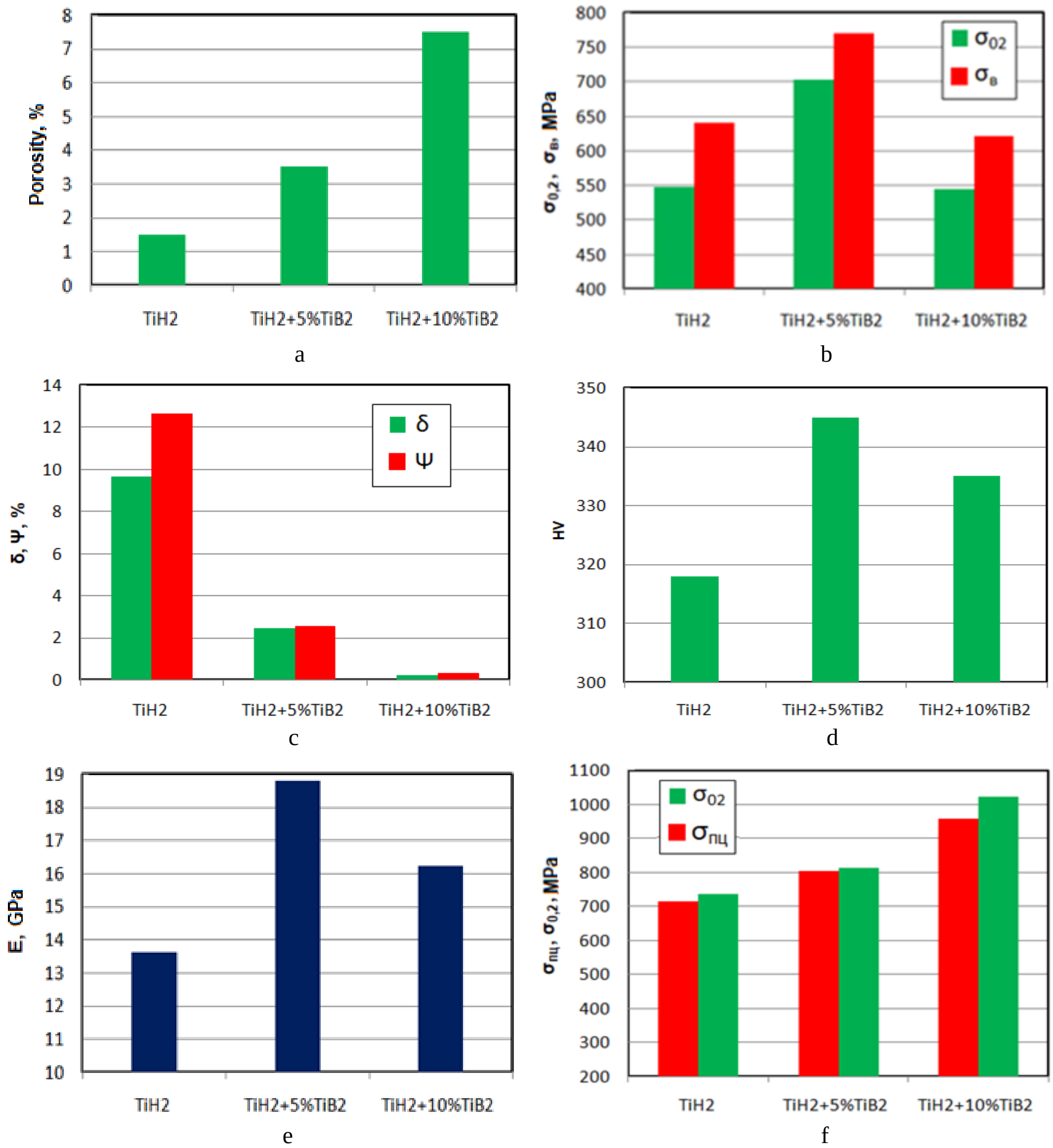


Fig. 4. *The effect of titanium diboride content in powder mixture on the basic mechanical characteristics of sintered alloys when tested for tensile strength (a, b, c, e), hardness (d) and compression (f)*

