Porous Ti6Al4V scaffolds directly fabricated by 3D fibre deposition technique: Effect of nozzle diameter

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3D porous Ti6Al4V scaffolds were successfully directly fabricated by a rapid prototyping technology: 3D fibre deposition. In this study, the rheological properties of Ti6Al4V slurry was studied and the flow rate was analyzed at various pressures and nozzle diameters. Scaffolds with different fibre diameter and porosity were fabricated. ESEM observation and mechanical tests were performed on the obtained porous Ti6Al4V scaffolds with regard to the porous structure and mechanical properties. The results show that these scaffolds have 3D interconnected porous structure and a compressive strength which depends on porosity at constant fibre diameters and on the fibre diameter at constant porosity. These Ti6Al4V scaffolds are expected to be constructs for biomedical applications.

1. Introduction

Titanium and its alloys are widely used in biomedical application for two reasons: their good corrosion resistance and their high strength-to-density ratio [1]. Therefore, various processing techniques have been used to make porous titanium scaffolds, such as: sintering of powders [2], electro sparkling Ti6Al4V powder [3], or plasma spraying of powder on a dense substrate followed by cutting off the porous layer [4], compressing and sintering of titanium fibres [5, 6] and polymeric sponge replication [7]. Most of these processes form structures with randomly arranged pores with a wide range of sizes, and have limited flexibility to control the pore architecture such as size distribution or interconnectivity and porosity.

During the past decade, rapid prototyping (RP) technologies have emerged as a revolutionary manufacturing process to build objects with predefined microstructure as well as macrostructure [2]. RP allowed researchers to design and fabricate regular and reproducible scaffolds with a completely interconnected pore network. Generally, polymer scaffolds can be directly fabricated by this technique for ceramic and metal scaffolds, a negative replica of the desired structure within a polymeric mould was created first by RP where after the scaffold is produced by casting with molten metal or ceramics suspensions, drying the structure, removing the organic mould by thermal decomposition, followed by sintering. Normally these are time consuming process [8, 9]. Although, selective laser sintering has been used to make ceramics and metal scaffolds directly, the material resolution is relatively low and supporting materials, necessary in overhanging structure, are not easy to remove completely [10].

Directly fabricating scaffolds by using a concentrated Ti6Al4V slurry and 3D fibre deposition offers a new approach for creating Ti6Al4V scaffolds. Development of porous scaffolds via 3D fibre deposition involves three major steps. The first step includes the selection of a Ti6Al4V powder slurry which can be extruded. The second step is feedstock development. The final step involves the 3D deposition of Ti6Al4V fibres. In this study, a 3D-plotting machine [11, 12] (Bioplottor, EnvisonTech GmbH, Germany) was used. A syringe containing slurry was mounted, and 3 axis motion is independently controlled by a computer system. By setting of the fibre diameter (FD), the length thickness (LT), fibre space (FS) and the fibre lay-down pattern, the elementary structure can be created. Fig. 1 shows this fabrication process of computer controlled 3D fibre deposition in the case of a simple "cross-bar" structure.

In a previous study, parameters of 3D fibre deposition were optimized and influences on the scaffold structure were discussed [13].

The purpose of this study was to fabricate scaffolds with different nozzle diameter and determine the result for the scaffold properties.

2. Materials and methods

2.1. Material

Ti6Al4V powders (Bongen Titanium (China) Co, ltd) with a mean diameter of 45 μm were used in this study. The Ti6Al4V powder (66.vol %) was mixed with an aqueous solution of binders (Methylcellulose (MC, Fisher Scientific B.V) and Stearic acid (Acros organics, USA)). The nozzle used to extrude the Ti6Al4V slurry fibres is a stainless steel hypodermic needle, shorted to

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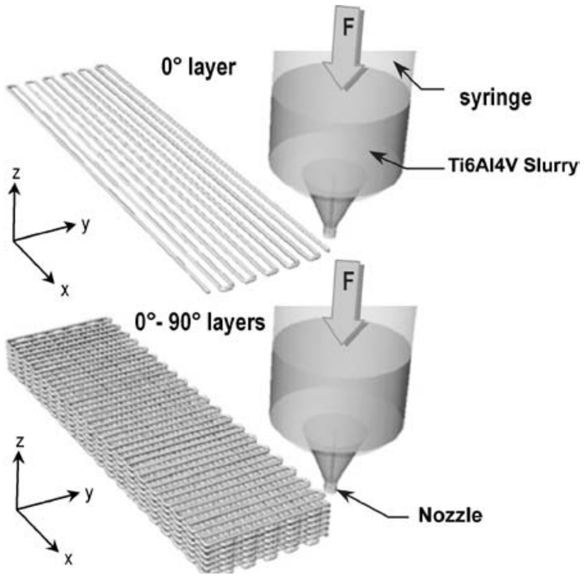

Figure 1 3D fiber depositing scaffold process.

a length of 16.1 mm. The nozzle size is expressed as an inner diameter of the nozzle. Nozzle diameters of 250, 400, 500 and 700 μm were used for this study.

