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Research Article

Porous Ti6Al4V Scaffold Directly Fabricated by Sintering: Preparation and *In Vivo* Experiment

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The interface between the implant and host bone plays a key role in maintaining primary and long-term stability of the implants. Surface modification of implant can enhance bone ingrowth and increase bone formation to create firm osseointegration between the implant and host bone and reduce the risk of implant losing. This paper mainly focuses on the fabricating of 3-dimensiona interconnected porous titanium by sintering of Ti6Al4V powders, which could be processed to the surface of the implant shaft and was integrated with bone morphogenetic proteins (BMPs). The structure and mechanical property of porous Ti6Al4V was observed and tested. Implant shaft with surface of porous titanium was implanted into the femoral medullary cavity of dog after combining with BMPs. The results showed that the structure and elastic modulus of 3D interconnected porous titanium was similar to cancellous bone; porous titanium combined with BMP was found to have large amount of fibrous tissue with fibroblastic cells; bone formation was significantly greater in 6 weeks postoperatively than in 3 weeks after operation. Porous titanium fabricated by powders sintering and combined with BMPs could induce tissue formation and increase bone formation to create firm osseointegration between the implant and host bone.

1. Introduction

The hip joint is a spherical joint between the femoral head and the acetabulum in the pelvis. It is a diarthrosis or synovial joint wrapped in a capsule that contains the synovial fluid (SF). The hip joint can transmit high dynamic loads and accommodate a wide range of movements because of the presence of the SF and the ball-in-socket geometry. Though its remarkable characteristics, the hip joint can be affected, more often in aged people, by chronic pain and diseases such as osteoarthritis, rheumatoid arthritis, bone tumors, or traumas. In these cases, the best clinical solution is the total hip replacement, a surgical procedure that replaces the unhealthy hip joint with an implant, preserving the synovial capsule [1–3].

Metallic materials are widely used for joint replacement and orthopedic and dental implants. Metals are more suitable for loading-bearing applications when compared with ceramics or polymeric materials because of their excellent mechanical property. Among various metallic biomaterials, titanium and its alloys are the most attractive metallic biomaterials for orthopedic and dental implants due to their excellent mechanical properties, biocompatibility, processability, and good corrosion resistance in recent years. However, a major problem about metallic implants in orthopedic surgery is the mismatch of Young's modulus between natural bone and bulk metallic biomaterials. Due to this mechanical mismatch, bone is insufficiently loaded and causes a problem of stress shielding. Stress shielding can lead to eventual loosening of the implant. In order to overcome this drawback, porous materials are used, which are increasingly attracting the interest of researchers as a method to reduce mechanical mismatches and achieve stable long-term fixation by means of full bone ingrowth [4–10]. A porous implant material with adequate pore structure and appropriate mechanical properties has been sought as the ideal implants for bone substitute.

Porous Ti6Al4V usually has a similar Young's modulus with nature, which is a more suitable material used as hip prosthesis implants. There have been various processes for

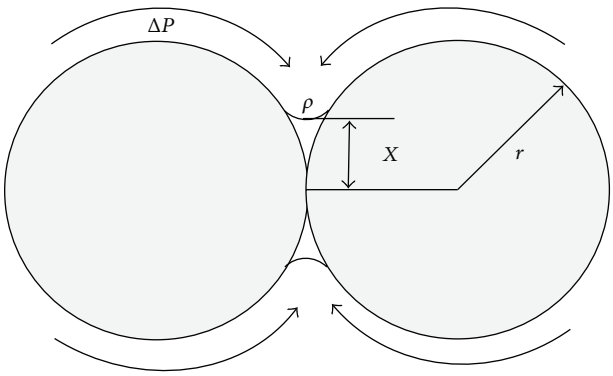


Figure 1: Solid phase sintering principle.

