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Advanced methods for consolidation of powder materials by impulse electromagnetic fields

E G Grigoryev

Merzhanov Institute of Structural Macrokinetics and Materials Science, Russian Academy of Sciences, Chernogolovka, 142432 Russia

E-mail: eugenrig@mail.ru

Abstract. Advanced technologies for the production of new materials and, in particular, nanostructured materials, using powder technologies require fundamentally new approaches for the formation and preservation of a given structural-phase state. Precision control of the state of materials in the process of consolidating powders of nanomaterials can be carried out using pulsed electromagnetic fields. The aim of the project is to study the effect of high-voltage and low-voltage pulsed electromagnetic fields in the technologies of powder consolidation. The experimental devices of spark-plasma sintering, flash-sintering, high-voltage consolidation and magnetic-pulse compaction are used in our laboratory for the production of advanced materials from metal powders, ceramic and composite powders. We can produce boron carbide, silicon carbide, uranium nitride, tungsten carbide - cobalt - diamond composites, tungsten heavy alloys, and others by electromagnetic methods of powder consolidation. Experimental results to consolidation metal powders, ceramic and composites powders by electromagnetic methods presage fruitful results.

1. Introduction

Advanced technologies of consolidation of powder materials, based on various techniques using of electric current pulses and mechanical pressure, are widely studied in many research laboratories [1]. The interest in these methods is motivated by their ability to consolidate a large variety of nanostructure powder materials to high densities within short periods of time, without having to increase initial grain sizes. The unique potential of pulse electric consolidation methods of powders is reflected in the ever-growing number of scientific publications studying these technological approaches. The wide range of possible electrical and mechanical treatment modes of powder has resulted in a large number of these methods, including high-voltage electric discharge consolidation (HVEDC, also sometimes referred to as capacitor discharge sintering (CDS)), spark-plasma sintering (SPS), magnetic-pulse compaction (MPC), etc. [1–3].

The advantages of these techniques can be exploited only through the optimization of the consolidation parameters since excessive energy dissipation during this type of processing can lead to the instability of the compaction process, to the formation of an undesirable heterogeneous material structure, and even to the destruction of the sintered specimens and of the equipment used. The time dependence of the associated thermal processes at the interparticle contacts plays a key role in electric pulse powder consolidation [4].

To obtain materials with required properties, one has to know the macroscopic processes occurring in the volume of a consolidated sample. Indeed, the kinetics of the consolidation of powder materials



in various electric field-assisted methods is significantly different, and their duration changes from several tens of minutes for electric-discharge sintering and spark plasma sintering [1–3] to several milliseconds for high voltage electric discharge consolidation [4] and magnetic-pulse compaction [5].

In this paper, we report the results of studying the multiscale physical processes during the powder consolidation by pulse electric current methods. These physical processes are taking into account both as a whole specimen and in the particle contact zones. Also, we describe the experimental equipment for implementation spark-plasma sintering, flash sintering, high-voltage electric discharge consolidation and magnetic-pulse compaction of powder materials.

2. Research method

A mathematical model describing the multiscale physical processes occurring under the powder consolidation by pulse electric current methods is based on the conservation laws of mass (1), momentum (2), energy (3) and the electrodynamics equations (4, 5) for consolidated powder.

$$\frac{\partial \rho}{\partial t} + \operatorname{div}(\rho \vec{v}) = 0 \quad (1)$$

$$\rho \left(\frac{\partial \vec{v}}{\partial t} + (\vec{v}, \nabla) \vec{v} \right)_i = \left(\frac{\partial \sigma_{ik}}{\partial x_k} \right) + F_i \quad (2)$$

$$\frac{\partial}{\partial t} \rho \left(\varepsilon + \frac{\vec{v}^2}{2} \right) = -\operatorname{div} \left(\rho \vec{v} \left(w + \frac{v^2}{2} \right) - (\vec{v}, \hat{\sigma}') - \kappa \nabla T \right) + \frac{\vec{j}^2}{\sigma} \quad (3)$$

$$\operatorname{rot} \vec{E} = -\frac{\partial \vec{B}}{\partial t}, \quad \operatorname{rot} \vec{H} = \vec{j}, \quad \operatorname{div} \vec{B} = 0, \quad (4)$$