2.2. Scaffold fabrication

Air pressure (pressure 3–5 bar) was applied to the syringe through a pressurized cap. The Ti6Al4V slurry is forced through the nozzle to be deposited on a stage as a fibre, which rapidly dries on the air, and the scaffold is fabricated by layering a 0–90° pattern of these fibres. Rectangular block models of 20 × 20 × 10 mm were loaded on the Bioplotter CAM software. After the porous Ti6Al4V scaffolds were dried for 24 h at room temperature (RT), they were debonded and sintered under high vacuum at 1200 °C for 2 h.

Ti6Al4V scaffolds were fabricated for three experiments. The first experiment (Experiment I) was to investigate the structure of scaffolds with different nozzle sizes and porosities. In this experiment, nozzle diameters of 250, 400, 500 and 700 μm were used. The second experiment (Experiment II) was to study the effect of fibre diameter at constant porosity on the scaffold mechanical properties. Nozzle diameters of 400, 500 and 700 μm were chosen (EII-1, EII-2 and EII-3). The third experiment (Experiment III) was to study the effect of porosity on the scaffolds' mechanical properties. A nozzle diameter of 500 μm was used to make scaffold with different porosities (EIII-1, EIII-2 and EIII-3) by varying the fibre space (200, 500 and 800 μm). Table I shows the scaffold group for this experiment. To ensure adhesion between the fibres in adjacent layers, the layer thickness was set invariably at about 0.8* nozzle size [14].

The parameters which determine the geometry and porosity of the 0–90° cross fibre structure are LT, FD and FS as defined before. Based on a pilot experiment, the parameters of scaffolds with different diameters but equal porosity were estimated (Table I) so that the influence of each parameter on mechanical properties, for instance, can be determined.

TABLE I Scaffold groups for experiment

Specimen group	Nozzle size (mm)	Lay-down pattern (mm)	Fibre space (mm)	Layer thickness (mm)
EII-1	0.4	0/90°	0.5	0.35
EII-2	0.5	0/90°	0.6	0.4
EII-3	0.7	0/90°	0.8	0.5
EIII-1	0.5	0/90°	0.2	0.4
EIII-2	0.5	0/90°	0.6	0.4
EIII-3	0.5	0/90°	0.8	0.4

2.3. Characterization

2.3.1. Viscosity

The rheological behavior of Ti6Al4V slurries was measured at RT with a viscometer (Brookfield Engineering Labs DV-II+ viscometer) with interval time at a speed of 10 rpm with a RV0 spindle.

2.3.2. Morphology

The structure was characterized by environmental scanning electron microscope (ESEM, XL-30, Philips, Eindhoven, The Netherlands), enabling the measurement of the actual of obtained values for FD, FS and LT.

2.3.3. Porosity

The actual Porosity (P) was calculated by measuring the apparent density (ρ_b =weight of sample/volume of sample) of sample by using the formula: $\rho = (1 - \rho_b/\rho_s) \times 100$, where ρ_s is the density of 100% dense material(4.5 g/cm^3). A total of 5 samples were measured.

2.3.4. Mechanical properties

Five samples from each kind of scaffold were randomly chosen. The compression tests of porous Ti6Al4V samples (4 × 4 × 6 mm, $n = 10$) were performed at room temperature with a crosshead speed of 1 mm/min (Zwick/Z050, Germany). The loading direction is in the Z-direction of the deposition process.

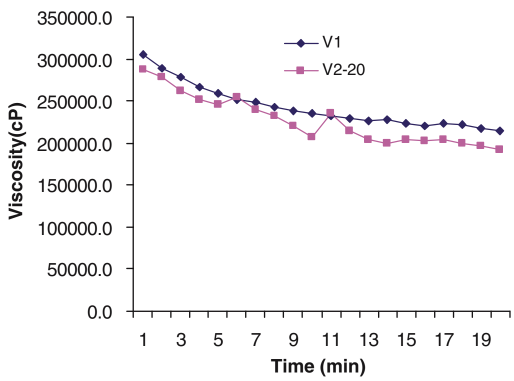

Figure 2 Viscosity of Ti6Al4V slurry with 80 wt.% concentration of powder. V2-20 means measurement after 20 mins of the first measurement.