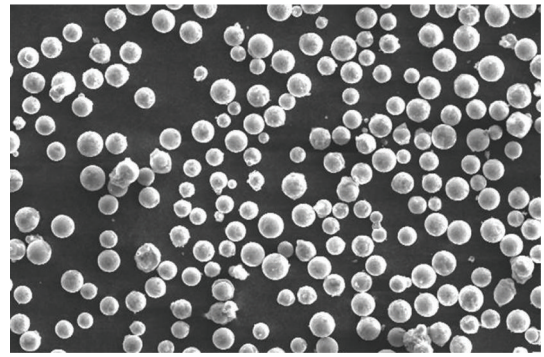


Figure 2: Morphology of Ti6Al4V powder when magnified 500 times using SEM.

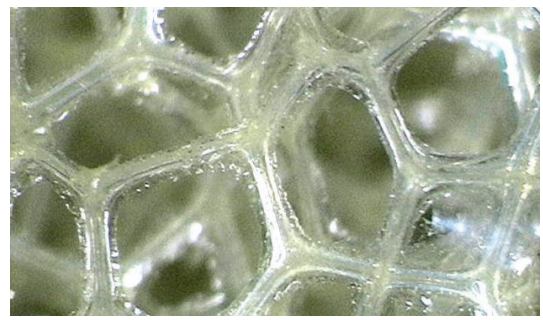


Figure 3: The photomacrograph of polyurethane sponge when magnified 50 times using optical microscope.

making open-cell titanium and its alloy foams, such as space holder method, combustion synthesis, plasma spraying, polymeric sponge replication, and powder sintering approach. In this study, Ti6Al4V implant with interconnected porous structure was fabricated by sintering. Regarding the procedures used to develope porous titanium structures, solid-phase sintering techniques have been proven to be more suitable than liquid-phase foaming. This is mainly due to the high melting point of titanium and its reactivity at high temperatures [7, 11]. The porous samples used in this work were therefore developed by powders sintering.

In this study, a method to fabricate porous Ti6Al4V is reported, by which the porosity and the pore structure of porous Ti6Al4V alloy that is similar to the trabecular bone structure are achieved. Also, the microstructure and mechanical property of porous Ti6Al4V are studied. The porous Ti6Al4V is processed to the surface of the implant shaft and was integrated with BMPs. An animal experiment is done in order to study the bone formation on the surface of the implant.

2. Materials and Methods

2.1. Materials. Ti6Al4V powders used in this study are provided by Northwest Institute for Nonferrous Metal Research, Xi'an. The Ti6Al4V particles are spherical in shape, which can pass through 300-mesh sieve. Silica sol (30 wt% SiO_2, <0.5 wt% Na_2O) was used as a binder. Polyurethane sponge was used as a carrier. BMPs are bought from China-Gen, Inc., Tianjin. The Ti6Al4V was fabricated by powders sintering in the lab. The artificial femoral handles were provided by Baimujinghang Company.

2.2. Fabrication of Scaffold. The Ti6Al4V power was mixed with a certain amount of silica sol. The slurry was stirred at room temperature. The Ti6Al4V foam is made of polyurethane sponge and Ti6Al4V slurry. A 50 mm × 20 mm × 10 mm volume of polyurethane (PU) sponge block was utilized in this study. The block was soaked in the Ti6Al4V slurry and then dried in the air for 24 hours. The dried porous Ti6Al4V form is first heated in vacuum furnace and then sintered at 1400°C for 2 hours to make the sponge disappear to fabricate the porous Ti alloy that is similar to the trabecular bone both in structure and elasticity modulus.

Figure 1 shows us the solid phase sintering principle. Ti-6Al-4V powders have small particle size, large specific surface area, and high surface energy. According to the minimum energy principle, the sintering process is a spontaneous irreversible process. According to the equal diameter sphere model proposed by G. C. Kuczynski, there are a positive pressure in the convex and a negative pressure in concave, so materials migrate from convex to concave spontaneously. Densification was achieved by mass transfer.

