Ti Matrix Syntactic Foam Fabricated by Powder Metallurgy: Particle Breakage and Elastic Modulus

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Titanium matrix syntactic foams have potential for orthopedic applications because of their good biocompatibility, corrosion resistance and ability of varying the elastic modulus. This paper studies the fabrication of Ti matrix syntactic foams embedded with ceramic microspheres (CMs) by a powder metallurgy method. The percentage of the crushed CMs during compaction was measured by the volume measurement and water absorption methods, and the elastic modulus of the Ti matrix syntactic foam was measured by compression tests. The effects of the Ti volume percentage and the compaction pressure on the percentage of crushed CMs and the elastic modulus were studied. For a given Ti volume percentage, the percentage of crushed CMs increases with increasing compaction pressure; for a given compaction pressure, the percentage of crushed CMs decreases with increasing Ti volume percentage. At a compaction pressure lower than 100 MPa, the elastic modulus increases with increasing Ti volume percentage and compaction pressure; at a compaction pressure above 100 MPa, further increases in Ti volume percentage and compaction pressure decrease the elastic modulus.

INTRODUCTION

Titanium is an excellent biomaterial for orthopedic 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 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 density and strength decrease. 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. Because bone is an anisotropic material and its mechanical properties vary throughout the body, a material with a variable elastic modulus would be an attractive candidate for implants.

Titanium matrix syntactic foams have recently been fabricated by a powder metallurgy method, with an aim for biomedical applications.[7] The preliminary results showed that the density, porosity, and compressive and flexural strengths of the Ti matrix syntactic foams are affected by the Ti volume percentage and the compaction pressure. Higher Ti volume percentages and compaction pressures resulted in higher compressive and flexural strengths. However, increasing the compaction pressure led to more crushed ceramic microspheres (CMs).

In this paper, the effects of the Ti volume percentage and the compaction pressure on the percentage of broken or crushed CMs and on the elastic modulus of the Ti matrix syntactic foam are studied in detail. A set of Ti matrix syntactic foams with different Ti volume percentages was fabricated with different compaction pressures. Two methods were used to estimate the percentages of crushed CMs in the Ti matrix syntactic foam samples. The elastic moduli of the samples were measured by compression tests.

See the sidebar for experimental procedures.

How would you…

…describe the overall significance of this paper?

This paper introduces a novel material we recently developed for biomedical applications. In order to meet the requirements of bone implant materials, the material needs to have a good structural integrity and possess a right balance between strength and stiffness. This paper addresses these critical issues by studying the integrity of the hollow ceramic particles and the elastic modulus.

…describe this work to a materials science and engineering professional with no experience in your technical specialty?

Ti matrix syntactic foams fabricated by powder metallurgy have an important advantage; the volume percentage of the Ti matrix can be varied in a wide range, which provides a means for regulating the elastic modulus. However, the compaction process involved in powder metallurgy can cause damages to the hollow ceramic particles. This paper studies the effects of Ti volume percentage and compaction pressure on the particle integrity and elastic modulus. The results show that a combination of high Ti volume percentage and low compaction pressure is beneficial.

…describe this work to a layperson?

A major problem for hip replacements is implant loosening, which is usually caused by the high stiffness of the implant materials. This paper studies a novel material designed for implant applications. The objective is to tailor the stiffness of the material by varying the proportions of the constituents and choosing the most appropriate fabrication conditions.