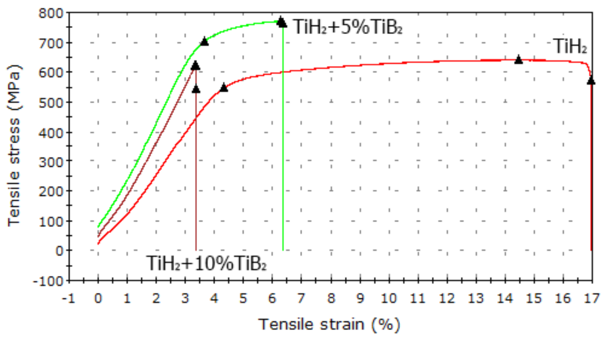
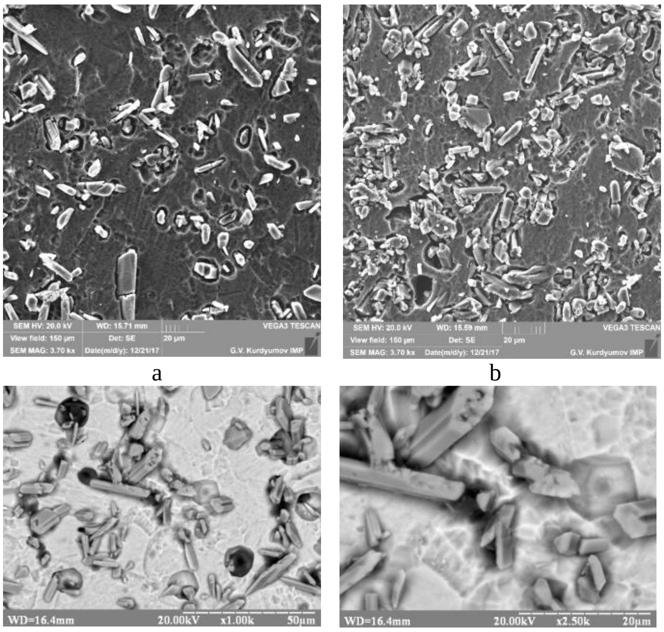


Fig. 5. *Typical deformation curves for sintered composites at tensile tests*

Taking into account fundamentally different mechanisms of tensile deformation for the investigated alloys of various component compositions, it is obviously of interest to compare their mechanical characteristics when tested for compression. As in can be seen from fig. 4,f, unlike tensile tests, in case of compression tests the value of both proportional limit and yield point monotonically grow with increase in the TiB_2 content in the mmixture despite the increase of porosity. This regularity, obviously, is due to considerably less influence of material porosity on the value of the compressing strength compared with the tensile strength due to decrease in porosity value during blanks upsetting, which is also confirmed by the results of theoretical works [17].

The analysis of the sintered specimens microstructure (fig. 6) showed that in the process of sintering, the titanium diboride inclusions actively interacts with the titanium matrix, resulting in formation of titanium monoboride particles, which mainly have a needlelike shape with cross section size of 1÷5 μm and a length of 10÷25 μm.

It is well known that one of the most effective methods for improving the basic mechanical and operational properties of sintered powder materials is to reduce porosity and provide favorable conditions for the intensification of intergranular diffusion processes between powder particles. In order to realize these effects, approaches are widely used, based on the employment of hot forging of porous powder preforms [18-21].



a b

c d

Fig. 6. *Microstructure of the composites made from powder mixtures with 5 % (a, c ,d) and 10 % (b)* TiB_2

Comparative estimation of the effect of porous preforms hot forging on the basic mechanical characteristics of the composite had shown, that this kind of treatment of sintered billets significantly increased the values of both proportional limit and yield point (fig. 7,a), and the hardness of the material (fig. 7,b).

