PRODUCTION OF HOMOGENEOUS COMPOSITE PRESS-POWDERS BASED ON ZRB_2 AND SIC FOR UHTCS

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Abstract. The aim of the research was to obtain superfine homogeneous composite powders, which, due to their unique composition and morphology, will be used to obtain ceramics with improved properties for the thermal protection of supersonic aircraft. The novelty of research was microstructure refining methods, selection of sintering additives/dopants and their combining action. As regards sintering additives and dopants, B_4C and graphite powder, carbon black and graphene structures were used. The use of the latter individually and in combination sintering additives was one of the innovations of the research.

1. Introduction

Materials working in the extreme environment are subject to permanent research. The extreme environment is the joint impact of temperature, chemically reactivity, mechanical stress, wear, etc. Such an environment is formed on the nose cone, wing leading edges and the propulsion system components of the hypersonic space vehicle (HSSV), generally during the HSSV's atmospheric re-entry and rocket propulsion. Already in its early reports, NASA underlined the necessity for rocket nozzles and thermal protection systems (TPSs) [1-3]. A particular attention was paid to the conditions associated with the entry into atmosphere, the rocket engine and the requirements to the materials of the leading edges and propulsion system of vehicles. A whole series of recommendations contained in the reports are still being prioritized for modern research. Studying the properties of refractory borides and carbides for hypersonic aerospace vehicles is a part of research carried out in the U.S.A. A great impact in the NASA reports was made in the definition of Ultra-High Temperature Ceramics (UHTCs), where zirconium and hafnium borides are the main candidates for HSSV. Based on the properties of zirconium boride ceramics, the ZrB_2-based UHTCs are rather promising in the TPS materials. Of special interest is the ZrB_2-SiC composite ceramics, on which a major part of the ZrB_2 research falls.

2. Experimental procedure

Milling and mixing of the powders were carried out in the tungsten carbide jar with zirconium oxide milling balls by the planetary micro mill PULVERISETTE 7 (FRITSCH, Germany). Zirconium oxide balls eliminated contamination of the powders with new elements. A novelty was the production of a multiphase mixture with equally distributed phases of different size and inhibitors, using ultrasonic, the planetary milling machine and granulator. Ultrasonic homogenizer TF-1000N and Granulator SD-1000 was also used. Production of ZrB_2 fine powders were carried out by carbo/borothermal reduction at 1500-1650°C in the high-temperature vacuum furnace FR210-30T-A-200-EVC (OXY-GON, USA) according to the following reaction: $2ZrO_2 + B_4C + 3C \rightarrow 2ZrB_2 + 4CO \uparrow$. In parallel, graphene structures were synthesized using modified Hummer's method; thereafter, reduction of the produced graphene oxide were carried out by chemical reagents, using ultrasonic and a microwave oven. Because graphene structures are characterized by the ability to aggregate, which prevent its homogeneous distribution in the matrix and have an affect the mechanical properties of the final product, graphene was prepared in the form of stable suspensions, for which an organic solvent was selected, where we placed graphene and treated by ultrasound for 1-2 hours. Graphene structures obtained in this way are ideal for use as additives in composites. A composite powder (mixture) with different stoichiometry - $ZrB_2 + SiC + B_4C(C)$ were produced. At first, we carried out powder milling and mechanic activation for production homogeneous mixtures of composite powders ($ZrB_2 + SiC + B_4C(C)$).

Next step was production of press-powders using sintering additives/dopants by nano mill, ultrasonic, and a granulator.

3. Results and discussions

In Fig. 1 the results of X-Ray, PSD and scanning microscopy of the ZB_2-powder are presented. The XRD shows a small amount of ZrC along with the ZrB_2, which was expected because carbon black was taken in excess. PSD da Scan indicate the superfine size of the powder.

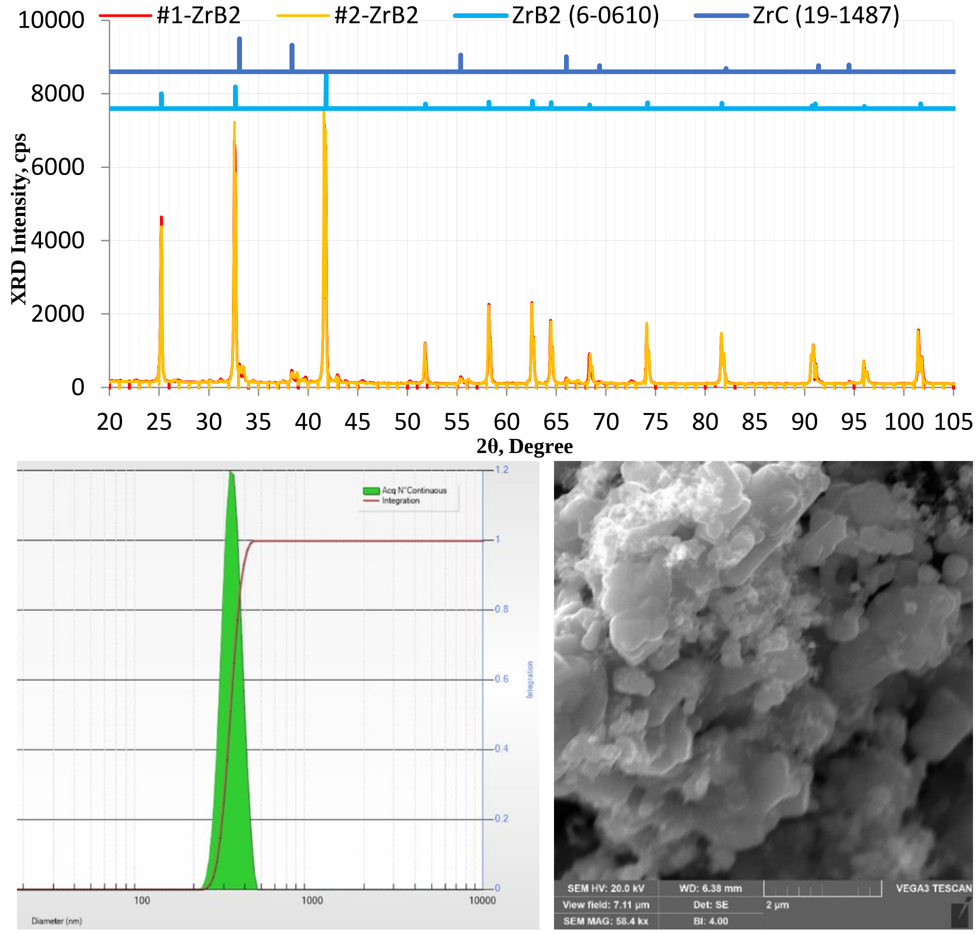

Fig.1. The results of X-Ray, PSD, and scanning microscopy of the ZB_2-powder

Conclusions:

A method has been developed of production of the superfine homogeneous composite press-powders based on ZrB_2 and SiC. For this, both methods of mechanical processing of powders and selection of sintering additives/dopants and their combining action were used. The method of obtaining super fine ZrB_2 powder is also developed, where, by adjusting the stoichiometry, the powder of the desired chemical composition is obtained.

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