

PRESSURE SINTERING KINETICS OF POWDER REFRACTORY-COMPOUND-BASED MATERIALS

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G. V. Samsonov initiated and subsequently implemented a broad study of the synthesis conditions, the structure and properties of refractory compounds showed that the synthesis of these compounds should be carried out at temperatures much lower than their melting point, and as a result to obtain these compounds in powder form. Therefore, all the powder processing into specimens and articles has to be carried out by powder metallurgy methods alone. Hitherto a systematic analysis of the compaction, pressureless and pressure sintering powders of refractory compounds are carried taking their structure into account.

This paper presents the results of research processes pressure sintering powders of refractory compounds made in the last two years. Within the framework of the theory of volume viscous flow for irreversibly compressible porous body, the experimental data on the change in the relative density of tungsten and titanium carbides powders during the isothermal pressure sintering, as well as a powder mixture of boron carbide with silicon carbide in isothermal and non-isothermal conditions of pressure sintering are analyzed.

Densification of the porous body occurs under action of Laplace's pressure, generated by surface tension, and applied pressure. The estimated mean value of Laplace's pressure was determined to be 5.8 MPa for tungsten carbide and 7.2 MPa for titanium carbide. The densification kinetics of tungsten carbide in the sintering range of 2100–2500 °C and titanium carbides in the sintering range of 2100–2700 °C are controlled by the mechanism of nonlinear steady-state creep, which occurs at a rate proportional to the fourth power of stress in carbide matrix forming porous material. The estimated values of activation energy for the powder particle power-law creep rate are 591 kJ/mol for WC and 573 kJ/mol for TiC during the pressure sintering in initial and intermediate stages. These values indicate that a climb dislocation mechanism controlled the creep, and the values are consistent with the activation energies of bulk diffusion in metal sublattice of carbides.

A diffusional creep controls the pressure sintering kinetics in a later stage.

The quantitative analysis of non-isothermal pressure sintering kinetics for boron carbide powder mixture with 20 wt. % of silicon carbide in the controlled heating mode was carried out. The isothermal sintering kinetics of the mixture at temperature of 2240 K under applied pressure of 36.1, 49.6, 63.2 and 72.2 MPa was carried out to determine the Laplace pressure. It was found that the kinetics is controlled by steady-state creep mechanism in matrix forming the porous body, with viscous flow rate proportional to the square of the stress. Relatively low value of the estimated Laplace pressure (5.6 MPa) explains the difficulties of obtaining the boron-carbide-based composites with pressureless sintering. According to the current values of temperature and sample height during the pressure sintering, the heating rate and the relative density derivatives in respect to temperature were determined that enabled to describe the pressure sintering kinetics in terms of the theory of viscous bulk flow of the porous body in a die. Estimated values of the activation energy of the intermediate and final stages of the composites pressure sintering for different heating rates ranges from 610 to 710 kJ/mol. These values indicate that the sintering kinetics is controlled by dislocation climb mechanism. Study of the structure and the fracture behavior of the sintered samples showed that they depend on the heating rate. The higher heating rate during the sintering of B₄C–20 % SiC powder mixture, the greater the heterogeneity in the distribution of structural components and the larger the fraction of transcrystalline fracture of sintered samples.

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