

# SYNTHESIS OF COMPOSITE MATERIAL ON THE BASIS OF TITANIUM SILICON CARBIDE BY MECHANICAL ACTIVATION AND SPARK PLASMA SINTERING

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Synthesis of powder composition for the subsequent formation of titanium silicon carbide is an urgent problem because ternary composition receive from elementary powders extremely difficult.

Most researchers followed mechanical activation and consolidation techniques to synthesize  $Ti_3SiC_2$  single phase by using several starting mixtures (e.g.,  $TiH_2/Si/C$ ,  $Ti/Si/C$ ,  $Ti/SiC/C$ ,  $Ti/Si/TiC$ , and  $TiC/Si$ ) and varying the composition of the starting reactant powders (e.g., Ti, Si, C, SiC, TiC, Al, etc.).

For manufacture of experimental samples of composite powders we used the following raw materials: powder titanium TPP-7 fraction less than 375 microns, technical silicon carbide powder particles less than 10 microns, graphite powder S-1.

Mechanical activation of powder mixture of  $Ti/SiC/C$  in a molar ratio of 3:1.25:0.75 performed in a planetary mill SAND. Specific surface of the original mixture is small (about  $2\text{ m}^2/\text{g}$ ), at the initial stage of activation is grinding components and specific surface increases up to  $10 - 12\text{ m}^2/\text{g}$ . Then in the process of formation of conglomerates and composite particles occurs reduction of the specific surface. At certain conditions (frequency of rotation of more than  $280\text{ min}^{-1}$ , duration more than 2 hours), the mixture occur solid-phase reactions leading to the formation of titanium carbide and titanium silicon carbide. This reduces the surface energy and total energy of the system, which is reflected in the reduction of the specific surface up to  $1.5 - 5\text{ m}^2/\text{g}$ .

The various modes of mechanical activation of powder mixture  $3Ti+1.25SiC+0.75C$  (mol.%) showed that in the course of processing formed composite particles containing chemical elements of ternary compounds Ti-Si-C.

According to the results of XRD the treated mixture corresponds to the composition of  $Ti_3SiC_2$ - $Ti_xC$ - $TiSi$ , and the line of pure titanium and carbon are not fixed.

Spark plasma sintering (SPS) of the mechanically activated powder composites was held on the devise Dr. Sinter SPS-1050b in graphite die at temperatures of  $1,200 - 1,300$  degrees C and a pressure of 30 MPa, isothermal holding 1, 5 and 25 min. Average heating rate was 80 degrees C/min.

The presence of phase  $Ti_3SiC_2$  experimentally confirmed on all samples after the above SPS-modes. In samples at a temperature of 1200 degrees C traced impure, quite porous structure, interspersed with grains SiC.

The silicon carbide grains are present in the sample structure, consolidated at 1300 degrees C/1 min, however, significantly fewer. The  $Ti_3SiC_2$  grains can be seen in the secondary electrons without prior etching of the surface of the samples obtained at a temperature 1300 degrees C and above. Revealed the existence of phase Ti-Si in the sample consolidated at 1400 degrees C/25min.

The typical structure of the experimental samples of the consolidated composite material presented on the fig. 1.

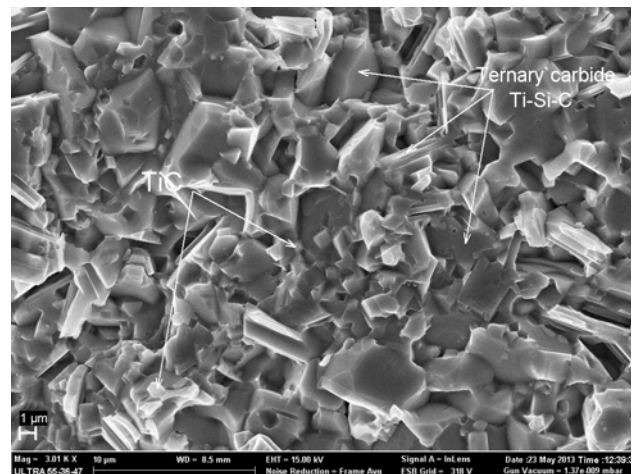


Fig. 1 FESEM image of a break sample of  $Ti_3SiC_2$  composite material (SPS 1300 degrees C/30 MPa/25 min) 3000×