A Study on the Manufacturing of Through-Hole Al_2O_3 Scaffolds with Ceramic Powders in Different Grain Diameters

Fwu-Hsing Liu, Chih-Yang Lin, Yu-Heng Liu, and Yunn-Shiuan Liao

***Abstract*—With the development of and demand for medical technology, the tissue engineering is attracting increasing attention. The traditional scaffold are usually made of metal materials, but more and more metal ions precipitate out as the inserting time increases. Therefore, there have been many researches on the manufacturing of scaffolds with ceramic materials in recent years. This research studies the process of manufacturing an Al_2O_3 ceramic scaffold with through-hole structure using 3D printing and SOL-GEL technology. And it uses Al_2O_3 ceramic powders in 2 different grain diameters to improve the scaffolds mechanical properties. The research results show the scaffolds compressive strength, bending strength and porosity are 155.22 Mpa, 35.71 Mpa and 47.24 % respectively when the powders in grain diameters 5 µm and 1 µm are mixed in the ratio 7:3 and under heat treatment at the temperature of 1500 ℃. Based on the material proportion and heat treatment temperature, the Al_2O_3 ceramic scaffold with through-hole structure is prepared. In the future, this research can further conduct cell culture and animal experiments to test its possibility of being applied to the tissue engineering.**

***Index Terms*—Rapid prototyping, sol-gel method, alumina, scaffold.**

I. Introduction

Porous Al_2O_3 ceramics have been widely applied in many fields, including ceramic filters, porous piezoelectric materials, electrodes in fuel cells, bone scaffolds and catalysts supports [1]-[4]. Among the various new technologies of porous material manufacturing, the layer manufacturing technique has attracted a lot of attention in recent years. Besides being as environmentally-friendly as freeze casting, it can be used to any material. For example, the shape and size of porous structure can be controlled precisely with ceramic materials, polymer materials, metal materials and composites [5], [6].

As one of the primary engineering ceramics with a very high melting point, the Al_2O_3 ceramic is able to replace metal in applications at high temperature, making itself an excellent fire resistant material [7]. And it has also been widely used in abrasive materials due to its hardness. Besides, as an inert material, it's corrosion resistant. It has been used to manufacture parts of the artificial limb and surgical instruments since 1970. Playing an extremely important role in tissue engineering, it can direct cells to cling on to the structure, making the structure become a tissue and organ in a specific shape. The scaffold must have high porosity, enabling the nutrient input and metabolite discharge from the interlinking holes and enabling the blood vessel and nerve around the damaged tissue to grow into the material at the same time.

The advantage of using the sol-gel method for the porous Al_2O_3 ceramic scaffold is that the lowered combination reaction temperature of materials enables the materials to realize combination reaction at a lower temperature, which is energy-saving. Many literatures show that the performance of the Al_2O_3 ceramics was improved through the concept of composite. For example, SiO_2 was added to the materials to become mullite through combination reaction. Furthermore, nanoscale or sub-micron SiC, SiN, MgO, Zr and metal powder were added to reinforce the structural performance of Al_2O_3 ceramics [8]-[10].

This paper combines 2 kinds of Al_2O_3 powders in different micron sizes to manufacture aluminum oxide ceramic scaffold, using the 3D printer assembled by our lab and the sol-gel method, hoping to realize the powder structure as shown using the ideal powder ratio and observe the its changes in mechanical property and microstructure at different sintering temperatures show in Fig. 1.

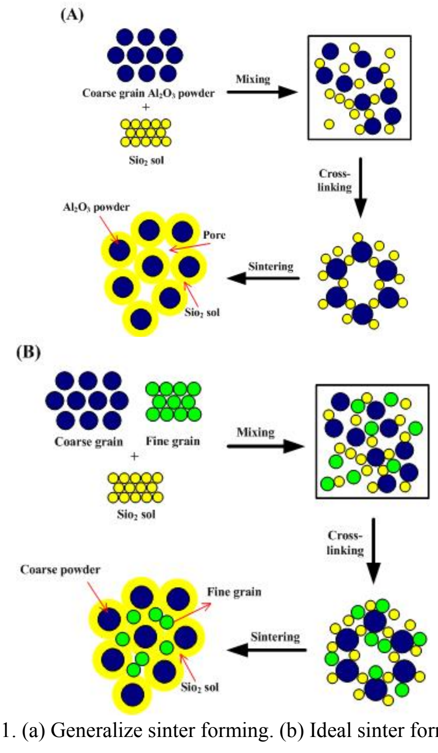

Fig. 1. (a) Generalize sinter forming. (b) Ideal sinter forming.

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II. Materials and Methods

A. Raw Materials

Two kinds of Al_2O_3 powders (marked AP-A, AP-B) are produced from the same company (Nippon Light Metal Company LTD., Japan), whose average particle size is 5.43 μm, 1.21 μm respectively. A typical SEM image of Al_2O_3 powder is shown in Fig. 2. The properties of the two types of powders are shown in Table I. SiO_2 sol (Nissan Chemical Industries LTD., Japan) were used in this study.

