Additive Manufacturing of Ceramic Products Based on Millimeter-Wave Heating

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Abstract. Additive manufacturing of ceramic articles making use of concentrated energy flows attracts the research interest worldwide. While the application of laser beams faces serious problems associated with high temperature of sintering and low thermal conductivity of ceramics, layer-by-layer sintering by focused millimeter-wave radiation appears to be a promising method of additive manufacturing. This paper describes the studies of fast millimeter-wave sintering of yttria-stabilized zirconia and hydroxyapatite ceramics. Coefficients of the millimeter-wave absorption have been determined in broad frequency and temperature ranges. Rapid sintering of compacted ceramics samples was accomplished using volumetric microwave heating in a work chamber of a 24 GHz / 5 kW gyrotron system. In addition, using a 263 GHz / 1 kW cw gyrotron millimeter-wave source and a purposely designed electrodynamic focusing structure, radiation intensities of up to 20 kW/cm² could be achieved, which was sufficient for fast localized heating of ceramic layers to the solidification temperature. The results of a study of the microstructure and mechanical properties of the sintered ceramics are presented.

1. Introduction

Additive manufacturing (AM) technologies are used more and more widely in various branches of industry due to their advantages distinguishing them from conventional ("subtractive") manufacturing techniques based on removal of material from the workpiece (e.g., cutting, milling, grinding). The recent advances in the basic principles of AM technologies, their approbation and practical implementation have resulted in the development and industrial introduction of a variety of devices for the manufacture of products for a wide application spectrum such as automotive, aerospace, engineering, medicine, biological systems, etc. A comprehensive description and discussion of principal approaches and methods used in the AM technological chains can be found in numerous recent reviews, e.g., [1–3].

Until recently the AM methods were primarily used for the fabrication of articles from plastics and metal powders. Respectively, the methods employed for the solidification at the final stage of fabrication, usually based on heat treatment, are focused on working with these materials.

In recent years an increasing attention is attracted to the use of AM methods for the fabrication of products from ceramic and composite materials. The interest in this problem is largely due to the fact that the conventional, "subtractive" technologies meet serious limitations when attempting to fabricate complex-shape products from materials possessing high hardness and predisposed to brittle failure. In
the application of AM methods to ceramic and composite materials, the basic principles and approaches remain unchanged (see, e.g., the reviews [4–6]). Clearly, the main distinguishing features when using these materials are related to the preparation of the feedstock possessing the necessary properties for the implementation of AM and to the solidification of the ceramic-based materials which usually requires temperatures far in excess of 1000 °C. One approach can involve preparation of a green body with a prescribed configuration according to a CAD-model and its solidification during a hold in a high-temperature furnace for several hours. Within another approach the article is fabricated layer by layer, and the solidification of the deposited layers of a ceramic-based material is accomplished via local heating with a concentrated energy flow – mostly by a laser beam.

The use of microwave radiation for the solidification of products fabricated by AM appears to be a promising alternative method. The volumetric absorption of microwave radiation (in most practically important ceramic materials) makes it possible to implement rapid heating of the articles with the rate of energy deposition not limited by thermal conduction. Studies indicate that the duration of the high-temperature stage of sintering to a close-to-theoretical density decreases by several times when using microwave heating [7]. Using quasioptical techniques, shorter-wavelength microwave radiation can be formed into a wave beam that can be utilized for sequential solidification of the layers via sintering or welding of powder compositions. Suitable for AM tasks is the use of millimeter-wave radiation (with frequencies on the order of or higher than 30 GHz). The existing millimeter-wave radiation sources, gyrotrons, have power on the order of 10 kW, which is sufficient for practical implementation of processes in industrial applications. Gyrotron-based systems developed for various applied tasks based on high-temperature processing of materials by intense millimeter-wave radiation are currently available [8, 9]. Good spatial uniformity of the microwave power density in the work chambers of the gyrotron-based systems operating at frequencies on the order of 30 GHz ensures fast and uniform volumetric heating of articles with the characteristic dimensions of several tens of centimeters. On the other hand, the radiation intensities on the order of $10^4$ W/cm$^2$ achievable in the gyrotron systems with a power of about 1 kW at frequencies 250 GHz and above can be feasible for the studies of layer-by-layer fabrication of articles using focused microwave beams.

