The Effect of Particle Distribution to the Sintering Properties of Al₂O₃ Ceramic Formed by Stereolithography

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The effect of particle distribution to the sintering properties of Al_2O_3 ceramic formed by stereolithography

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Abstract: This work studied the effect of particle size gradation on the structure and sintering properties of alumina ceramics manufactured by stereolithography after sintering, including room temperature flexural strength, sintering shrinkage, porosity and microstructure morphology. Seven different particle size gradations from coarse to fine were studied, and each particle size gradation was coarse particles: fine particles=6:4. With the increase of the sintering temperature, the degree of sintering and the bonding strength of the interface increased, the shrinkage in all directions, and the bending strength and the closed porosity increased, the open porosity decreased. For the same sintering temperature, the flexural strength and shrinkage in all directions decreased and the open porosity increased with the increase of particle size. The ratio of the height (4mm) shrinkage of each particle size to the length (50mm) shrinkage is about 1.5 times. Due to the presence of the lateral edge resin layer, the width shrinkage of the coarse particles was different from the length shrinkage but closer to the height shrinkage. The width shrinkage gradually approached the length shrinkage as the particle size decreased. When the gradation ratio is the same, and the particle size was ten times different ($30\mu\text{m}/5\mu\text{m}$, $3\mu\text{m}/0.5\mu\text{m}$; $20\mu\text{m}/3\mu\text{m}$, $2\mu\text{m}/0.3\mu\text{m}$), there was a big difference. When the sintering temperature was above 1500°C , the differences of sintering properties of the comparison groups are 2~10 times. Considering the alumina-based ceramic core, 20/3 has been evaluated comprehensively. It maintains excellent flexural strength and porosity under the condition of slight shrinkage.

Key words: Particle distribution; Alumina Ceramic; Stereolithography; Sintering properties

1. Introduction

Alumina ceramic has always received much attention because of many advantages, like excellent chemical stability and high-temperature properties [1–3]. However, the traditional manufacturing methods, such as hot pressure injection, are challenging to produce alumina ceramic cores with complex cross-sections. Simultaneously, the

manufacturing cycle of a ceramic core's mould is long, significantly increasing the time cost and expense of fabrication. To address those questions, additive manufacturing technology has been introduced recently to prepare alumina-based ceramic cores [4–6].

Compared to the traditional fabrication methods, the additive manufacturing (AM) technique is a unique manufacturing philosophy and has more advantages, such as precise structures, the flexible preparation of highly complex-shaped, and a short production cycle time [4,6–9]. For the fabrication of ceramics, several additive manufacturing techniques have been explored recently. As one of these techniques, stereolithography additive manufacturing technology [10–13], including stereolithography equipment (SLA) and digital light processing (DLP), is up-and-coming for the production of ceramic cores due to its advantages in the manufacture of ceramics with complex geometries and high precision. However, due to the inconsistency between the vertical packing density and the horizontal direction, there is delamination in additive manufacturing [5,14]. Print delamination makes it easy to crack after sintering, which dramatically affects the sintering performance of ceramics and results in differences in mechanical properties of ceramic cores with those formed by traditional methods[15,16]. Therefore, it is necessary to study the sintering performance by adjusting the sintering temperature, particle gradation and other methods.

It is known that the effects of sintering processes, such as sintering temperature, soaking time, on the performance of Al_2O_3 ceramic by stereolithography based additive manufacturing have been extensively developed by the academic community. Wu et al. investigated the effects of the drying process and debinding process on the microstructure and densification of Al_2O_3 ceramic and produced dense and defect-free Al_2O_3 ceramic parts through stereolithography [17,18]. A method for preparing fine and complex Al_2O_3 ceramic lattices by additive manufacturing based on stereolithography technology was developed in Shuai's work[19]. Their work found out the optimized process parameters during debinding and sintering. Schmidt et al.[20]

proposed dense and crack-free, highly complex porous mullite ceramics using stereolithography by firing a mixture of photosensitive polysiloxane and alumina powder. Li et al. [21,22] discussed the effects of the sintering process on the microstructure and mechanical properties initially. They produced Al_2O_3 samples with a flexural strength of 33.7 MPa and open porosity of 37.9 % using stereolithography based additive manufacturing. The introduction of fine grains or sintering additives on the alumina ceramic obtained by stereolithography based 3D printing process was studied in Zhang's work[23]. A defect-free Al_2O_3 ceramic lattice structure with high precision and high compressive strength was obtained.

