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Influence of electron-beam processing mode on the sintering of alumina ceramics

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Abstract. The article presents the results of electron beam sintering of alumina ceramic in the so-called forevacuum pressure range (5-20 Pa). Ceramic was irradiated by a focused electron beam generated by the forevacuum plasma electron source. Various modes of irradiation are considered without and with scanning of the electron on the ceramic surface. It is shown that using electron beam scanning increases the homogeneity of the sintered material. The modes of irradiation allowing to obtain dense ceramics with a homogeneous density distribution over its volume are given.

1. Introduction

Ceramic based on aluminium oxide is widely used in power-stressed operation due to its high hardness, heat resistance, chemical inertness, as well as availability. The technology of creation of ceramic materials consists of the main operations: production of powders with the necessary composition, preliminary compaction (pressing) of blanks and sintering. Traditionally, micron powders of technical purity, mechanical compaction and thermal sintering are used. Obtaining ceramics with improved properties is possible by using nanoscale powders with chemical state control and using such technologies as sintering in HF and microwave fields, the Spark Plasma Sintering (SPS) method [1, 2], laser sintering [3], electron beam sintering [4-5] and using special methods of pre-compaction [6, 7]. As shown by the previous experiments [8], the usage of an electron beam allows produce quite successful sintering of aluminum oxide ceramics. However, the issue of influence of electron beam processing mode on sintered materials properties was not given enough attention. This article presents a study of the effect of electron beam scanning modes of the sintered surface on the properties of the sintered sample.

2. Experimental setup

For experiments on electron-beam sintering and optimization of electron-beam irradiation regimes a forevacuum plasma electron source was used [9]. Forevacuum source of electrons is capable process dielectric materials (in particular ceramics) without additional methods to reduce the charge on the irradiated surface [10]. The scheme of the experimental setup is shown in figure 1.



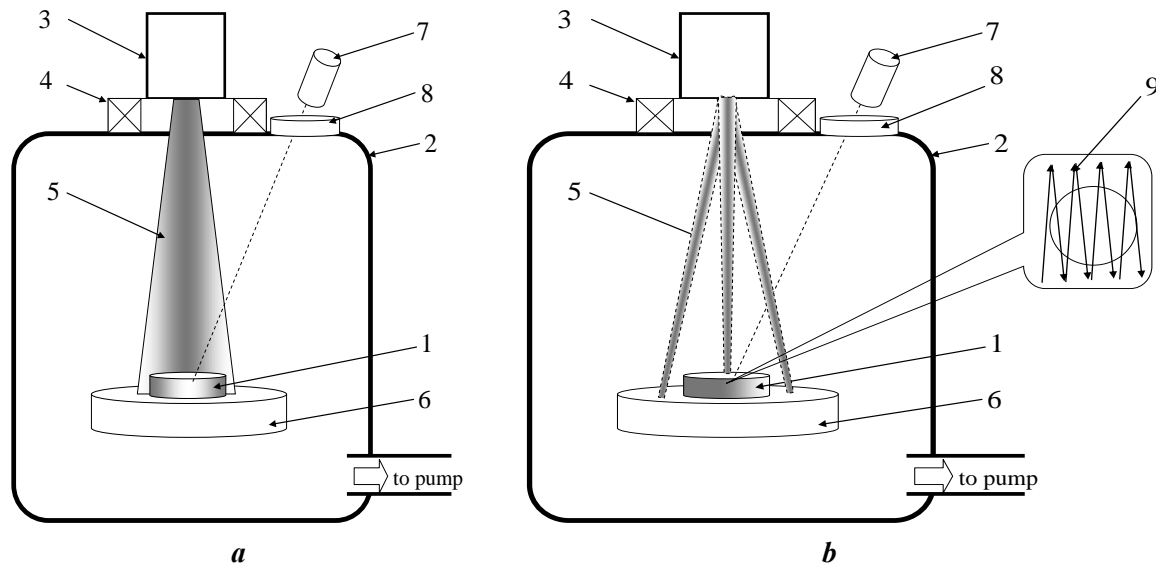


Figure 1. Scheme of electron-beam sintering of alumina ceramics in two regimes: a - without scanning, b - using scanning of electron beam. 1 – sintered sample, 2 – vacuum chamber, 3 – plasma electron source, 4 – focusing and deflection system, 5 – electron beam, 6 – collector, 7 – pyrometer, 8 - quartz glass, 9 – beam trajectory during scanning.

