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Effect of SBF on Cyclic Compression Behaviour of Porous Titanium Component for Implant Application

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Abstract: In the recent years, porous structure is being drawn attention to the researcher for implant application for superior characteristics over bulk materials. The aim of this study is to evaluate the cyclic compression behaviour of porous titanium components in simulated body fluid (SBF). Porous titanium component developed by replica impregnation method was taken for study. Compression tests in air revealed that the yield strength of the porous body is 8MPa on average and elastic modulus is around 180MPa which is compatible to cancellous bone application. After 10% strain porous structure deformed plastically producing a long plateau region. Compressive fatigue tests revealed that at higher stress level porous titanium failed earlier in SBF than in air. In contrast, fatigue limit of porous substrate is 2 MPa which was not affected by SBF medium. After 10 million cycles in SBF, Calcium Phosphate layer was partially formed on the surface of porous titanium by re-precipitation from SBF. EDS analysis showed that the Ca/P atomic ratio was 1.44 which is near to beta TCP and HA phase and these phases are beneficial for bone tissue ingrowth.

Keywords: SBF, Cyclic Compression Behaviour, Porous Titanium, Implant Application, Corrosion Resistance, EDS Analysis, Osteoconductivity, and Bioactive Coating

1. Introduction

Titanium and its alloy are being widely used for prosthetic implant material due its superior mechanical properties and corrosion resistance. Though their elastic modulus (110GPa) is lower than other metallic material but still much higher than human bone which generates stress shielding effect during gait which cause pain to the patients [1]. The stress shielding effect is responsible for implant loosening and revision surgery and also causes bone resorption. So porous structures of metallic material are being developed by different method such as replica impregnation, additive manufacturing, 3D printing, selective laser melting etc. [2]. The porous structures can be either open cell or close cell. The open cell porous structure not only helps for bone ingrowth but also provides space for fluid transformation which provides long term stability. Some researchers reported that the pore size ranging from 100 to 400 mm was effective for bone formation whereas pore size below this range limits the ingrowth of bone tissue, while pore size much higher than this range make no significant difference or even increase the possibility of fibrous tissue formation [3-6]. SEM and EDX analysis revealed cell morphology were identical for HAp and HAp/amino acid ligands complex coating [7]. Such characteristics are desirable for the success of implant biomaterial coating that can preserve both antibacterial property and cell adhesion behavior. Significant research works have been done to manufacture porous scaffold by different method and their mechanical behaviour as well as compression fatigue behaviour was evaluated for long term durability [8-13]. These studies were confined to air environment only and effect of SBF was studied only by static immersion without applying any load. However, very few literatures were found to evaluate the effect of body fluid on cyclic compression behaviour of developed implant material which may reduce the fatigue limit. Thus, our study focused on particularly the effect of simulated body fluid on cyclic compression behaviour of porous titanium scaffold developed by replica impregnation method. Surface morphology before and after fatigue test was evaluated using digital microscope, scanning electron microscope along with EDS analysis and mapping.

2. Experimental Procedure

2.1. Specimen Preparation

Commercial pure porous titanium bar was fabricated by replica impregnation method having 90% porosity similar to cancellous bone [7]. After that porous titanium specimen was prepared by wire cutting having 9 mm height and 9 mm dia. The chemical composition of porous titanium is presented on table 1.

Table 1. *Chemical compositions of commercially pure titanium (wt.%) [7].*

Chemical Compositions	Percentage of wt.
Fe	0.033
H	0.006
N	0.009
C	0.005
O	0.113
Ti	Balance

2.2. Monotonic Compression Test

Compression tests for porous Ti specimens were conducted using a servo hydraulic fatigue testing machine (load cell capacity; 30 kN, Shimadzu Co. Ltd., Japan). The crosshead speed was -1 mm/min and the maximum displacement was -5 mm. Data was collected using PCD-300 series software system and recording frequency was 50Hz.

2.3. Cyclic Compression Test in SBF

Compressive fatigue tests in SBF were conducted for porous titanium using the same fatigue machine used for the compression tests. The tests were carried out by controlling the maximum compressive stress with a loading frequency of 10 Hz and stress ratio 10. Fatigue limit was defined by the number of cycles when cumulative compressive displacement of the porous specimen reached to 10%. The applied maximum stress was equal to $0.75\sigma_y$, $0.5\sigma_y$, $0.25\sigma_y$. Fatigue limit was calculated for 10million cycle. For fatigue testing in simulated body fluid (SBF), a cylindrical SBF holder was assembled with fatigue testing machine. SBF was prepared according to the composition given by Tadashi Kokubo *et. al.* [14] presented in table 2. The tests were carried out using the same conditions as those in air. The temperature of SBF medium was maintained at 37±1°C using a temperature controller (MTCS 15-BN) and pH was maimed at 7.4. The SBF medium was changed every three days to avoid depletion calcium phosphate ions in the solution.

