Mechanical Evaluation of Open Pore Titanium Foam Obtained by Space Holder Method

M.M. El-Saies^{1, []}, Ibrahim Hassan², M. T. El-Wakad³, A. H. El-Shazly⁴



Abstract In the process of powder metallurgy, titanium powder is compressed into a green body and sintered into a net shape. Investigations were done into how the processing factors affected the physical, microstructural, and mechanical qualities. In the current study, titanium foam with adjustable porosity was created utilizing a newly invented powder metallurgy technique called the space-holder approach. The stress-shielding effect, that encourages bone resorption around implants, is primarily caused by the high Young's modulus of titanium relative to the surrounding bone. Growing scientific and technological interest has been paid for to the development of titanium foam for implants that have a low Young's modulus. This study's objective is to assess the commercial viability, industrial applicability, and potential technology transfer of several powder metallurgy methods. Under various strain rates, titanium foam of varying relative densities was analyzed for their compressive deformation behaviors. Foam's plateau stress, Young's modulus, and energy absorption all increase with increasing relative density, as predicted by power law correlations. Nonetheless, densification strain increases proportionally with increasing relative density. The strain rate compassion and the strain rate sensitivity parameter of these foams were also studied. This research not only traces the origins of the mechanical properties of titanium foams, but also demonstrates the substantial practical value of the new method of measuring porosity.

Keywords: titanium foam, powder metallurgy, space holder method, mechanical properties.

1. Introduction

mechanical, Biological, and thermal properties must all be maximized in a candidate implant material. An ultimate interface between the body and implant calls for biocompatibility. Implants need to be designed with the best possible balance of mechanical properties to prevent damage from fatigue. protecting phenomenon and avoiding cracks under static and cyclic loads, to avoid destruction to the surrounding bone and/or tissue during the implantation process, low thermal conductivity is necessary. Metals with a high tensile strength-to-density ratio, high corrosion resistance, fatigue resistance, high crack resistance, and hence on are titanium and titanium alloys. Many novel physical, mechanical, thermal, electrical, and acoustic properties, as well as low densities, are among the many advantages of metal foams. Both conventional and AM techniques for producing titanium foams are discussed. Mixing powder, compression molding, and sintering are the three main steps in the powder sintering process. In most cases, densification is performed at room temperature, while sintering is performed at elevated temperatures, both at atmospheric pressure and in a thoroughly monitored atmosphere structure. As previously mentioned, Titanium foams used in biomedical implants have porosity for the purpose of lowering the implant's modulus of elasticity to values more in line with the underlying bone. And because bones are naturally porous, it promotes cellular attachment and growth even more effectively than other materials [1,2]. Some of these methods are competitive with the Space holder process in terms of managing the quantity and shape of pores, as dimensional control clearly as the of economically produced parts. Since the pore formers in the space holder process don't meet the

Received: 28 February 2023/ Accepted: 02 April 2023 Corresponding author, M. M. El-Saies, marwaelsaies@gmail.com ¹Chemical Engineering Department, Higher Institute of Engineering and Technology (HIET), Alexandria, Egypt ²Department of Biomedical Engineering, Faculty of Engineering, Helwan University, Helwan, Cairo, Egypt ³Faculty of Engineering and technology, Future University,Cairo, Egypt

⁴Chemical and petrochemicals Engineering Department, Egypt-Japan University of Science and Technology, New Borg El-Arab City, Alexandria, Egypt.

metal, they can be simply washed away. The quantity and geometry of the spacers used to reinforce a structure determine its permeability. The SH is picked because it meets the following requirements: (i) it can be obtained in a variety of sizes and shapes; (ii) it is inexpensive; (iii) it does not react with Ti; (iv) it leaves behind few residues; (v) it can be processed easily; (vi) it is not toxic; and (vii) it is not easily deformed. SH is typically eliminated when subjected to solvents or heat. Powder metallurgy was used to create this titanium foam product with a 65% porosity through compaction and pressure working. Many industries, from medicine to aerospace, anticipate huge benefits from this breakthrough in manufacturing technology. The ability to tailor the metal foam's modulus of elasticity by changing the pore volume, fraction, and dimensional distribution to match the bone's is a major benefit because it greatly lessens the shielding phenomena. Longer-lasting porous titanium prosthetic implants have been shown to confirm this property [3]. To effectively distribute loads and prevent stress shielding, the Youngs modulus of an implant material ought to be as close to that of human bone as possible [4]. Bone tends to thicken and thicken in the direction of the applied load, making the bone stronger [5]. The relative Youngs moduli of an implant and bone determines how the load is shared between the two. Because the implant will be under more stress than the bone around it, the bone around it will weaken. This is especially true if the implant's modulus of elasticity is greater than that of the bone. More stress is absorbed by a larger gap in Young's moduli, which accelerates bone weakening. This results in the implant becoming less stable in the bone and eventually breaking due to aseptic loosening [6]. Currently, many implant materials are stronger than bone, so stress shielding is almost always an issue [7]. The pore morphologies of titanium foams varied using a well-known technique called the space holder method, which permits fine tuning of porosity and achievable pore content structure. Mechanical testing and X-ray computer microtomography were applied to determine the foams' properties.