The sample of aluminum oxide ceramics 1 was a disk with the diameter of 14 mm and the thickness of 3 mm. The disk was pressed from an aluminum oxide nanopowder with the average grain size of 30 microns. The sample was installed in the holder placed in a vacuum chamber 2, which was pumped out to the pressure of 3 Pa. Then helium was injected into the chamber to the pressure of 30 Pa. The plasma electron source 3 was installed on the upper flange of the vacuum chamber and was equipped with a system of electromagnetic focusing and deflection 4 of the electron beam 5. The sample was heated for 25 minutes with increasing beam power. The accelerating voltage in the experiments increased from 5 to 12 kV. The deflection system control the position of electron beam on the sample surface. Also the deflection system allows to the electron beam scan the sample surface along the raster with the specified geometric dimensions (from 5*5 to 25*25 mm) at the frequency of 1 to 1000 Hz. The temperature control of the sintered sample was carried out by a non-contact method using an infrared pyrometer Marathon MM (Raytec, USA) 7 with a measurement range of 600-3000 °C. Thermal radiation from the sample was captured by the pyrometer located behind the quartz glass 8 with a bandwidth corresponding to the working band of the pyrometer wavelengths. Sintering of samples was carried out at the constant temperature of 1350 °C and the holding time of 15 minutes according to two different modes. Mode 1 – the sintering was carried out without the electron beam scanning by the electron beam with the diameter of 15 mm covered the all surface of the ceramic sample (Fig. 1 a). Mode 2 – the sintering was produced by the electron beam with the diameter of 0.5 mm with the included scanning system with a frame drawing frequency of 100 Hz. The scanning area 9 was 15*15 mm and was larger than the transverse dimensions of the sintered sample.

To study the microstructure of sintered samples, the JSM-7500FA scanning electron microscope (JEOL, Japan) was used. Mass measurement was carried out by electronic scales with the measurement accuracy of 0.01 gm. The sample density was determined from the measured value of the mass and the geometric dimensions of the sample measured by a micrometer with the accuracy of 0.1 μm.

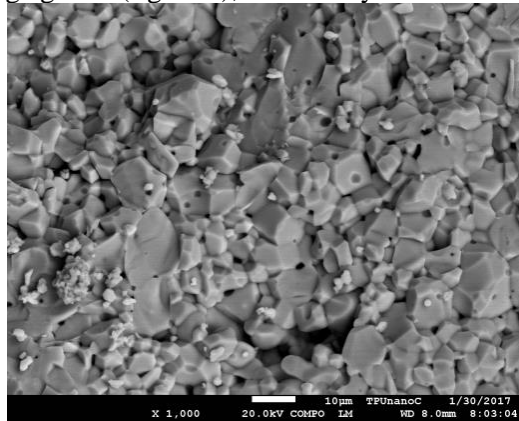
3. Experimental results and discussion

During the electron beam sintering the volume of the samples decreases. The change in the sample dimensions is presented in table 1. The density increases by amount of 16-17% of the original value in the non-sintered state. The results presented in table 1 indicate the compaction of the ceramic material after electron beam sintering, that is an indirect confirmation of the sintering process.

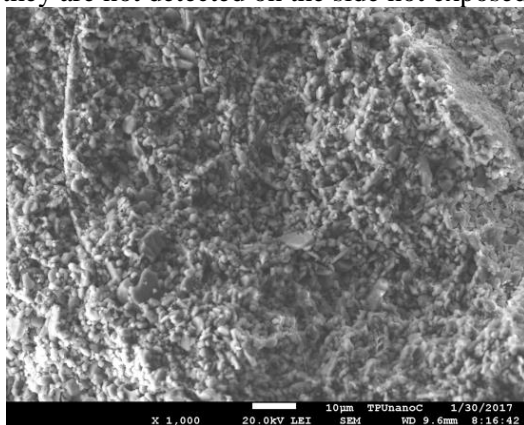
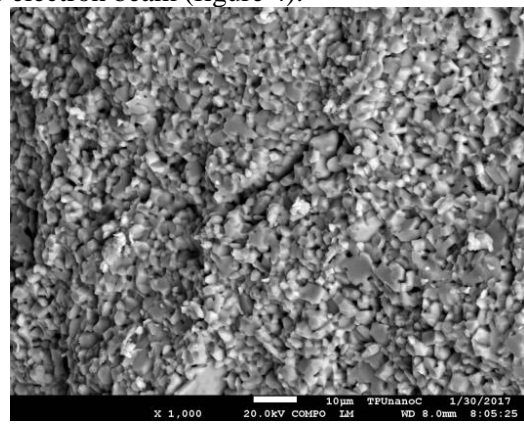
Table 1. Geometrical parameters of samples.

