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## Volume change of macropores of titanium foams during sintering

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**Abstract:** The porosity of titanium foams obtained from the space holder technique was theoretically analyzed in the cases of volume shrinking, retaining and expanding during sintering. The relationship between porosity and spacer content was compared under different conditions. The kind of volume change of macropores during sintering was discussed. The results indicate that the relationship between porosity and spacer content depends on the decreased volume of macropores and the volume of micropores in cell-walls in the first case, while the porosity will be greater than the spacer content for the other two cases. It proves that the volume change of macropores during sintering decreases based on theory and practice.

Key words: porous material; titanium foam; space holder technique; sintering; volume change; porosity

## **1** Introduction

Titanium foams are a kind of novel functional materials. They combine the advantages of porous structure and titanium. Compared with traditional dense titanium, they are attractive for aerospace, automobile, biomedical and chemical catalytic applications, due to their excellent mechanical properties, superior corrosion resistance and good biocompatibility [1]. It is well known that all these properties directly depended on their pore structure. Hence, it is important to study the pore structure of titanium foams.

The salient structural features of a metal foam are its porosity, cell topology (open cells, closed cells), cell size and cell shape and anisotropy [2]. Among them, the porosity was considered as the most important feature [3]. Its value is always designed to be equal to the spacer content when the titanium foams were obtained from the well established space holder technique. This method utilizes a fugitive solid material to create the desired macropores. The fugitive solid material was the so-called space holder such as familiar carbamide [4], ammonium bicarbonate [5] and sodium chloride [6]. Recently, there have been new space holders as starch [7], saccharose [8] and cenosphere [9]. In the authors' previous work, titanium foams with porosity in the range of

50.2%-71.4% were successfully prepared by using acicular carbamide as a space holder when the spacer content was in the range of 60%-80% [10]. However, the results show that the final porosity of the foams was lower than the spacer content. It is similar to the cases when the spacer size was varied in Ref. [11]. However, there is still a lack of a further study to provide a more detailed analysis from the point of view of the volume change of macropores during sintering. TORRES et al [12] speculated that it was the consequence of metallic framework shrinkage during sintering. Furthermore, this view was referenced from Ref. [13], which showed axial and radial shrinkages of the measured titanium foams after sintering. As a result, the porosity was lower than the spacer content. On the other hand, while this work puts forward that the macropores remain nearly unchanged or even tend to grow during the sintering process, the other researchers' work indicated that the macropores shrink their volume during sintering with the result of the porosity lower than the spacer content [14]. However, it is unknown by far that what kind of volume change of macropores would occur during sintering and what kind of relationship between the porosity and the spacer content will be obtained.

Therefore, the aim of the study was to determine the volume changes of macropores that occur during sintering when using the space holder technique. Given

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that a considerable number of experiments had been conducted in the authors' previous work, part of their results will be used in this work instead of conducting new experiments and other authors' works will also be combined to provide a detailed analysis.

## 2 Processing and characterization of titanium foams

Initially, the pre-calculated amount of titanium powders and spacer particles (Fig. 1 in Ref. [10]) was mixed using a mortar for 2-3 min. Then, the mixture was uniaxially cold pressed at ~200 MPa using a cylindrical steel die (diameter 16 mm; height 50 mm) to obtain green compacts. The dwelling time is set to be a fixed value of 45 s. Subsequently, two steps of heat treatment were applied to these compacts in a carbon vacuum furnace as follows: Firstly, a low heating rate was used to remove spacer particles at 460 °C under vacuum  $(10^{-1}-10^{-2} \text{ Pa})$ , followed by furnace cooling. Secondly, with argon of 99.99% purity protective atmosphere to avoid oxidation, the scaffolds were sintered at 1250 °C for 2 h, followed by furnace cooling (for details, Figs. 2 and 3 in Ref. [10]). Figure 1 shows the relationship between the porosity and spacer content in the case that the content or size of space holder particles varied in Refs. [10,11].

Figure 2 shows the macromorphology and micro morphologies of sintered foams and green compact. The three sintered foams were the parallel samples (i.e., the



**Fig. 1** Porosity of titanium foams prepared with different spacer contents and sizes in Refs. [10,11]

same starting materials and preparation process). It can be seen that the external heights of sintered foams were lower than those of green compact, regardless of whether the direction is parallel or perpendicular to that of compact pressing (Fig. 2(a)). It is assumed to be a consequence of metallic framework shrinkage during sintering. The SEM image of surface morphology of a green compact show that it contains titanium powders and spacer particles (Fig. 2(b)). The compact with spacer particles completely removed was called scaffold. The experimental results indicated that providing an accurate characterization of a spacer particle and its removal hole



**Fig. 2** Macromorphology and micromorphology of titanium foams before and after sintering: (a) One green compact and three sintered parts with same spacer content; (b) SEM image showing surface morphology of green compact; (c) BES image showing surface morphology of green compact; (d, e) SEM images of foam sample structures from surface (Directions of white and black arrows represent formation of macropores and micropores in sintered foams, respectively. SEM and BES images of green compact well depict morphology of hole generated from spacer particle removal)

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