EXPERIMENTAL METHODS

Fabrication of Specimens

The raw materials used in fabricating the Ti matrix syntactic foam samples are a 99.4% pure Ti powder supplied by Active Metals Ltd, U.K., and a CM powder supplied by Envirospheres Pty Ltd, Australia. Figure A shows the morphology of the two powders. The Ti powder particles have an irregular shape and are smaller than 45 μm, with a nominal average particle size of 30 μm. The CMs have a nearly spherical shape, a composition of ~60% SiO_2, ~40% Al_2O_3 and 0.4–0.5% Fe_2O_3 by weight, and an apparent density of 0.8 g/cm^3. The CM powder used in this study has a particle size range of 125–250 μm, with approximately 65% porous CMs and 35% hollow CMs.[8] The CM particles have a compressive strength roughly of 45 MPa.[9]

A powder metallurgy process was used to manufacture the Ti matrix syntactic foam samples.[7] The Ti powder and CMs were first blended uniformly into a series of mixtures with Ti volume percentages of 40%, 50%, 60%, 70%, and 80%, excluding air. Each mixture was divided into five equal parts, which were compacted at pressures of 45, 70, 100, 150, and 200 MPa, respectively. The compacted samples were sintered at 1,200°C for 1.5 hours in a vacuum furnace with a vacuum of 1×10^{-4} mbar and then cooled to room temperature in the same furnace. The as-fabricated Ti matrix syntactic foam samples had a cylindrical shape with a diameter of 50 mm and a height of 10 mm.

Estimation of Percentage of Crushed CMs by Volume Measurement Method

The breakage of CMs during compaction would result in a decrease in the volume of Ti/CM compact. The percentage of the crushed CMs can be estimated from the difference in the actual volume of the sample, before sintering, and the ideal volume of the sample if no CMs had been crushed. The difference between the ideal volume and the actual volume is roughly equal to the volume of the air contained in the CMs that have been crushed during compaction. Therefore, the ratio of this decreased volume to the total volume of air contained in all the CMs before compaction is equal to the ratio of the crushed CMs to all the CMs.

The percentage of crushed CMs can be expressed as Equation 1, where V_I is the ideal volume of the compact, V is the actual volume of the compact, and V_A is the total volume of air, or pores, in the CMs. (All equations are shown in the table on page 39.)

The actual volume of the compact, V, was obtained by direct measurements using Archimedes' method. The total volume of pores in the CMs, V_A, was calculated by Equation 2, where W_{CM} is the weight of the CMs in the compact, ρ_{CM} (= 0.8 g/cm^3) is the density of the CMs, and ρ_{SC} (= 2.65 g/cm^3) is the density of the solid ceramic in the CMs.

The ideal volume of the Ti/CM compact, V_I, is the sum of the volume of the Ti matrix and the volume of the CMs without any breakage, and can be calculated by Equation 3, where V_{TM} represents the volume of the Ti matrix in the compact. V_{TM} was estimated by measuring the volume of a pure Ti powder with the same mass, compacted under the same pressure, as the Ti/CM compact. This estimation was based on the assumption that the compaction pressure distributed uniformly within the compact, i.e., the pressures applied onto the CMs and the Ti matrix were the same and equal to the compaction pressure. In other words, the deformation of the Ti particles in the Ti matrix was assumed to be the same as in the pure Ti powder during compaction, as the porosity levels in the Ti matrices in both cases are expected to be the same.

Estimation of Percentage of Crushed CMs by Water Absorption Method

Assuming that the pores in the Ti matrix and the voids in the crushed CMs in a Ti/CM compact are interconnected, and the uncrushed CMs are intact and cannot be infiltrated by water, the volume of the compact can be expressed as the sum of the volumes of the solid Ti, intact CMs, solid ceramic part of the crushed CMs, and interconnected porosity. The volume of the compact is shown in Equation 4.

Re-arranging Equation 4 gives the percentage of the crushed CMs as given in Equation 5, where W_{Ti} is the weight of Ti in the compact, ρ_{Ti} (= 4.5 g/cm^3) is the density of Ti, and V_P is the interconnected porosity in the compact.

To measure the interconnected porosity, V_P, the compact sample was first dehydrated in a furnace at 200°C for 3 hours to remove any moisture. The weight of the sample was measured immediately after being taken out of the furnace. The sample was then immersed into water for 5 minutes. The weight of the sample together with the absorbed water was measured. The difference between the weights of the sample after and before water absorption was the weight of the water in the interconnected pores. Because water can wet both Ti and the CMs, the interconnected pores in the sample can be filled effectively with water due to the capillary phenomenon. The volume of the absorbed water was therefore the volume of the interconnected pores in the sample.

