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Microstructures and Mechanical Properties of Porous Titanium Compacts Prepared by Powder Sintering *1

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Using pure Ti powder with particle sizes from 300 to 500 μm prepared by the plasma rotating electrode process (PREP), porous pure Ti compacts for biomedical applications were synthesized by powder sintering, and microstructures and mechanical properties of the compacts were investigated in this study. Porous compacts having porosity of 19–35 vol% are successfully fabricated by controlling sintering condition. It is found that Young's modulus and compressive yield strength decrease linearly with increasing porosity, and porous Ti compacts having porosity of about 30–35 vol% exhibit identical Young's modulus of human bone.

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1. Introduction

Metals and alloys have been widely used as implants for artificial hard-tissue replacement. Among them, pure Ti is often employed in place of human bone because of excellent biocompatibility, resistance of corrosion reaction and light weight. However, critical problems caused by the mismatch of Young's modulus between implant (110 GPa for Ti) and bone (12–23 GPa) are still unsolved.[1)] One way to alleviate the problems is, therefore, to reduce Young's modulus of pure Ti by introducing pores, thereby minimizing damages to tissues adjacent to the implant and eventually prolonging device life time.[2)] In addition, low Young's modulus of porous Ti compacts encourages regular bone cell growth, while its porosity enables bone cell in-growth.[3)]

To fabricate sintered porous Ti compacts having properties similar to those of human bone, the microstructures and mechanical properties such as Young's modulus, yield strength, bend strength of the sintered porous Ti compacts are investigated at room temperature.

2. Experiment Procedure

Pure Ti particles were produced by the plasma rotating electrode process (PREP) under the operating conditions of plasma arc current of 80 A and anode rotating speed of $150\,s^{-1}$ in an Ar atmosphere. They were filled between two graphite punches in a graphite mold. Ti thin foils were used so that the powder was not brought into direct contact with the graphite punches and mold. To attain uniform compaction of the powder in the mold, ultrasonic vibration for 43.2 ks followed by pre-pressing at 70 MPa for 0.6 ks was applied before sintering. Ti powder was sintered by holding for 7.2 ks with heating/cooling rate of 0.17 K/s in a vacuum of 1×10^{-3} Pa without applied pressure at temperatures 1173, 1373 and 1573 K, which are above the β-phase transition temperature (1156 K). Also, Ti powder was sintered under applied uniaxial punch pressures of 5 and 10 MPa at 1173 K for 7.2 ks and of 7 kPa at 1373 K for 7.2 ks. Microstructures of sintered porous Ti compacts were observed with an optical microscope (OM) and a scanning electron microscope (SEM). The porosity of the porous compacts was measured by the Archimedes' technique after coating the specimen surfaces with paraffin to prevent penetration of water into the specimen. The open porosity was evaluated by measuring the weight and volume of paraffin penetrating into the porous compacts when they were boiled in paraffin. Compression tests were carried out to determine Young's modulus, 0.2% offset yield stress and bend strength at a cross-head speed 0.05 mm/min at room temperature. A size of test specimens for Young's modulus and 0.2% offset yield strength was about $9 \times 9 \times 25\,mm^3$, and specimen size for bend test was about $6 \times 6 \times 28\,mm^3$. The surfaces of these test specimens were mechanically polished with SiC paper #4000. In order to determine Young's modulus, electrical resistance strain gages (base width: 5.2 mm/base length: 16 mm) were mounted on two lateral surfaces to measure longitudinal elastic strain in compression. The test was performed in an elastic range up to a maximum load of 1000 N.

3. Results and Discussion

Pure Ti powder particles produced by PREP were entirely spherical and their sizes were distributed in the range of 280 to 880 μm with a mean diameter of about 419 μm. The obtained powder was sieved to use similar sizes of particles in the range of 300 to 500 μm with a mean diameter of 374 μm, as shown in Fig. 1(a). Figure 1(b) shows a SEM micrograph of spherical particles obtained after sieving.

