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Synthesis titanium foam by space holder technique

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Abstract

The main property required of metallic biomaterials such as Titanium foam for biomedical applications is to be a biocompatible material. Good mechanical properties, osseointegration, high corrosion resistance, and excellent wear resistance are required. Furthermore, it is crucial for improving the biological and mechanical properties of the highly porous structure of titanium foams as implant materials for dental implants. Owing to its outstanding mechanical properties, low density, and high chemical resistance, titanium-based foams have various promising applications. This paper aims to highlight the significance of porosity in the blend of dental implants of Titanium foam with bone tissue and the space holder fabrication method presently being studied. It has been found that the preservative built-up technique is promising for controlling both pore size and shape to achieve the optimum biological properties of Titanium foam. Titanium foam has been successfully synthesized through the space holder method.

The properties of the space holder content on the porosity and compression mechanical properties were studied. The titanium foam samples were investigated with scanning electron microscopy (SEM) and X-ray diffraction (XRD). It was proved that pore sizes, porosity gradients, and geometries can be efficiently measured by fluctuating the space holder sizes, pressing order. The sintering time and temperature are often optimized to minimize/remove the larger pores. Sintering needs to be done over a longer time to further densify the material and increase the mechanical property values.

Keywords: biomaterials, titanium foam, dental implant, space holder, osseointegration.

1. Introduction

Metallic foam has designed a new category of materials. Associated with bulk metals, these extremely lightweight materials have an inimitable blend of properties. Titanium metals are currently the focus of exact dynamic research and growth activities. The possessions of the structure and composition on the properties of porous titanium for biomedical applications have been considered and recognized by various researchers. Titanium-based porous materials can be used in structural applications and medical implants in order to their good corrosion resistance, admirable mechanical possessions, and great biocompatibility. Additionally, previous research has acquainted that the open cellular structure of the porous titanium passes the ingrowth of new bone tissue and the transport of body fluids [1]. Because of its very high melting point (1670°C) and high reactivity with other elements [2] at the liquid state, the production method of titanium foam has engrossed on the powder metallurgy route to evade a liquid state. There are several methods to produce porous metallic foam including powder sintering, solid-state foaming, and rapid prototyping [3].

ISSN: 2785-9541 VOLUME 5, ISSUE 1, 2022, 67 – 79. www.egyptfuture.org/ojs/

Powder sintering has been widely used because the material blending and compaction processes provide customization of fabric compositions, mechanical properties, and shapes so as to provide implants with the required characteristics. Powder sintering has its recompenses over other synthesis methods. The method is extremely simple and economical. Conveniently, a newly settled method of powder metallurgy using a space-holder technique has arisen to the forepart [4].

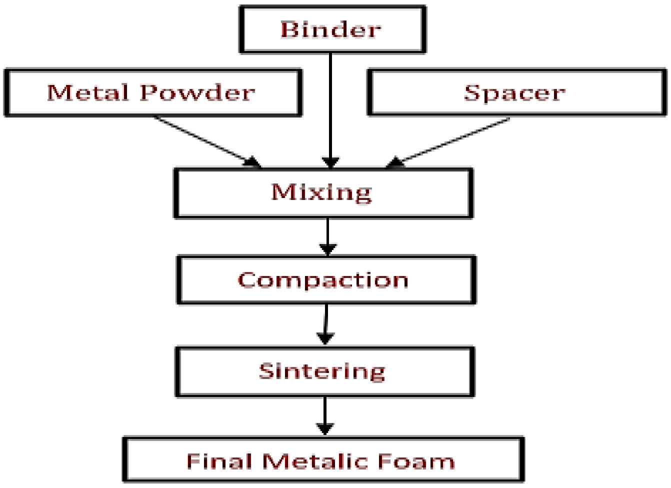

Figure 1: The schematic process of space-holder technique.

Powder metallurgy is a procedure wherever a material powder is compressed as a green form and sintered to a disposable figure at higher temperatures [5]. A powder metallurgy sintering process was used. At first, a sequence of powder compaction researches was carried out to investigate the compression behaviors of a variety of titanium. As revealed in Fig. 1, the space holder technique contains five major steps: powder selection, mixing, compaction, sintering, and spacer removal. Firstly, metal and foaming agent powders must be chosen. Subsequently selection, they must be mixed entirely to declare the homogeneousness of the combination. Control over this procedure hinge on how acceptable the powders are mixed.