TABLE I: Properties of Al_2O_3 Powders

Sample	Average particle diameter (um)	Particle size distribution (um)
AP-A	5.43	4.77－6.41
AP-B	1.21	0.92－1.87

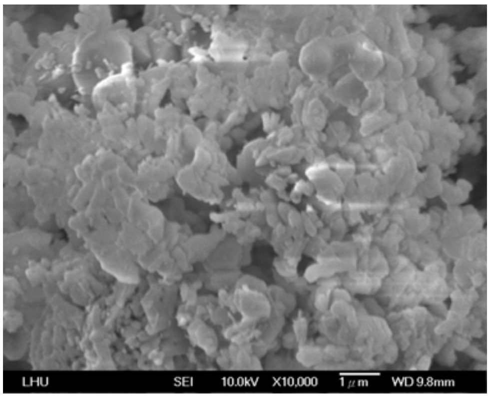

Fig. 2. A typical SEM image of Al_2O_3 powder.

B. Materials Proportion and Analysis

AP-A and AP-B aluminum oxides were mixed with silica gel in a certain ratio and they were kept mixing with zirconia balls for one hour at the ball milling speed of 200 rpm per minute. Their mechanical properties were tested with the universal tester respectively, in which the bending strength (a long test piece in 40 mm × 3 mm × 4 mm) and compressive strength (a round test piece in Φ 6mm × 4mm) were tested with the three-point bending test. For porosity test, the method of Archimedes and the DI water were used to measure the changes of the test piece (10 × 10 × 4 mm) in weight before and after being vacuumized [11]. As for the micro-structure analysis, the scanning electron microscope was used to observe the surface morphology. In the tests above, the sintering speeds of all test pieces that had gone through heat treatment were 5 ℃/min and their heating temperatures were 900, 1100, 1300, 1500 ℃ respectively. They were cooled to normal temperature after being heated for 2 hours. Every test data were based on an average of 5 samples. Their heating temperatures are shown in Fig. 3.

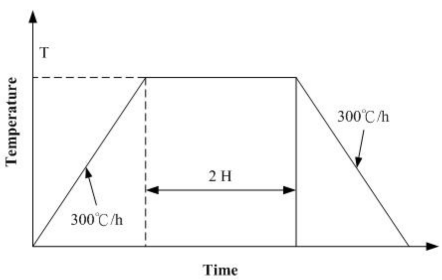

Fig. 3. Heating programs of test sample.

C. Selective Laser Gelling

The test pieces and porous Al_2O_3 ceramic scaffold needed by the mechanical property test were manufactured using the RP machine with the selective laser gelling. This machine includes a laser scanner, a CO_2 laser tube, a controller and a construction zone, etc. Deferent the traditional laser sintering, selective laser gelling achieves curing through the sol-gel method instead of fusing and sintering powers directly. The level of laser that can achieve curing depends on the laser power density (J/mm^3) [12]. The parameters it includes are the laser power P, the laser scanning velocity V, the scan interval S and the thickness of a layer. The ideal laser power density parameters are shown in Table II.

TABLE II: Ideal SLG parameters Used to Produce Green Parts

Parameter	Value
Laser power (p)	2.8 W
Scan speed (v)	130 mm/s
Scan spacing (s)	0.1 mm
Layer thickness (l)	0.1 mm

III. Results and Discussion

A. Structural Analysis of Al_2O_3 Powder

The mechanical properties of Al_2O_3 ceramics have a lot to do with the heat treatment temperature. With the rise in temperature, the mechanical properties would improve sharply. The test pieces went through heat treatment at 900 ℃. The temperature was increased to 1500 ℃ with 200 ℃ as an interval. The data show the mechanical properties at 1500 ℃ are apparently higher than those at 900 ℃, which conforms to the heat treatment mechanism of aluminum oxide ceramics. Table III shows the bending strengths, compressive strengths and porosities of AP-A and AP-B ceramic structures at 1500 ℃.

TABLE III: Mechanical Properties of Single-Module Powders

Sample	Bending strength (Mpa)	Compressive strength (Mpa)	Porosity (%)
AP-A	26.19	98.80	57
AP-B	31.74	106.75	52

B. Composite Structure Analysis of Al_2O_3 Powder

AP-A and AP-B powders were mixed in a certain ratio (Table IV) and the mechanical property was observed after heat treatment (Table V). According to the data, the mechanical property didn't show any difference after 10% AP-B powder was added. After 20% AP-B powder was added, both the bending strength and compressive strength were better that those of AP-A and AP-B. When 30% AP-B powder was added, the maximum value of the mechanical property was reached and the bending strength and compressive strength reached 35.71 Mpa and 155.22 Mpa respectively. However, after 40% and 50% AP-B powders were added respectively, the mechanical strength dropped.

However, the porosity analysis presents a steady distribution of values. Since the grains of AP-B powder were smaller than those of AP-A, the specific surface area of the test piece increased as more and more AP-B powders were added, making the porosity drop. It dropped by 8.65 from when 10% AP-B powder was added to when 30% AP-B powder was added. However, it dropped slowly from when 30% AP-B powder was added to when 50% AP-B powder was added. The porosity' s dropping with the adding of finer powders had something to do with the increase of the specific surface area, but the bending strength and the compressive strength were not consistent with the porosity.