Measurements of Elastic Modulus

The Ti matrix syntactic foam samples were first cut into cuboid specimens with a width of 10 mm, depth of 10 mm and height of 20 mm. The surfaces of the specimens were ground using 1,200 dpi grit sandpaper to remove any surface defects. Static compression tests were conducted on a universal testing machine (Instron 4505) with a cross-head speed of 0.5 mm/min. For each set of samples produced under the same condition, five specimens were prepared and tested.

During each compression test, a series of unloading and reloading routines were carried out at the strains of 0.4%, 0.6%, and 0.8% to determine the elastic modulus values. The gradients of the unloading curves of each sample were averaged to give the elastic modulus of the sample.

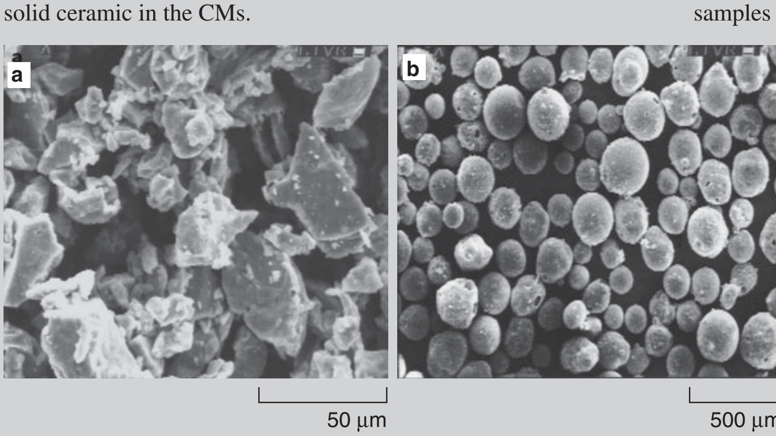

Figure A. SEM images showing the morphology of the (a) Ti powder and (b) CM powder used to fabricate the Ti matrix syntactic foams.

Equations

$$\phi = \frac{V_I - V}{V_A} \qquad (1)$$

$$V_A = \frac{W_{CM}}{\rho_{CM}} - \frac{W_{CM}}{\rho_{SC}} \qquad (2)$$

$$V_I = V_{TM} + \frac{W_{CM}}{\rho_{CM}} \qquad (3)$$

$$V = \frac{W_{Ti}}{\rho_{Ti}} + (1-\phi)\frac{W_{CM}}{\rho_{CM}} + \phi\frac{W_{CM}}{\rho_{SC}} + V_P \qquad (4)$$

$$\phi = \left(\frac{W_{Ti}}{\rho_{Ti}} + \frac{W_{CM}}{\rho_{CM}} + V_P - V\right) \Big/ \left(\frac{W_{CM}}{\rho_{CM}} - \frac{W_{CM}}{\rho_{SC}}\right) \qquad (5)$$

RESULTS AND DISCUSSION

Structure

Figure 1 shows the optical micrographs of the cross-sectional surfaces of the Ti matrix syntactic foam samples with five different nominal Ti volume percentages of 40%, 50%, 60%, 70%, and 80%, fabricated by four different compaction pressures of 45, 70, 100, and 150 MPa.

Figure 1 is divided into two regions by a solid line depending on the appearance of the Ti matrix. On the left of the line, the Ti matrix has a rough surface after the polishing, indicating a more porous structure. On the right of the line, the Ti matrix exhibits a flat, smooth surface after the polishing, indicating a more dense structure. It is shown that higher compaction pressures result in a more dense structure, because of decreased amounts of air voids in the Ti matrix. Increasing the Ti volume percentage in the syntactic foam also results in a more dense structure because of better bonding between the Ti particles. To achieve a dense Ti matrix, the samples with lower Ti volume percentages require higher compaction pressures, and the samples with higher Ti volume percentages require lower compaction pressures.