Figure 2 shows OM micrographs of polished cross-sections of the compacts sintered at 1173 and 1573 K without pressure and at 1173 K under 10 MPa (abbreviated hereafter as 1173 K/10 MPa). Since the number of interparticle contacts in the compacts sintered at 1173 K is quite less than that of the other compacts in this section, as shown in Fig. 2(a), pores seem to be interconnected three-dimensionally. On the other hand, many necks, which are produced by contact with adja-

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cent particles, are observed in the compact sintered at 1573 K (Fig. 2(b)). At 1173 K/10 MPa, neck growth assisted by pressure is clearly seen in spite of low sintering temperature (Fig. 2(c)).

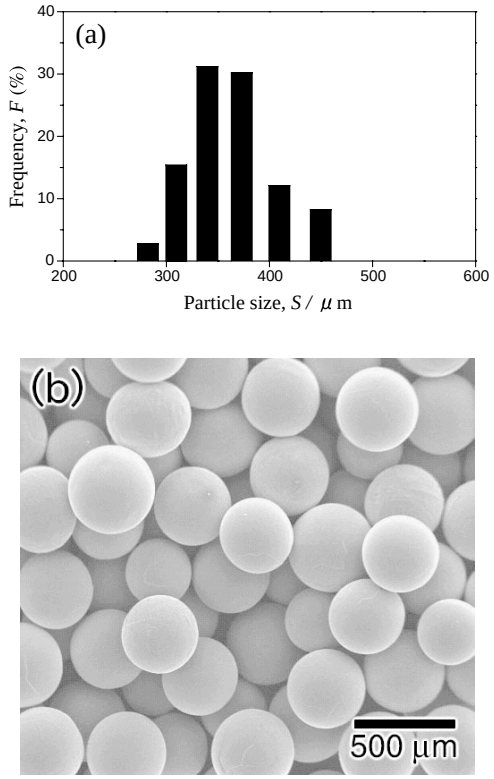


Fig. 1 (a) Size distribution of PREP Ti powder after sieving (vertical axis shows the frequency in volume) and (b) SEM micrograph of sieved PREP Ti powder.

Figure 3 shows SEM micrographs of fracture surface of the specimens sintered at 1173, 1573 K and 1173 K/10 MPa, where these specimens were fractured by bending to observe the fracture surface as a function of sintering condition. At 1173 K, no apparent traces indicating particle deformation are observed on the surface. At 1573 K, traces corresponding to decohesion of interparticle contacts and contact zones are locally observed. In contrast, at 1173 K/10 MPa, the number and area of contacts per particle are found to increase noticeably, as compared with the compacts sintered at 1173 and 1573 K without pressure. It is evident from Figs. 2 and 3 that the behavior of densification, or the porosity of the sintered compacts, strongly depends on sintering condition.

Table 1 shows the volume percent of porosity of the sintered compacts, where the total porosity is equal to the additive of the open and closed porosity. At 1173, 1373 and 1573 K without pressure, the total porosity of porous compacts is larger than 30 vol%, and open porosity is almost 100%, suggesting that most pores are interconnected through channels. It should be noted that the present compacts with the porosity greater than 30 vol% are promising for biomaterial applications, since ideal porosity of implant materials is in the range of 30–90 vol%.[4] Young's modulus values of porous compacts and non-porous bulk Ti are plotted against porosity in Fig. 4, where 3 measurements for each sample are averaged. Unfortunately, mechanical properties could not be measured for porous samples sintered at 1173 K without pressure because of particle chipping during polishing and cutting. As expected, the experimental results reveal that Young's modulus of porous specimens decreases with increasing porosity. By using the method of least squares a straight line is obtained