$$\vec{F} = [\vec{j}, \vec{B}], \quad \vec{j} = \sigma (\vec{E} + [\vec{v}, \vec{B}]) \quad (5)$$

where ρ is the density, \vec{v} is the velocity, $\hat{\sigma}$ is the internal stress tensor, ε is the internal energy, w is the enthalpy, $\hat{\sigma}'$ is the viscoplasticity tensor, T is the temperature, \vec{j} is the electrical current density, \vec{E} , \vec{H} are the tensors of the electrical and magnetic fields, respectively, \vec{B} is the magnetic field induction, \vec{F} is the Ampere force; k is the thermal conductivity; σ is the conductivity of the powder material. The system of equations (1–5) should be supplemented by the state equations of the powder material and electrodes-punches material. It is assumed that the electrode-punch material obeys Hooke's law. We used a visco-plastic material model [13] for the description of the powder compaction process.

$$P = \frac{2}{3} \sigma_T \ln \frac{\alpha}{(\alpha-1)} - \frac{4}{3} \eta \frac{\dot{\alpha}}{\alpha(\alpha-1)} - \frac{\rho_m b^2}{3(\alpha_0-1)^{2/3}} \frac{d}{d\alpha} \left\{ \frac{\dot{\alpha}^2}{2} [(\alpha-1)^{-1/3} - \alpha^{-1/3}] \right\} \quad (6)$$

where: P is the compaction pressure, σ_T is the yield stress of powder material, η is the viscosity of a powder material, b is the initial size of pores, ρ_0 , ρ_m are the initial and theoretical density of powder material, respectively, $\alpha = \rho_m / \rho$ ($\dot{\alpha} = d\alpha/dt$, $\alpha_0 = \rho_m / \rho_0$).

Set of equations (1–6) with the appropriate initial and boundary conditions allows the establishment of the laws governing the process of powder consolidation by pulse electric current of conductive powder materials. Preliminary numerical estimates can simplify the solution of system (1–6) while maintaining an acceptable accuracy of the results. Joule energy input into the powder sample was determined by the parameters of the pulse current: amplitude $J_0 \leq 500$ kA, pulse frequency $\omega \sim 10^4 \div 10^5$ Hz, pulse duration $\tau_0 < 10^{-3}$ s. Cooling time in the consolidated sample is determined by

the heat sink in the electrodes-punches and the die, and depends on the thermal conductivity of the material of punches and dies, as well as on the geometrical dimensions of the sample. The duration of the compaction process of the powder material depends on the parameters of the pressing system, which creates pressure applied to the consolidated sample.

The simulation of the thermal processes in the interparticle contacts has identified the critical amplitude of the pulse current density, at which there is an electric thermal explosion of contact [4]:

$$j_0 = \sqrt{\frac{2\zeta\sigma}{\rho h} T_b^2} \quad (7)$$

here: j_0 is the pulse electric current amplitude, $\zeta \leq 1$, σ is the Stefan–Boltzmann constant, T_b is the boiling point of the material, ρ is the electrical resistivity of the contact spot, and h is the thickness of the contact area between two powder particles.

Equation (7) is obtained from the analysis of the heat balance at the initial period of time when the pulse electric current density rapidly increases during the pulse (at its leading edge). The heat balance assumes the equivalence of the Joule heat generation rate and the heat dissipation by heat transfer through radiation. Criterion (7) is obtained based on equations (1–5) for the temperature of the interparticle contact zone. If under the maximum value of the electric current density j_0 , the contact temperature reaches boiling point (or exceeds it), then this results in a contact thermal explosion. Figure 1 shows the experimental points for SPS of heat-resistant steel (EP-741) powder (under a load of 10^{-2} N between two particles) and the respective calculated data based on equation (7) for the current amplitude at the explosion contact as a function of particle size [4].