2.3. Microstructure and Mechanical Property Test. The microstructure of porous Ti6Al4V was observed by SEM (XL30 S-FEG), the mechanical property was tested by compression test using Mini Bionix (MTS 858), and the porosity was tested by mercury porosimeter.

The porous Ti6Al4V was cut into a block with a size of 15 mm × 15 mm × 10 mm for SEM test. Five samples were used for testing the porosity of porous Ti6Al4V by mercury porosimeter. Five samples were used for testing the mechanical property by compression test.

2.4. Animal Experiment In Vivo. The BMPs directly composite with porous titanium of the implant surface through chemical infiltration method. The porous Ti6Al4V was immersed in the rhBMP-2 solution (rhBMP-2 in PBS) with

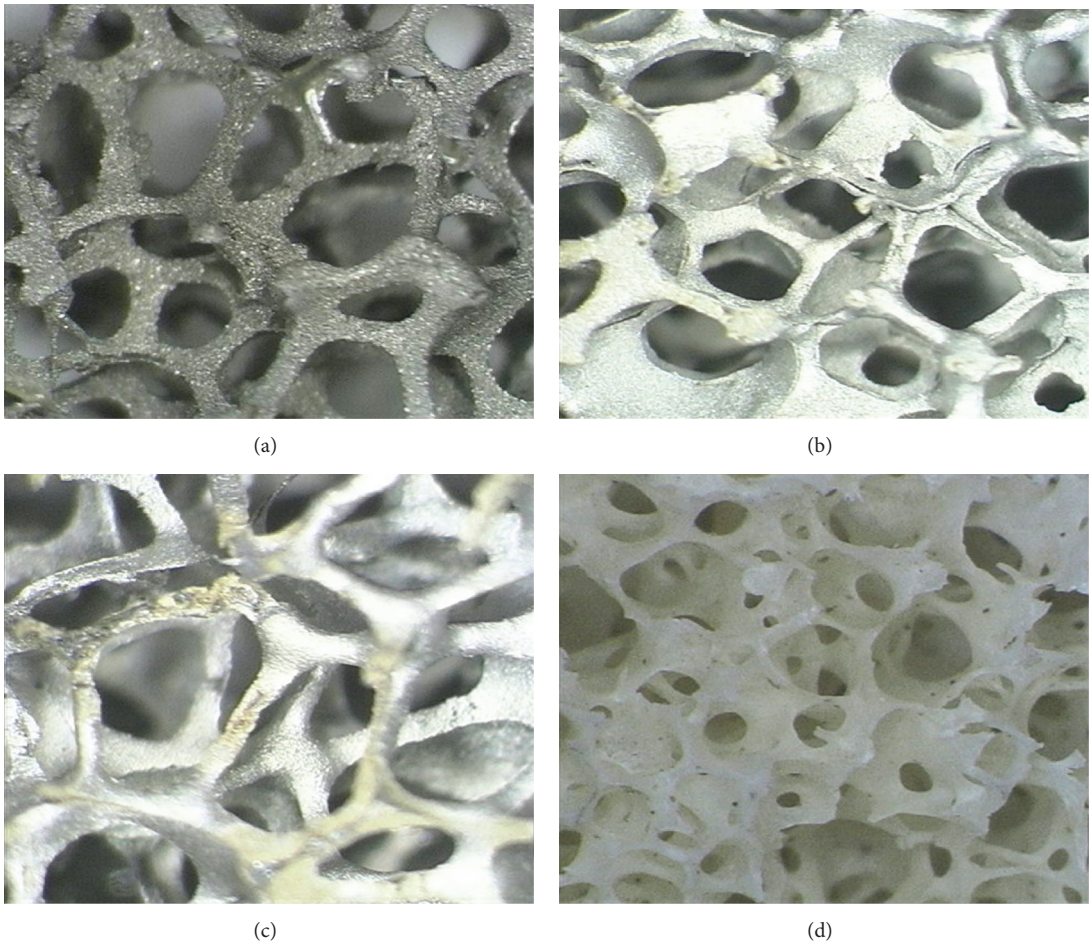


Figure 4: The macrostructure of the porous Ti-6Al-4V alloy ((a), (b), and (c)) and the trabecular bone structure of femoral head (d).