2. Methods

2.1. Preparation of titanium foam

As raw materials and space fillers, titanium powder and sodium chloride were selected. The titanium powders used had a (99.5%) purity range of 50 μ M to 200 μ M and were purchased from Bayville Chemical Supply Company Inc. in the United States. The purity of the sodium chloride particles (99%). Titanium powders were combined with sodium chloride in proportions of 15, 25, and 35% wt, resulting in foams designated as F1, F2, and F3 after processing.

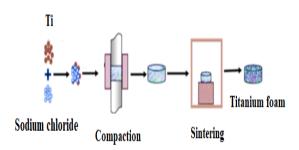


Fig. 1 Processing of titanium foam.

We chose NaCl as a space holder due to its high-water solubility, complete inertness with titanium, and extremely low toxicity. In foams used for biomedical applications, it is crucial that small amounts of space holder remain. represents the particle size of titanium powder. In a mortar, the powdered titanium and sodium chloride were combined after being weighed. Following this, the mixture was poured into a steel mold with an inner diameter of 50 mm. The filled mold was then placed on a press under 300 MPa of pressure for 1 minute to undergo cold pressing. The green compacts were subsequently subjected to a two-step heat treatment. The first step was to put the green compacts in a furnace and heat them to 450°C. Once the furnace cooled to room temperature, the compacts were removed. The second step involved placing the preforms without spacers into a sintering furnace and heating them for one hour at 1100 °C. The processing of titanium foam is depicted in Fig 1. For the mass volume method, the mass and shape sizes were substituted into the equation (1) below to calculate porosity [8]. Were, P: porosity; m: mass, g; V: volume, cm³; ρ_s : substrate density, g/cm^3 .

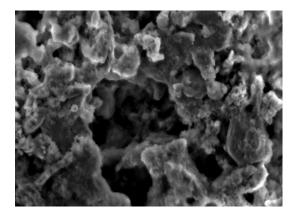
$$P = 1 - \frac{m}{\nu\rho} \tag{1}$$

2.2. Characterization of titanium foam and Mechanical Testing

The titanium foam samples were ultrasonically cleansed in deionized water for 15 minutes. Then, alternately add ethanol for cleansing and saturating. Finally, place them in an oven to dry them for future use. Using a scanning electron microscope, the internal structure and cell wall morphology of the titanium foams were studied. Ti foam samples (5 mm in diameter and 10 mm in height) subjected to a room temperature compression test with a crosshead speed of 0.005 mm/s to evaluate their mechanical properties. It is known that raising the strain rate during the compression test has no major effect on Young's modulus, but increasing the strain rate results in a large increase in compressive strength [9]. Hence, the test speed was set as low as possible in this investigation. To get the stress-strain curves of the specimens, each specimen was subjected to progressively increasing stress until failure was seen. The moduli of the samples were determined by measuring the slopes of the linear parts of the stress-strain curves while simultaneously measuring the compressive strengths at the greatest stress levels.

3. Results and Discussion

The SEM topographic view reveals that the microstructure of the porous material is composed of micropores, which are predominantly spherical, and macropores. It is able to be noted that the macropore walls are rough due to the nature of titanium powder, which is an advantageous property for osseointegration of implants [10].



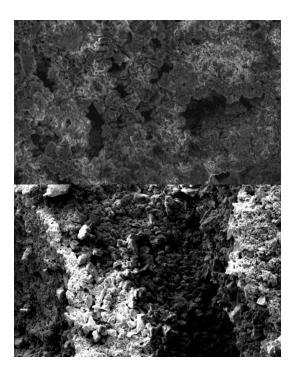


Fig. 2 SEM of (A) F1 (x 2000 and 5μm), (B) F2 (x 500 and 50μm) ad (C) F3 (x 2000 and 200μm)

Fig 2 illustrates the optical micrographs of samples F1, F2, and F3 with 15%, 25%, and 35% NaCl additives, respectively. Micrographs depict pores as dark regions. These numbers indicate that the titanium matrix has acquired a near-complete degree of densification.