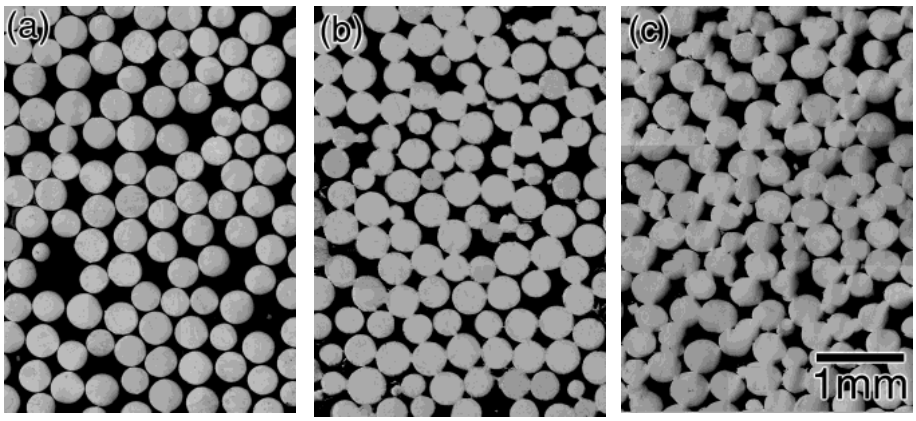


Fig. 2 OM micrographs of polished cross-section of porous Ti compacts sintered at (a) 1173 K, (b) 1573 K and (c) 1173 K/10 MPa.

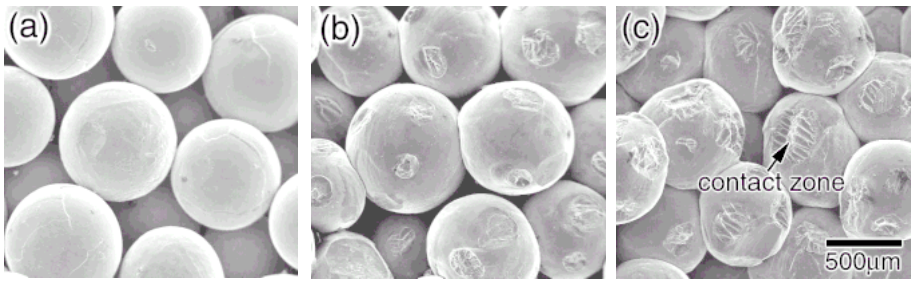


Fig. 3 SEM micrographs of fracture surface of porous Ti compacts sintered at (a) 1173 K, (b) 1573 K and (c) 1173 K/10 MPa.

Table 1 The total porosity, open porosity and closed porosity of porous Ti compacts sintered at 1173 K, 1373 K, 1573 K, 1173 K/5 MPa, 1173 K/10 MPa and 1373 K/7 kPa.

	Sintering conditions					
	1173 K	1373 K	1573 K	1173 K/5 MPa	1173 K/10 MPa	1373 K/7 kPa
Total porosity (%)	35.1	33.4	31.7	19.1	19.1	26.3
Open porosity (%)	100	100	99.8	94.5	94.3	95.3
Closed porosity (%)	0	0	0.2	5.5	5.7	4.7

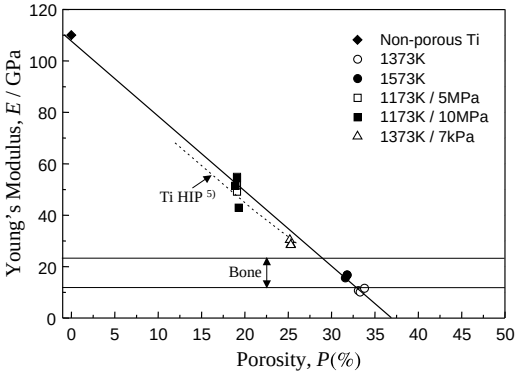


Fig. 4 Young's modulus vs. porosity of porous Ti compacts sintered at 1373 K, 1573 K, 1173 K/5 MPa, 1173 K/10 MPa and 1373 K/7 kPa.

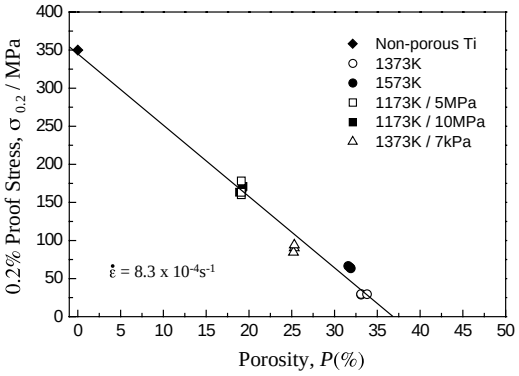
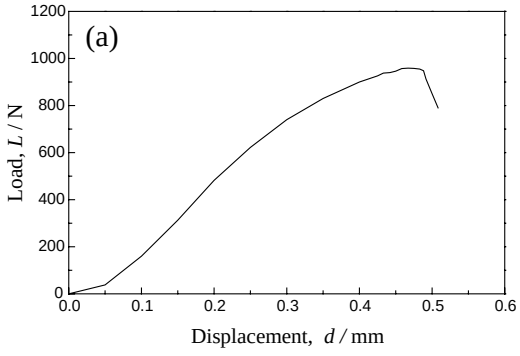


Fig. 5 0.2% offset yield strength vs. porosity of porous Ti compacts sintered at 1373 K, 1573 K, 1173 K/5 MPa, 1173 K/10 MPa and 1373 K/7 kPa.