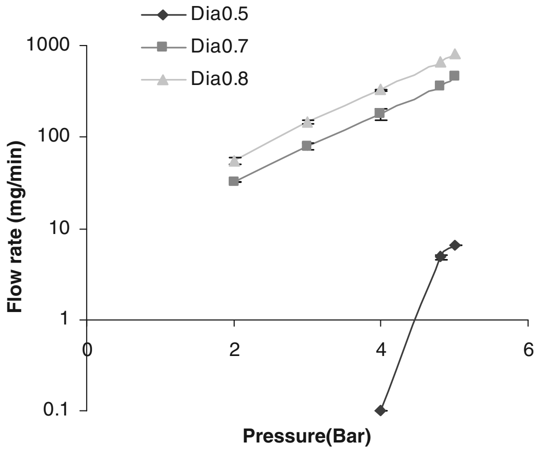

Figure 3 Flow rate varied with nozzle diameter at different pressure.

3. Results and discussion

3.1. Viscosity

The viscosity of Ti6Al4V slurry is shown in Fig. 2. The viscosity decreases with increase of time. It can be seen that when the measurement is repeated after 20 min, the same tendency of decreasing viscosity is observed. It reveals that the slurry displays a thixotropic behaviour. It is of importance to be able to deposite a fibre without supporting material when bridging gaps and this is favoured by the thixotropic behaviour of the slurry which means that a reduction in viscosity occurs when shaken, stirred, or otherwise mechanically disturbed, but at rest the slurry recovers in time, eventually reaching its original viscosity. Fig. 3 shows the flow rate as a function of nozzle diameter and pressure. It appears that the flow rate is not linearly proportional to the pressure, and thus not in agreement with the Hagen–Poiseuille equation [15]. This is to be expected due to non Newtonian behaviour of particulate slurries.

TABLE II Measurement of structural features of Ti6Al4V scaffold

Specimen group	Fibre diameter (μm)	Fibre space (μm)	Layer thickness (μm)	Porosity (%)
EII-1	369 ± 13	381 ± 16	284 ± 22	56 ± 1.5
EII-2	478 ± 18	374 ± 18	352 ± 25	55.5 ± 1.2
EII-3	570 ± 19	402 ± 9	402 ± 29	55 ± 1.1
EIII-1	455 ± 18	176 ± 14	343 ± 29	36 ± 2.2
EIII-2	465 ± 15	391 ± 5	347 ± 26	55 ± 1.4
EIII-3	485 ± 13	556 ± 13	358 ± 31	62 ± 0.7

3.2. Morphology

Fig. 4 shows the scaffold made at different nozzle diameters. The use of a finer nozzle allowed the fabrication of scaffolds with a larger pore size, under similar setting of the fibre space, and having a higher surface area to volume ratio which may be of importance for cell adhesion in tissue engineering application.

Fig. 5 shows the scaffolds with varying porosity as result of changing fibre space (FS) at constant fibre diameter (FD). It can be seen that with decreasing the fibre space the structure becomes denser so that porosity of scaffold decreases. To increase the porosity, the FS can be increased but not indefinitely as slacking will occur when the strength of the extruded fibre becomes insufficient to bridge a wide gap.

Based on the ESEM measurement, features of the obtained scaffold are given in Table II. Compared with the original setting (Table I). FD is near to the inner size of nozzle, but FS and LT deviate more as a result of the sintering process at elevated temperature.

3.3. Porosity

The porosity of the scaffolds in Experiment II showed little variations under measurement (Table II). The