Besides, the current research on particle size in ceramic stereolithography is mainly focused on the impact of particle size on the slurry. Since DLP printing has fluidity requirements for the slurry and the requirements for the layering thickness of the printing process, the particle size and the volume fraction of the ceramics must be adjusted to meet them. It is believed that controlling the particle size distribution may be an effective way to meet the requirements of the suspensions and the SLA process [17]. In particular, Subbanna et al. [24] and Tari et al. [25] have shown that it is possible to obtain high-density green parts using a bimodal particle size distribution in which fine and coarse powders are combined at an appropriate ratio. Zhou et al. [26] discovered the dispersant, volume fraction of ceramic powder and powder diameter could influence the viscosity of the suspension and obtained a complex impeller by using a new aqueous ceramic suspension with a wide ceramic particle-size distribution. On the other hand, researchers like Wu[17] have studied the effect of particle size on some mechanical properties of alumina ceramics fabricated by 3D printing based on stereolithography initially. Especially, Nakajima et al.[27] investigated the effects of grain size on the spinel and mullite crystallization kinetics during sintering. However, the interactions among sintering, particle size distributions and sintering performance (including microstructure, mechanical properties, and so on) during the stereolithography of Al_2O_3 ceramic have not been systematically analyzed yet.

In this work, the influence of particle size and sintering temperature on the sintering performance of alumina ceramics formed by stereolithography was systematically explored and studied. Seven kinds of alumina gradation particle sizes that can meet the requirements of digital light processing were printed. Then the green parts were degreased and sintered at different sintering temperatures. The properties of sintered samples such as flexural strength, porosity, shrinkage, and pure interlayer shear strength were developed, and microstructure morphology was observed. The sintering densification process with different particle sizes and the comprehensive performance of the 3D printing process alumina ceramics were revealed.

2. Materials and experiments

2.1 Materials and the fabrication of green parts

Ceramic suspensions used in the stereolithography comprised curable monomers or oligomers, photoinitiators, dispersants and ceramic powders[28]. In this study, 1,6-hexanediol diacrylate (HDDA, Alfa Aesar Co., Ltd.) and ethoxylated pentaerythritol tetraacrylate (PPTTA, DSM-AGI Co., Ltd.) were used as the monomers. 2,4,6-Trimethylbenzoyl diphenylphosphine oxide (TPO, BASF (China) Co., Ltd.) was used as the photoinitiator. The volume-loading rate of alumina powder (Al_2O_3 , Zhengzhou Non-ferrous Metals Research institute Co., Ltd of CHALCO) was approximately 56%. The purity of Al_2O_3 powder is no less than 99.99%.

According to the requirement of additive manufacturing layering (in this article, the thickness of each layer is 100 μm), the coarsest particles were selected with $D_{90} < 100\mu\text{m}$, particles with D_{50} of 30 μm . The coarser particles in each gradation were determined to be 30 μm , 20 μm , 10 μm , 5 μm , 3 μm , 2 μm . Some scholars found that the mass fraction ratio of coarser particles to finer particles of 6:4 was a better choice[29]. Due to the fluidity and solid content requirements of the slurry, great efforts were tried to achieve a close-packed inter-particle with coarser particles as the skeleton when the slurry is configured. Considering the densest packing of plane and space (Fig. 1), the ratios of

the radius of the coarse and fine particles are 6.46 and 4.45, respectively. The corresponding particle size gradation was designed with around six as the ratio of the radius of the coarse and fine particles. The finest particle was selected as 0.3 μ m. Based on the above, the experimental comparison groups were set as 30/5, 3/0.5 and 20/3, 2/0.3. In addition, to compare the identical coarse particles and the same fine particles, the comparison groups were selected as 10/2, 5/2, 5/0.8 in the middle. The materials used for the fabrication of each type of samples are listed in Table 1.

Figure 1 a) the densest packing of the plane; b) the densest packing of space

The powder and resin were mixed in a ball mill for 12 h in order to thoroughly mix uniformly. Then the slurry was vacuum defoamed for 2 minutes to obtain the final alumina ceramic slurry for manufacturing. The green parts of alumina were formed by a 3D printer (Auto-ceraM, Beijing Ten Dimensions Technology Co., Ltd.) through the alumina slurry exposed to a UV light source with a wavelength of 405nm layer by layer. The layer thickness was 100 μ m.