This paper discusses the possibilities of using millimeter-wave radiation in the processes of consolidation of hydroxyapatite and yttria-stabilized zirconia powders and densification of powder compacts. These materials are candidates for bioceramics fabrication due to their bio-compatibility and good mechanical properties. The following results are reported:

1. measurement of absorption coefficients for microwave radiation of frequencies 24 GHz and 100–200 GHz as a function of temperature and porosity of the powder compacts;
2. comparative study of the sintering kinetics of powder compacts under 24 GHz microwave heating and conventional heating;
3. analysis of the microstructure and the measurements of microhardness and fracture toughness of the sintered materials;
4. demonstration of rapid heating of hydroxyapatite powder layers by focused radiation at a frequency of 263 GHz.

2. Experimental

The following powders were used in the experiments: hydroxyapatite (Ca$_{10}$(PO$_4$)$_6$(OH)$_2$, HA) – GAP-85d (Polistom, Russia), yttria-stabilized zirconia (ZrO$_2$ + 3 % Y$_2$O$_3$, YSZ) – TZ-3YE (Tosoh, Japan). The mean particle size of the YSZ powder was 40 nm, specific surface area 16 ± 3 m$^2$/g. According to the X-ray phase analysis data the YSZ powder consisted of a mixture of the tetragonal (87.5 wt. %) and monoclinic (12.5 wt. %) phases. The particle size of the HA powder was in the range 0.7 – 2 µm, and the specific surface area was 3 – 10 m$^2$/g.

The samples in the shape of disks, 8 mm in diameter and about 2.5 mm in thickness, were compacted by uniaxial pressing under a pressure of 200 MPa. The relative densities of the obtained compacts were 48.3% (3YSZ) and 48.0% (HA) of the theoretical values which are equal to 6.05 g/cm$^3$ and 3.16 g/cm$^3$, respectively.
The experimental studies of sintering and measurements of the absorption coefficient for the 24 GHz microwave radiation were carried out on gyrotron systems with the maximum power 2.5 and 5 kW (automatically regulated). The compacted samples were sintered in the work chamber in air at normal atmospheric pressure. The samples were positioned in the middle of a channel with a diameter of 10 mm drilled through a block of low-absorption porous alumina (AL-30, ZIRCAR Ceramics, U.S.A.). In the sintering experiments the temperature of the samples were measured by two unshielded B-type thermocouples. One thermocouple head was inserted into a channel drilled in the center of the disk to half of its thickness, which provided data about the maximum temperature of the sample during its volumetric microwave heating. The head of the other thermocouple touched the disk surface at a distance of 0.6 mm from its edge. The constant rate of heating was sustained by automatically regulating the microwave power input to the work chamber based on a feedback from the measured temperature reading. The preset rate of heating varied in the range 10 – 100 °C/min, and the maximum temperature of heating – in the range 1000 – 1400 °C. There was no isothermal hold at the maximum temperature. The changes in the diameter of the disks were monitored by processing their infra-red images obtained using a monochrome digital camera (NET 3iCube, 1/2 inch CMOS sensor with a horizontal and vertical resolution of 1280 × 1024 pixels, a maximum frame rate of 60 frames per second and a maximum data transfer rate of 5 GBit/s). Automatic brightness control of the images from the camera, transfer and recording of the sequence of images to a computer, and the display on the monitor were carried out using the software Stream Pix 7 (NORPIX, Canada). For comparison of the regimes and results of microwave and conventional sintering, some of the samples were heated in a resistive furnace (Termokeramika, Russia).

The density of the sintered samples was determined by Archimedes weighing in distilled water. The microstructure was studied by scanning electron microscopy (JEOL JSM-6390 LV). The phase composition of the sintered samples was analyzed by X-ray diffractometry (RigakuUltima IV). The microhardness and fracture toughness of the sintered samples were measured on the mechanical tester(Struers Duramin-5). Before measurements one side of the sintered samples was grinded off to a depth of about 100 µm and polished with diamond paste.

The heating of HA powder layers by focused radiation was carried out on a gyrotron system operating at a frequency of 263 GHz [9].

3. Measurements of microwave absorption coefficients in 3YSZ and HA powder compacts at frequencies 24 GHz and 100 – 200 GHz

At the 24 GHZ frequency the measurements of the microwave absorption coefficients were accomplished by the calorimetric method. The microwave power absorbed in the sample was determined based on the difference between the heating and cooling rates and the microwave power density at the time instants of intentional abrupt change of the microwave power at different sample temperatures.

