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STRUCTURE OF A CERAMIC MATERIAL DEVELOPED BY LASER PROTOTYPING TECHNIQUES

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Abstract. The structure of a ceramic material manufactured by laser sintering was studied by scanning electron microcopy (SEM) and X-ray microtomography. It was shown that the calcination mechanism in this material is the formation of "bridges" between agglomerates of ceramic powder with a typical size of ~ 14 μm. These agglomerates were composed of nanolayers as a result of the powder pretreatment technique used. Calcination was found to be of good quality.

1. INTRODUCTION

Manufacturing of new generations of modern turbine machines requires new materials to be used in their construction, in turbine rotors, combustion chambers, heat exchangers, and gas manifolds. Development of new materials able to withstand long-term mechanical loading at temperatures of 1200-1400 °C could provide an opportunity to significantly increase the turbine efficiency coefficient and to thereby decrease the nitrogen oxides emission. Ceramics are usually considered as the most promising materials for this task, and their application in turbine construction has received a lot of interest during the last few decades, see e.g. [1-4].

The main advantages of ceramic materials include their heat resistance, thermal stability, and high compression strength. They, however, have a number of disadvantages: low stability against cracks, impacts and high temperature gradients, and insufficient bending strength. For this reason, new techniques should be applied so as to take advantage of the positive properties of ceramic materials while minimizing the effects of their negative properties [5,6].

However, the basic problem in application of ceramic materials to gas turbine construction is the difficulty to manufacture components of complex geometry such as those which include internal cavities and orifices of changing curvature. An evident choice for the manufacturing of such components would be to use diamond tools, but they are not effective when manufacturing components of complex geometry, and their use is also time consuming and expensive. An alternative choice would be the laser prototyping technique widely used when producing metal components of complex geometry from a metal powder. At present a number of companies (CONCEPT Laser GmbH, Germany; Phenix Systems, France; Aspect, Inc., Japan; 3D Systems, USA) already apply laser sintering for producing various types of metallic machinery, and it has also been proposed to apply it to ceramic components. However, a direct production of ceramic

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components by laser sintering has not been successful, and the use of ceramic powders with polymeric binding agents does not provide high enough thermal strength for the components. Also, it has been characteristic of such components that they shrink non-uniformly, which is a typical reason for their fracturing. It is evident that non-uniform shrinking makes it impossible to retain the geometry of the component.

A solution to the above problems is development of a new type of ceramic material which will meet the following requirements: possibility to calcinate the material by laser sintering, stability of the geometrical dimensions of components made of this material during laser sintering and the following heat treatment (no shrinking), and strength characteristics of these components, which satisfy the construction requirements. To this end ceramic powders suitable for laser prototyping techniques were developed by the “Glass and Ceramics” company, and the “Boyko Center” then modified the laser prototyping procedure so as to be suitable for this new material. Ceramic microturbines with a power of 2 kW were thus manufactured by laser sintering using a modified version of a Phenix-PM100 device [7,8]. We report here the results of an analysis of the structure of this new ceramic material sintered by laser prototyping techniques.

2. SAMPLE PREPARATION AND EXPERIMENTAL TECHNIQUES

Nanostructured ceramic powders based on SiC-Si-Al_2O_3 system for laser sintering were produced by the “Glass and Ceramics” company. The complex procedure of their manufacturing process includes mechano-activating ball milling using planetary mills, vacuum calcination, disintegration, and mesh classification. The fraction with the particle size <40 μm was finally used in laser sintering, and the average particle diameter as determined by PSD analysis (Horiba LA 950, volume distribution) was ~ 14 μm. Laser sintering was done using a Phenix PM-100 device modified for ceramic powders, and synthesis was made in dried argon at room temperature. A standard 50 W CO_2 laser with a focal shift of 50% was used, and the thickness of the layer calcinated with the laser was ~ 50 μm. Samples made for the structural analyses were disks whose diameter was 10 mm and height 3.5 mm. A more detailed report of the procedures used in sample preparation is given in [7,8].

SEM analysis of samples’ structures was performed using a Supra 40 electron microscope and applying standard measurements procedures.

X-ray tomography is a nondestructive imaging technique in which the three dimensional structure of the sample is reconstructed from two dimensional X-ray projection images [9]. The measured absorption of X-rays is based on the linear absorption coefficients and the thicknesses of the material components along the X-ray paths, which allow us to resolve the distributions of components of different linear absorption coefficients inside the sample [9]. By adjusting the X-ray energy with respect to sample composition and size, the sensitivity of the measurement can be optimized. Also, increasing the number of projection images enhances the quality of the reconstructed three dimensional distribution of linear absorption coefficients. In the tomographic analysis an Xradia MicroCT-400 device was used. The maximum energy of the X-rays was 40 keV, and 1505 projection images of each sample were recorded for the reconstructions. The pixel size in the projections and the reconstructed images was 1.17 μm, which is close to the maximum resolution that can be reached by conventional X-ray CT scanners.

