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Effects of SiC Nanoparticles on the Properties of Titanium-Matrix Foams Processed by Powder Metallurgy

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Abstract: Metal-matrix foams are used widely for structural applications such as impact energy absorption, vibration resistance and weight reduction. In this study titanium nanocomposite foams with different porosity percentages were produced using TiH_2, as foaming agent, by powder metallurgy technique. At first, raw materials including titanium powder and different weight percentages of SiC nanoparticles were mixed and then different amounts of TiH_2 were added to the mixture. The mixture was compacted at 200 MPa. The samples were heat treated in two stages, first at 400 °C for 1 h, as a partial sintering, and then at 1050 °C for 2 h, as foaming treatment. Mechanical and structural properties such as compressive strength, energy absorption, porosity percentage and relative density of samples were measured and compared together. Thermo gravimetric analysis (TGA), differential thermal analysis (DTA), scanning electron microscopy (SEM) and X-ray diffraction (XRD) were performed on foaming agent and samples. The results showed uniform distribution of SiC nanoparticles in titanium matrix and also homogenous pore structure. It was concluded that with increasing SiC weight percent, relative density is increased to 0.43 in the sample with 1.5 wt % SiC. Besides, the measured compressive strength of samples was in the range of 14.4–32.3 MPa. Moreover, it was concluded that the energy absorption of samples increases with increasing SiC nano particles up to 33.09 MJ/m^3.

Keywords: foam; titanium-matrix composite; SiC nanoparticles; Ti-SiC; powder metallurgy

1. Introduction

Metallic foams are a group of materials with good thermal, mechanical, physical and electrical properties, which have already been developed due to their good potential for different applications [1]. The majority of the metal-matrix foams developed so far are used for structural applications such as impact energy absorption or weight reduction [2]. Titanium-matrix foams are one of these materials. Many investigators have conducted much research on porous titanium-matrix foams. Ahn et al. [3] and Xue and Zhao [2] produced highly porous titanium-matrix foams by powder metallurgy and showed that they have good mechanical properties. Fan et al. [4] found that the graded porous titanium scaffold could be used for engineering scaffolds in load bearing conditions. Dunand [5] described different ways of processing of titanium foams. Moreover, Aşık and Bor [6] discovered that mechanical properties of foams are in the suitable range for many applications. They suggested that, because of the novel mechanical properties, such as good strength and stiffness, low density and good corrosion resistance, the materials are very useful in many applications such as aerospace and submarine vehicles. The other usages of these foams are in implants and orthopedic applications because of their excellent biocompatibility and mechanical properties and chemical resistance. Dunand [5] reported that titanium

foams, in addition to excellent mechanical properties, have biocompatibility. Moreover, Gu et al. [7] found that porous Ti alloys show excellent bioactivity. Xiong et al. [8] fabricated porous titanium implants by three-dimensional printing.

The tribological and mechanical properties of titanium foams can be improved by reinforcing them with ceramics. Nano particles such as SiC improve noticeably the strength of titanium foams, especially at high temperatures [9].

The particles characterized in previous studies as reinforcement of titanium foams are ceramics, oxides, and intermetallic compounds. Ceramic reinforcement particles have been used in the studies of Choe et al. [10] who worked on TiC, Lee et al. [11] on TiN, Yoshida et al. [12] on TiO_2, Karbalaei Akbari et al. [13] on TiB_2, Liang et al. [14] on Si_3N_4, and Poletti et al. [9] on SiC. Eriksson et al. [15] produced Ti-TiB_2 composites by spark plasma sintering and Godfrey et al. [16] investigated microstructure and tensile properties of mechanically alloyed Ti-6A1-4V with boron oxide additions. Oxide reinforcement particles such as Al_3O_2 and R_2O_3 (with R = rare element) have been used by Liu et al. [17] and Hieda et al. [18], respectively. Moreover, the effects of intermetallic compounds additions such as TiAl and Ti_5Si_3 particles have been investigated by Ma et al. [19] and Sumida and Kondoh [20].

Titanium-matrix foams have recently been produced through powder metallurgy methods [2]. Because of high melting point of titanium (1670 °C), it is difficult to produce foam in liquid state. Besides, high vacuum and high temperature processing equipment is required for casting titanium foams. However, with powder metallurgy method, titanium foams can be produced at low temperatures and with low costs [5].

