

# Surface Modification of Porous Titanium with Rice Husk As Space Holder

Jitendra Kumar Kar

Department of Basic Science, Aryan Institute of Engineering & Technology, Bhubaneswar

Anchal Paramguru

Department of Basic Science, Capital Engineering College, Bhubaneswar

Dr. Paramananda Jena

Department of Basic Science, Raajdhani Engineering College, Bhubaneswar

---

## ABSTRACT

Porous titanium was characterized after its surface modification by acid and alkali solution immersion. The results show that the acid surface treatment caused the emergence of flocculent sodium titanate and induced apatite formation. The surface modification of porous titanium promotes biological activation, and the application of porous titanium is also improved as an implant material because of the existence of C and Si.

**Keywords:** Porous titanium Surface modification Microstructure XPS

---

## I. INTRODUCTION

Porous titanium has been proposed as a bone defect repair material, to achieve an adequate bone bonding degree, materials must have a good biological activity [1–3]. Dense titanium has the disadvantages of poor biological activity and of a mismatch with the bone elastic modulus [4–7]. Surface modification technologies can improve the biological activity of the porous titanium, improving the degree of binding and reducing immune rejection [8]. Alkali-base surface modification can allow the formation of a layer of bone-like apatite on the titanium surface, providing the structural conditions for bonding of the bone tissue on the implants and obtaining a good biological activity.

This paper adopts the method of powder metallurgy to prepare porous titanium materials, using rice husk as the space holder and acid treatment to improve the bioactivity of the porous titanium material surface.

## II. EXPERIMENTAL

The samples used in the experiments are porous titanium. The kinds of sample were immersed in 10 ml of the acid and alkali solution (1:6 concentration ratio) and placed in the drying box at 70 °C for 5 h. Finally, the surface of the sample was cleaned with ethanol and deionized water, and then dried for 1 h at 60 °C. Fig. 1 shows the immersion of the sample in the alkali solutions, which was carried out in such a way as to optimize the contact.

## III. RESULTS

Fig. 2 (a-c) shows the microstructure of porous titanium before (a) and acid and alkali treatments (b-c). Fig. 2 (d) shows after the acid and alkali treatments EDS in (c) spot “①”. From Fig. 2 (a) it can be seen that the sample surface is very smooth, the aperture of the pores ranges between 10 and 200 nm. The pores present mainly a small and irregular single-hole aperture. Si atoms present in rice husk play an important role in bone formation and mineralization, Silicon can promote osteoblast proliferation, differentiation and collagen production, silicon can help human bone cell culture, leading to increased bone formation [9]. Fig. 2(b) shows the micro morphology of treated sample, from which it can be seen that flocculent material is more abundant in the convex portions of the surface than in the flat parts. Fig. 2(c) shows the presence of deep cracks and the irregular floc morphology. It also shows that the increase of flocculent material with acid and alkali soaking contact area, evidenced by the fine acicular substance in the flat area with a characteristic dimension of about 4 nm. The EDS scan analysis of the flocculent material is reported in Fig. 2 (d), and shows that the elements present are C, O, Na, Ti and a small amount of

S. A preliminary analysis suggests that these substances may derive from  $\text{Na}_2\text{TiO}_3$  and rutile, generated during the treatment of the material.

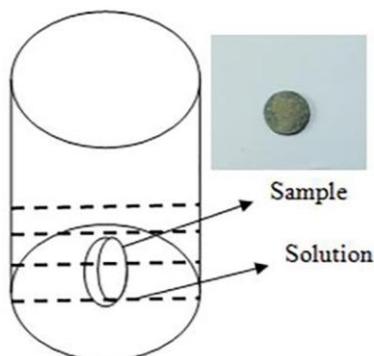


Fig. 1. Schematic diagram of the alkali immersion sample.