Table 1: Chemical composition of Ti6Al4V powder.

O max	H max	N max	C max	Si max	Fe max	Al max	V max	Ti
0.13	0.012	0.03	0.08	0.04	0.25	5.5–6.5	3.5–4.5	Bal

concentration of 1000 ng/mL for 24 hours and then dried in the air for 4 days at the temperature of 4°C. Prune the three-dimensional interconnected porous titanium into the size of the slot, and weld it on the slot of the dog femoral prosthesis handle with a laser welding. Implant the dog femoral prosthesis handle twice.

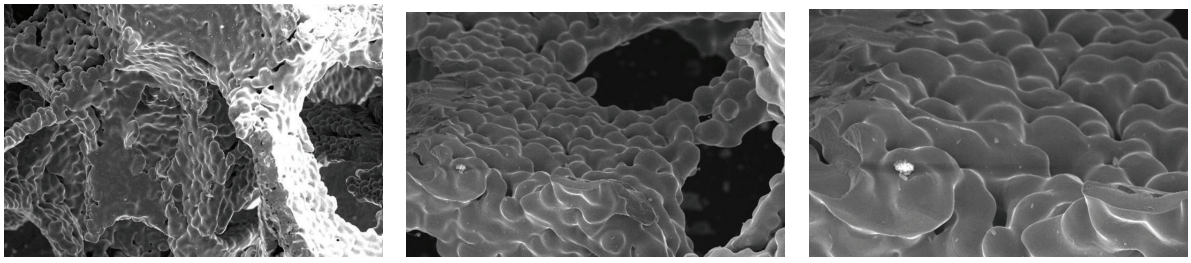
Inject tetracycline fluorescent tags in the vein in the first 3 days of a week before taking the specimens. Cut the upper femur with a saw, and observe the combing of prosthesis and femur combining with the naked eye. Cut the specimens into small blocks with the size of $1 \times 1 \times 0.5$ mm and dehydrate, embed, section, stain, then observe them under microscope.

3. Results and Discussions

3.1. Characteristics of Materials. Figure 2 illustrates an image of Ti6Al4V powder under SEM. It shows that the particles have both a highly spherical shape and a smooth surface. The size of Ti6Al4V powder in this paper is in the range of 5–30 μm. Table 1 shows the chemical composition of the Ti6Al4V powder. It shows that the aluminum, vanadium; and titanium are in the main proportion.

Figure 3 is the photomacrograph of polyurethane sponge used in this article. The PU sponge has a porosity of 78–80%, and macropores are in the range of 600–1200 μm.

3.2. Characteristics of Scaffold. The macrostructure of the porous Ti-6Al-4V alloy obtained by sintering of Ti-6Al-4V powder presents a fully interconnected pore network (Figures 4(a), 4(b), and 4(c)), which is similar to the trabecular bone structure of femoral head (Figure 4(d)). The microstructure of the porous Ti-6Al-4V alloy is shown in Figure 5. It shows that particle bonding is achieved by neck growth through



(a) (b) (c)

Figure 5: The microstructure of the porous Ti-6Al-4V alloy when magnified with different times using SEM ((a) 200 times, (b) 500 times, and (c) 1000 times).