Table 2. *Amount of chemical for 1L SBF preparation [13].*

Chemical	Amount (gm)
NaCl	8.035
$NaHCO_3$	0.355
KCl	0.225
$K_2HPO_4.3H_2O$	0.231
$MgCl_2.6H_2O$	0.311
$CaCl_2$	0.292
Na_2SO_4	0.072
Tris	6.118
HCl (mol/L)	39+5

2.4. SEM Observation

The surface morphology was observed using a microscope camera (VHX-1000). Scanning electron microscopy (SEM) (JSM-6306A, JEOL Ltd., Japan) was conducted. Elemental composition after SBF testing was also carried out by EDS analysis.

3. Result and Discussion

3.1. Surface Morphology

From digital microscope observation (figure 1 (a)) significant pores were detected on the surface of the porous titanium. SEM observation showed the wavy pattern of the surface and the pore size was measured 10-20mm whereas average pore size was 300-600 mm.

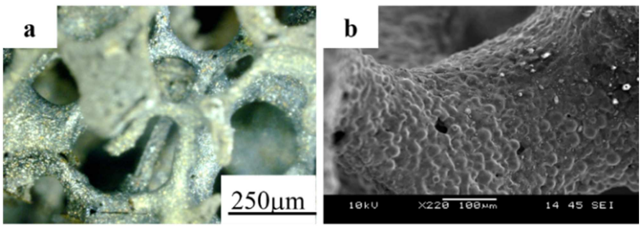

Figure 1. *Surface morphology of porous pure titanium (a) Optical observation (b) SEM observation.*

3.2. Monotonic Compression Test

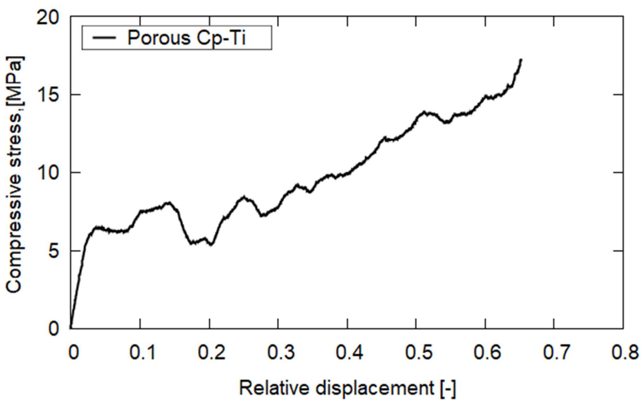

Figure 2. *Compressive stress vs relative displacement curve.*

From the compressive stress vs compressive relative displacement graph shown in figure 2, it can be observed that 0.2% proof strength of porous titanium is around 8MPa. Compressive elastic modulus of porous titanium was 180MPa which is identical to the one of cancellous bone (100-1000MPa) [15]. The long plateau region showed the load drop by strut breaking and again load rising due to support by other struts. After 10% increment of the strain, the porous body entered to the plastic region. This is why 10% strain was chosen as the breaking point during fatigue test.

3.3. Effect of SBF on Cyclic Compression Loading

The S-N curve (figure 3) showed that at higher stress level the porous titanium scaffold failed much earlier in SBF than ambient condition. The fatigue limit was found 2MPa which was not affected by Simulated Body Fluid. Figure 4 showed the progressive compressive strain of the porous component with increment of cycle. At initial stage (Stage I) compressive strain increased rapidly due to applied maximum stress. After reaching the saturation point, strain increased gradually (Stage II). When reaching at the critical point strain increased drastically at the final stage (Stage III) leading to final fracture of the porous body. Similar result was observed by other researchers also [11].