Fig. 3 shows the X-ray Photoelectron Spectroscopy(XPS) spectra of various elements on the surface of porous titanium after the alkali treatments. The main components of the sample surface are five elements: Ti, O, C, Na and Ca. The binding energies of Ti2p3/2 and Ti2p1/2 are 458.27 eV and 464.27 eV, respectively, which correspond to the chemical valence of Ti<sup>4+</sup>; and the binding energy of O1s is 531.39 eV, corresponding to the chemical valence state of O<sup>2-</sup> in X-Ti-O. The corresponding material should therefore present a titanate structure. The binding energy of C1s is 284.7 eV, corresponding to the carbides in the valence state C<sup>-4</sup>, which here corresponds to TiC. The wear resistance of porous titanium is increased as a bone implant material attributable to the presence of TiC. Na1s had a single peak at 1071.78 eV, corresponding to the Na<sup>+</sup> valence state. Ca2p has a double state position in Ca2p3/2 (346.62 eV) and Ca2p1/2 (350.15 eV), which corresponds to the Ca<sup>2+</sup> state. In addition, Ca is not the main component of the reagents used in the experiment, but it is largely present on the surface of the sample. To understand this result, tests were carried out on pure NaOH, in which the content of Ca<sup>2+</sup> was found to be 0.01%. A 0.0005% Ca content in the NaOH solution is sufficient to induce apatite formation [10], therefore it can be inferred that the Ca<sup>2+</sup> found on the surface of the sample after alkali heat treatment derives from the NaOH solution.

#### IV. CONCLUSION

After the treatment of porous titanium sample, the following conclusions can be drawn. Porous titanium samples after acid and alkali treatment present a smooth surface with flocculent material. The presence of acicular nanometric material was analyzed by XPS and found to have a high Ca and C element content. The presence of Ca element will induce apatite formation, increasing the biological activity of the porous titanium surface, and the wear resistance of porous titanium is increased as a bone implant material.

#### ACKNOWLEDGMENTS

The authors thank the editor and the anonymous reviewers for their helpful comments and suggestions. The author would like to thank the financial support from National Science Foundation of China (51505433).