The energy balance equation is:

\[
C_s m \left( \frac{dT}{dt} \right)_{b(a)} = \alpha \Pi_{b(a)} S_{\text{sample}} - P_{\text{loss}}
\]

where \( C_s \) is the specific heat capacity of the material of the sample, \( m \) is the mass of the sample, \( \left( \frac{dT}{dt} \right)_{b(a)} \) is the heating/cooling rate, \( \alpha \) is the integral absorption coefficient, \( \Pi_{b(a)} \) is the microwave power density incident on the sample, \( S_{\text{sample}} \) is the surface area of the sample, \( P_{\text{loss}} \) is the power in heat losses, the subscripts \( b \) and \( a \) denote the instants of time just before and just after the intentional change of the microwave power, respectively.

The integral absorption coefficient can be expressed from (1) as
The values of the microwave power density $\Pi_{b(a)}$ are determined from the gyrotron parameters with the help of calibration experiments. After the integral absorption coefficient is obtained, it is possible to make a rough estimate of the absorption coefficient per unit length $\gamma$:

$$\gamma = \frac{\ln(1 - \alpha)}{h_{\text{sample}}}$$  \hspace{1cm} (3)

where $h_{\text{sample}}$ is the thickness of the sample. The change of both $S_{\text{sample}}$ and $h_{\text{sample}}$ with time due to the sintering process was taken into account in the calculations.

The temperature dependencies of the absorption coefficient $\gamma$ for both materials are presented in figure 1.

![Figure 1. The absorption coefficient for HA (a) and 3YSZ (b) vs. temperature: raw sample (solid lines), sintered sample (dashed lines). The monoclinic phase content for 3YSZ (b) is shown by dotted line.](image)

The non-monotonic temperature dependence of $\gamma$ for 3YSZ is possibly associated with the monoclinic to tetragonal phase transformation. It can be seen in figure 1(b) that the decrease in the monoclinic phase content coincides with a peak in the absorption coefficient.

In the frequency range 100 – 200 GHz the dielectric properties of HA were determined using a spectrometer based on a high-Q ($Q_0 \sim 10^6$) open Fabry–Perot resonator [10]. The measurements of the dielectric properties of materials by this method are based on determining the shift in the central frequency of the resonance curve and the change in its width when the sample is placed in the resonator. The HA samples with a diameter of 26–32 mm and a thickness of 2 mm, sintered to different densities, were annealed in a resistive furnace immediately before measurements in order to exclude the influence of the adsorbed water. An annealed plane-parallel ceramic sample was positioned in the central section of the resonator. The measurements were carried out at resonance frequencies of the sample (when the sample thickness is equal to an integer number of half-
wavelengths in the material). The method was capable of determining the refraction index, \( n \), to an accuracy of better than 0.1\% and the dielectric loss tangent, \( \tan \delta \), to an accuracy of 5–10\% in the studied frequency range. The accomplished measurements have shown that the dielectric loss tangent increases almost twofold as the density of the material grows. For example, at a frequency of 180 GHz the green sample with a relative density of 48\% had \( \tan \delta = 4.5 \times 10^{-3} \), whereas the sintered sample with a density of 98\% had \( \tan \delta = 8.3 \times 10^{-3} \). The refraction index \( n \) in this density range changed linearly from 1.68 to 3.07. Based on these results, it was possible to obtain the upper-bound estimate of the absorption coefficient for the radiation intensity in the HA powder at room temperature: \( \gamma \approx 0.5 \text{ cm}^{-1} \).

The results of the dielectric loss measurements vs. the radiation frequency for a fixed density of samples suggest that the frequency dependency in the studied range is weak. For example, when changing the frequency of electromagnetic radiation from 108 to 190 GHz, the dielectric loss tangent measured on a sample with a relative density of 70\% varied from \( 3.3 \times 10^{-3} \) to \( 4.5 \times 10^{-3} \), and the refraction index \( n \) was equal to 2.39 in the entire frequency range.

4. Microwave sintering of 3YSZ and HA powder compacts at a frequency of 24 GHz

Shown in figure 2 are the dependencies of the relative density of HA (figure 2 (a)) samples on their sintering temperature: under microwave heating at a rate of 50 °C/min with no isothermal hold; under conventional heating at a rate of 2 °C/min with a hold for 2 hours at the sintering temperature. Similar dependencies for 3YSZ samples are shown in figure 2 (b): under microwave heating at a rate of 100 °C/min with no isothermal hold; under conventional heating at a rate of 2 °C/min with a hold for 30 minutes at the sintering temperature. These results demonstrate that the sintering of both materials is greatly enhanced in the case of microwave heating. It can be seen that although the duration of the microwave sintering process is much shorter, equal density values are obtained at temperatures that are about 100 °C smaller than in the case of conventional sintering. The time needed to achieve a relative density on the order of or higher than 95\% in the described microwave sintering experiments was shorter by a factor of \( 10^2 – 10^3 \) compared to conventional sintering.

