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Fabrication and characterization of titanium foam using powder compaction route and ammonium carbonate as a spacer

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Abstract
The increasing interest in titanium foam can be attributed to its unique mix of a porous structure and titanium material. The utilization of the space holder technology in fabricating this material was prevalent due to its cost-effectiveness, ease of operation, and ability to manipulate the pore structure and qualities freely. Titanium foam was manufactured using space holder techniques, employing ammonium bicarbonate (NH_4HCO_3) as a sacrificial material. A detailed investigation was conducted to examine ammonium bicarbonate's impact on titanium's characteristics. The specimens were subjected to characterization of pore characteristics and compressive properties using scanning electron microscopy (SEM). The findings indicate that the porosity of the titanium foam may be effectively adjusted by varying the quantity of (NH_4HCO_3) introduced. Additionally, the use of (NH_4HCO_3) does not impact the microstructure and phase components of the Titanium foam.
***Keywords**: Space Holder, Titanium Foam, Ammonium Carbonate, Compaction Route.*

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I. INTRODUCTION

Titanium and its alloys have been widely recognized as highly effective biomaterials despite their relatively high stiffness compared to cortical bone. This characteristic has been observed to contribute to failures in prosthetic implants, mostly due to t hestress shielding that arises between the implant and the bone [1]. Nevertheless, the mechanical properties of these materials can be adjusted and customized by utilizing cellular solids or foams, which involve manipulating factors such as open and closed porosity, pore size distribution, and pore morphology [2]. In the context of biomaterials applications, it has been observed that a porosity level of 50% exhibits mechanical qualities that are comparable to those of cortical bone. Still, a porosity level of 80% aligns with the behavior exhibited by trabecular bone. In addition, the presence of porosity is advantageous for facilitating the osseointegration process of the implant, which is a crucial determinant of the implant's long-term dependability [3]. The foams exhibit a notable strength-to-weight ratio, high stiffness, and effective impact energy absorption properties. Recently, there has been a significant focus on titanium foams due to their prospective utilization in structural capacities within the aerospace and marine sectors [4]. The titanium's high melting point and exceptional corrosion resistance are well-suited for applications involving elevated temperatures, such as catalyst substrates or heat exchangers. Cellular titanium foams with an open structure are currently employed in orthopedic and dentistry implants due to their exceptional mechanical characteristics and compatibility with biological systems [5]. According to reference [6], closed-cell metallic foams exhibit superior modulus, strength, and impact energy absorption properties. It is worth noting that the processing of titanium foams through the liquid metallurgical technique poses challenges due to the element's high melting point and its chemical reactivity with ambient gases.

In contrast, these materials have undergone processing using different methods within the field of powder metallurgy. In recent studies, various authors have employed a straightforward powder metallurgy technique to explore viable manufacturing routes to produce dependable titanium foams. Titanium foams have been effectively manufactured using space-holding materials, such as NaCl or other soluble substances. [7- 8]. Among these options, the most frequently employed method for sintering powder preforms involves using a gas-blowing agent or solid space holders. Several other materials have been used to produce foams, including polymeric polymers, magnesium [9], and urea [10]. One of the benefits associated with utilizing space holders is the ability to modify the pore morphology and porosity levels by manipulating space holder type and number. The space holder method is straightforward and cost-effective for producing titanium foam. The careful selection of suitable space holders is crucial because of their significant impact on the final properties of specimens, including cell shape, cell size, and porosity. Considering this, the synthesis of titanium metal foam was conducted in the present study utilizing starch as the solid-state space holder. The investigation focused on analyzing the foam's pore structure and its response to compression.

Ammonium bicarbonate (NH_4HCO_3) is widely regarded as an excellent space-holder material for porous titanium and titanium alloys. This is mostly owing to its capacity to undergo complete decomposition at very low temperatures, preventing undesirable reactions with the host particles. Numerous studies have investigated the impact of varying quantities of space holders on ammonium bicarbonate (NH4HCO3) properties when used as a space-holder material through the powder metallurgy technique.

The current study involves the fabrication of porous Titanium with different porosity levels by the space holder compaction technique. Ammonium bicarbonate (NH_4HCO_3) is utilized as the space-holder material.