Table 1

Part	Group preparation	Group abbreviation	D50 of coarse particles(μ m)	D50 of fine particles(μ m)	Ratios of mass fraction
1	30 μ m/5 μ m	30/5	30	5	6/4

	20μm/3μm	20/3	20	3	6/4
	3μm/0.5μm	3/0.5	3	0.5	6/4
	2μm/0.3μm	2/0.3	2	0.3	6/4
2	10μm/2μm	10/2	10	2	6/4
	5μm/2μm	5/2	5	2	6/4
	5μm/0.8μm	5/0.8	5	0.8	6/4

2.2 Debinding and Sintering Process

The green bodies were debinded and sintered in a muffle furnace (HEFEI KE JING MATERIALS TECHNOLOGY CO., LTD. China). Firstly, the green parts were heated to 200 °C in 180 minutes, sustained at 200 °C for 180 minutes. This procedure was repeated until 600 °C, and the samples were kept at 600 °C for 120 minutes. Secondly, the samples were heated to 900°C for 40 minutes and held for 40 minutes. Then the samples were controlled to heat to the sintering temperature T (T=1350, 1400, 1450, 1500, 1550, 1600, 1650°C) at a heating rate of 5°C/min, and sustained for 120 minutes. Finally, the sample was cooled to 800°C at a rate of 6°C/min and then cooled in the furnace. The debinding and sintering atmosphere was air.

2.3 Characterization of the Samples

The sintering shrinkage of samples was measured by the following formula using a vernier calliper, with an accuracy of 0.02mm:

$$\delta = \frac{L - L_1}{L} \times 100\% \quad (1)$$

Where δ is the shrinkage of the sintered samples (%), L is the length of the models before the sintering (mm), and L_1 is the length of the models after the sintering (mm).

A procedure defined by the test method for bulk density, apparent porosity and actual porosity of dense shaped refractory products (GB/T 2997-2015), based on Archimedes' principle[30], was used to calculate the bulk density and apparent porosity of the

alumina specimens after sintering. The accuracy of the balance was 0.0001 g (JJ124BC, G&G, America). The theoretical density of the Al₂O₃ was calculated as 3.96 g/cm³[31]. The specific calculation formula is as follows:

$$\begin{aligned}
 p_o &= \frac{m_2 - m_1}{m_2 - m_3} \times 100\% \\
 D &= \frac{m_1 \times D_{\text{water}}}{m_2 - m_3} \times 100\% \quad (2) \\
 p_c &= 1 - \frac{D}{D_{\text{Al}_2\text{O}_3}} - p_o
 \end{aligned}$$

Where p_o is the open porosity of the sintered samples (%), p_c is the close porosity of the sintered samples (%), D is the bulk density of the sintered samples (g/cm³), m_1 is the mass of the dry samples (g), m_2 is the mass of the wet weight of the samples (g), m_3 is the mass of the buoyant weight of the samples (g), D_{water} is the density of the water (g/cm³), and $D_{\text{Al}_2\text{O}_3}$ is the density of the alumina ceramic (g/cm³).

The flexure strength of the sintered samples was tested with an electronic universal testing machine (WDW-10, Jinan Hensgrand Instrument Co., Ltd., China) using the three-point flexure method [32]. The loading speed was 0.5 mm/min, and the span was 30 mm. The specific calculation formula of the flexure strength is as follows:

$$\sigma_f = \frac{3FL}{2bd^2} \quad (3)$$

Where σ_f is the bending strength (MPa), F is the maximum load (N), L is the span of fixture (mm), b is the width of the sample (mm), and d is the height of the sample (mm).

The microscopic morphology was observed by SEM (GeminiSEM 300, ZEISS, Germany). And the surface roughness morphology was followed by a white light interferometer (ZYGONexView, AMETEK, Germany).

3. Results and discussion

3.1 Effect of particle size on flexural strength of alumina ceramics formed by

stereolithography

According to Fig. 2, except that the bending strength of 2/0.3 decreased at 1650°C, the bending strength of each grain size increased with the increase of the sintering temperature. At the same sintering temperature, the bending strength increased with the decrease of the grain size. The rule was consistent with traditional mould forming ceramic manufacturing.

