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MECHANICAL AND BIOLOGICAL PROPERTIES OF TITANIUM SYNTACTIC FOAMS

XB Xue[1], YY Zhao[1], V Kearns[2], RL Williams[2]

[1]Department of Engineering, University of Liverpool, L69 3GH, UK
[2]Clinical Engineering, School of Clinical Sciences, University of Liverpool, L69 3GA, UK

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Abstract

Titanium syntactic foam is a novel composite material with hollow ceramic microspheres embedded in titanium matrix. This paper reports on the preliminary studies on the mechanical and biological properties of Ti syntactic foam manufactured by powder metallurgy. The density and porosity were measured. The compression, three-point bending, indirect and direct contact cell culture tests were conducted. The density and porosity varied with compaction pressure. However, higher pressure resulted in a large number of crushed microspheres. The apparent modulus, compressive and flexural strength increased with increasing sintering temperature or sintering time. Indirect cell contact tests demonstrated that the titanium syntactic foams were non-cytotoxic. In direct cell contact tests the cells attached and spread well on the surface of titanium syntactic foams. These data suggest that this material warrants further investigation in bone replacement applications.

Introduction

Ti is an excellent biomaterial for orthopaedic and dental applications because of its good biocompatibility and corrosion resistance. Previous research has shown that Ti surfaces can support cell growth and differentiation [1]. However, the surveys conducted by Havelin et al. [2] and Malchau et al. [3] showed that more than two thirds of all revisions of femural implants were caused by implant loosening, i.e., disruption of the implant/bone or cement interface [4]. A major factor in implant loosening is caused by stress-shielding, a phenomenon in which the physiological load applied to the bone is reduced due to the presence of an implant with a stiffness greater than that of bone [5, 6]. With stress shielding, the bone becomes less dense and weaker. Implant materials with an elastic modulus similar to that of human bone are thus desirable in implant applications, so that the distribution of loads in the bone is not altered. Bone is an anisotropic material and varies in its mechanical properties throughout the body. Therefore, a material that can be designed with to have a variable elastic modulus would be an attractive candidate for implants.

This paper investigates a novel material, Ti matrix syntactic foam, for implant applications. In Ti syntactic foam, ceramic microspheres with hollow or porous structure are embedded in a Ti matrix. The existence of two solids, ceramic and Ti, and air in the syntactic foam, allows a wide range of elastic moduli to be produced. This characteristic, together with the biocompatibility of the Ti matrix, makes Ti syntactic foam a potential implant material. In this study, a set of Ti syntactic foams were fabricated in different manufacturing conditions. Their density, porosity, elastic modulus and strength were measured, and their biological properties were evaluated.

Experimental Procedure

Materials and Fabrication

The raw materials used for fabricating the Ti syntactic foam samples were titanium powder with 99.4% purity, supplied by Active Metals Ltd U.K., and ceramic microspheres, supplied by Pty Ltd Australia. The Ti powder particles have an irregular shape and an average particle size of 30 μm. The ceramic microspheres have a composition of ~60% SiO_2, ~40% Al_2O_3 and 0.4-0.5% Fe_2O_3 by weight, an apparent density of 0.8g/cm^3 and an average particle size of 150 μm.

Ti syntactic foam samples were fabricated by a powder metallurgy process. Ti powder and ceramic microspheres were first blended uniformly, with a Ti:ceramic microspheres volume ratio of 4:6, 5:5, 6:4, 7:3 or 8:2. Each mixture was divided into five equal parts and compacted at pressures of 45MPa, 70MPa, 100MPa, 150MPa and 200MPa, respectively. The compacted samples were sintered at 1200 ℃ for 1.5 hours in a vacuum furnace with a pressure of 1×10^{-4} mbar and then cooled to room temperature in furnace. A set of Ti syntactic foam samples with different volume percentages of Ti and different compaction pressures were thus obtained.

