Research, modeling and optimization of processes for obtaining nanocomposites based on oxygen-free and oxygen matrices

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Abstract. Modern technologies require structural materials capable of operating at high temperatures, with high strength, wear resistance, hardness, heat resistance, crack resistance for use in the aviation, space, defense and chemical industries. Ceramics has thermal and chemical resistance, but low strength and crack resistance.

The relevance of the work. Therefore, the creation of Al_2O_3-$ZrO_2(Y_2O_3)$-CNT functional ceramic matrix composites with unique performance characteristics is an urgent task. The SiC-$MgAl_2O_4$-CNT composite will have a high operating temperature under conditions of thermal erosion wear, since $MgAl_2O_4$ will reduce the SiC sintering temperature and provide protection against SiC oxidation throughout the volume of the material at elevated temperatures, and the CNT reinforcement will provide the material with the necessary strength, hardness, high thermal erosion indicators. Mechanical activation and sintering of SiC and B powders will create conditions for obtaining SiC-B ceramic matrix composite with unique heat-resistant properties for use in space technology.

The aim of the work conducting experimental studies and creating a mathematical description of the production of composites (based on oxygen and oxygen-free matrices) in order to determine the optimal conditions for obtaining ceramic matrix composites with desired properties: for the Al_2O_3-$ZrO_2(Y_2O_3)$-CNT composite obtained by spark plasma sintering: porosity $< 0{,}2\%$, bending strength > 850 MPa, microhardness $\geq 17{,}7$ GPa, crack resistance coefficient > 7 MPa·$m^{1/2}$; for composite SiC-$MgAl_2O_4$-CNT obtained by spark plasma sintering: porosity $< 0{,}5\%$, bending strength > 500 MPa, microhardness ≥ 26 GPa, crack resistance coefficient > 6 MPa·$m^{1/2}$; for the SiC-B composite obtained by spark plasma sintering: porosity $\leq 1\%$, bending strength > 200 MPa, microhardness > 26 GPa, crack resistance coefficient ≥ 5 MPa·$m^{1/2}$, weight loss (heat resistance) $\leq 0{,}2\%$.

Tasks solved in the dissertation work:

- conducting experimental and analytical studies:
    - carrying out experimental studies on obtaining a powder of a eutectic composition in the $0{,}5Al_2O_3$-$0{,}42ZrO_2$-$0{,}08Y_2O_3$ system by the method of heterophase coprecipitation;
    - obtaining alumina-magnesia spinel;
    - study of spark plasma sintering of Al_2O_3-$ZrO_2(Y_2O_3)$-CNT, SiC-$MgAl_2O_4$-CNT, SiC-B composites;
- on the basis of a mathematical model of the grinding process, the determination of the optimal conditions for the grinding process of silicon carbide to obtain a given value of the particle size;
- construction of a mathematical model of the process of spark plasma sintering and conducting a computational experiment and determining the optimal conditions for obtaining of ceramic matrix composite Al_2O_3-$ZrO_2(Y_2O_3)$-CNT, SiC-$MgAl_2O_4$-CNT, SiC-B.

Scientific novelty. Based on the methods of mechanics of heterogeneous media, thermodynamics of irreversible processes, a mathematical model of the process of baking ceramic composites was obtained, including: the ratio of the balance of the number of pores by size, considering the phenomena: connection ("healing") of pores, coalescence of pores during baking processes; the balance of the balance of the number of grains in the composite, which observes the phenomenon of recrystallization of grains during the baking process.

To solve the equation for changing the distribution of pores and grains in size (integro-differential values of random derivatives of the 1st order), a "Z" scheme and a mirror "Z" scheme, the product of the second order of approximation both in time and in size, were obtained.

Based on experimental studies and mathematical modeling, the optimal sintering conditions for three composites based on oxygen and oxygen-free matrices, which made it possible to improve the properties of these composites: Al_2O_3-$ZrO_2(Y_2O_3)$-CNT bending strength 998 MPa, crack resistance coefficient 7,3 $MPa{\cdot}m^{1/2}$, porosity 0,12%; SiC-$MgAl_2O_4$-CNT bending strength 515 MPa, crack resistance coefficient 7,2 $MPa{\cdot}m^{1/2}$, porosity 0,26%; SiC-B weight loss (heat resistance) ~ 0,19%, microhardness 26,4 GPa, crack resistance coefficient 5,1 $MPa{\cdot}m^{1/2}$.

For the sintering of the SiC-$MgAl_2O_4$-CNT composite, a study of the study of CNTs on the porosity of the composite was revealed. For the baking process of SiC-B, the results of the analysis of boron additions on the rate of change of pores and grains of the composite during baking were revealed.

Practical significance. Software has been created, consisting of software modules, which makes it possible to simulate the stage of grinding the initial powders, the spark sintering process (consisting of 2-3 stages, depending on the type of composite). The optimal conditions for obtaining composites Al_2O_3-$ZrO_2(Y_2O_3)$-CNT, SiC-$MgAl_2O_4$-CNT, SiC-B are determined.

The main provisions for the defense:

- The results of experimental studies on the production of powders: powder of eutectic composition in the system of aluminum oxide and zirconium dioxide, stabilized with yttria; alumina magnesia spinel powder;
- A generalized mathematical model for sintering ceramic matrix composites based on oxygen and oxygen-free matrices, including: 1) the equation for changing the pore size distribution density, considering the mechanisms of pore "healing" and the phenomenon of pore coalescence; 2) the equation for changing the grain size distribution in the composite due to the phenomenon of recrystallization during the sintering process;
- Difference schemes: "Z"-scheme and "mirror Z"-scheme, which are absolutely stable and approximate the equations of the mathematical model with the 2nd order in time and size;
- Mathematical model of 2-stage process of spark plasma sintering of Al_2O_3-$ZrO_2(Y_2O_3)$-CNT composite;
- Mathematical model of 3-stage process of spark plasma sintering of SiC-$MgAl_2O_4$-CNT composite;
- Mathematical model of 2-stage process of spark plasma sintering of SiC-B composite;
- Mathematical model of silicon carbide grinding;
- The results of the computational experiment and the obtained optimal conditions for obtaining composites: Al_2O_3-$ZrO_2(Y_2O_3)$-CNT, SiC-$MgAl_2O_4$-CNT, SiC-B;
- Results of a computational experiment and the obtained optimal conditions for grinding silicon carbide powder.