The possibilities of dimensional electron-beam processing as applied to selective sintering of oxide ceramics in the fore-vacuum pressure range

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Abstract. The paper presents the results of studies devoted to an effect of deviation and scanning parameters on the value of power density of a focused electron beam generated by a plasma electron source in the forevacuum pressure range. It is shown that the change in the beam power density does not exceed 20% with an angle of beam deviation within 20 degrees in the investigated pressure range of 6-8 pascals in air. The results of selective sintering of powder system of zirconia-alumina by an electron beam generated by a forevacuum plasma electron source are demonstrated.

1. Introduction

Ensuring of effective precision electron-beam processing of products is largely connected with the achievement of high power density of the electron beam in a local area [1]. One of the known examples of the precision application of an electron beam is the EBM technology, which is used to create products with an arbitrary shape from titanium alloys [2]. However, as shown in [3], when irradiation of metal powders is used in this technology at working pressures of $10^{-1}$-$10^{-2}$ Pa, there is a problem of spread of the powder particles caused by the process of their charging by the electrons. The solution of this problem consists in preheating the volume of powder to the sintering temperature of metal particles. The usage of high-temperature ceramic powders for the synthesis of three-dimensional products is complicated by the need to attract some special measures to neutralize the accumulated negative charge [4]. In this case, it is advisable to use a forevacuum plasma source [5], which generates an electron beam at the pressures from one to hundreds of pascals. During working in this pressure range, almost complete neutralization of the accumulated negative charge is realized by a flow of ions from the plasma formed by ionization of residual gas by the beam electrons and ignition of a glow discharge between the irradiated surface and walls of a vacuum chamber [6, 7].

Effective focusing of an electron beam in the region of high pressures in the forevacuum range is primarily complicated by the processes of scattering of the electrons on molecules of residual gas.
Despite this, we were able to obtain the electron beam with a diameter less than 0.6 mm and provide the power density of $10^4$-$10^5$ W/cm$^2$, which was sufficient to "drill" holes in high-temperature ceramics [8]. However, in order to achieve a high quality of electron-beam synthesis, it is necessary to preserve specific parameters of the electron beam effect when a deflection of the beam is occur. Thus, the purpose of the investigations was to demonstrate the possibility of direct electron-beam dimensional processing by the forevacuum plasma electron source and investigate the possibility of selective synthesis of a ceramic powder of the zirconia-alumina system by this source.

2. Scheme and technique of experiment

The scheme of the experimental setup, the technique of electron-beam sintering and the measurement of beam sizes are shown in Figure 1. The electron beam $I$ is generated by the forevacuum plasma electron source $2$, whose operation principle is based on emission of electrons from plasma of a glow discharge with a hollow cathode. The parameters of the electron beam, which were used in the experiments, are the following: the accelerated voltage $U_a$ is $12 - 16$ kV, the beam current $I_b$ is up to $15$ mA; the pressure in the vacuum chamber is controlled by feeding working gas (air) and is within $P = 6$-8 Pa. In more detail, the design and features of functioning of this electron source are presented in [9]. The magnetic lens $3$ performs focusing of the electron beam. Using the deflection system $4$ consisting of two pairs of magnetic coils, the beam deflection from the original direction and its scanning are performed. The current signal of the deflection system is controlled by an electronic system for formation and deflection of an electron beam designed and manufactured at NPT TETA [10] for electron-beam welding setups.

![Figure 1](image_url)

**Figure 1.** Scheme of the experiment and illustration of the technique of electron beam size measuring (a – scheme of beam size measuring and powder sintering; b – beam measurement plane (top view): 1 – electron beam; 2 – forevacuum plasma electron source; 3 – focused magnetic lens; 4 – deflection system; 5 – position and cross-section form of electron beam without deflection; 6 – position and cross-section form of electron beam when it is deflected at angle $\alpha$; 7 – horizontal plate of the beam size measurement system; 8 – pair of gaps for measuring minimum size of beam $d_1$; 9 – pair of gaps for measuring maximum size of beam $d_2$; 10 – collector for beam current measuring; 11 – sintered ceramic powder; 12 – graphite crucible; 13 – Faraday cylinder; x, y – directions of beam deflection.

The investigation of cross-sectional size change of the electron beam during its deviation by an angle $\alpha$ (Figure 1 a) is carried out by the "deflection" method [11]. Due to changing the form of electron beam cross-section from round ($\alpha = 0$, 5 in Figure 1 b) to oval ($\alpha > 0$, 6 in Figure 1 b), the beam power density is determined by equation: $q = \frac{4 \cdot U_a \cdot I_b}{\pi d_1 d_2}$, where $d_1$, $d_2$ – minimum and maximum cross sizes of the beam respectively, which are measured in the mutually perpendicular directions (Figure 1 b).