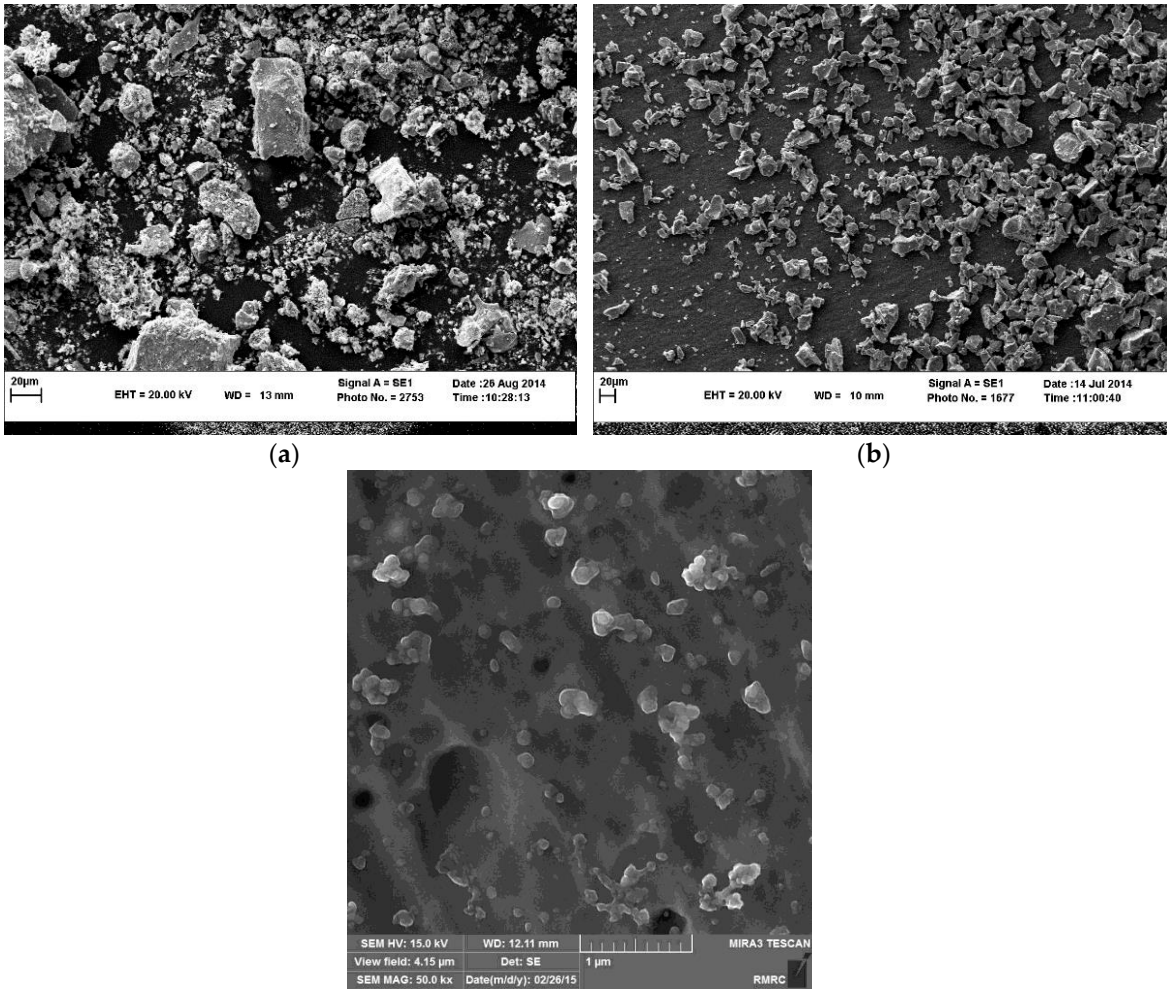
The primary results showed that the percentage of titanium and ceramic nanoparticles affect the porosity, density and compressive strength of titanium-matrix foams [2]. It has been reported that with increase in the percentage of nanoparticles the density and compressive strength increase [21].