Figure 2 bending strength of a):30/5, 20/3, 3/0.5, 2/0.3; b): 10/2, 5/2, 5/0.8

Figure 3 Compare the SEM of 2/0.3 sintering at a) 1600°C and b) 1650°C

Since finer grains show higher surface energy and grain activity, the finer the grain, the easier the sintering and densification [23]. Therefore, as a whole, as the particle size decreased, the flexural strength at room temperature gradually increased, which also was observed from the change in the degree of particle sintering in Figure 4. For the 10/2, 5/2, and 5/0.8 groups, the particle size change had a more negligible effect on the

bending strength, and the impact of sintering temperature was more prominent. This showed that for alumina particles when the coarse particles were in the range of 5-10 μ m, the bending strength was not sensitive to the particle size gradation. For the 20/3, 2/0.3 and 30/5, 3/0.5 comparison groups, the change in particle size had a more significant impact, indicating that the larger the adjustment ratio of the particle size gradation, the greater the effect on the bending strength.

After the sintering temperature of the 2/0.3 group reached 1500°C, the flexural strength showed a significant increase. It is known that the surface energy of the ceramic particle is the driving force for sintering. The sintering of ceramics results from the migration of ceramic particles under the action of surface tension[33]. The bending strength of the 2/0.3 group decreased at 1650°C. Through Figure 3, we found that the sintering degree increased when the sintering temperature reached 1650, and abnormal grain growth occurred. Although its density was high, the increase in crystal grains greatly impacted the bending strength.

Figure 4 SEM of different particle sizes at different sintering temperature

Four stages of the sintering process were found in Figure 5, which were named the particle rearrangement stage (stage1), the large particle absorption stage (stage2), the pore discharge stage (stage3), and the abnormal crystal grain growth stage (stage4). The schematic diagram of stages was shown in Figure 4. In the stage of particle rearrangement, the degree of particle sintering was low, and the original morphology of the particles was maintained. When the large particles absorb the tiny particles, the

sintering neck between the large and small particles expanded, and the small particles began to fuse to the large particles. The large and small particles were merged in the pore discharge stage, and the crystal grains were relatively uniform. As the degree of sintering increased, the grain boundary slipped, and many pores were discharged. At this stage, the degree of densification grew rapidly, and the porosity quickly decreased. When most of the pores were removed, as the degree of sintering further increased, abnormal growth of crystal grains occurred, which affected the mechanical properties of ceramic. The stages of each particle size at different sintering temperatures were shown in Table 2.

Figure 5 The sintering process

Table 2

	1350℃	1450℃	1550℃	1650℃
30/5	Stage1	Stage1	Stage2	Stage2
20/3	Stage1	Stage2	Stage2	Stage2
10/2	Stage1	Stage2	Stage2	Stage3
5/2	Stage1	Stage2	Stage2	Stage3
5/0.8	Stage1	Stage2	Stage2	Stage3
3/0.5	Stage1	Stage2	Stage2	Stage3
2/0.3	Stage1	Stage2	Stage3	Stage4

From Figure 4 and Table 2, it could be found that both groups of 30/5 and 20/3 only reached stage2, indicating that the degree of sintering of both groups was low. Also, due to the grain size, the grains of the two groups were large, which explained why the strength of these two groups was low. In the micro-topography, although 30/5 and 20/3

both reached the second stage at 1650°C, the degree of fusion of large and small particles was different. As marked by the yellow circle, the interface of 30/5 particles was still clear, while the interface of 20/3 particles disappeared. This was the reason why the flexural strength of 20/3 was significantly higher than 30/5 at 1650°C. The change process of 10/2, 5/2, and 5/0.8 was similar. The final stage and state were close. And their average grain size was all about 3-5 μ m. So their flexural strength changes were identical. For 3/0.5 and 2/0.3, the sintering speed of 2/0.3 was significantly faster than 3/0.5, especially at 1550°C, 2/0.3 entered the pore discharge stage, while 3/0.5 was still in the stage of large particles absorbing small particles. This explained that after 1500°C, the bending strength of 2/0.3 increased, much higher than 3/0.5.

3.2 Effect of particle size on anisotropic shrinkage of alumina ceramics formed by stereolithography

It is generally believed that the shrinkage in the height direction is more significant than that in the length and width directions. Some scholars thought that due to the existence of the interlayer interface, the resin distribution in the height direction was more than that between the layers, and the shrinkage space of the particles after degreasing was also larger[34]. In addition, different curing depth conditions in height, width, and length also led to different shrinkage in different directions. However, not all particle size grading groups showed compliance with this conclusion.