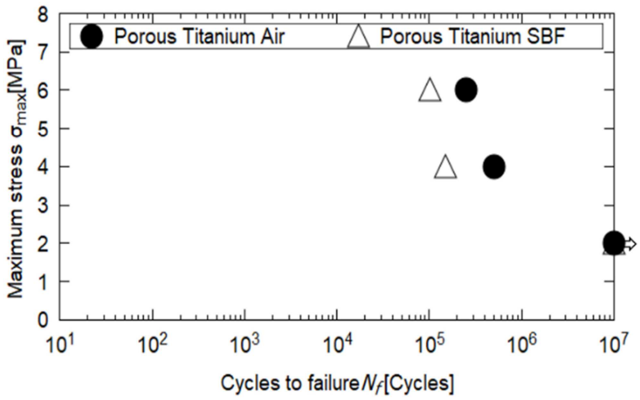

Figure 3. *Compressive fatigue lives of porous titanium in air and in SBF.*

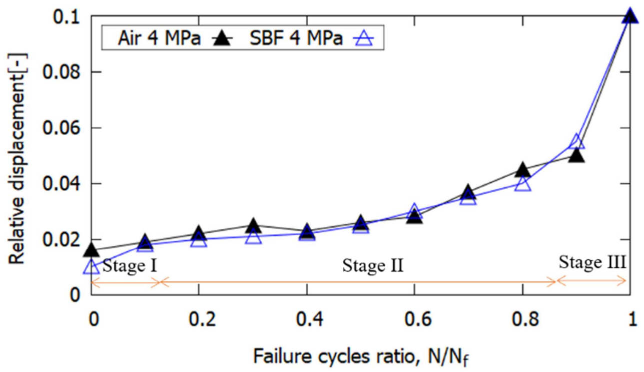

Figure 4. *Accumulation of compressive relative displacement during fatigue tests in Air and SBF.*

3.4. SEM Observation of Fracture Surface After SBF Testing

From fracture surface observation (figure 5) it was found that fracture generated from the pores remained in the porous titanium surface especially from the meshing point in struts which causes stress concentration during cyclic loading. From the figure 6 it is clear that calcium phosphate layer partially formed on the surface of porous titanium component even though under cyclic loading in Simulated Body Fluid. From EDS analysis in figure 7 and mapping in figure 8, the presence of calcium phosphate was confirmed and calcium phosphate (Ca/P) atomic ratio was calculated 1.44, which is near to b-TCP and HA phase and these phases are highly recommended for osteoconductivity [16]. This result clearly proved the biocompatibility of porous titanium components developed by replica impregnation method.

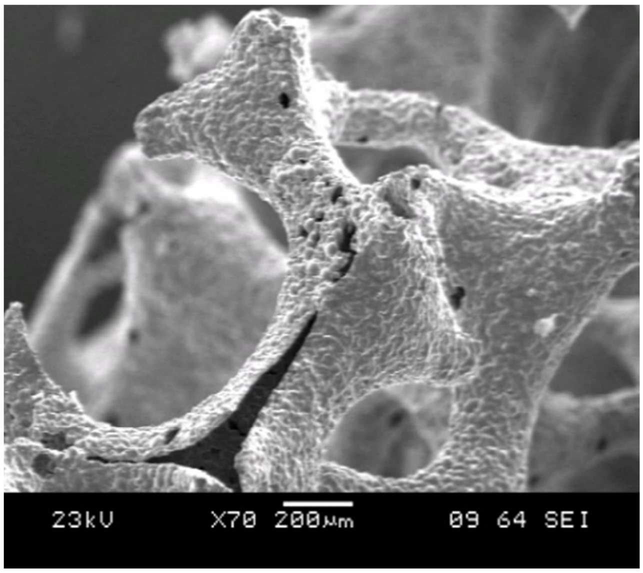

Figure 5. *SEM observation of cracking from microspores leading to fracture of trunk.*

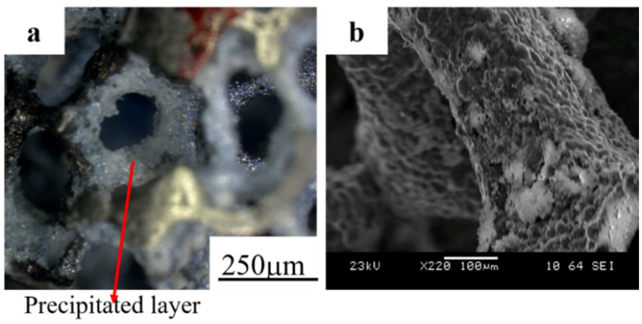

Figure 6. *Optical image (a) and SEM image (b) of precipitated surface after fatigue test at 2 MPa in SBF.*