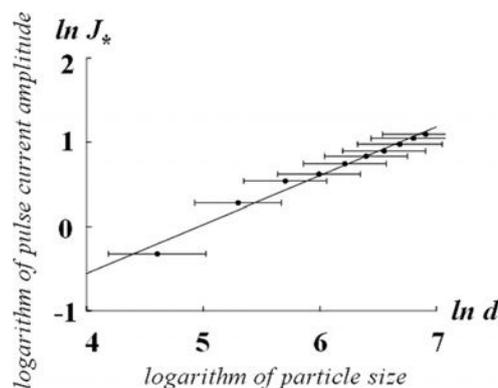


Figure 1. Experimental points and the theoretical prediction for the pulse current amplitude J^* of the contact explosion as a function of particle size d .

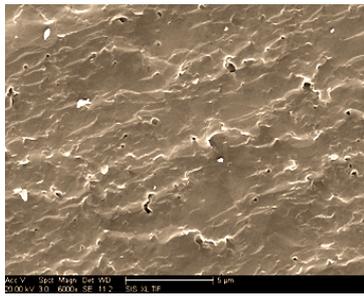
The simulation results indicate the possibility of the localization of heat in the interparticle contacts for certain parameter values of the pulse electric current. An upper critical level has been determined for the pulse current amplitude beyond which the interparticle contacts in powder material disintegrate via an electrothermal explosion.

For the implementation spark plasma sintering, microwave sintering, flash sintering, high voltage consolidation and magnetic pulsed compaction of powder nanostructured materials we used experimental equipment available in our lab.

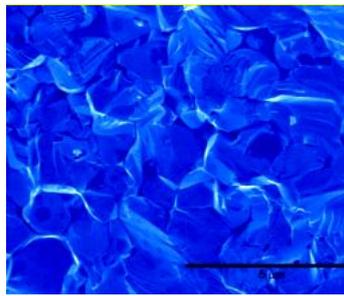
3. Results of the study

We have done (by SPS) the processes of consolidation of high-strength high-refractory compounds, maintaining the grain size of the powders (figure 2):

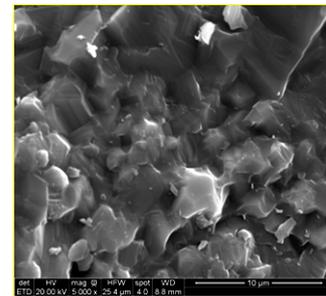
We have conducted successful experiments on consolidation using spark plasma sintering, and high voltage compaction of iron-titanium composite powders, of vanadium carbide powders, tantalum powders, zirconium nitride powders, alumina powders, ferritic-martensitic steels (ODS) powders with unique radiation-protective properties, tungsten carbide tools (figure 3).



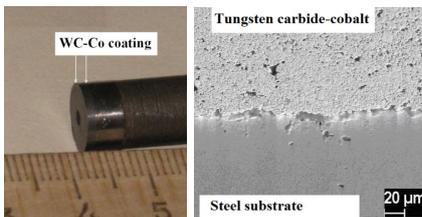
Boron carbide (15 min)



Zirconium carbide (5 min)



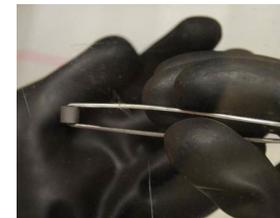
Silicon carbide (less than 1s)

Figure 2. Most significant experimental results of the study.

Tungsten carbide (WC) tools



Tungsten carbide + diamond tools



Uranium mononitride

Figure 3. Most important application results.

We have shown the possibility of manufacturing samples with relative density of 85–95% for titanium nitride and uranium mononitride in less than 1 s. The resulting samples had a fine-grained microstructure with a grain size of about 1–2 μm .

4. Conclusions

For both techniques (SPS and HVEDC) there is an upper level for the local Joule heating of the inter-particle contacts beyond which the processing instability may occur. While for HVEDC the duration of a single pulse and the amplitude are process controlling parameters, SPS apparently can be controlled by multiple pulse on and off frequency. It is shown that an optimum current amplitude and pulse time are necessary to generate sufficient heat for producing strong inter-particle joining and to avoid local overheating phenomena. An important expression for the critical amplitude of the pulsed electric current is derived. This upper critical level has been assessed for the high voltage pulse current amplitude beyond which the inter-particle contacts in powder material disintegrate by an electro-thermal explosion.

Experimental results to consolidation metal powders, ceramic and composites powders by electromagnetic methods presage fruitful results.

Acknowledgments

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