Figure 1 is also separated into two regions by a dash-dot line, depending on the shapes of the CMs in the Ti matrix. In the samples on the left of the line, most CMs remain spherical. In the samples on the right of the line, significant deformation of the CMs is observed. The CMs are clearly intact in the samples fabricated with a compaction pressure of 45 MPa. In the samples fabricated with higher compaction pressures, however, more CMs are crushed and lost from the cross-sectional surface, leaving holes in the matrix. This becomes more serious for the samples with lower Ti volume percentages.

Percentage of Crushed CMs

Figure 2 shows the relationship between the percentage of crushed CMs and the nominal Ti volume percentage at different compaction pressures. The results obtained by the volume measurement and water absorption methods show a similar trend. The percentage of crushed CMs decreases with increasing the Ti volume percentage and increases with increasing the compaction pressure. For the samples with a low Ti volume percentage, the CM particles are more easily to be crushed because more CMs are in direct contact with each other and the Ti matrix cannot offer sufficient protection to the CM particles during the compaction. For samples with a high Ti volume percentage, more CM particles in the sample can be protected by the flowing, rearranging and deforming the Ti particles. Thus the percentage of crushed CMs is decreased. However, this protection from the Ti particles is limited for samples fabricated with a compaction pressure over 150 MPa. Even if the Ti volume percentage is increased to 80%, the volume percentage of crushed CMs in the samples is still higher than 50%.

Figure 2 also compares the values of the percentage of crushed CMs obtained by the two methods. For the samples fabricated with a low compaction pressure, the values obtained by