ISSN: 2785-9541 VOLUME 5, ISSUE 1, 2022, 67 – 79. www.egyptfuture.org/ojs/

The next step is compacting the powders into a mold under measured pressure. Depending on what kind of space holder (e.g., magnesium [6], sodium chloride [7], polymers [8,9], camphene [10], ammonium hydrogen carbonate [11] and ice [12]) is used in the process, the following step can be moreover sintering or removal of the space holder [13]. Space-holder techniques offer a route to controlling porosity parameters such as pore morphology and percentage [14]. Significant exertions and developments in this field are commonly predictable. The performance of titanium foam via the space holder technique offers flexibility and a cost-effective substitute. Among the different techniques, there are encouraging manufacturing processes where low compaction pressures are working as a consequence of higher porosities and a lower young's modulus can be attained. Loose sintering is an attractive method to form titanium foam samples. Commonly, as showed in tab.1 there are two categories of space holders: those that can be removed thermally when the temperature is higher and those that are dissolved and removed by a solvent. This study objectives to determine an approach to combining Ti powder with a powder salt bead space holder, in an effort to enhance the reproducibility of mixing and hence of the porosity and interconnectivity in the resulting porous part. Porous Ti structures made by this route have been characterized and their structures are presented.

Table 1: Space holder in titanium foaming [15].

Spacer	*Removal method*
Mg	*Thermal*
Carbamide	*Thermal*
Sod. Chloride	*Solvent (water)*
Corn starch dextrin	*Solvent (Water)*
saccharose	*Solvent*
NaF	*Solvent*

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Therefore, within the above framework, this study has been resulted with a summary of the most promising technique according to industrial viability, economic profits and duplicability, realizing an optimum equilibrium between mechanical properties and bio functional performance. To satisfy this combination of requirements, we used the space holder method, which has the advantage of ensuring controllable porosity, pore size, and pore distribution, while providing the ability to machine the compact composed of Titanium powder and spacer particles. As shown in Fig. 2. The core of the technique is that mechanically unchanging and adequately strong Ti/Nacl compacts are gained, which varieties it possible to first machine a compact to the chosen form. Removal of NaCl from the machined part by solvent using water is then carried out as a next processing stage. To conclude, sintering consequences in a net-shape Ti foam.

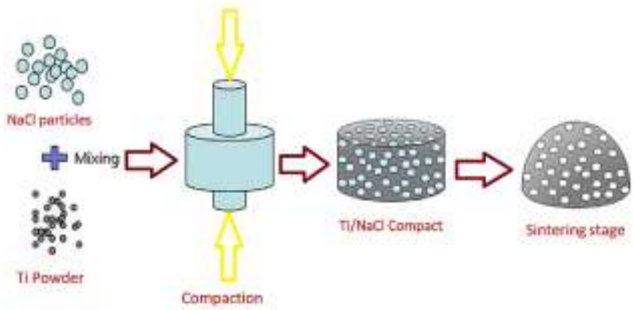

Figure 2: Synthesis of titanium foam.

2. Methods

2.1. Raw Materials

The titanium powders were used with (~99.5%) purity with50µM - 200µM as shown in Tab.1, supplied by Bayville Chemical Supply Company Inc., USA. The sodium chloride particles (~99%) purity shown in Tab. 2

ISSN: 2785-9541 VOLUME 5, ISSUE 1, 2022, 67 – 79. www.egyptfuture.org/ojs/

2.2. Titanium and Sod. Chloride (NaCl) blend compaction

Mixtures of Ti and NaCl powders were methodically mixed by dipping for 15 min. All mixtures were pressed using cold compacted in a die made of high-strength steel at a controlled pressure 400Mpa. Pressure is pragmatic for one minute in order to ensure efficiency and homogeneously pressing. For the powders binding, 2-3 droplets of ethanol per 10 g of powders mixture were enhanced. As uniaxial pressure from the top was pragmatic to flattened powder mixture to form green compacts, the green compacts will have mixed density dispersal due to the resistance between die walls and mixture form and likewise between the powder particles.

Table 2: Characteristics of titanium powder.