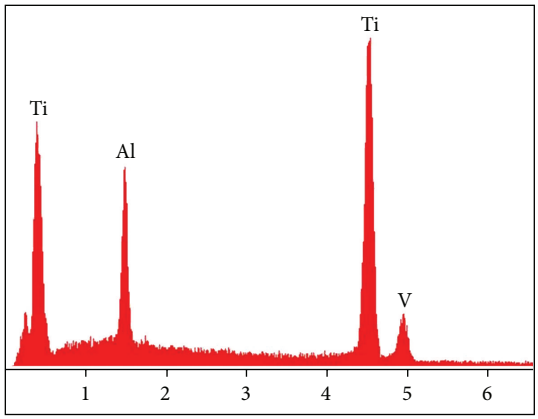


Figure 6: The energy spectrum analysis of the porous Ti-6Al-4V alloy.

a solid-state diffusion process. The formation of necks between particles is evidence of good sintering conditions. It can be seen that both macro- and micropores exist in the porous Ti-6Al-4V alloy. SEM shows that the micropore size of porous Ti6Al4V alloy ranges from 300 μm to 600 μm.

The energy spectrum analysis of the porous Ti-6Al-4V alloy is done using SEM, and the result is given in Figure 6. It shows that the absorption peak of titanium element, aluminium element, and vanadium element can be observed, and the intensity of the absorption peak ratio is consistent with that of Ti-6Al-4V alloy.

The elasticity modulus of the porous Ti-6Al-4V alloy was tested by MTS 858 Mini Bionix (Figure 7). The elasticity modulus of Ti-6Al-4V alloy is 0.6~0.7 GPa and similar to that of trabecular bone structure, whose elasticity modulus is 0.1 GPa~2 GPa.

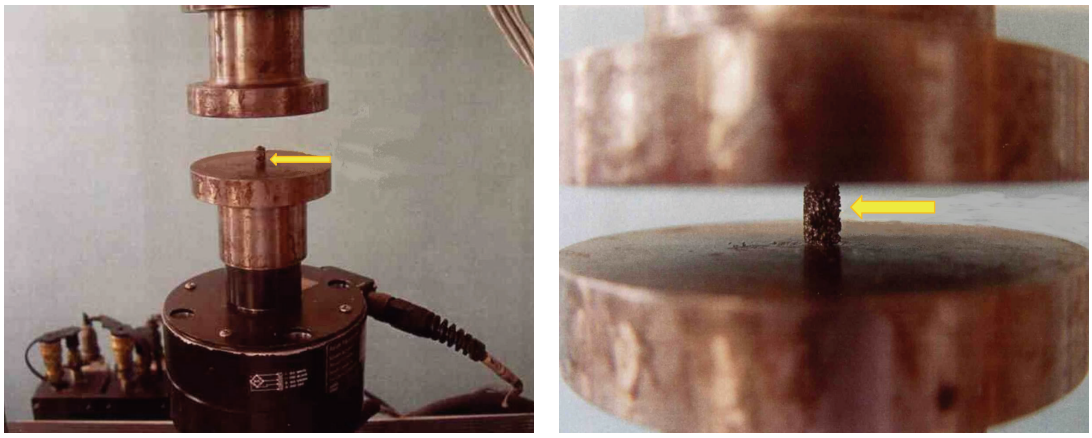
The porosity of the porous Ti-6Al-4V was analyzed by mercury porosimeter. The porosity is in the range of 50%–60%, and the average pore diameter is 300~600 mm.

Porous titanium presents diminished mechanical properties, and porous titanium implant helps reduce the stiffness mismatch between implant and bone tissue for a similar elasticity modulus with trabecular bone structure, thus reducing "stress shielding" and achieving stable long-term fixation. A porous implant material with adequate pore structure and appropriate mechanical properties has been sought as the ideal bone substitute.

The processing of sintering can be improved by changing the sintering temperature, the type of binder, the size of PU sponge, and so on in order to fabricate a kind of porous Ti alloy with uniform interconnected porous and less closed pore.