![Figure 2](image-url)

Under the described experimental conditions, microwave volumetric heating accompanied with heat removal from the surface of the samples gives rise to a significant temperature non-uniformity in the volume of the samples. The observation of the samples undergoing microwave sintering with the
help of an infrared digital camera, along with the two-channel temperature measurements, has made it possible to analyze the dynamic temperature distribution during the process.

Figure 3. Temperatures of the center ($T_c$, solid line) and edge ($T_{edge}$, dashed line) of the sample and the input microwave power (dotted line) vs. time during microwave heating of a HA sample. The heating rate at the final stage of the process – 50 °C/min, no isothermal hold.

During the heating of both HA and 3YSZ samples, at temperatures about 520 – 550 °C a glowing area with a size on the order of several millimeters is observed (using the infrared camera) on the surface of the samples. Its brightness rapidly increases with further increase in temperature. Concurrently, the automatically regulated microwave power input to the work chamber is reduced to sustain the prescribed heating rate (figure 3). The localized glow and the automatic reduction of power, while the heat removal from the sample grows with temperature, evidence in favor of the development of a thermal instability in the sample associated with an increase in the effective high-frequency electric conductivity with temperature. Since the temperature at which the glow arises and the corresponding maximum of the input power occurs is the same for both materials, it is reasonable to assume that the microwave radiation is absorbed by the water adsorbed on the powder particle surfaces. The water desorption process causes automatic reduction of power required to sustain the prescribed heating rate. With further increase in the temperature of the sample center, $T_c$, the temperature of its periphery, $T_{edge}$, begins to lag behind noticeably, because the decrease in the power causes a decrease in the rate of heating of the thermal insulation arrangement surrounding the sample. The temperature difference $\Delta T = T_c - T_{edge}$ along the sample radius reaches about 200 °C and even more. Since the thermal conductivity of porous powder compacts is low, the temperature partially equalizes (i.e., $\Delta T$ decreases) only upon the start of the densification process (at $T_c \sim 800$ °C).

In principle, it is possible to reconstruct the temperature distribution over the sample surface using the brightness distributions on the images obtained with the infrared camera, with a help of a calibration procedure. It should be noted that the density curves shown in figure 2 are plotted as a function of the temperature measured in the center of the samples, $T_c$, which corresponds to the maximum of the temperature distribution in the sample. Not only this temperature is lower by more than 100 °C compared to the conventional sintering process, but the material is also sintered to full density in the peripheral regions of the samples, where the temperature is at all times lower than the temperature of the densification onset in the case of conventional sintering.

The microstructure images obtained by SEM on broken HA samples sintered under microwave (a) and conventional (b) heating are shown in figure 4. The microstructure of the sample sintered under
microwave heating to a density of about 94.0 % represents a dense packing of rounded grains with a size of 2–4 µm fused to each other (figure 4 (a)). The grain structure on the broken surface of the conventionally sintered sample (with a density of 93.4 %) cannot be seen (figure 4 (b)). The microstructure of the latter sample represents a dense packing of fused regions which have irregular shapes and a size of about 10–15 µm.

Figure 4. SEM microstructure images of broken surfaces of HA samples obtained by (a) microwave sintering, heating rate 10 °С/min, maximum temperature 1280 °С, no isothermal hold; (b) conventional sintering, heating rate 2 °С/min, maximum temperature 1250 °С, isothermal hold for 2 hours.

The microstructure images obtained by SEM on broken 3YSZ samples sintered under microwave (a) and conventional (b) heating are shown in figure 5. The microstructure of the sample sintered under microwave heating to a density of 99.8 % represents a dense packing of rounded grains with an average diameter about 0.5 µm (figure 5 (a)). In the sample obtained by conventional sintering (density 99.1 %, figure 5 (b)) the particles have approximately the same average size, yet they are fused together into aggregates, about 2–3 µm in size.