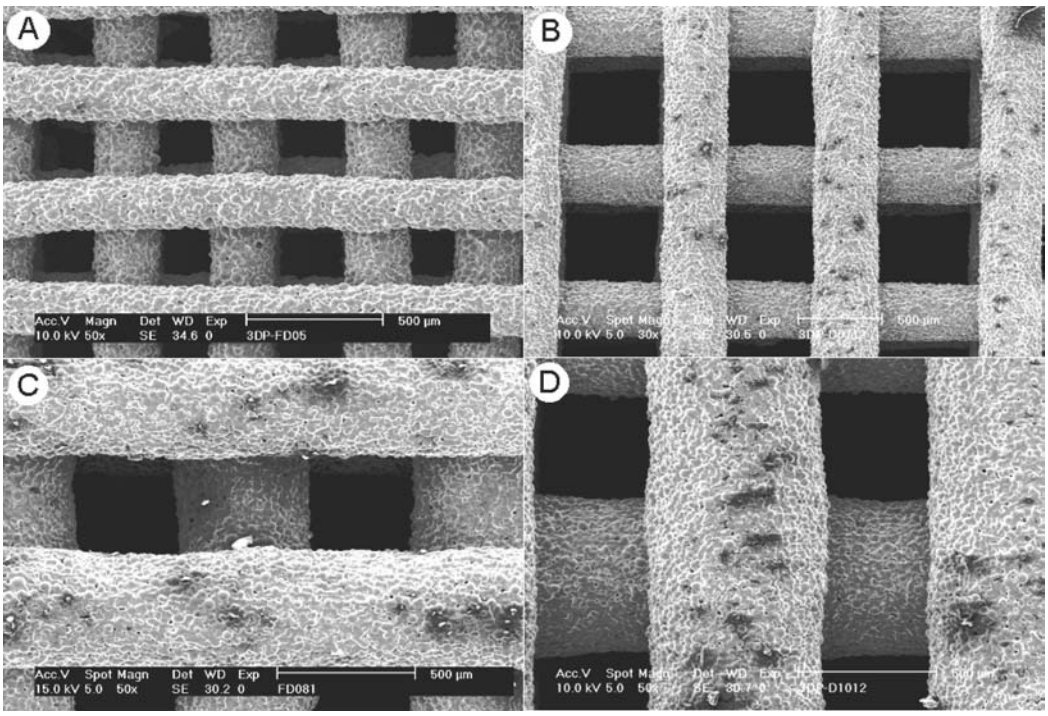

Figure 4 Ti6Al4V scaffold made by different nozzle diameter provide the unity measure for the diameters indicated in (a) 250, (b) 400, (c) 500, (d) 700.

