

ULTRA-RAPID MICROWAVE SINTERING¹

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We report the results of studies of microwave sintering of powder materials in the regimes with high heating rates and zero hold time at maximum temperature. Microwave processing of compacted samples based on Al_2O_3 , ZrO_2 , Y_2O_3 , MgAl_2O_4 , and $\text{Yb}:(\text{LaO})_2\text{O}_3$ ceramics was carried out using a gyrotron system operating at a frequency of 24 GHz with an output power of up to 6 kW [1]. The system is equipped with a fast computer control subsystem for automatic regulation of the microwave power and data acquisition.

The heating regimes used in the experiments were of two types: 1) constant-rate heating at 50...200 °C/min, and 2) heating at a fixed microwave power level. In the latter case very high heating rates, up to thousands °C/min, were achieved via a controlled use of the overheating instability, also known as thermal runaway. The volumetric absorption of intense microwave radiation resulted in a very rapid densification with the duration of the high-temperature stage of sintering on the order of one to several minutes.

In the rapid microwave heating regimes the effective high-frequency conductivity of the materials increased sharply at a certain threshold temperature. This suggests that the ultra-rapid sintering occurred via grain-boundary softening and formation of transient liquid phases. The indications of the presence of such phases can be observed in the microstructure images. The effect of ultra-rapid sintering is demonstrated to depend on the intensity of microwave radiation. The absorbed microwave power density required for the transition to the ultra-rapid sintering is on the order of 10...100 W/cm³ for a broad class of the materials [2, 3]. The obtained results suggest that ultra-rapid microwave sintering proceeds via essentially the same mechanism as the so-called flash sintering that occurs in the presence of ac / low-frequency dc electric field [4]. However, the implementation of the volumetric energy deposition by means of microwave processing is more advantageous from the technology scaleup viewpoint, since it does not require application of electrodes to supply electric current to the articles undergoing sintering [5].

The materials obtained by ultra-rapid microwave sintering possessed uniform structural and functional properties. The $\text{Yb}:(\text{LaO})_2\text{O}_3$ laser ceramics samples were uniformly translucent, which suggests that they were sintered to full density with very low residual porosity [6]. The obtained MgAl_2O_4 samples exhibited good dielectric properties in the millimeter-wave and terahertz range [7].

Microwave sintering of metal powders can also be feasible provided that the metal particles have poor electric contact between them (e.g., due to the presence of native oxide layers on the particles). Although the overheating instability does not develop in metals because of the decreasing dependence of electric conductivity on temperature, ultra-rapid microwave sintering regimes are still possible via purposeful use of resonance effects [8].

REFERENCES

- [1] Bykov, Yu., et al. // IEEE Trans. on Plasma Science. – 2004. – V. 32. – P. 67-72.
- [2] Bykov, Yu.V., et al. // Materials. – 2016. – V. 9. – P. 684 (1–18).
- [3] Bykov, Yu.V., et al. // Technical Physics. – 2018. – V. 63, No. 3. – P. 391-397.
- [4] Raj, R. // J. Am. Ceram. Soc. – 2016. – V. 99. – P. 3226-3232.
- [5] Bykov, Yu.V., et al. // RU Patent 2592293, priority date March 02, 2015.
- [6] Bykov, Yu.V., et al. // J. Am. Ceram. Soc. – 2015. – V. 98. – P. 3518-3524.
- [7] Egorov, S.V., et al. // Radiophys. Quantum El. – 2017. – V. 59. – P. 690-697.
- [8] Rybakov, K.I., Buyanova, M.N. // Scripta Mater. – 2018. – V. 149. – P. 108-111.

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