Figure 5. SEM microstructure images of broken surfaces of 3YSZ samples obtained by (a) microwave sintering, heating rate 10°C/min, maximum temperature 1350°C, no isothermal hold; (b) conventional sintering, heating rate 2°C/min, maximum temperature 1400°C, isothermal hold for 30 minutes.
| Material / Sintering method | Relative density, % | $H_\mu$, GPa | $K_{1C}$, MPa·m$^{1/2}$ |
|----------------------------|---------------------|---------------|-------------------|
| HA / Microwave              | 95.1                | 4.6           | 0.7               |
| HA / Conventional           | 93.4                | 4.5           | 0.6               |
| 3YSZ / Microwave             | 98.6                | 13.5          | 2.8               |
| 3YSZ / Conventional         | 99.1                | 13.6          | 3.1               |

The results of the measurements of Vickers microhardness $H_\mu$ and fracture toughness $K_{1C}$ of the samples sintered under rapid microwave and conventional heating are listed in Table 1. The measurements on 3YSZ samples were carried out under a load of 1 kg, and on HA samples under a load of 0.3 kg. The mechanical properties were measured on five sintered samples. Five measurements were accomplished on each of the five samples.

An analysis of the results listed above suggests that the values of the microhardness and fracture toughness of the microwave sintered HA and 3YSZ samples virtually coincide with the respective properties of the samples obtained by conventional sintering, in spite of the fact that the sintering time differs by a factor of about $10^3$. However, for the HA samples both parameters are noticeably (by 30–60 %) lower than the values known from the literature [11]. Since the mechanical properties are low in the samples obtained by both microwave and conventional sintering, the reason apparently lies in the properties of the HA powder used in this work. For the 3YSZ samples, the values of the microhardness and fracture toughness of the samples obtained by rapid microwave sintering fall in the range of values that are typical for the conventionally sintered ceramics [12].

1. **Sintering of HA powder layers using 263 GHz millimeter-wave radiation**

The HA powder layers were heated by focused radiation from a gyrotron system with a maximum power of 1 kW at a frequency of 263 GHz [9]. The quasi-optical radiation transmission line [13] ensured focusing into a spot with a size of about 2.5 mm, which provided an intensity of up to 20 kW/cm$^2$. The powder was poured loosely into a crucible made of an alumina-based thermoinsulating material.

![Figure 6](image_url)

**Figure 6.** (a) Schematic of the experimental configuration for the heating of HA powder layers with a focused millimeter-wave radiation beam at a frequency of 263 GHz: 1 – powder layers, 2 – movable crucible with the powder, 3 – focusing mirror; 4 – millimeter-wave radiation, 5 – optical camera, 6 – setup for crucible displacement. (b) A result of heating of HA powder layers with a scanning focused 263 GHz millimeter-wave radiation beam with an intensity of 2 kW/cm$^2$: 1 – powder layers, 2 – consolidated portion of the powder; 3 – crucible.
material (AL-30, ZIRCAR Ceramics, U.S.A.) with the external dimensions 57 × 21 × 12 mm. The dimensions of the pit in which the powder was filled were 57 × 12 × 2.5 mm. The crucible with the powder was positioned in the work chamber of the gyrotron system so that the radiation focal point was close to the surface of the powder (figure 6 (a)). The scanning of the radiation wave beam over the surface of the powder was implemented by the displacement of the crucible. An optical camera was used for the observation of the heating and consolidation of the powder layers.

Figure 6 (b) shows a result of heating of the HA powder by a millimeter-wave radiation beam with an intensity of about 2 kW/cm² (the output power of the gyrotron was 100 W). The crucible with the powder was being displaced relative to the wave beam focal point at an average velocity of 0.46 mm/s. The displacement started after a 5 second hold for the initial heating of the powder. The displacement was controlled to ensure uniform glowing of the heated powder.

As seen from figure 6 (b), as a result of heating localized consolidation has been achieved along the entire depth of the powder layer in the region that was exposed to the focused millimeter-wave radiation. The width of the consolidated region was 3–5 mm.

5. Conclusion
The main findings of the study described in this paper can be summarized as follows.
- The dependencies of the dielectric properties of HA and 3YSZ compacts on temperature and porosity have been obtained for the frequencies 24 GHz and 100 – 200 GHz.
- The HA and 3YSZ samples have been sintered under 24 GHz microwave heating at rates of 50 and 100°C/min with no hold time. The final densities were close to the theoretical values for both materials.
- During microwave sintering the same density values were achieved at temperatures of about 100°C lower compared with conventional sintering, despite a significantly shorter (by a factor of 10² – 10³) sintering time.
- A microstructure study on HA and 3YSZ compacts has demonstrated a finer grain structure in the microwave heated compacts in comparison to the conventionally heated ones, due to a shorter sintering time.
- The microhardness and fracture toughness of the microwave sintered samples are close to those of the conventionally sintered ones despite a significantly shorter heating time and the absence of an isothermal hold.
- The feasibility of localized consolidation of HA powder layers by a scanning focused microwave radiation beam at a frequency of 263 GHz has been demonstrated.

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