Characteristics	*Description*
Chemical Formula	*Ti*
Melting point	*1,660 °c (3,020 °f) - lit.*
Density	*4.5 g/ml at 25 °c (77 °f)*
Particle size	*50µm - 200µm*
Molecular weight	*47.87 g/mol*

Table 3: Characteristics of sodium chloride (NaCl)

Characteristics	*Description*
Chemical Formula	*NaCl*
Melting point	800.7 °c (1,473.3 °f)
Density	2.17 g/cm^3 *at 25 °c (77 °f)*
Molecular weight	*58.443 g/mol*

2.3. Titanium foams

Subsequently density measurement of the Ti/NaCl mixtures, the NaCl phase was dissolved in water to acquire Ti foams. Complete removal of the NaCl was detected, and foam metal density measured representative that no closed porosity was existing. NaCl space-holder removal was near complete. The cold compacted pellets were characterized in terms of their dimension and density. Then cold compacted pellets were dried in a furnace at 250 C for 1.5 hours to eliminate moisture and rise strength.

ISSN: 2785-9541 VOLUME 5, ISSUE 1, 2022, 67 – 79. www.egyptfuture.org/ojs/

After that sintering stage was coming. Titanium is a highly reactive metal at the raised temperatures. It eagerly responds to atmospheric gases at high temperatures. Consequently, to evade objectionable reactions of Ti with gases, sintering of compacted green forms was used by inert gas. In this procedure as soon as, green compacts are held in a furnace at a high temperature, metallurgical bonds form between titanium particles and the compaction process takes place. By using a parameter of sintering time and temperatures. There was four different holding times of 2, 4,6 and 8 hours were considered for this work and two sintering temperatures of 1000 ºC and 1500 ºC were measured.

2.4. Porosity characterization.

Scanning electron microscopy (SEM) was used to examine of the Titanium powder, NaCl partials and foam forms which was sintered at different temperatures. The phases in Ti-NaCl compacts, green bodies, and sintered samples were identified with X-ray diffractometer with Cu Ka radiation which was used to acquire information on the full elemental composition of sintered final Ti foam forms. Superficial porosities of Ti foams sintered at changed temperatures were calculated through Archimedes principle. For each sintered Ti foam form, dry weight (W_1) was measured using a digital balance. The titanium foam form was formerly flooded in water to be boiled for 2 h, before saturated for another 24 h. Weight of Ti foam adjourned in water (W_2) was detected. Next it was recovered out of water and spoiled dry via a tissue paper to remove extra water, it was directly re-weighed (W_3). Porosity was considered using the following formula:

Apparent porosity (%) = $\frac{W3-W1}{W3-W2}\times 100\%$

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3. Results and discussion

We selected NaCl as space-holder because of its high solubility in water, complete inertness with titanium, and very low toxicity. It's important if small amounts of space holder persist in the foams used as biomedical application. Fig.3 displays the particle size of the titanium powder. It shows that the particles are in agglomerated form and these particles are then handled for synthesis of foam.

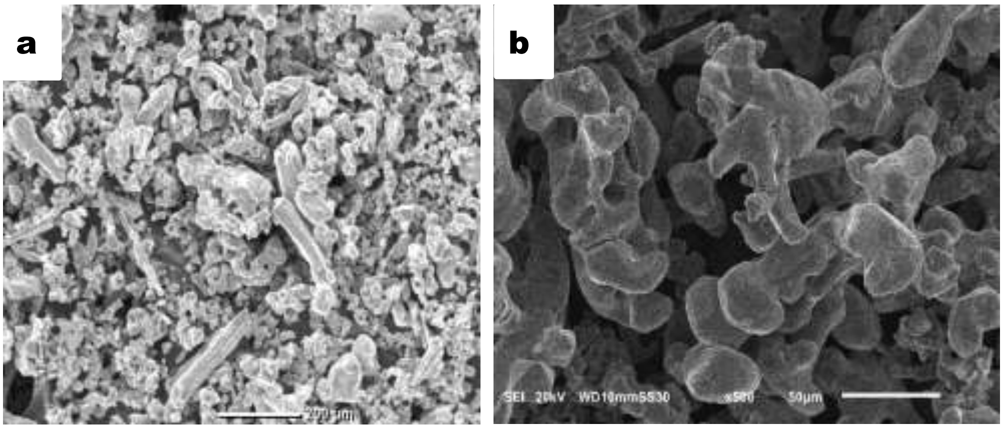
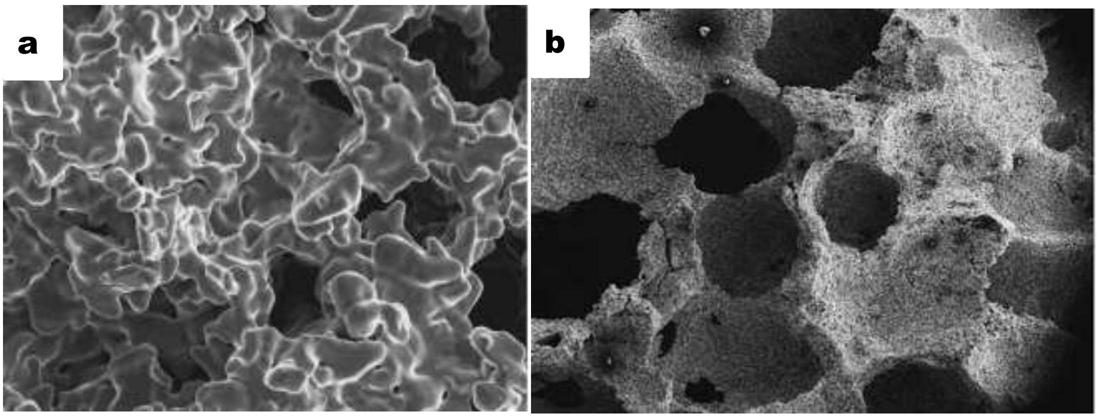