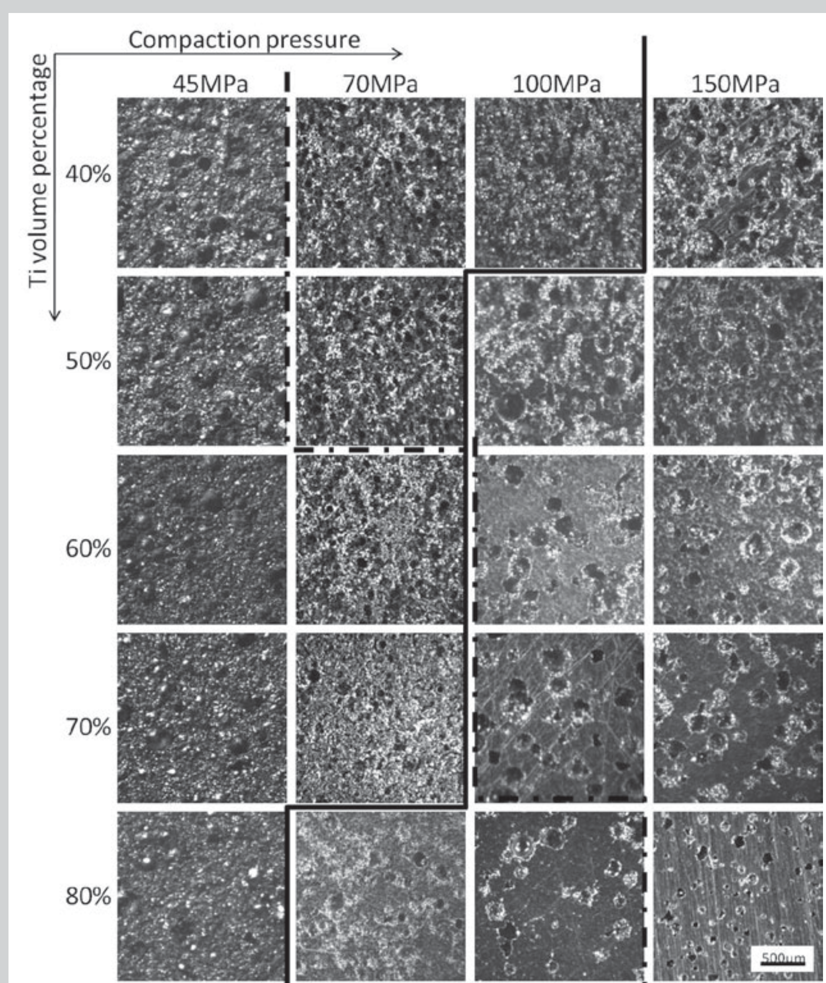

Figure 1. Optical images of the Ti matrix syntactic foam surfaces with a series of Ti volume percentage of 40%, 50%, 60%, 70%, and 80%, fabricated with compaction pressures of 45, 70, 100, and 150 MPa.

the water absorption method are higher than those obtained by the volume measurement method. For the samples fabricated with a high compaction pressure, however, the values obtained by the water absorption method are lower than those obtained by the volume measurement method. The difference is due to the different degrees of accuracy of the two methods at different compaction pressures.

The accuracy of the volume measurement method depends on the estimation of the volume of the Ti matrix in a Ti/CM compact. Optical micrographs of the Ti matrix in a syntactic foam and a sintered Ti specimen processed under the same compaction and sintering conditions showed that the porosity levels in the two cases are similar. Therefore, it is reasonable to estimate the volume of the Ti matrix in a Ti/CM compact from the volume of a pure Ti compact. However, the volume of the Ti matrix in a Ti/CM compact can be different from that of the Ti powder with the same mass, compacted under the same pressure, due to different stress conditions in these two situations. The CMs have a high Young's modulus than the Ti matrix and thus bear a higher load than the Ti matrix. Increasing the compaction pressure can significantly increase the densification of the compact, leading to a more uniform stress distribution in the Ti matrix. Therefore, the volume measurement method is likely to have a relatively high accuracy when the samples are fabricated with a high compaction pressure.

The accuracy of the water absorption method depends on the extent of the water infiltration within the sample. Although water wets the ceramic and Ti, the air in the voids can still prevent the water from full infiltration. The infiltration of the water is easier in a more porous Ti matrix than a dense one. The samples manufactured by low compaction pressures are more porous, and the CMs are less likely to fracture, which may result in water infiltration into the CMs even if they are not crushed. The water absorption method therefore has a higher accuracy for samples fabricated with a low compaction pressure.

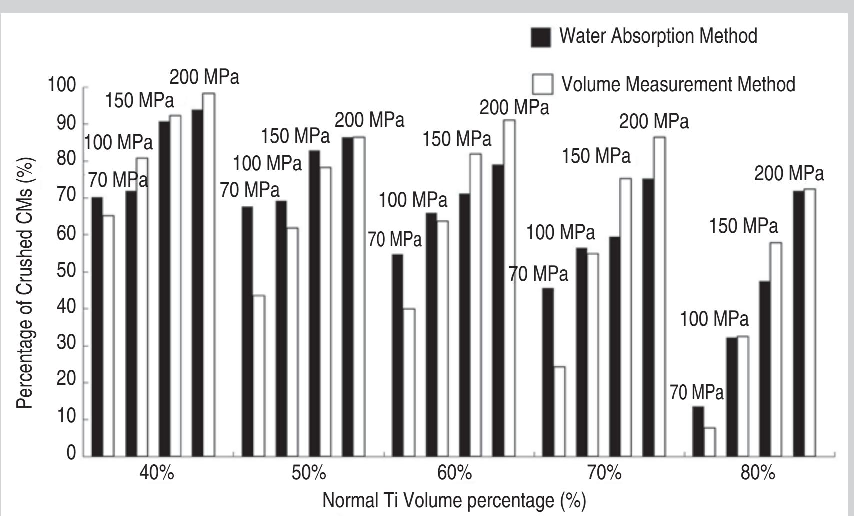

Figure 2. Relationship between the percentage of crushed CMs, estimated by the volume measurement and water absorption methods, and the nominal Ti volume percentage, at different compaction pressures.