3. RESULTS AND DISCUSSION

Results of the SEM analysis are shown in Fig. 1. Inspection of Fig. 1a indicates the presence of agglomerated objects with typical dimensions of 1 to 20 μm, which is agreement with the average particle size of the powder used in the manufacturing, ~ 14 μm, as well as with the calcinating effect produced by the laser beam with a focus of <400 μm. Obviously, the maximal temperature is achieved in the region near the center of the focus (note that this region is quite a bit smaller than the area illuminated by the laser beam). This was the reason for the high quality of calcination obtained near the center of the focal point. Fig. 1b shows a magnified image of the central region, which indeed indicates that the ‘baking’ mechanism is the formation of ‘bridges’ between agglomerated particles. The typical length of these bridges is a bit smaller than the typical linear dimension of the agglomerates (~ 10-15 μm), while their typical width is 2-4 μm, and occasionally even less.

Figs. 1c and 1d demonstrate the nanostructure of the agglomerates. It is evident from Fig. 1d that agglomerated particles are formed by pseudo-layers with a typical thickness of 30-50 nm. It is also evident that these layers consist of nanoparticles of similar dimensions. This observation supports the conjecture that the observed layers are monolayers of nanoparticles.

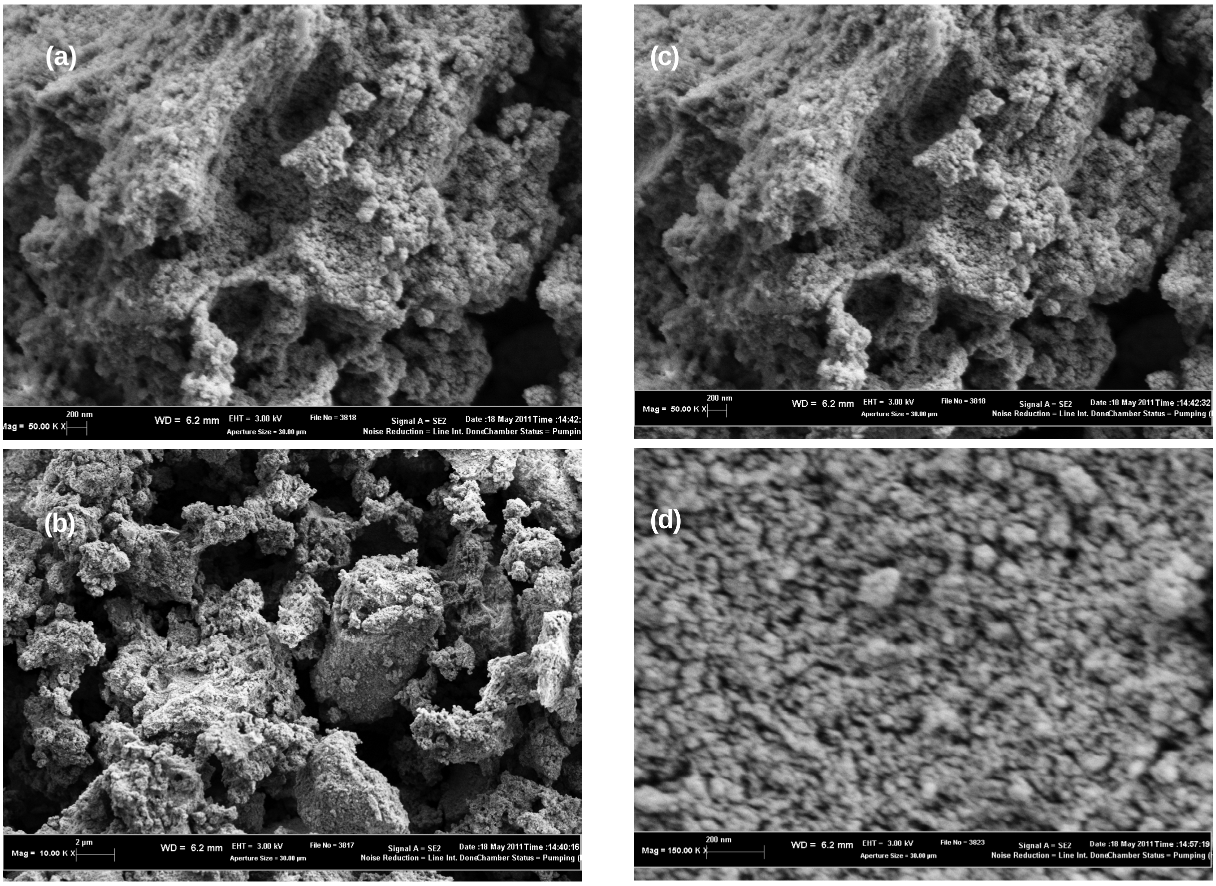

Fig. 1. SEM examination of the final ceramics structure.

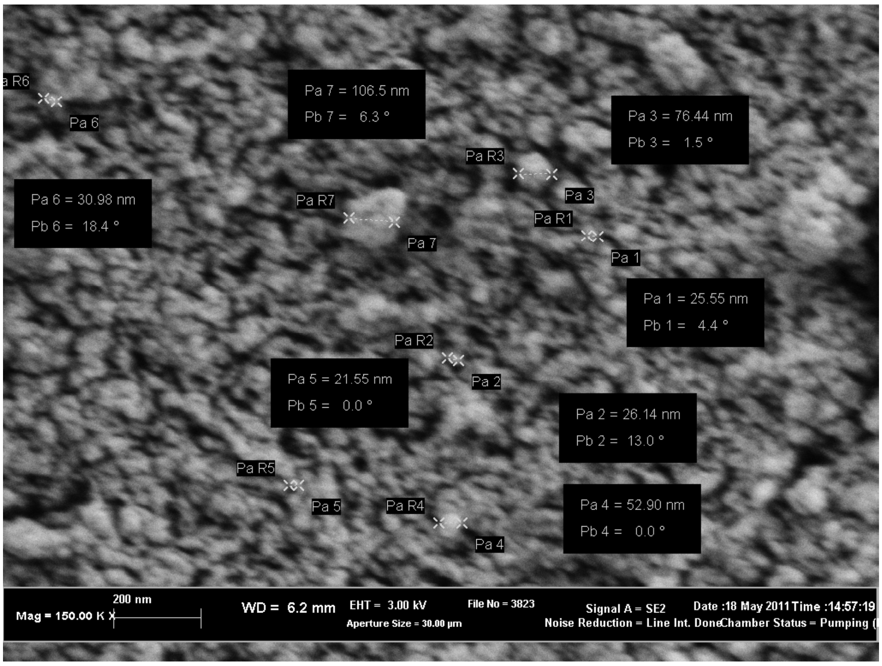

Fig. 2. Typical characteristics of nanoobjects determined by SEM.

Fig. 2 displays typical dimensions of the nanoparticles that form the agglomerates. It lends support to the conclusion above of the typical size of these particles: most particles in this figure have dimensions of 30-50 nm, although larger structures (up to ~ 120 nm) also appear. We can attribute the

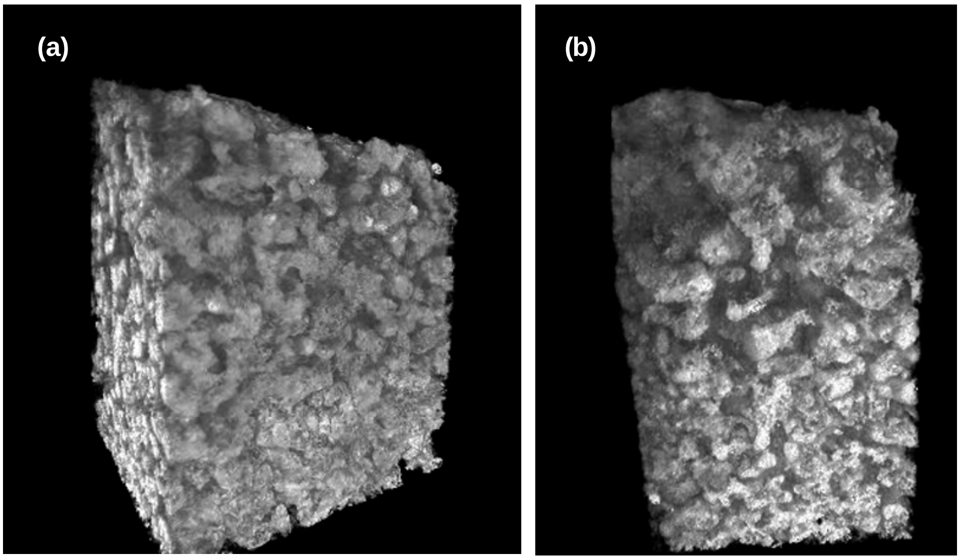

Fig. 3. Results of X-ray tomography: (a) Volume rendered visualization of a sample of (0.5x0.5x0.8) mm^3; (b) a view from different angle to the volume rendered image of Fig. 3a.