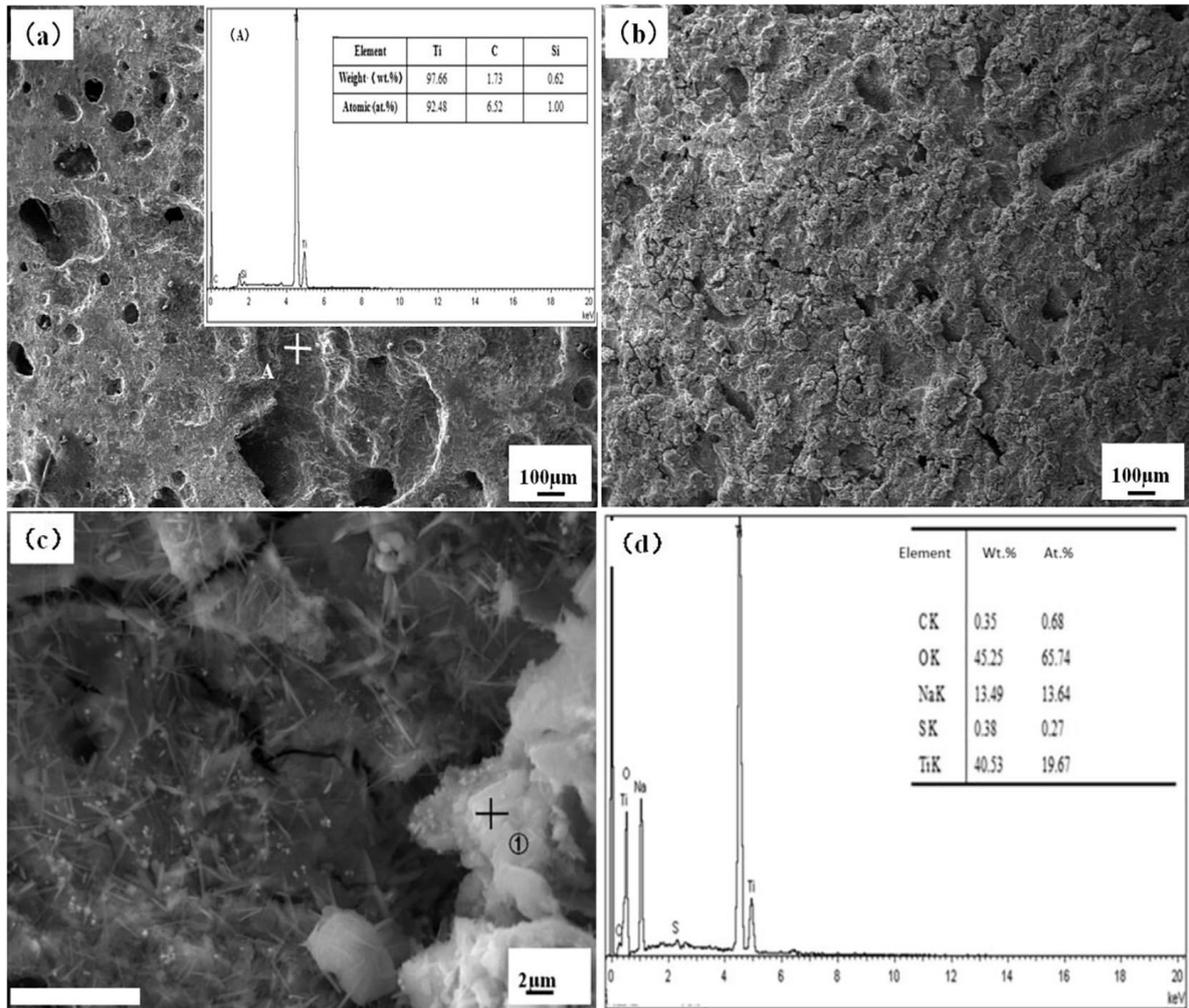


Fig. 2. SEM images of porous Ti materials before (a) and after (b,c) alkali treatment and EDS (c).