Elastic Modulus

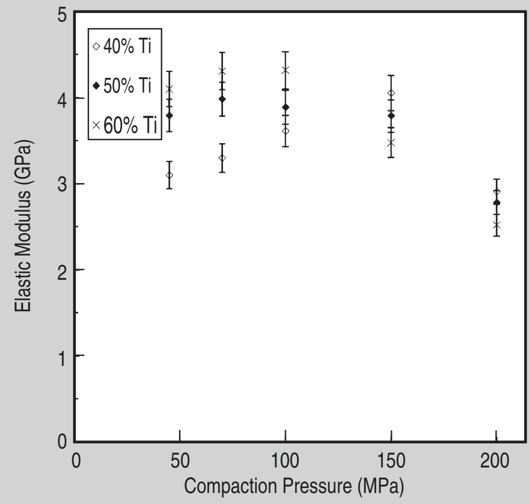

Figure 3. Elastic moduli of Ti matrix syntactic foams with different Ti volume percentages, fabricated at different compaction pressures.

Figure 3 shows the elastic moduli of the Ti matrix syntactic foams with different Ti volume percentages, fabricated at different compaction pressures. In the low compaction pressure range, increasing the Ti volume percentage can increase the elastic modulus of the Ti matrix syntactic foam. When the compaction pressure is over 100 MPa, however, increasing the Ti volume percentage decreases the elastic modulus of the Ti matrix syntactic foam.

The complex effects of the compaction pressure and the Ti volume percentage on the elastic modulus of the Ti matrix syntactic foam are a result of breakage of CMs. If all the CM particles had remained intact during the fabrication process, increasing the Ti volume percentage would have resulted in a decrease in the elastic modulus of the Ti matrix syntactic foam, because the CM particles have a higher elastic modulus than the Ti matrix. Increasing the compaction pressure can result in a denser Ti matrix; as a consequence, the elastic modulus of the Ti matrix syntactic foam would have been increased. In practice, the CM particles are quite brittle, and some CM particles can be crushed during the compaction process. These crushed CMs can significantly affect the elastic modulus of the Ti matrix syntactic foam, depending on the internal structure of the CM particles.

Increasing the compaction pressure has two different effects on the elastic modulus of the Ti matrix syntactic foam. On the one hand, a specimen fabricated with a higher compaction pressure has more crushed CM particles, leading to a lower elastic modulus. On the other hand, it is more dense, leading to a higher elastic modulus. At a relatively low compaction pressure, fewer CM particles are crushed. The as-produced syntactic foam has a high elastic modulus. When the compaction pressure reaches 100 MPa, the volume of the crushed CMs in the sample becomes very high. The decrease in the elastic modulus due to the crushed CMs becomes greater than the increase due to the densification effect. Overall, the elastic modulus of the sample is decreased.

The effect of the Ti volume percentage has a similar trend. Increasing the Ti volume percentage decreases the volume of crushed CM particles, and therefore increases the elastic modulus of the Ti matrix syntactic foam. However, because the elastic modulus of the Ti matrix is lower than that of the CM particles, increasing the Ti volume percentage can also decrease the elastic modulus. The overall effect depends on the compaction pressure. At a relatively low compaction pressure, the amount of crushed CMs has the dominant effect; increasing the Ti volume percentage increases the elastic modulus. At a high compaction pressure, the amount of crushed CM particles becomes very large and the effect of the Ti matrix becomes dominant; as a result, increasing the Ti volume percentage decreases the elastic modulus.

CONCLUSIONS

For a given Ti volume percentage, the percentage of crushed CMs increases with increasing the compaction pressure; for a given compaction pressure, the percentage of crushed CMs decreases with increasing Ti volume percentage. The elastic modulus of the Ti matrix syntactic foam was measured by compression tests. At a compaction pressure lower than 100 MPa, the elastic modulus increases with increasing Ti volume percentage and compaction pressure; at a compaction pressure above 100 MPa, further increases in Ti volume percentage and compaction pressure decrease the elastic modulus.

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