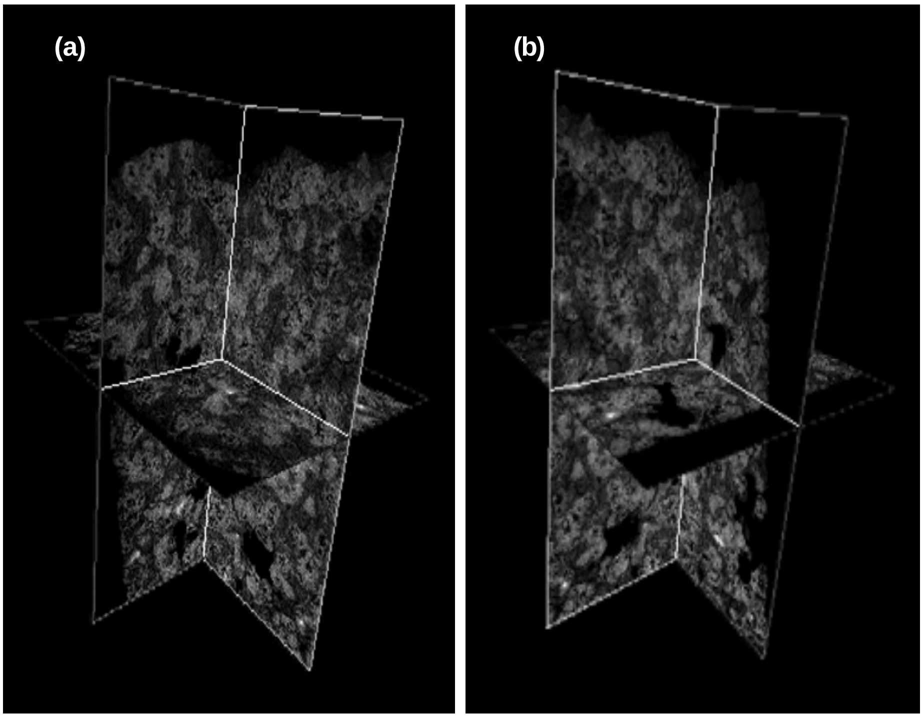

Fig. 4. (a) Visualized orthoslices of the sample shown in Fig. 3. Grayscale values are proportional to the local absorption coefficient for X-rays; (b) as in Fig. 4a, but different orthoslices.

observed structure of the system to the procedures applied in the preparation of the powder: it most probably results from compactification and mechano-activation caused by ball milling.

The same samples were also analyzed with X-ray microtomography. Figs. 3a and 3b demonstrate the typical cross-sectional structure inside a (digitally cut) sample, while Fig. 4 displays similar information from selected ortoslices through the sample, and shows even better the different material components. Fig. 3 supports the conclusion of the bridging of agglomerates induced by 'baking' with laser. It is evident from Fig. 4 that the new ceramic material manufactured by laser sintering is fairly compact. A more detailed report on the distribution of porosity and different material components in these samples will be given in a forthcoming publication.

The main conclusion based on the SEM and X-ray tomography results is that the new ceramic material produced is compact enough for construction components. There exist in the material regions of high density, probably formed in the regions of maximal laser power, while there also exist porous regions, probably formed outside the centers of the focal points in the laser sintering process.

4. CONCLUSIONS

The structure of the new ceramic material synthesized by laser prototyping techniques was analyzed by SEM and X-ray tomography. It was shown that this material is composed of grains with a typical dimension of about 14 mm. The basic mechanism of ceramic formation induced by the applied laser 'baking' is the formation of calcination bridges between powder agglomerates. These agglomerates are composed of layers with a typical thickness of ~ 30-50 nm, produced by the complex powder treatment procedure used.

The ceramic material produced by laser sintering techniques is compact enough for the requirements of a construction material for microturbines. The density of the ceramic material was not, however, homogeneous. There existed regions of high density alternating with porous regions. It was conjectured that the porous regions were caused by application of relatively low laser power in the peripheral parts of the focal point areas. For this reason, application of lasers of higher power is a possible way to increase the strength of the ceramic material produced in this way by laser sintering.

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