Figure 6 height shrinkage/length shrinkage with different particle size in all sintering temperature

Figure 7 height shrinkage/width shrinkage with different particle size in all sintering temperature

Figure 8 Width shrinkage/length shrinkage with different particle size in all sintering temperature

Figure 9 Length shrinkage

Figure 10 Width shrinkage

Figure 11 Height shrinkage

The overall trend of the three shrinkage rates increased with the decrease of the particle

size of the graded particles (Fig.8&9&10), and the difference between 30/5 and 20/3 was slight. When the size of large particles was within 10um, the shrinkage rate had been significantly improved. The 10/2 and 5/2 groups had a small difference in the height shrinkage, while 5/0.8 showed an increase, indicating that the height shrinkage rate was more affected by the size of the small particles. In addition, the 2/0.3 gradation group also showed a big difference from the other groups, indicating that when the particles were fine to a certain extent, there was a significant manifestation in the shrinkage.

The ratio of the height shrinkage to the length shrinkage of each particle size was about 1.5 (Fig. 5), which clarified that the sintering temperature and particle size have little effect on the ratio. As shown in Fig.6, the shrinkage in the width direction showed consistency with the shrinkage in the height direction in the 30/5 and 20/3 groups. As the particle size decreased, the shrinkage in the width direction gradually approached the shrinkage in the length direction. Some scholars have learned that due to the 3D printing process, there would be a certain degree of resin enrichment between layers [35]. Since the height shrinkage and the length shrinkage maintained a fixed ratio, we believed that there was a certain degree of resin enrichment between layers for each particle size group. Regarding the change in the width shrinkage in this work, it is believed that the coarse particles were difficult to be cured on the outer edge and would fall off during cleaning, leaving the edge resin layer. The two groups of 20/3 and 30/5 have a certain thickness of the edge resin layer, and the sintering shrinkage is greatly affected by the resin layer. The schematic diagram of the model is shown in Figure 11. It is assumed that particles cured less than 2/3 of the particle radius will fall off during cleaning. Taking 20/3 as an example, its ΔR is about 13um. The fine particles are easily embedded in the edge resin, and the edge resin layer is thin. Taking 2/0.3 as an example, its ΔR is about 1.3um.

$$\alpha' = \frac{2\Delta R + (X - 2\Delta R) \times \alpha}{X} \quad (4)$$

$$2\Delta R = (X - 2\Delta R) \times \alpha \text{ (More than ten times)} \quad (5)$$

If Eq. 5 is applicable, then $\alpha' = \alpha$.

Since $\alpha > 1\%$, when $X=50\text{mm}$ (length), Eq. 5 is applicable for all particle sizes. As a result, it can be considered that α is the length shrinkage rate. The calculation shows that the length and width shrinkage of coarse particles are different, while the length and width shrinkage of fine particles are the same. The consistency of the height shrinkage and the width shrinkage of the coarse particles indicates that the proportion of the resin layer in the two directions is the same.

Figure 12 Schematic diagram of edge resin layer model

The roughness of the layered side could also support the above conclusion. Fig. 12 was the side roughness of 2/0.3 and 20/3 in unsintered and sintered at $1350^{\circ}\text{C}/1450^{\circ}\text{C}/1550^{\circ}\text{C}$ for two hours. Before sintering, 2/0.3 and 20/3 had a particular layered structure, and the layered structure of 20/3 was not apparent due to scattering and edge particle falling. After sintering, the layered side structure of 2/0.3 was distinct, while the side surface of 20/3 was uniform and had no layered structure. It indicated that the layered structure of coarse particles was mainly composed of resin, and the resin was removed after sintering, so there was no layered structure. In contrast, the layered structure of fine particles contained fine particles, and the layered structure remained after debinding and sintering.

Figure 13 The roughness of the layered side before and after sintering

3.3 Effect of particle size on porosity of alumina ceramics formed by stereolithography

With the increase of the sintering temperature, the open porosity of each particle size decreased, and the closed porosity increased. At the same sintering temperature, the open porosity increased with the increase of the particle size. (Fig.14 & Fig.15)

a) 2µm/0.3µm

b) 3µm/0.5µm

c) 5μm/0.8μm

d) 5μm/2μm

e) 10μm/2μm

f) 20μm/3μm

g) 30μm/5μm

Figure 14 Porosity of all particle distribution

Figure 15 Comparison of the effective porosity

The open porosity of the 30/5 and 20/3 groups was significantly different from the other groups. For these two groups, the particle size was the main factor affecting their porosity. For the rest of the groups, at 1350°C-1500°C, the distinction between the groups was not great. After 1500°C, the 2/0.3 group showed an apparent rapid decline in open porosity, and the 3/0.5 group only showed a rapid decrease at 1650°C, which was consistent with the change in strength. At the same time, it also showed that the temperature at which the surface energy and grain activity of different fine particles showed prominent sintering ability was different. The finer the particles increased the sintering degree, the lower the sintering temperature.