Figure 3: SEM images of Titanium powder: (a) 200µm (100× magnification); (b) 50µm (500× magnification)



ISSN: 2785-9541 VOLUME 5, ISSUE 1, 2022, 67 – 79. www.egyptfuture.org/ojs/

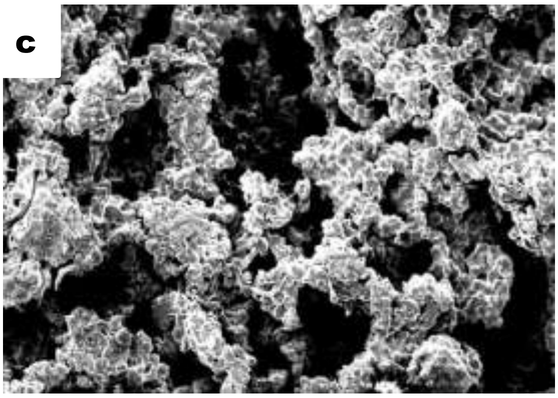


Figure 4: SEM images of Titanium foam: (a) After sintering 60% and 50µm; (b) 300µm, (c)80% and 300µm.

Titanium foams with different stages of porosity were effectively produced by adding several ratios of space holder (NaCl). For each ratio of NaCl to titanium, three samples in the same range of porosity were invented, and all of them were used for examination. Porosity defines most foam applications and is therefore one of the furthermost significant features.

The measured porosity of samples was in the range of 60–80%. The pore morphology of the produced titanium foams is shown in Fig. 4. It is pure that there are two classes of pores: interconnected pores, which are called macropores, and micropores. The open cellular structure of the titanium produced is clearly shown in Fig. 4. The interconnected macropores are a result of NaCl decomposition. The sizes of these pores range from 50µm to 300µm. Nevertheless, there are similarly some micropores in the assembly.

Titanium powders were estimated for any contamination before processing. Actually, the purpose of XRD is to confirm that the compound used after sintering is exactly similar to that before sintering. The form of titanium before sintering was selected as a reference pattern for comparison. The peaks shown at the top of Fig. 5 are the titanium peaks fitting to the samples before sintering. Compared with the pre-sintering pattern, the peaks didn't change, as shown at the bottom of Fig. 5, indicating that there was no change or contamination after sintering.