TABLE IV: COMPOSITION OF CERAMICS POWDER (WT%)

Sample	AP-A	AP-B
1	90	10
2	80	20
3	70	30
4	60	40
5	50	50

TABLE V: MECHANICAL PROPERTIES OF COMPOSITE-MODULE POWDERS

Sample	Bending strength (Mpa)	Compressive strength (Mpa)	Porosity (%)
1	29.09	100.45	55.89
2	33.29	122.16	51.94
3	35.71	155.22	47.24
4	31.07	132.90	46.88
5	27.16	119.88	45.79

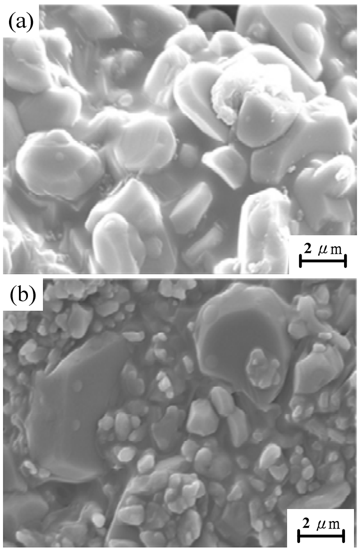

Fig. 4. (a) AP-B heat treatment 1500℃, (b) AP-B mixed AP-A heat treatment 1500℃.

C. Micro-Structure Analysis

Fig. 4 (a) shows the microstructure of AP-A at 1500℃, from which many electron holes can be seen, which is a big factor that deteriorates mechanical properties. And the sizes and distribution of grains are volatile. Fig. 4 (b) shows the microstructure of the mixed grains of AP-A and AP-B at 1500℃, from which we can see electron holes left by AP-B grains filling AP-A grains. The structures of the 2 kinds of grains are diversified. Compared with those in Fig. 4 (a), AP-A grains grow obviously in size.

The distribution of grains and whether the structure has cracks have a great effect on mechanical properties. If the grains are evenly distributed and the microstructure is free from cracks, the engineering properties will be good. From the ratio in which the composite is added, adding 30% AP-B powder at 1500℃ is ideal. If the heat treatment temperature is too low, the reaction temperature will not be reached; on the contrary, the grains will grow so big in size that they will collide with other grains and damage each other, making the mechanical properties drop.

IV. POROUS Al_2O_3 CERAMIC SCAFFOLD MANUFACTURING

To make cells grow into the structure needed and direct cells to grow and differentiate, we need a porous structure to simulate the environment of extracellular matrix in the living organism so as to make cells move in, proliferate and reproduce in the porous scaffold. The outside dimension of the porous Al_2O_3 ceramic scaffold in this experiment was designed to be 10 mm × 10 mm × 6 mm and there were 9 square through-hole structures 1mm on a side inside, as shown in Fig. 5 (a). A porous scaffold made of 100% AP-A powders with the laminating machine assembled and appropriate machining parameters was used as the control group. And another porous scaffold was made with powders in the ideal ratio. Fig. 5 (b) shows the finished product of the porous composite scaffold.

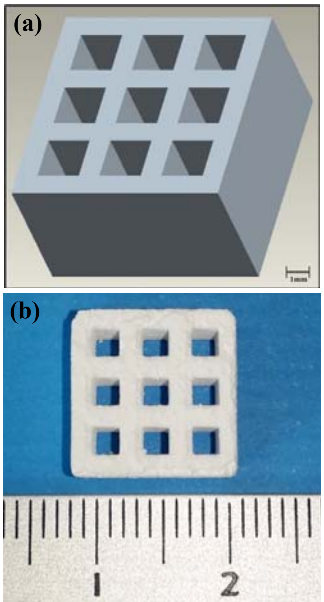

Fig. 5. A through-hole Al_2O_3 ceramic scaffold. And (a) 3D CAD, (b) an actual part.

V. CONCLUSION

This paper hopes to improve the mechanical properties of the scaffold by making it with Al_2O_3 powders in different grain diameters with the sol-gel method in the proper ratio. The research results show the when the ratio of AP-A to AP-B is 7:3, the mechanical properties are the best, with the compressive strength, bending strength and porosity being 155.22 Mpa, 35.77 Mpa and 47.24 % respectively.

On the manufacturing of the traditional scaffolds, salt fractionation, freeze-drying method and gas foaming all have problems, such as uneven distribution of holes, uncontrollable microstructure and pore size distribution constraint. However, the porous scaffold made with the laminating machine assembled can control the pore shape and size, which proves the scaffold made with the RP machine has high stability and reproducibility. Besides having interlinking pores, the ideal porous scaffold needs to have good mechanical properties.

In future researches, the mechanical properties of the porous scaffold will be tested and compared with those of the traditional scaffold to make the scaffold able to bear the damages caused by the external force in the cell culture environment and the surgical inserting process.

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