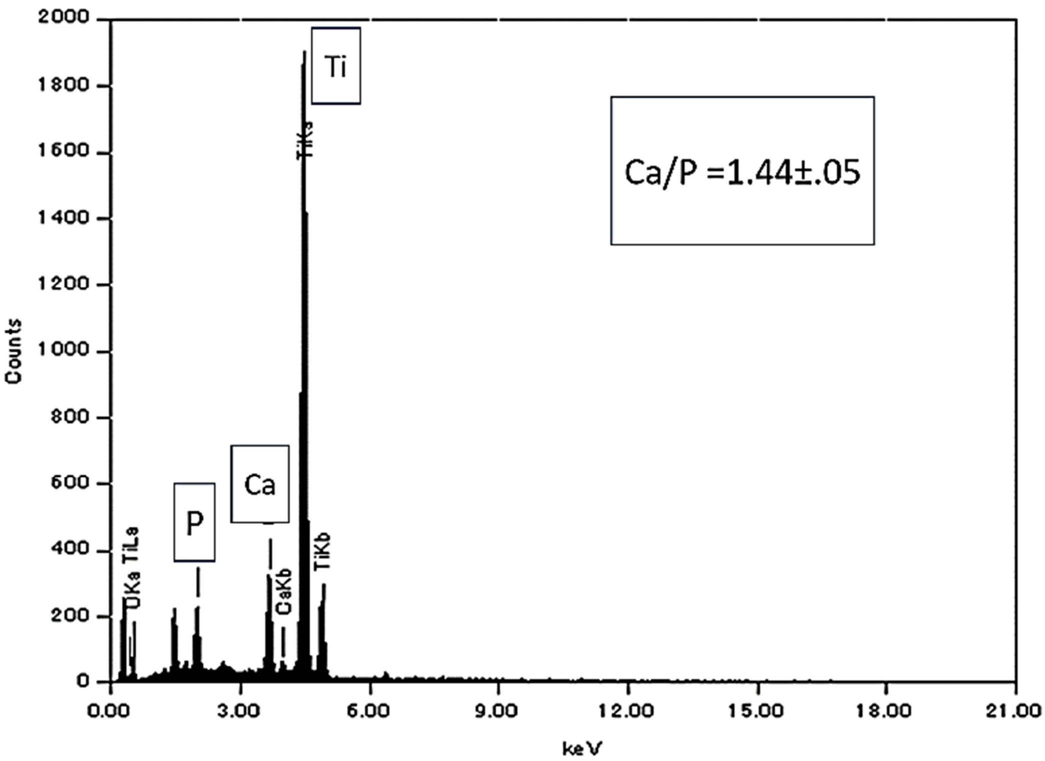

Figure 7. *EDS analysis precipitated surface in the region of figure 6 (b).*

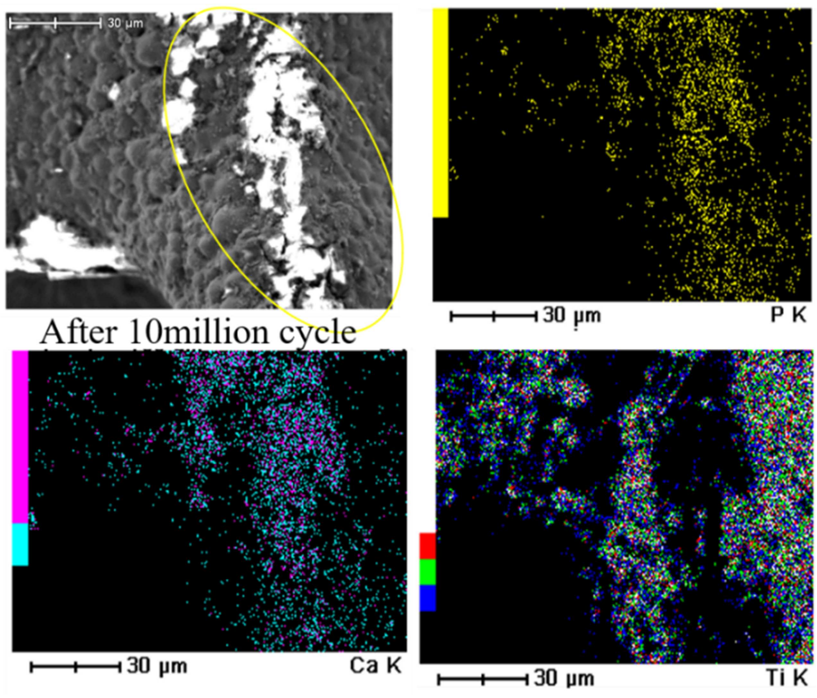

Figure 8. *Mapping for Ca, P content after cyclic loading testing at 2MPa in SBF.*

4. Conclusion

Monotonic compression test revealed that yield stress of porous titanium developed by replica impregnation method is compatible with cancellous bone. Fatigue limit was found 2MPa which was not affected by SBF environment. Though the porous component in SBF was subjected to cyclic loading, calcium phosphate particles can be precipitate partially which indicates high biocompatibility. Finally, it can be concluded that porous titanium components studied here can be an excellent candid for implant application. Further modification of porous titanium surface by applying some bioactive coating can be more beneficial bone tissue adhesion and osseointegration.

Conflicts of Interest

The authors declare that they have no competing interest.

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