Density and Porosity

The density of each Ti syntactic foam sample was determined by the mass of the sample divided by its volume, which was measured by the Archimedes method. The porosity of the samples is the volume percentage of air in the sample and can be estimated by:

$$\mathrm{P} = \frac{V - \frac{W_{Ti}}{\rho_{Ti}} - \frac{W_c}{\rho_{sc}}}{V} \% \qquad (1)$$

where V is the volume of the Ti syntactic foam sample, W_{Ti} is the mass of Ti, ρ_{Ti} is the density of Ti (4.5 g/cm^3 [7]), W_c is the mass of ceramic microspheres and ρ_{sc} is the density of solid ceramic in the microspheres. ρ_{sc} is estimated to be 2.9g/cm^3 from the composition of the microspheres.

Static Compression Tests

Before the compression tests, the Ti syntactic foam specimens were cut to produce samples with dimensions of 10×10×20mm. The compression tests were conducted in an Instron machine with a cross-head speed of 0.5mm/min. During the compression tests, a series of unloading and reloading routines were carried out at 0.4%, 0.6% and 0.8% strains to obtain the elastic modulus values.

Three Point Bending Tests

The Ti syntactic foams specimens were cut to produce samples with dimensions of 10×5×40mm for three point bending tests. The surfaces were polished with 1200 grit papers to remove any surface defects. The tests were conducted on the Instron machine at a cross-head speed of 0.3mm/min. The flexural strength of each specimen was calculated by:

$$\sigma = \frac{3FL}{2bd^2} \tag{2}$$

where F is the maximum load, L is the span length (L=36mm), b is the width of specimens (b=10mm) and d is the depth of specimens (d=5mm).

***In Vitro* Biocompatibility**

Indirect and direct contact cell culture tests were conducted with Ti syntactic foams. Samples were placed in an autoclave and sterilized in 121 ℃ high pressure steam. Indirect contact tests were carried out according to ISO 10993-5. Samples were placed in sealed plastic vessels and incubated in complete culture medium (Dulbecco's Modified Eagle Medium, DMEM, containing 1% penicillin/streptomycin and 10% foetal calf serum) at 37 ℃ for 72 hours. The extract was collected and diluted with complete culture medium and used at a ratio of 1:1, 10:1, 100:1. Positive and negative controls were 100% D-MEM and 100% extraction, respectively. Saos-2 osteocarcinoma cells were seeded at a density of 5×10^4/well in a 24-well plate and grown for 72 hours. A viability assay (Invitrogen, Paisley, UK) was performed according to the manufacturer's instructions. Cells were observed using an inverted fluorescent microscope (Axiovert 200, Carl Zeiss Ltd).

Cells were also cultured in direct contact with the foams. 5×10^4 cells in a volume of 5μl complete culture medium were seeded onto the surface of the samples. The samples were incubated for 20min and then 1ml of medium was added to cover the surfaces of the sample. After 48 hours, the samples were fixed in 2.5% gluteraldehyde for 30 minutes then dehydrated for 30 minutes in each of 70%, 90% and 100% ethanol, before being critical point dried. Finally, the samples were gold coated and for observation by SEM (Hitachi S-2460N).

Results and Discussion

Structural Properties

Fig. 1 shows a cross-section of a typical specimen of the Ti syntactic foam. Ti syntactic foam generally has a homogeneous structure, with ceramic microspheres surrounded by the Ti matrix and distributed uniformly in the whole sample. Fig. 2 shows the density and porosity of the Ti syntactic foams fabricated with different compaction pressures. Generally, higher compaction pressure increased the densification of the Ti matrix, therefore, increased the density and decreased the porosity. However, if the compaction pressure was too high, a considerable proportion of the ceramic microspheres were crushed during compaction. Smaller Ti particles also benefited the densification. A higher volume percentage of Ti in Ti syntactic foam resulted in increased densities and decreased porosities of the foams. Compared with compaction pressure, the effects of Ti volume percentage were much less significant.