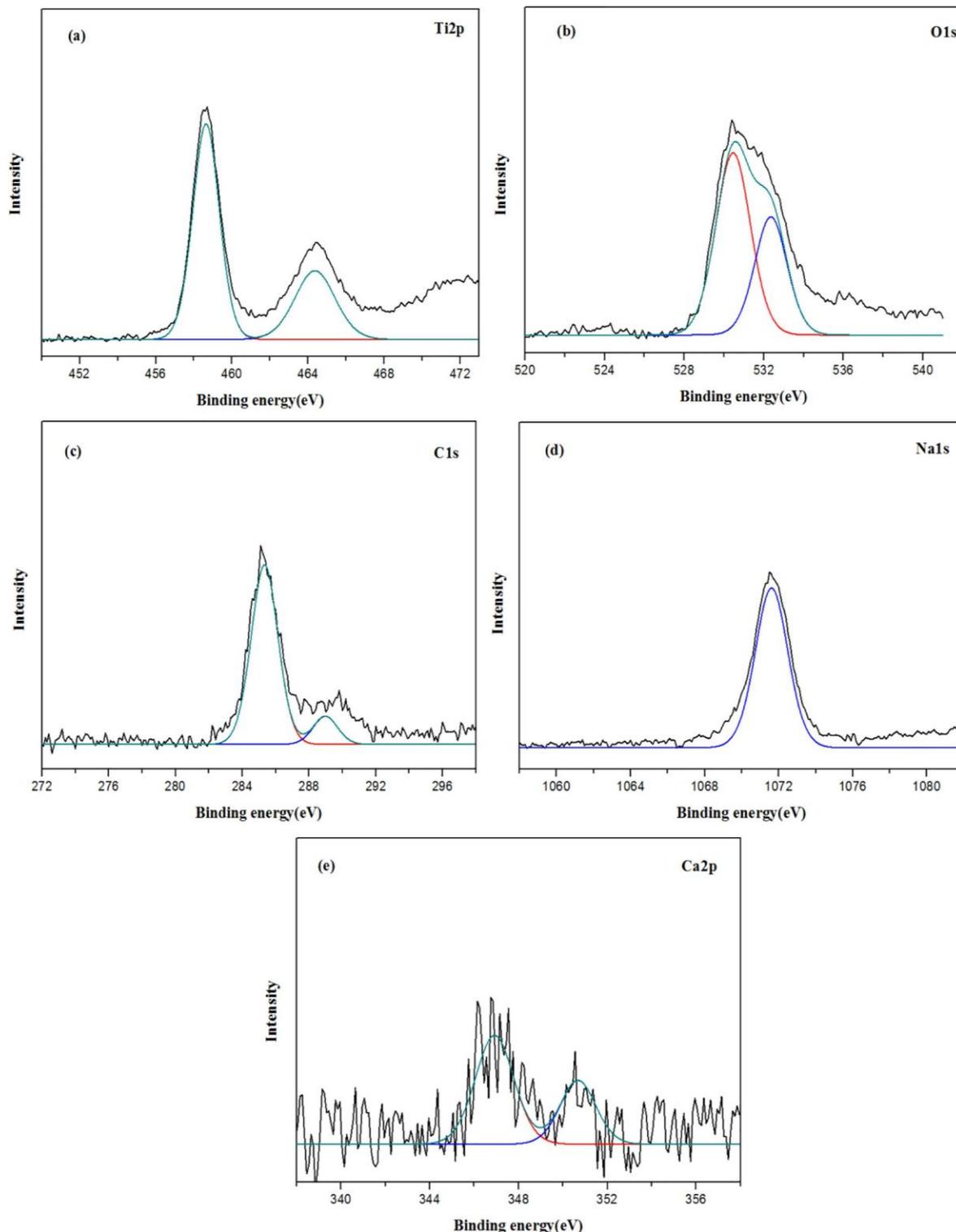


Fig. 3. XPS analysis results of alkali-heat treated specimen: (a) Ti 1 s binding energy range, (b) O1s binding energy range, (c) C1s binding energy range, (d) Na1s binding energy range, (e) Ca2p binding energy range.

### REFERENCES

- [1]. Diller DE. Measurements of the viscosity of compressed gaseous and liquid nitrogen + methane mixtures. *Int J Thermophys* 1982;104(3):143–52.
- [2]. Chung DDL, Fernando JA. Pore structure and permeability of an alumina fiber filter membrane for hot gas filtration. *J. Porous Mate* 2002;9:211–7.
- [3]. Ismagilov AAKZR, Shkrabina RA. Porous alumina as a support for catalysts and membranes. Preparation and study. *React Kinet Catal Lett* 1997;60:225–9.

- [4]. Boomsma K, Poulikakos D, Zwick F. Metal foams as compact high performance heat exchangers. *Mech Mater* 2003;35:1161–9.
- [5]. Shimizu T, Matsuura K, Furue H, Matsuzak K. Thermal conductivity of high porosity alumina refractory bricks made by a slurry gelation and foaming method. *J Eur Ceram Soc* 2013;33:3429–32.
- [6]. Barcellos WM, Souza LCEO, Saveliev AV, Kennedy LA. Ultra-low-emission steam boiler constituted of reciprocal flow porous burner. *Exp Therm Fluid Sci* 2011;35:570–7.
- [7]. Thamaraiselvi TV, Rajeswari S. Electrochemical behaviour of alkali treated and hydroxyapatite coated 316 LVM. *Trends Biomater Artif Organs* 2005;18(2):242–8.
- [8]. Amin YS, Ahmadi SM. Effects of bio-functionalizing surface treatments on the mechanical behavior of open porous titanium biomaterials. *J Mech Behav Biomed* 2014;36:109–19.
- [9]. Xynos I, Edgar A, Buttery D. Gene-expression profiling of human osteoblasts following treatment with the ionic products of Bioglass 45S5 dissolution. *J Biomed Mater Res* 2001;55:151–7.
- [10]. Kizuki T, Takadama H, Matsushita T, Nakamura T. Effect of Ca contamination on apatite formation in a Ti metal subjected to NaOH and heat treatments. *J Mater SCI-Mater M* 2013;24:635–44.