In the present study, titanium-matrix foams reinforced with SiC nanoparticles have been produced using TiH_2 particles, as foaming agent, by powder metallurgy method. It is expected that SiC particles improve the compressive strength and energy absorption and other properties of titanium foams.

2. Experimental Procedure

2.1. Materials

Commercial titanium powder (99.9% purity), SiC nano particles (99% purity) and TiH_2 powder (98% purity) as foaming agent were supplied by Iran Nanosany Corporation (Tehran, Iran). Morphologies of the powders are shown in Figure 1. The titanium powders with irregular shape were lower than 60 μm, with a nominal average size of 45 μm. The SiC particles were characterized with spherical shape, apparent density of 3.5 g/cm^3 and size between 40 nm and 70 nm. Particle size distribution of SiC powder is shown in Figure 2. TiH_2 powder with an average particle size of 40 μm was used in this study.



(**a**) (**b**)

(**c**)

Figure 1. SEM (Scanning Electron Microscopy) images showing the morphology of the: (**a**) Ti powder; (**b**) TiH_2 powder; and (**c**) SiC nanoparticles.

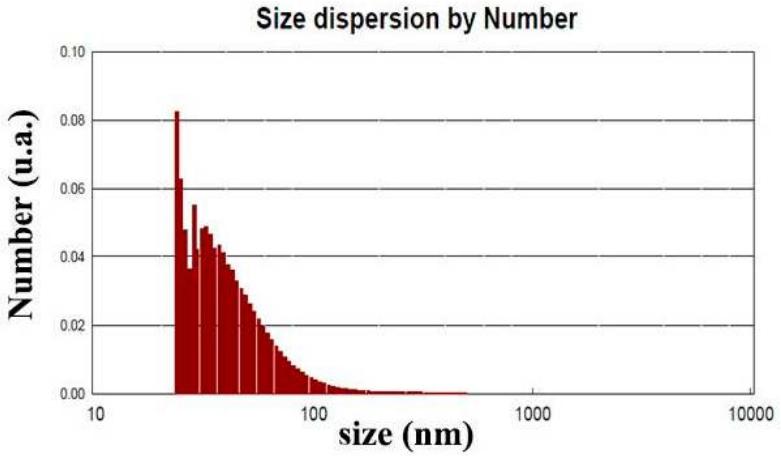


Figure 2. PSA (Particle Size Analysis) of SiC nano powders.

2.2. Procedure

In this study, powder metallurgy process was used to fabricate titanium-matrix foam. First, titanium powders with different SiC weight percentages (0.5% and 1.5%) were uniformly blended.

Then, each mixture was divided into three parts and each part was blended in a Lab mechanical blender with 5 wt %, 10 wt % and 15 wt % TiH_2, separately. Additionally, pure titanium foams were produced for comparison purpose.

In the next step, the mixtures were compacted at 200 MPa pressure. The compacted samples were first sintered at 400 °C for 1 h, then at 1100 °C for 2 h under argon atmosphere and then furnace cooled to room temperature. The fabricated titanium-matrix foams had a cylindrical shape with 10 mm diameter and 15 mm height. Porosities of the fabricated foams were measured according to Archimedes' rule.

The microstructure of titanium-matrix foams were characterized by a field emission scanning electron microscopy (FESEM) MIRA3 TESCAN (TESCAN, Brno-Kohoutovice, Czech) equipped with an energy dispersive X-ray spectrometry (EDS). The thermal characterization of TiH_2 was analyzed by thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) techniques.

Relative density and porosity content of samples were measured using Equations (1) and (2):

$$\text{Relative density} = (\rho_f/\rho_s), \tag{1}$$

$$\text{Porosity (\%)} = [1 - (\rho_f/\rho_s)] \times 100, \tag{2}$$

in which, ρ_f is the apparent density of nanocomposite foams obtained using Archimedes method and ρ_s is the theoretical density of the samples.

Compressive strength of each sample was measured according to JIS H 7902 standard (2008–09) using a universal testing machine ZWICK (Z250) (ZWICK, Ulm, Germany). To avoid buckling of samples during pressure test, the ratio of height to diameter of samples chosen was 1.5. The load was applied parallel to the long axis of samples and the strain rate was selected as 10^{-3} s^{-1}.