Hence, the titanium foam's mechanical anisotropic qualities can be attributed to its porous structure, which resembles those of bone. In addition, the interconnectedness of the pores, which is essential for cell proliferation, is visible at higher magnification. Changing the particle size of NaCl and the ratio of Ti to NaCl in the Ti-NaCl combination affects the pore size and porosity.

The porosity amount and shape depend on the powder sizes and their proportions in the combination; hence the porosity structure varies considerably between the three samples [11]. The compaction and sintering behaviors of the various powder quantities in the combination also affects the foam processing. All specimens had a significant percentage of pores by volume. If there is a large real proportion of open porosity, it is likely that the already-formed open channels can be connected to channels outside the polished surface. varying the amount of additive has a substantial effect on the volume fraction of pores [12].

The XRD profile of titanium foam is depicted in Fig 3. The foam contains titanium and titanium dioxide, as determined by XRD examination. In the foam, there were no traces of sodium (Na) or chlorine (Cl). This demonstrates that high-temperature and water treatment for 24 hours totally separates NaCl from Ti foam.

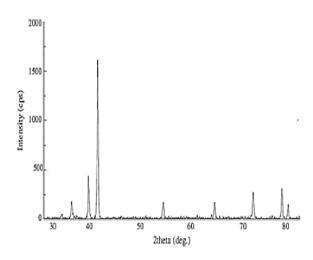


Fig 3 XRD of titanium foam.

Microhardness tests allowed for an assessment of the effect that the foaming phenomena had on the mechanical properties of the alloy, using the same samples and sections as those evaluated under a light microscope. The indenter makes it easy to see that the pits are significantly bigger, which is in keeping with the higher volume and proportion of porosities. Microhardness measurements of Ti foam have been recorded at values between 108.9 and 234.3 HV.

Titanium foam was discovered to have an overall average microhardness value of 172.14 HV, lower than that of titanium metal utilized in industry.

Table 1 Micro hardness values of Titanium foam for 180mins.

Microhardnes s (HV)	Load Applie d (gm)
108.9	300
127.8	300
169.7	300
220.0	300
234.3	300

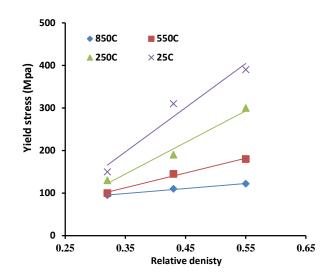


Fig 4 variation in yield stress with respect to relative density and temperature of testing.

For three groups of porosity volume percent, the compressive strength values of foams with distinct pore geometries are shown. The evolution of foam compressive strength at three different specific gravities revealed that compressive strength diminishes as the mean aspect ratio of the cells increases.

There are three unique regions on the stress-strain curves of sintered Ti foam samples at different temperatures for a given specific gravity: linear elastic, plateau, and compression. A flattening of the compression curve is seen in stress-yielding sintered specimens. The voltage drop effect is found to be more pronounced at lower temperatures than to higher ones because of the lower slip system in Ti. At higher temperatures, a different mechanism contributes to the Ti foam's deformation. Figure displays a scatter plot of the calculated Y_s and E_f values against the relative densities (R.D) (3 and 4). It has been shown that Y_s and E_f both grow with R.D, whereas Y_s and E_f both reduce with temperature.

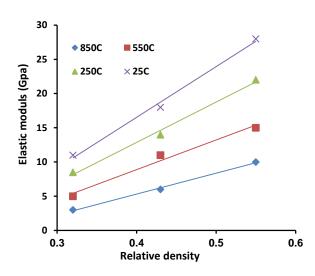


Fig 5 Modulus of elasticity vs relative density at a range of test temperatures.

Since the cell walls have ruptured or failed, and plastic deformation has taken place, there is a significant disturbance in the plateau region. Each individual layer of these cell walls may be damaged with relative ease by shearing and fracturing, and the cell walls can collapse at room temperature with little effort. Due to their increased flowability under light loads, foams' cell walls begin to disintegrate when their temperature is raised.

During the process of sharing and breaking, the cell walls are distorted incrementally. This results in a nearly flat and smooth plateau area. It turns out that the test temperature has a big impact on the plateau region's length and slope.

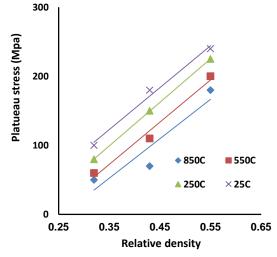


Fig. 6 Typical plateau stress versus density at various temperatures.