The measurements of the cross sizes of the beam is carried out by the following way: the electron beam is focused on the plate 7 which is perpendicular to the propagation direction of beam, then the electron beam is deflected by an angle $\alpha$ along the x axis (Figure 1 a) from the initial position. The
electron beam consistently intersects one of two pairs of the narrow measuring gaps 8, 9 with the width of 0.2 mm located on a special displacement device. One pair of gaps is located along the x axis, the other is perpendicular to it along the y axis. The beam electrons that have passed through the gaps are received by the collector 10, a current signal from which is fed to an oscilloscope. When the beam intersects the gaps 8 along x, the beam size $d_1$ is determined. Accordingly, the size $d_2$ is determined during crossing the pair of gaps 9 along the y axis. The values of $d_1$ and $d_2$ are estimated as $d_{1,2} = L \tau / T$, where $L$ – the distance between gaps, $T$ – time interval between amplitude values of the signal on the oscillogram, $\tau$ – peak width on the oscillogram at half-height.

The electron-beam sintering of the powder 11 (zirconia-alumina system with a weight rate 6:1 the average particle size is 1-10 μm) located in a cavity of the graphite crucible 12 in the free-filling state is performed by the beam scanned the surface of the powder in the form of a raster with sides 10x10 mm with the frequency of 60 Hz. To avoid appearance of cracks in the volume of the sintered sample, controlled warming and cooling after processing are implemented with a rate of change of power density 2 W/cm$^2$·min. The dwell time at the fixed beam parameters during sintering is 30 minutes.

The morphological features and elemental composition of a surface of the sintered samples were studied by scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS) at the JSM-7500FA (JEOL, Japan).

3. Experimental results

As it is shown in [8], the increase in pressure unambiguously leads to the beam broadening. It is obvious that this influence is expressed to a greater extent with the larger scattering cross section of gas molecules. In particular, it is shown in Figure 2 that when the air pressure in the working chamber is increased from 6 to 8 Pa, the beam power density decreases almost fivefold due to the increase in beam geometric sizes. However, despite the scattering processes that negatively affect on focusing of the electron beam, when the beam deflection is performed within 20 degrees, the reduction in the power density does not exceed 20% from the initial value. Such small decrease in the power density of narrow-focused beam indicates the possibility of efficient dimensional precision processing of high-temperature dielectrics.

Figure 2. Dependences of the electron beam power density $q$ on the deflection angle $\alpha$ for different gas pressures $P$ ($U_b = 16$ kV, $I_b = 5$ mA, working gas – air): 1 – $P = 6$ Pa; 2 – $P = 7$ Pa; 3 – $P = 8$ Pa.

As a demonstration of the effectiveness of using the narrow-focused electron beam generated by the forevacuum plasma source, the ceramic powder of the zirconia-alumina system with a weight ratio of 6:1 was selectively sintered. The SEM images of surface of the sintered samples with different integrated power densities and EDS data are presented in Figure 3. Analysis of the obtained results and their comparison with the known ratio of the components of the initial powder mixture (6:1), allow us to conclude that significant alumina segregation occurs on the pore surfaces or the zirconia particles after the action of the electron-beam sintering. This process is quantitatively expressed by the conditional value of segregation coefficient $k$ of the alumina, which is defined as the ratio of the excess founded by the EDS method to the zirconia content of the initial mixture. Figure 3.1 shows that the part of alumina is sintered in a solid phase with insufficient intensity of the impact, and the effects of grain-boundary slip during sintering of zirconia particles are not completely realized. In the case of excessive intense melting of the alumina, there is a restriction of the sintering zirconia particles together (Figure 3.3). In this case, the zirconia particles are separately distributed in a liquid matrix of
the alumina, but their amount is not sufficient to form a continuous ceramic layer on the surface of the sample.

Figure 3. SEM images of surfaces of the sintered ceramics samples, obtained in backscattering electrons: light gray areas – ZrO₂; gray areas – Al₂O₃; dark gray fields – deepening of surface relief and pores: 1 – I = 80 W/cm², Uₐ = 12 kV, C Al₂O₃ = 42.99 mas.%, C ZrO₂ = 57.01 mas.%, k = 0.33; 2 – I = 125 W/cm², Uₐ = 12 kV, C Al₂O₃ = 53.66 mas.%, C ZrO₂ = 46.34 mas.%, k = 0.46; 3 – I = 170 W/cm², Uₐ = 12 kV, C Al₂O₃ = 54.42 mas.%, C ZrO₂ = 45.58 mas.%, k = 0.47.

In the case of the optimal mode for the electron-beam sintering (Figure 3.2), the surface has dense interphase boundaries and a complex grain structure. In this structure, in addition to individual equiaxed grains of aluminum oxide and ensembles of extended grains of zirconia, two-phase grains with a core of zirconium dioxide encased in an alumina shell are observed.

4. Conclusions

Thus, the results of the investigations indicate the possibility of efficient dimensional processing of high-temperature dielectrics by the electron beam generated by the plasma source of electrons in the forevacuum pressure range. In addition, the possibility of selective electron-beam sintering of a zirconia-alumina powder is demonstrated.

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