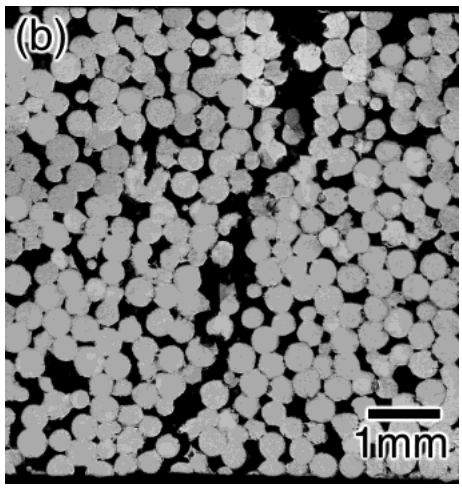


Fig. 6 (a) Displacement-load curve and (b) OM micrograph after bend test of porous Ti compact sintered at 1373 K/7 kPa.

and the extrapolation of Young's modulus to zero results in porosity of about 37 vol%. As can be seen in Fig. 4, data by Ledbetter[5] are in fairly good agreement with the present result, although they have measured Young's modulus in a narrow range indicated. It is very interesting to note that Young's modulus of the present compacts with the high porosity levels from 30 to 35 vol% is almost the same as that of human bone. 0.2% offset yield stress of the porous compacts is plotted against porosity in Fig. 5. Although the compacts were strained in compression up to about 5.1% plastic strain, no detectable cracking or buckling was caused. Yield stress linearly decreases with increasing porosity, and the line extrapolated by least squares fitting intersects abscissa at the porosity of about 37 vol%. These results shown in Figs. 4 and 5 suggest that porous compacts having porosity greater than about 37 vol% cannot be fabricated when the sizes of initial powder particles are in the range of 300 to 500 μm.

Figure 6(a) shows a typical displacement-load curve obtained by three-point bending test for the compact with porosity of 26.3 vol% which was sintered at 1373 K/7 kPa. The curve shows linear elastic deformation followed by plastic yielding and strain hardening up to a peak load. Although a load drop was observed after showing the maximum load, catastrophic fracture was not caused. Therefore, plastic deformation of the porous compacts is achieved in contact regions. The measured bend strength is 150.2 MPa. This value is very close to the bend strength of human bone (156.9 MPa).[6] OM micrograph after the bend test is shown in Fig. 6(b) indicating the crack propagation at the surface on which maximum tensile stress is applied in bending. A main crack was observed to initiate by stretch and fracture of interparticle contacts.

It is most likely that the number and size of interparticle contacts and contact regions, which are controlling factors of mechanical properties, are strongly affected by initial particle size as well as sintering condition. In addition, these factors controlling pore size or surface roughness are associated with

biocompatibility.[7] Further studies are needed to clarify the effect of initial powder particle size on mechanical properties and biocompatibility for biomaterial applications.

4. Conclusions

Microstructures and mechanical properties of porous Ti compacts prepared by sintering PREP powder with sizes of 300–500 μm are investigated in this study. The results obtained are summarized as follows:

(1) Sintered Ti compacts with porosity of 19 to 35 vol% are fabricated by controlling sintering condition such as temperature and pressure.

(2) Number and size of interparticle contacts and contact areas depend on sintering condition.

(3) Young's modulus and compressive yield strength of porous Ti compacts decrease with increasing porosity.

(4) Young's modulus of porous Ti compacts with porosity of 31.6 vol% and 33.4 vol% is similar to that of human bone.

(5) Bend strength of porous Ti compact with porosity of 26.3 vol% is similar to that of human bone.

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