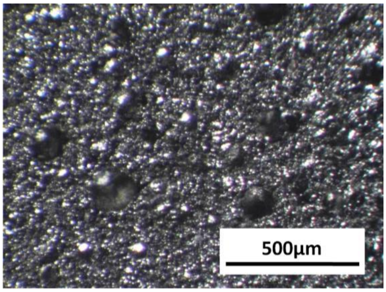

Fig. 1. Optical micrographs of a cross section of a typical Ti syntactic foam.

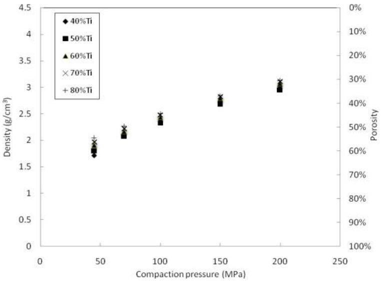

Fig. 2. Densities and porosities of Ti syntactic foams.

Elastic Modulus and Strength

The elastic modulus of Ti syntactic foam was obtained from the compressive stress-strain curves of the samples (a typical curve is shown in Fig. 3). A series of unloading and reloading cycles were carried out at different strains. The slope of the unloading curve was taken as the elastic modulus. Table 1 shows the elastic moduli of the Ti syntactic foams fabricated at a compaction pressure of 45MPa with different Ti volume percentages measured at different strains. Compared with pure Ti, Ti syntactic foam had a much lower elastic modulus, because of the existence of hollow ceramic microspheres and air void. The elastic modulus was comparable with that of cement but lower than that of cortical bone. The elastic modulus generally increased slightly with the volume percentage of Ti in the Ti syntactic foam. It also increased with increasing strain.

Table 1. The elastic moduli of Ti syntactic foams fabricated at a compaction pressure of 45MPa at different strains.

Ti%	Elastic modulus (GPa)		
	0.4% Strain	0.6% Strain	0.8% Strain
40%	2.6	3	3.42
50%	2.7	3.1	3.7
60%	2.65	3.67	4.35
70%	2.88	3.65	4.25
80%	2.96	3.96	4.4
Pure Ti [7]	115	-	-
Cement [8]	2	-	-
Cortical bone [8]	20	-	-

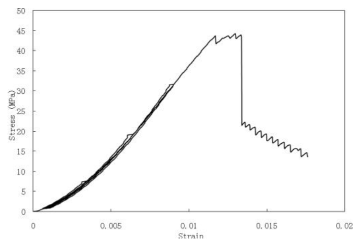

Fig. 3. Compressive stress-strain curve of Ti syntactic foam with 60% volume percentage of Ti fabricated at a compaction pressure of 45MPa.

Fig. 4 shows the effect of compaction pressure on compressive and flexural strengths. The embedded ceramic microspheres and air void resulted in a reduction in the compressive and flexural strengths in comparison with pure Ti [7]. By increasing the compaction pressure, the compressive and flexural strength can be increased. This is because increasing compact pressure increased the contacts between the Ti particles and therefore the bonding strength of the Ti matrix was increased during sintering. Fig. 5 shows that extending the sintering time also enhanced the compressive and flexural strengths.

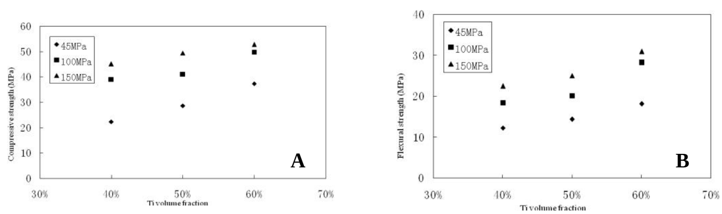

Fig. 4. Variations of (A) compressive and (B) flexural strength of Ti syntactic foams with volume percentage of Ti fabricated at different compaction pressures.