II. METHODOLOGY

The study utilized titanium powder, with a purity of 99.9%. The average particle size of the titanium powder was measured to be 35 µm. This study employed ammonium bicarbonate (NH4HCO (3as a space holder. The mean diameter of ammonium bicarbonate particles was determined to be 300µm. The ammonium bicarbonate powder was provided by (Chema Jet Co., Egypt). This study used a polyvinyl alcohol (PVA) solution consisting of 5 wt.% PVA and 95% water were employed as an organic binder to impart adequate strength to cold compacted green pallets. The titanium alloy powders were manually blended in the presence of a five wt% binder, followed by compaction using a hydraulic press with a pressure of 550 MPa. The weight ratios between titanium powder and space-former were selected for final porosities ranging from 60% to 80%. The green pellet has a diameter of 25mm. The resulting green compact was then subjected to a two-step heat treatment.

The initial stage involved subjecting the compacts to a temperature of 70 °C for 2 hours to remove the space-holder material. Subsequently, the deals were further heated to 120 °C for 1 hour in a vacuum furnace to eliminate any remaining moisture.

During the second phase, the compact was subjected to a temperature of 1200°C and maintained at this temperature for 2 hours under an argon atmosphere within a tubular furnace to mitigate the oxidation of the pellets. To ensure the structural integrity of the compact during the melting and evaporation of the space holder, a constant heating and cooling rate of 5°C/min was maintained. Subsequently, the deal was allowed to cool to room temperature. The porosity of the sintered sample was determined using the Archimedes principle, while the pore structure was analyzed using a scanning electron microscope (SEM).

III. RESULT AND DISCUSSION

A green titanium pellet was manufactured utilizing the space holder approach, as shown in Figure 1. The shots had nominal porosities of 60% and 80% and were formed by combining titanium powder with ammonium bicarbonate. Following the removal of the space holder, the brown titanium pellets exhibited sufficient strength to be manipulated. Following the sintering process, as depicted in Figure 1**,** the specimens retain their porous structure and demonstrate satisfactory integrity without any noticeable alterations in size when observed. Two samples of sintered foams with nominal porosities of 60% and 80% were subjected to triplicate measurements to ascertain the overall open porosity. The ultimate porosity consistently exhibited a modest decrease compared to the projected value derived from including space-holder weight during the initial formation of the green pellet. However, it is important to note that the porosity exceeds 60% in all instances, a threshold widely recognized as the minimal requirement for the cortical bone to possess appropriate mechanical characteristics and for osseointegration. Consequently, the overall open porosity attained successfully encompasses the intended objective, with a maximum porosity level reaching 80%.

Figure 2 displays scanning electron microscopy (SEM) images of titanium foams sintered with varying nominal porosities of 60%, 70%, and 80%. The low-magnification image revealed that the macropore size ranged between

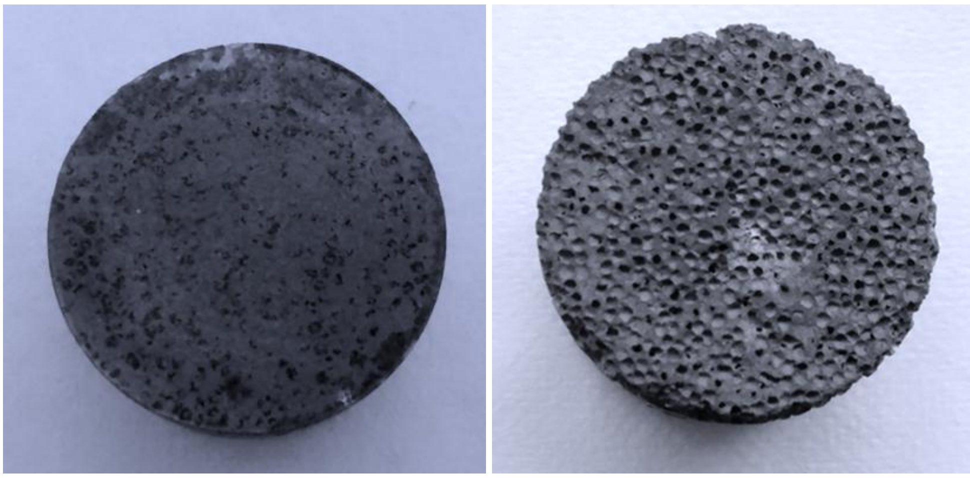

Figure 1: Green compact pellet and titanium foam after sintering.

About 300µm, which aligns with the initial size of the NH_4HCO_3 space holder crystals. This observation suggests that the macropores do not undergo any collapse or substantial reduction in size throughout the compaction, NH_4HCO_3, or sintering stages. Holes with a diameter over 300 µm are essential for facilitating direct osteogenesis in the implant since they e**nable** vascularization. The high-magnification photo reveals the occurrence of partial melting and sintering of the titanium powders. The macropore-wall structure of the three porosities under consideration has a rough surface and comprises sintered powders with favorable metallurgical joining.

