Preparation and mechanical properties of SiC-TiN composite

A V Leonov, M A Sevostyanov, A S Lysenkov, A S Baikin, M G Frolova and A M Tsareva
Baikov Institute of Metallurgy & Materials Science (IMET RAS), Moscow, Leninskiy prospect 49, Russia
Email: a_leonov.imet@list.ru

Abstract. A method has been developed for the production of a SiC-TiN composite material based on combining the processes of hot pressing and reaction sintering. The main difference of the method is the use of pure titanium as initial components. The mechanical properties and microstructure of the obtained samples were studied. X-ray phase analysis showed the absence of pure titanium and carbotitanium in the samples. Investigations of the influence of the type of sintering additive on the structure and properties of the resulting material were carried out.

1. Introduction
Recently a lot of attention in materials given to new superhard composite materials. Materials based on the silicon carbide (SiC) is widely used in this area. Silicon carbide has unique combination of chemical, physical, electrical and magnetic properties. Because of these properties SiC is widely used in many industry areas and products such as: heating elements, nuclear power plant details, turbine and pump details, etc. One of the most important advantages of silicon carbide is high wear resistance and heat resistant. [1] However, it is not possible to obtain the SiC material with theoretical density without additives. It has been reported that some additives can enhance the SiC material and improve its properties. Composite material based on silicon carbide with titanium nitride (TiN) is perspective. TiN is a material with good chemical and thermal stability, high hardness and thermal conductivity. TiN additives in SiC materials also reduce the electrical resistance of the material, which will make possible to apply the method of electro-erosion cutting for processing process. [2]

Obtaining dense, high-quality materials based on refractory compounds requires a long exposure at high temperatures (> 2000°C). To reduce the firing temperature of the SiC-TiN material, sintering additives of various systems, such as Y2O3-Al2O3-ZrO2 and Y2O3-Al2O3, can be used. In the current literature there are data on composite SiC-TiN materials [3,4], however for the production of such materials an expensive powder of titanium nitride powder is used. It is possible to simplify the production of the SiC-TiN composite material by carrying out the nitriding of pure titanium during the sintering of the ceramic material.

2. Experimental procedure
SiC obtained by the SHS synthesis method (<25 μm), Ti powder obtained by sputtering in a plasma torch (<25 μm) were used. As a sintering additive, the addition of the Y2O3-Al2O3-ZrO2 system, obtained by the plasmchemical method (Ya1Zr), and also Y2O3-Al2O3 (3: 5) (YAG), obtained by solid-phase synthesis, were used. 7 different compositions were prepared, differing in the amount of Ti and also in the form of a sintering additive.
The compositions are listed in Table 1. Components were mixed in a Pulverisette 6 planetary mill in teflon beakers for 1 hour in isopropyl alcohol medium with a material: balls ratio of 1:10. As grinding bodies were balls of zirconia. After mixing, the resulting powders were dried in a drying oven at a temperature of 80 °C, and then sieved through a sieve having a cell size of 63 μm and then hot-pressed. The blank was a disc d = 25 mm, the weight of the sample was 10 g. The firing was carried out in a nitrogen medium in a graphite mold. The firing was carried out stepwise. The first exposure was carried out at a temperature of 1600 °C for 30 minutes. Then the temperature was raised to 1850 °C and a second exposure was carried out for 30 minutes. The pressure was 30 MPa. The samples were then removed from the mold and the samples obtained were mechanically processed before testing and measurement.