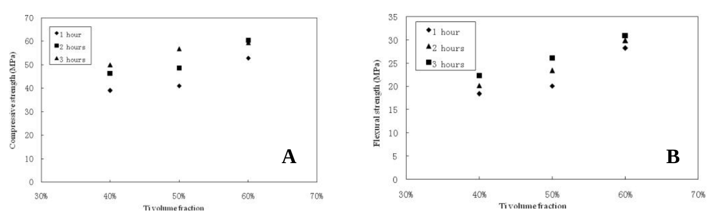

Fig. 5. Variations of (A) compressive and (B) flexural strength of Ti syntactic foams with volume percentage of Ti fabricated at different sintering times.

In Vitro Biocompatibility

The live/dead cell culture assay allowed qualitative assessment of the cytotoxicity of the Ti syntactic foams. After 72 hours of cell growth in medium containing extracted material from the Ti syntactic foams, the vast majority of the cells remained alive (green) with only a small number of dead cells observed (red) (Fig. 6b). The cellular response was similar to that in the control, which was shown in Fig. 6a.

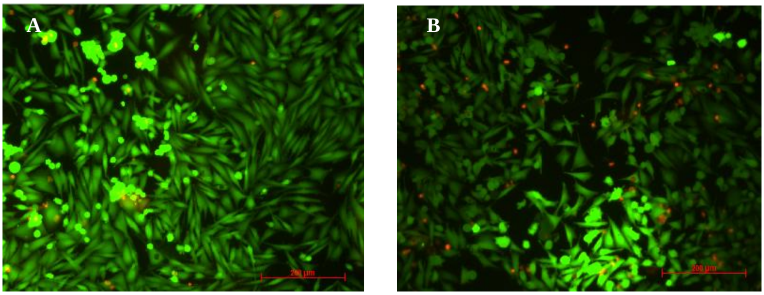

Fig. 6. Cells cultured in (A) 100% DMEM medium and (B) 10% extraction of Ti syntactic foam with a Ti volume fraction of 40%. (green = live cells; red = dead cells)

Fig. 7 shows the examination of the cell attachment to the Ti syntactic foams after 48 hours. It demonstrated that the cells attached to the Ti matrix were generally greater in number and had a more spread morphology than those attached to the ceramic microspheres.

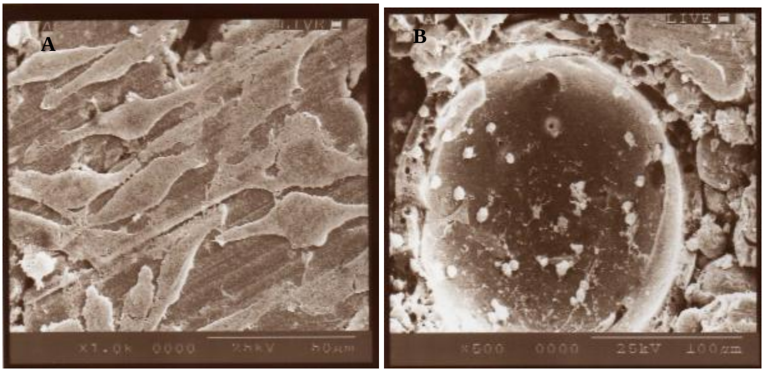

Fig. 7. Cells cultured on the surface of (A) Ti matrix, and (B) ceramic microsphere.

Conclusion

Titanium syntactic foams with a uniform structure were fabricated by powder metallurgy. Higher compaction pressure or higher volume percentage of Ti resulted in higher density and lower porosity, but the effect of Ti volume percentage was less significant. The elastic modulus, compressive and flexural strengths of Ti syntactic foam were much lower than those of pure Ti. However, these properties could be improved by increasing compaction pressure and sintering time. *In vitro* tests demonstrated that cells spread more readily on Ti matrix than on ceramic microspheres. Overall, Ti syntactic foams were not cytotoxic.

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