It thus follows that the relationship is a power law. Figure 6 shows that as R.D. increases, the σ_{pl} does as well, but that as test temperature rises, the σ_{pl} falls. Following the flat section, the compacted region started. Considering that these compression temperatures are slightly and early very much below recrystallization, dynamic expansion is not conceivable. When exposed to a compression test at temperatures ranging from room temperature to 850°C [13], the flexibility of the foams increases because of the ease with which dislocations can move due to the decrease in yield strain. In this procedure, foam samples are transformed into solids while the cell walls are crushed uniformly in successive layers.

4. Conclusion

Mechanical and microstructural changes were investigated in this work. After much deliberation, we arrived at these findings. The study's findings demonstrate that titanium foam may be successfully manufactured by the powder metallurgy method with sodium chloride as a space holder. If the right porosity is selected, the necessary mechanical qualities can be achieved. Titanium foam can be made to mimic the qualities of either cancellous bone (foam with a porosity of 80%) or cortical bone (foam with a porosity of 50%), depending on what is needed. An average porosity of 60-65 % is chosen and analyzed in further depth for implant applications in the dentistry field. Because of the presence of non-spherical spacer particles in the studied foams, they are anisotropic. When the powder combination is compacted, the space holder particles reorganize themselves, resulting in a non-isotropic distribution of the cell walls. Titanium foams have greater strength perpendicular to the direction of compression and less strength along the compression axis. Compression is where the material really shines, because even with very massive plastic deformations, the underlying structure remains intact. The foams' interconnected porosity makes it a good permeability to body fluids like blood.

References

[1] C. Torres-Sanchez, F. R. A. Al Mushref, M. Norrito, K. Yendall, Y. Liu, and P. P. Conway, "The effect of pore size and porosity on mechanical properties and biological response of porous titanium scaffolds," *Mater. Sci. Eng. C Mater. Biol. Appl.*, vol. 77, pp. 219–228, 2017.

[2] W. Xu *et al.*, "Porous Ti-10Mo alloy fabricated by powder metallurgy for promoting bone regeneration," *Sci.*

China Mater., vol. 62, no. 7, pp. 1053-1064, 2019.

[3] S. J. Hollister, "Porous scaffold design for tissue engineering," *Nat. Mater.*, vol. 4, no. 7, pp. 518–524, 2005.

[4] H. U. Cameron, I. Macnab, and R. M. Pilliar, "A porous metal system for joint replacement surgery," *Int. J. Artif. Organs*, vol. 1, no. 2, pp. 104–109, 1978.

[5] N. H. Hart, S. Nimphius, T. Rantalainen, A. Ireland, A. Siafarikas, and R. U. Newton, "Mechanical basis of bone strength: influence of bone material, bone structure and muscle action," *J. Musculoskelet. Neuronal Interact.*, vol. 17, no. 3, pp. 114–139, 2017.

[6] H. J. Rack and J. I. Qazi, "Titanium alloys for biomedical applications," *Mater. Sci. Eng. C Mater. Biol. Appl.*, vol. 26, no. 8, pp. 1269–1277, 2006.

[7] Q. Chen and G. A. Thouas, "Metallic implant biomaterials," *Mater. Sci. Eng. R Rep.*, vol. 87, pp. 1–57, 2015.

[8] X. Jian, L. Yongning, L. Yong, Q. Guibao, and L. Jinming, "The application of model equation method in preparation of titanium foams," *J. Mater. Res. Technol.*, vol. 13, pp. 121–127, 2021.

[9] N. Tuncer and G. Arslan, "Designing compressive properties of titanium foams," *J. Mater. Sci.*, vol. 44, no. 6, pp. 1477–1484, 2009.

[10] F. Jones, "Teeth and bones: applications of surface science to dental materials and related biomaterials," *Surf. Sci. Rep.*, vol. 42, no. 3–5, pp. 75–205, 2001.

[11] C. E. Wen, M. Mabuchi, Y. Yamada, K. Shimojima, Y. Chino, and T. Asahina, "Processing of biocompatible porous Ti and Mg," *Scr. Mater.*, vol. 45, no. 10, pp. 1147–1153, 2001.

[12] N. Murray, "Microstructure evolution during solid-state foaming of titanium," *Compos. Sci. Technol.*, vol. 63, no. 16, pp. 2311–2316, 2003.

[13] H. Bafti and A. Habibolahzadeh, "Production of aluminum foam by spherical carbamide space holder technique-processing parameters," *Mater. Eng.*, vol. 31, no. 9, pp. 4122–4129, 2010.