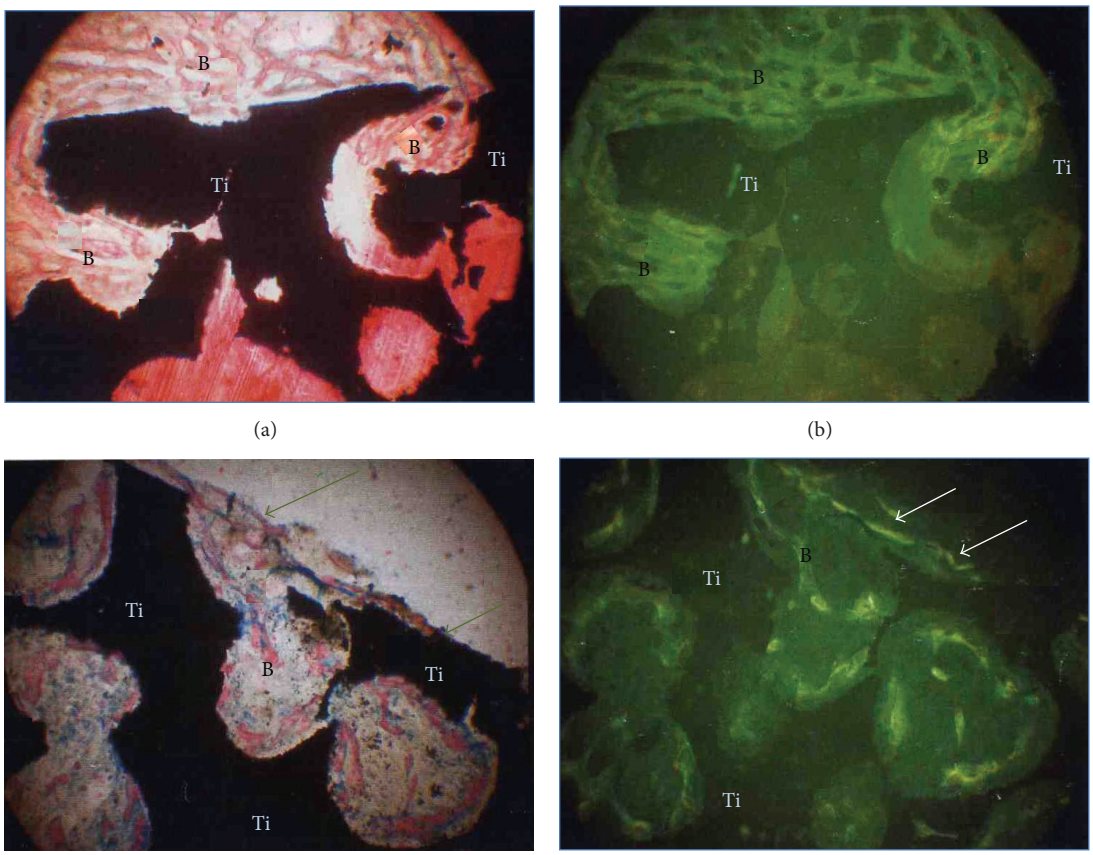
3.3. In Vivo Bone Repair. BMPs are a group of growth factors also known as cytokines and as metabologens. Originally discovered by their ability to induce the formation of bone and cartilage, BMPs are now considered to constitute a group of pivotal morphogenetic signals, orchestrating tissue architecture throughout the body. Because of the interconnected pore structure and larger porous, the bone can grow into the porous materials easily. By combining with BMPs, the porous materials can speed up the early bone ingrowths and improve the amount of bone formation, making the bond between the prosthesis and the bone tighter. The purpose of this experiment is to make the artificial femoral handle surface patch a three-dimensional interconnected porous titanium which composites BMPs and to observe bone ingrowth *in vivo*.