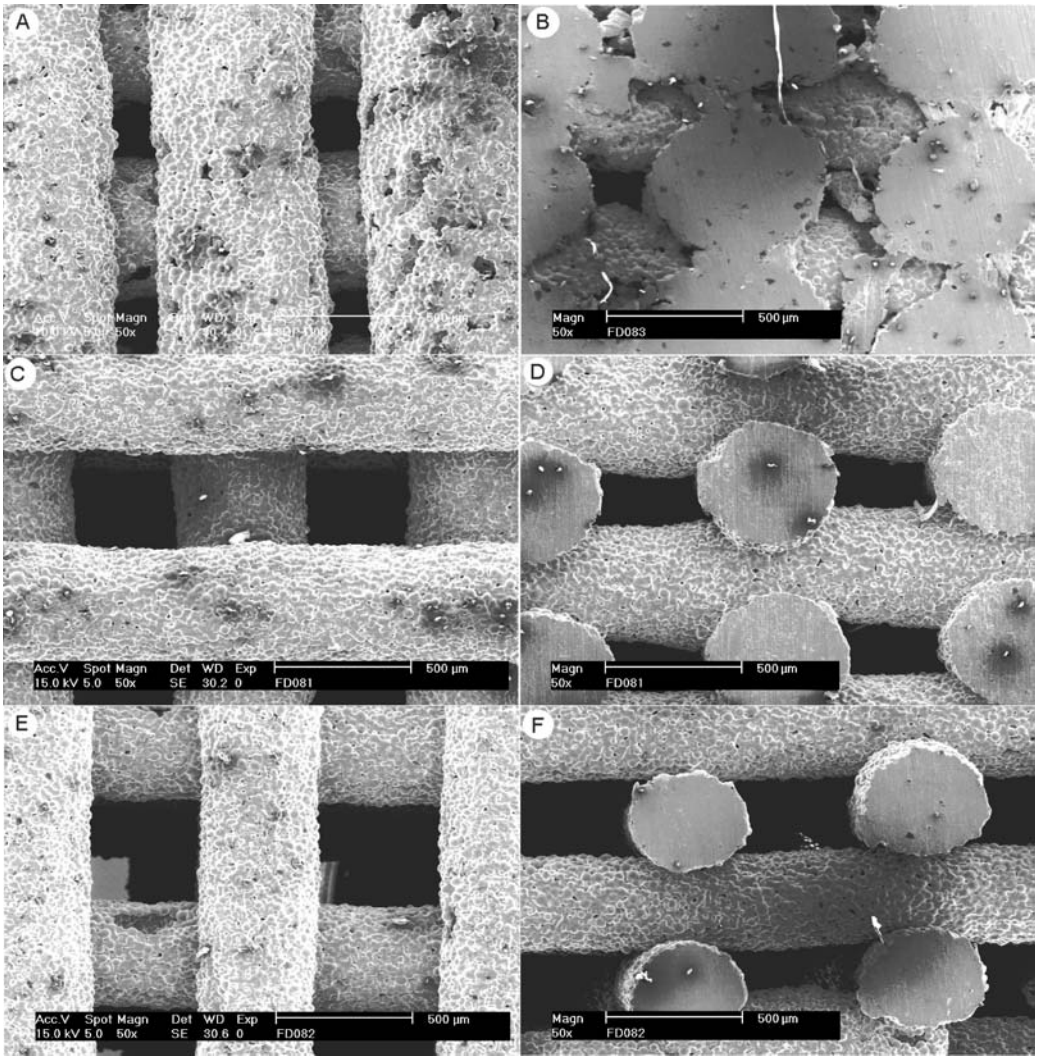

Figure 5 Effect of fibre space on the scaffold processing (a) 200, top-view, (b) 200, side view of cross-section, (c) 500, top-view, (d) 500, side view of cross-section, (e) 800, top-view, (f) 800, side view of cross-section.

results showed that 3D fibre deposition enables the production of highly reproducible scaffold.

3.4. Mechanical properties

The compressive strength of scaffolds made with different nozzle sizes is shown in Fig. 6. In the porosity range of 54–58% scaffolds with the smallest fibre diameter had the highest compressive strength (432 ± 30 MPa). As compressed under z-direction, it is the fibre joint of adjacent layers that bear the loading force. A larger number of fibre joints can indeed be expected to strengthen the scaffold structure. Fig. 7 shows the compressive strength of scaffolds at different fibre space. It can be seen that the compressive strength decreases with increasing fibre spacing. It reveals that the compressive strength varies as function of porosity and

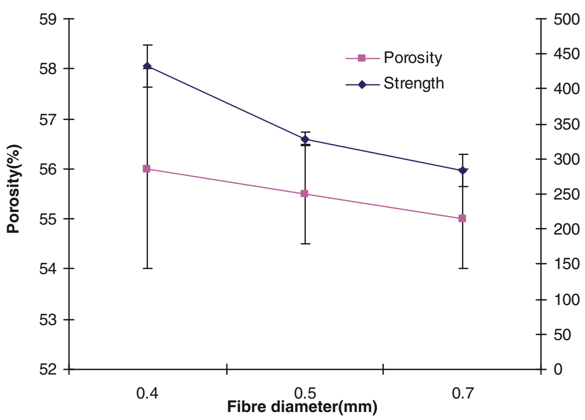

Figure 6 Strength varies with fibre size.

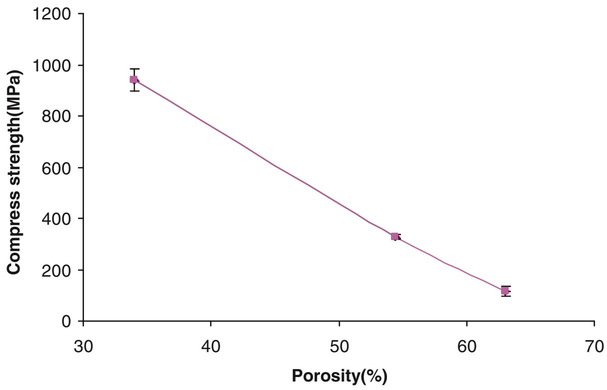

Figure 7 Strength varies with fibre space (porosity).

consequently increasing porosity at constant fibre diameter. This is in agreement with Gibson and Ashby's model which gives an exponential relationship between porosity and mechanical strength [16]. It appears, however, that also a structural component like the fibre diameters in these scaffolds are of influence on its mechanical properties.

4. Conclusion

Porous Ti6Al4V scaffolds were successfully directly made by a 3D fibre deposition technology. The scaffold possesses in principal fully interconnected pore networks, and highly controllable porosity and pore size. Scaffolds with different morphology can be made by this technique. The compressive strength was found to be higher in scaffolds with finer fibres under similar porosity. At equal fibre diameter, the strength decreases rapidly with increasing porosity. This technique offer attractive opportunities for designing and desktop manufacturing of biomedical scaffolds and applications in tissue engineering and orthopaedic implants.

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