INTERNATIONAL JOURNAL OF
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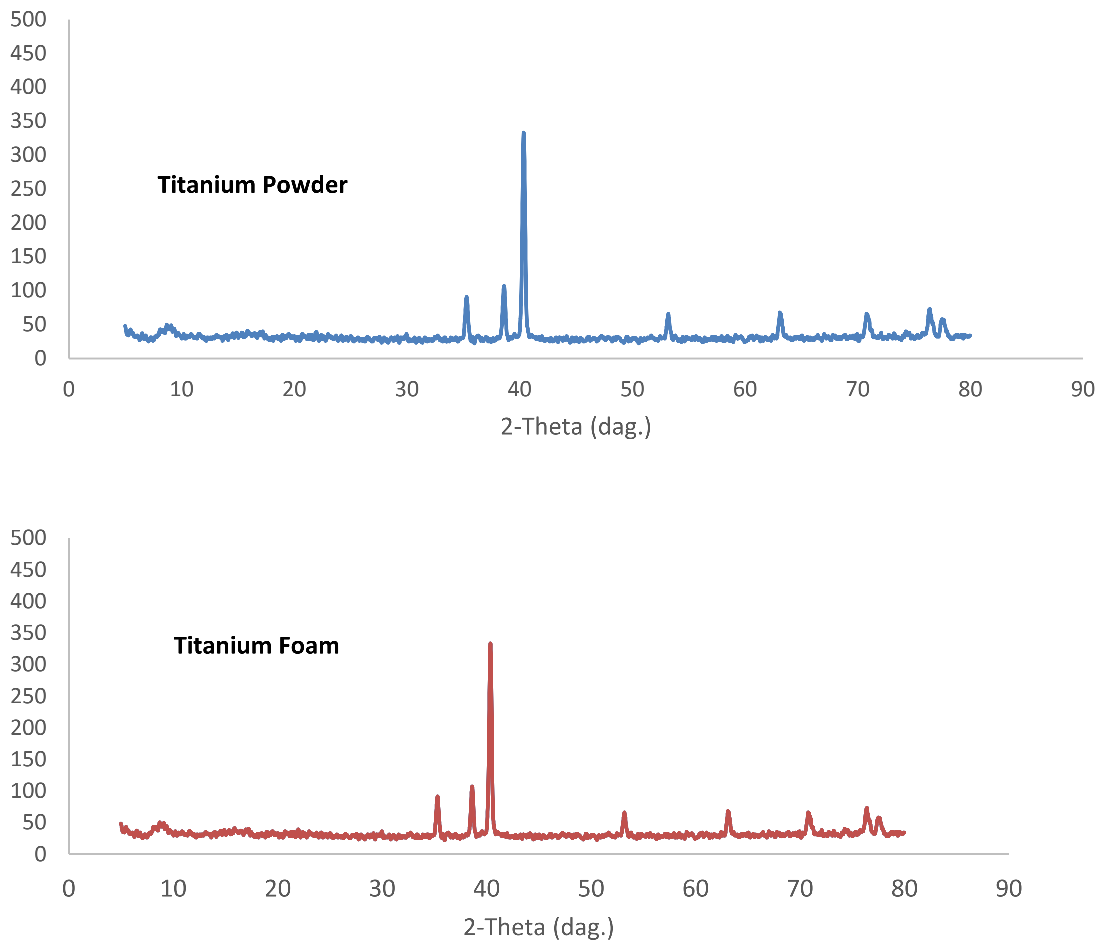

Figure 4: XRD of Titanium powder and foam.

The XRD analysis also illustrates the presence of Titanium dioxide phase in the foam. And there was any of sodium (Na) and chlorine (Cl) were obtained in the final Ti foam. This approves that NaCl is completely separated out from Ti-foam by high temperature water treatment for a period of 24 hours.

ISSN: 2785-9541 VOLUME 5, ISSUE 1, 2022, 67 – 79. www.egyptfuture.org/ojs/

4. Conclusion

Titanium is usually utilized in dental applications and might be fabricated from numerous predictable and rapid techniques, looking at the sort of materials used or the kind of pore structures needed. This study determines the submission of powder sintering using common salt (NaCl) as an area holder material to supply titanium foam. additionally, the effect of porosity and pore size on foam behaviors.

NaCl with well-measured sizes and high biocompatibility could be a perfect space holder which is solely removed. Titanium scaffolds with different levels of porosity and ranging pore sizes were synthesized.

Ti foam with open porosity within the range of 70–80% has been made. By combining the Ti powder into NaCl, by mixing followed by compaction and sintering, porous structures with uniform density, pore sizes, and predictable level of connectivity are produced, showing important development on the structures made by predictable powder mixing procedures. From XRD it's concluded that foam contains mainly titanium and titania, no clue of sodium (Na) and chlorine (Cl) were found. That meant NaCl is totally separated out from Ti-foam by high-temperature water**.**

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