The loosening of prosthesis and host bone is a fatal weakness about artificial joint replacement, which is the result of the failure fusing between prosthesis and host bone. In order to promote bone ingrowth in prosthesis, the researchers use a number of methods of modifying the surface of the prosthesis to achieve osseointegration [11–16]. In this article, porous Ti6Al4V alloy fabricated by powders sintering is used. The results are shown in Figure 8. Porous titanium combined with BMPs was found to have large amount of fibrous tissue with fibroblastic cells. Three weeks later, bone ingrowth was found reaching half of the integrated material layer, and the bone tissue is relatively dense under light microscope with 40 times enlargement factor (Figure 8(a)). There are more active bone tissues in the porous of the Ti alloy under the fluorescence microscope (Figure 8(b)). Six weeks later, bone ingrowth was found reaching the full integrated material layer, and the new bone tissues grow along the hole wall under light microscope with 40 times enlargement factor (Figure 8(c)). The activity of the new bone tissues is confirmed under the fluorescence microscope



(a) (b)

Figure 7: The mechanical property test using MTS 858 Mini Bionix.

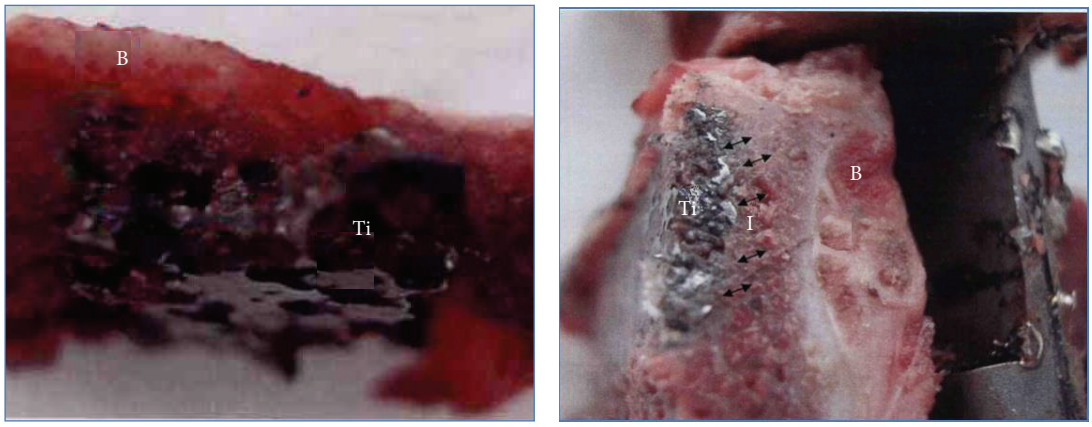


(a) (b)

(c) (d)

Figure 8: Image of bone tissue specimens gotten from BMPs and the porous titanium composite prosthesis, 3 weeks ((a), (b)) and 6 weeks ((c), (d)) under microscope with a 40 times enlargement factor (the right one is fluorescence-labeled, B is the bone, and the arrow is the interface between porous titanium and the prosthesis).

(Figure 8(d)). Ti porous materials achieve part osseointegration after three weeks (Figure 9(a)) and complete osseointegration after six weeks (Figure 9(b)). Bone formation was significantly greater in 6 weeks postoperatively than in 3 weeks. After combining with BMP, implant shaft with surface modification of porous titanium had bone formation in the interface that was found to increase with time. Six weeks after operation, bone ingrowth had reached the whole integrated



(a) (b)

Figure 9: Image of osseointegration. The osseointegration of BMP + porous titanium after 3 weeks (a) and 6 weeks (b) since the operation (the arrows mean the osseointegration of BMPs and bone; B is bone; Ti is titanium; I is the interface).

material layer. Two fixation mechanisms of mechanical interlock and chemical bond in the implant-bone interface may meet the demand of early fixation.

4. Conclusion

We can get the conclusion as follows.

(1) 3D interconnected porous titanium had a similar structure with cancellous bone, and its elastic modulus was similar to cancellous bone.

(2) After combining with BMP, implant shaft with surface modification of porous titanium had bone formation in the interface that was found to increase with time. Six weeks after operation, bone ingrowth had reached the whole integrated material layer.

(3) Porous titanium fabricated by powders sintering and combined with BMP could induce tissue formation and increase bone formation to create firm osseointegration between implant and host bone.

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