The primary surface diffusion mechanism is dominated at low temperatures, while surface diffusion cannot make the pores disappear. At high temperatures, the grain boundary diffusion mechanism and the volume diffusion mechanism change to the dominant mechanism, and both mechanisms produce a concentric contraction. When the grains grow up, the grain boundaries move across the pores, and many pores

disappear in the places swept by the grain boundaries so that the density has been significantly improved. [36]

In most cases, the change in sintered porosity is measured by the change in open porosity. With the gradual increase of the degree of sintering, some of these pores are filled, and the other part is transformed into isolated or closed pores. Therefore, as the sintering temperature increases, each particle size group showed a trend of decreasing open porosity and increasing closed porosity.

At the beginning of sintering, a part of the connected pores can be wholly and quickly filled, so the open porosity decreases quickly. When only closed pores remain, the number of closed pores is small on the one hand, and the closed pores are difficult to disappear on the other hand. This slows down the process of densification. We can see that only the two groups 2/0.3 and 3/0.5 had a density of 90% when sintered at 1650°C, indicating that they had entered the later stage of sintering at this time [37,38]. Other samples were still in the middle of sintering when sintered at 1650°C. Therefore, each particle size gradation showed an increase in the rate of porosity change as the sintering temperature increased.

4. Conclusion

In this research, alumina ceramics formed by stereolithography were debinded and sintered. Systematic sintering performance experimental study was carried out with seven different particle size gradations under different sintering temperature. The room temperature flexural strength, shrinkage rate in each direction, and closed porosity of each particle size group increased with the increase of sintering temperature. The open porosity decreased with the growth of sintering temperature. For the same sintering temperature, the flexural strength and shrinkage in all directions decreased with the increase of particle size, and the open porosity increased with the addition of particle

size. The sintering process can be divided into four stages: rearrangement of particles, absorption of small particles by large particles, discharge of pores, and abnormal growth of crystal grains. Each particle size group was at different stages at different sintering temperatures. The coarser the particles, the lower the sintering degree, and the earlier the stage at 1650°C. Only the 2/0.3 group reached the stage of abnormal grain growth at 1650°C. The ratio of height shrinkage to length shrinkage rate was about 1.5, and the width shrinkage gradually approached from the height shrinkage to the length shrinkage as the particle size decreased. Compared with 30/5 and 3/0.5, 20/3 and 2/0.3, when the sintering temperature was above 1500°C, the difference in flexural strength, porosity and shrinkage rate could reach 2~10 times. For 10/2, 5/2, and 5/0.8, the effect of particle size was smaller than that of sintering temperature, and the impact of sintering performance fluctuated within 10%-50%. The 2/0.3 group showed the significant influence of fine particles during sintering. When the sintering temperature was higher than 1500°C, its various sintering properties had been greatly improved. In particular, for the alumina-based core, in this study, the optimal particle size gradation is 20/3, the sintering temperature is 1500°C, the holding time is two hours, the length shrinkage is 1.84%, the width shrinkage is 2.43%, the height shrinkage is 2.34%, bending strength is 29.195MPa, open porosity is 32.98%, closed porosity is 1.44%.

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Figures

Figure 1

a) the densest packing of the plane; b) the densest packing of space

Figure 2

bending strength of a):30/5, 20/3, 3/0.5, 2/0.3; b): 10/2, 5/2, 5/0.8

Figure 3

Compare the SEM of 2/0.3 sintering at a) 1600℃ and b) 1650℃

Figure 4

SEM of different particle sizes at different sintering temperature

Figure 5

The sintering process

Figure 6

height shrinkage/length shrinkage with different particle size in all sintering temperature

Figure 7

height shrinkage/width shrinkage with different particle size in all sintering temperature

Figure 8

Width shrinkage/length shrinkage with different particle size in all sintering temperature

Figure 9

Length shrinkage

Figure 10

Width shrinkage

Figure 11

Height shrinkage

Figure 12

Schematic diagram of edge resin layer model

Figure 13

The roughness of the layered side before and after sintering

Figure 14

Porosity of all particle distribution

Figure 15

Comparison of the effective porosity