MECHANICAL TREATMENT OF COMBINE (ZRB_2-SIC)-POWDERS FOR ADDITIVE MANUFACTURING OF HARD CERAMIC COMPOSITES

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The refractory compounds are the basic components of materials used in high-temperature engineering, such as thermal protection of space vehicles, electronics, etc. Among materials with high melting temperature, a special attention is paid to ZrB_2-based composites but due to a very high melting point the manufacturing of products based on these materials are difficult because a treatment of materials are almost impossible due to a high hardness. The new technique called additive manufacturing may be used for making samples with finely shapes and sizes after sintering process however it is needs to prepare such powders before forming and sintering.

It is known, that addition of SiC to ZrB_2 increases the density of sintered materials due to smaller melting temperature of SiC. The ZrB_2-SiC composites are usually obtained under pressing at temperatures higher than 2000 ^oC [2], and to decrease the sintering temperature, the powders undergo mechanical treatment in high-energy ball-milling. In this case, subsequent sintering will be activated due to increased number of defects, acceleration of diffusion processes etc., so sintering can be carried out during SPS-process. Unfortunately, data on the influence of mechanical treatment on the properties of powders and the process of subsequent hot pressing are poorly investigated. So, the aim of this paper is to study the influence of mechanical treatment of ZrB_2-SiC powders on their properties and properties of ceramic composites sintered by hot pressing.

The research was carried out using powder mixtures of ZrB_2 (d50 = 2.5 μm) and SiC (d50 = 4.2 μm) with SiC content of 10, 15 and 20 vol.%. The powders were mechanically treated in a planetary mill with acceleration of approximately 30g with time up to 20 minutes. Hot pressing of ceramic composites was carried out at the temperature of 1800 °C and pressure of 50 MPa with isothermal sintering for 30 minutes. X-ray with CuKα radiation was used to study structure, phase content and coherently diffracting domains (CDD). Scanning electron microscope Tescan VEGA-3SBH was used to determine the structure and average grain size.

It have been shown that increasing of treatment time are accompanied by increase of relative density, in addition, the morphology of particles has appreciably changed, from separate particles in the beginning state up to the formation of agglomerates in the end of treatment. The X-ray phase analysis of mixtures has shown that during the treatment there were no changes; addition of SiC to the mixture leads to the occurrence of its peaks. With increased treatment time, we have found a broadening of peaks due to increasing number of lattice defects and decrease of CDD or grain size from 46 down to 37 nm.

After sintering phase content did not change and the increase of treatment time in the planetary mill before sintering have no effect on CDD of sintered materials. This means that all defects are annealed during sintering process.

Addition of SiC leads to essentially increased sample density: its value goes up to 99% of a theoretical one for a powder with 20% of SiC, as compared to ZrB_2 going not higher than 76%.

It have been shown that relative density change of sintered materials are well described by a simple function like $Y = A*x^n$, where parameter n characterizes the speed of density change and according results addition of SiC to the mixture leads to up to four-fold decrease of **n**-value. This is due to that all defects accumulated during mechanical treatment are annealed during sintering and there are no changes of CDD values in sintered ceramics.

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