Table 1. Compositions of different samples and processing conditions

| Material       | mass %  | Processing conditions |
|----------------|---------|-----------------------|
|                | SiC     | Ti   | YAG  | YAIZr | T1=1600, 30 min | T2=1850, 30 min |
| SiC-Ti-0-1     | 93      | 0    | 7    | 0     |
| SiC-Ti-10-1    | 83      | 10   | 7    | 0     |
| SiC-Ti-20-1    | 73      | 20   | 7    | 0     |
| SiC-Ti-0-2     | 93      | 0    | 0    | 7     |
| SiC-Ti-10-2    | 83      | 10   | 0    | 7     |
| SiC-Ti-20-2    | 73      | 20   | 0    | 7     |
| SiC-Ti-40-2    | 53      | 40   | 0    | 7     |

The density of the material was determined by hydrostatic weighing in water, while the true density was calculated by the additivity rule. For bending tests, specimens were produced. The tests were carried out on an Instron 3382 unit with a load cell of 100 kg. For microhardness measurements and microscope studies, metallographic samples were made. Microhardness was measured on a Wolpert Wilson Instruments 401/402-MVD unit with a load of 0.98 N and. Microstructure studies were carried out on a Scanning Electron Microscope (SEM) of the Tescan Vega II SBU (Tescan, Czech Republic). For X-ray phase analysis, the Ultima IV (Rigaku, Japan) was used.

### 3. Results and discussion

The main task of the work was to carry out the nitration of the initial titanium in the billet during sintering. The firing regimes of the SiC-TiN composite were selected [5]. According to X-ray analysis, the material obtained is represented by two main phases of SiC and TiN (Figure 1). Titanium in its pure form, as well as carbotitan, which could have formed during firing in a graphite mold, was not detected.

Investigations of the influence of the type of sintering additive and the amount of titanium in the composition on the mechanical properties of the material and the microstructure were carried out. Data are presented in Table 2.

Table 2. Mechanical properties of materials

| Material       | Density, g cm³ | Relative density | Bending stress, MPa | Microhardness, GPa |
|----------------|----------------|------------------|---------------------|--------------------|
| SiC-Ti-0-1     | 3.11           | 0.85             | 225.46              | 9.45               |
| SiC-Ti-10-1    | 2.98           | 0.87             | 238.00              | 10.73              |
| SiC-Ti-20-1    | 2.89           | 0.8              | 263.14              | 12.80              |
| SiC-Ti-0-2     | 3.07           | 0.83             | 190.73              | 10.11              |
| SiC-Ti-10-2    | 2.95           | 0.86             | 175.38              | 11.14              |
| SiC-Ti-20-2    | 2.81           | 0.77             | 166.15              | 13.27              |
| SiC-Ti-40-2    | 3.25           | 0.81             | 153.91              | 21.83              |
Figure 1. XRD patterns of SiC-Ti-10-1 (1) and SiC-Ti-20-1 (2)

It can be seen from the studies carried out that the use of the sintering additive YAG leads to a greater strength value than the use of the YAlZr additive. The bending strength of the material using the YAG additive is in the range of 230-270 MPa, while for the YAlZr additive, within the range of 150-170 MPa. The relative density of the resulting materials is not high and on the average is 0.82. As can be seen from the microphotographs of the microstructure presented (Figure 2), the increased bending strength of the samples with the addition of YAG is associated, first of all, with the shape of inclusions of titanium nitride (light areas in the figure). Image (a) shows that TiN in the material is distributed in the form of elongated inclusions, while in the images (b) and (c) TiN has a basically spherical shape.

An increase in the amount in the initial components of titanium, and as a consequence, an increase in the amount of titanium nitride leads to an increase in the microhardness of the samples. A particularly sharp increase in microhardness was found in the SiC-Ti-40-2 sample.
Figure 2. Microstructure of SiC-based composites: (a) SiC-Ti-20-1; (b) SiC-Ti-20-2; (c) SiC-Ti-40-2

4. Conclusion
The samples of the composite ceramic material SiC-TiN were obtained. The prospect of introducing titanium nitride into a carbide-silicon matrix was shown. The introduction of TiN led to increase the microhardness of the material. The technology of obtaining the material by combining the processes of hot pressing and reaction sintering has also been studied. For the production of titanium nitride, hot pressing was carried out in a nitrogen atmosphere, which resulted in the simplification of manufacturing technology and the cheaper material due to the use of cheaper components. The use of the YAG additive is more promising for obtaining a stronger material.

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