	Mode 1 (no raster)		Mode 2 (100 Hz scan rate)	
	before	after	before	after
Weight, gm.	1.14	1.09	1.19	1.14
Thickness, mm	2,8	2.7	3	2,8
Diameter, mm	14.20	13.10	14.15	13.25
Density, g/cm ³	2.57	2.99	2.54	2.97
Density, %	1	1.16	1	1.17

Despite the almost identical external parameters of ceramic samples, the internal structure of the samples after sintering is significantly different for various modes. The structure of the sintered samples in mode 1 is heterogeneous in the thickness of the sample. In the central part of the sample surface we observed the formation of large grains (figure 2), which may indicate overheating in this part.

**Figure 2.** Microphotography of the central part of the sintered sample in mode 1

Near the irradiated surface of the samples sintered in mode 1 large grains are not detected (figure 3), as they are not detected on the side not exposed by the electron beam (figure 4).

**Figure 3.** Microphotography of the irradiated part of the sintered sample in mode 1**Figure 4.** Microphotography of the non-irradiated part of the sintered sample in mode 1

These facts indicate an inhomogeneous heating of the sample volume, which can lead to such significant differences in its internal structure. Microphotographs of the internal structure of the samples sintered in mode 2, i.e. which are made with scanning of the surface by the narrowly focused electron beam, are presented in figures 5-7.

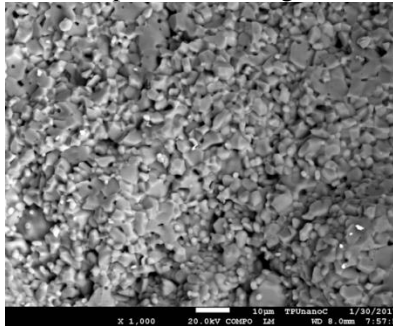


Figure 5. Microphotography of the central part of the sintered samples in mode 2

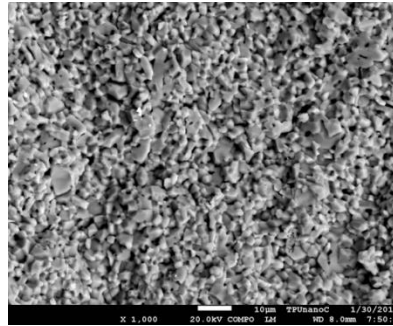


Figure 6. Microphotography of the irradiated part of the sintered samples in mode 2

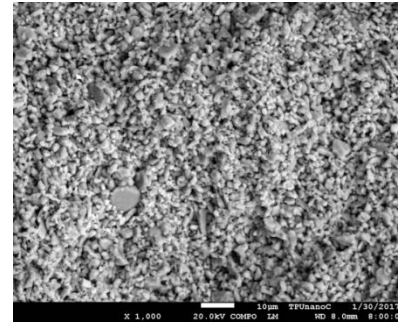


Figure 7. Microphotography of the non-irradiated part of the sintered samples in mode 2

In the samples sintered in mode 2 there is no formation of large grains and there is an initial stage of sintering, figure 5. The grain size distribution is more homogeneous, see figure 5 and 6, which seems to be related to the irradiation conditions and heating. In the case of irradiation by the electron beam with a diameter comparable to the diameter of the sintered sample, due to the Gaussian distribution of the current density across the beam section [11], the sample is heated unevenly with a maximum in its central part. Despite the existence of time intervals between beam hits one point of the sample surface, scanning on the surface by the narrow beam still contributes to a more homogeneous heating and, consequently, sintering.

4. Conclusion

Obtaining a homogeneous sintered sample of aluminum oxide ceramics is possible when a focused electron beam is used as a heating source and its scanning of the ceramic surface by this beam is ensured. It is shown that at a scanning frequency of 100 Hz it is possible to obtain a homogeneous sintered sample while maintaining a small grain size.

Acknowledgments

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