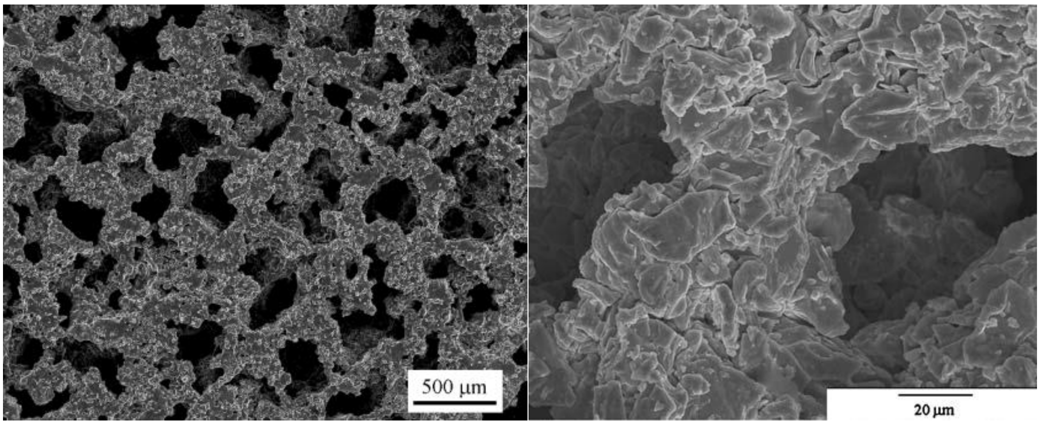

Figure 2: SEM of titanium foam at different magnifications.

Figure 3 illustrates both elastic and plateau zones in the sintered compact. The stress at which the table was seen, referred to as the plateau stress, was roughly 275 MPa. Additionally, the energy absorbed up to a strain level of 25% was estimated to be around 55 MJ/m3. Their study documented a plateau stress of about 150 MPa for titanium foam with a porosity level of 46%. Furthermore, the presence of foam indicated the existence of a well-defined plateau region. However, this phenomenon was observed only when the porosity level was beyond the threshold of 60%. The elevated stress level seen in this investigation, compare**d** to the findings published in previous work [9], may be related to the presence of finely dispersed micro-porosity zones with a morphology inside the matrix. From a mechanical perspective, porous patches (as seen in Figure 3) can lead to an anisotropic response because of the increased stress concentration at sharp tips and edges.

Nevertheless, in the current study, this element did not hurt the properties, such as plateau stress and energy absorption capacity of the sintered foam. This observation is further supported by the comparatively higher parameter values (280 MPa) obtained in this study, in contrast to those associated with pores of comparable porosity levels [9]. Hence, the treated foam exhibits a distinctive amalgamation of elevated plateau stress and exceptional impact energy absorption properties, suggesting enhanced potential for various applications [11].

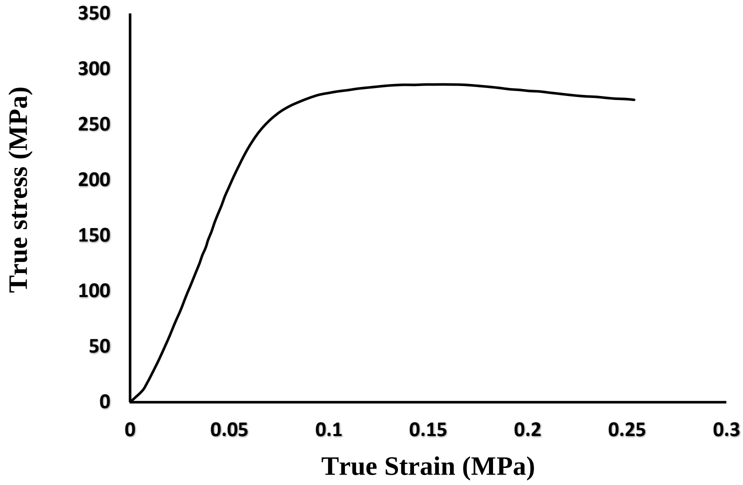

Figure 5: Stress–strain diagram for the titanium foam.

IV. CONCLUSION

In the present work, a methodology aimed at elemental metal powder can produce Titanium foams. The utilization of ammonium bicarbonate particles as a space holder in the production of titanium foam has been observed.

The management of foam density can be achieved by manipulating the space holder content within the green compact. The size of foam cells can be regulated by using the dimensions of the space holder.

Titanium foams with a porosity of 80% have been effectively manufactured using ammonium bicarbonate as a spacer.

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