It is noteworthy that the effect of hot forging to a large extent is shown for a composite obtained from the mixture with a higher content of titanium diboride (10%). The observed regularity is due to the following reasons. As it was shown above (fig. 4a), the increase the content of boride component in the alloy composition results in a corresponding increase of the composite porosity after sintering as well as associated with the latter mechanical characteristics (fig. 4,b,d,e). At the same time, the use of hot forging provides obtaining of practically non-porous (with porosity of 0,5 ÷ 1,0 %) powder materials. Thus, after hot forging, the effect of residual porosity on the properties of the alloy, which is manifested for sintered materials, is eliminated, and the mechanical characteristics of such alloys will be determined (at the same other conditions) mainly by the content of the high modulus boride component, the dispersion of the structure and the state of the intergranular boundaries.

Taking into account the known influence of hot forging on the structure dispersion and the intensification of intergranular diffusion, we can conclude that the increase in the properties of the alloy with a minimal content of the boride component (5% TiB_2), with a small initial porosity after sintering (≈1.5%) is due mainly to these factors, whereas for the billets with 10 % TiB_2 significant increase in properties is due as well to the effect of removing residual porosity during forging.

4. Conclusion

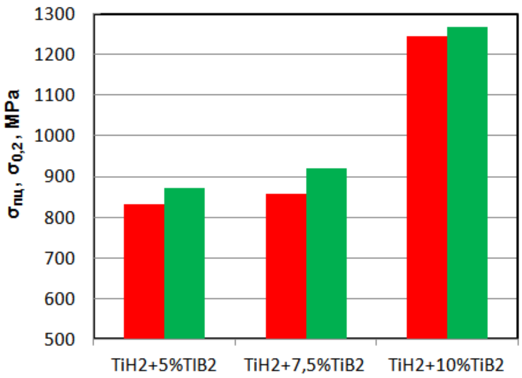
1.Sintering of the samples made from a powder mixture TiH_2-TiB_2 is accompanied by active interaction of diboride particles with titanium matrix, as a result of which the the formation of needle-like titanium monoboride particles in the titanium matrix phase takes place.

2. Porosity of the sintered samples increases with the increase of titanium diboride content in the initial mixture from 1.5 % for samples without boride inclusions, to 7.5 % for materials with 10 % TiB_2 due to development of Frenkel effect at sintering.

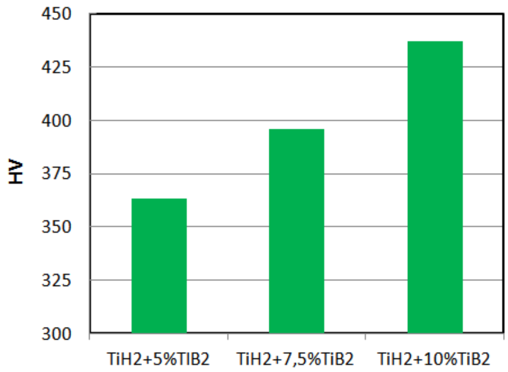
3. The values of tensile strength, hardness and elastic modulus, despite some porosity growth of the sintered alloy, increase with the insertion of 5 % of TiB_2 powder into the mixture, while increasing the content of the high modulus component to 10% leads to a decrease in the level of these characteristics. The plasticity of sintered alloys monotonically decreases with increasing content of the boride component.

4. Unlike tensile tests, at compression tests values of limit of proportionality and yield point increase monotonically with an increase of TiB_2 content despite the increase in porosity of the latter.

5. The use of hot forging of sintered powder preforms leads to increase of strength properties and hardness of the composites.



a



b

Fig. 7. *The effect of titanium diboride content in powder mixture on proportional limit and yield point at compression testing (a) and hardness (b)*

5. References

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