Energy absorption of the samples was determined using calculation of the area under stress-strain curve up to their own densification strain according to JIS H 7902 and Equation (3):

$$\text{Energy absorption} = \int_0^{\varepsilon D} \sigma d\varepsilon \ (MJ/m^3). \tag{3}$$

3. Results and Discussion

3.1. TGA Results

Figure 3 shows thermal analysis results of TiH_2 powders. As is shown, the decomposition of TiH_2 occurs in two steps. The first step happens at 450 °C and the second occurs at around 580 °C. However, as the DTA results show the decomposition at 450 °C is so week that there is no peak in the TGA curve. Most of the TiH_2 decomposition occurs at 580 °C, so a strong peak is observed in the TGA curve. Finally, the decomposition finishes at 630 °C. Therefore, the best temperature for foaming is above 630 °C, at which TiH_2 can completely decomposes. Due to the two possible non-equivalent locations of hydrogen atoms in Ti structure, one in tetrahedral, and the second in octahedral structure, the double-peak curve is formed [22]. As is shown in the TGA curve, the TiH_2 powder is oxidized during sintering at 1000 °C and an oxidized film forms on the TiH_2 powders during metal foaming.

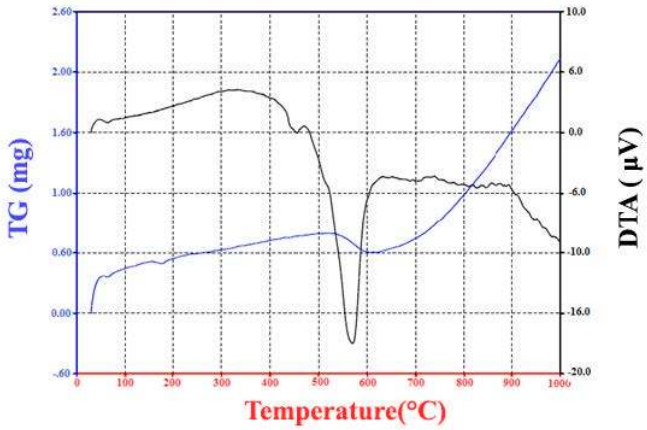


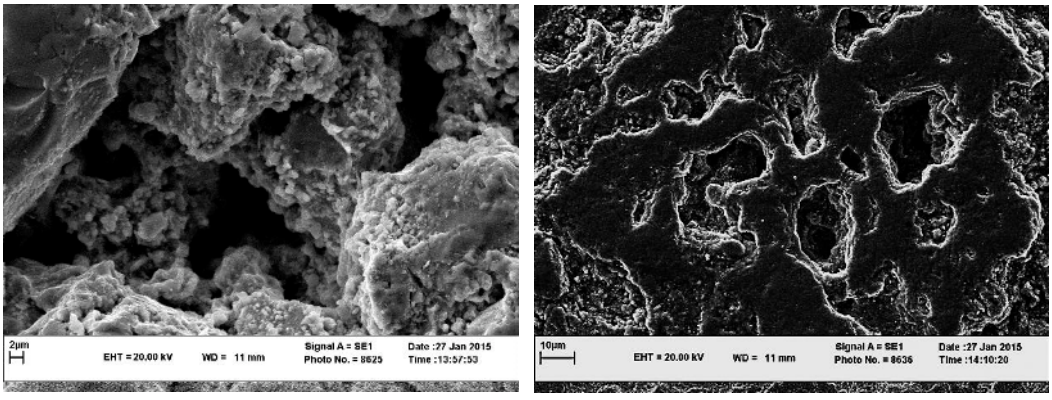
Figure 3. DTA (Differential Thermal Analysis) and TGA (Thermo Gravimetric Analysis) of TiH_2 powder.

3.2. Characterization of Produced Foams

Figure 4 shows SEM images of titanium-matrix foams with different percentages of SiC (0.5 and 1.5 wt %) sintered at 1050 °C. As shown in the figure, the specimens have structures at which pores are uniformly distributed. Size of the porosities is in the range of 20–150 μm and the structure is closed cell. SEM images show uniform distribution of nano particles in the samples. EDS results indicate that the foams contain two phases: Ti and SiC, as shown in Figure 5.

XRD pattern of titanium-matrix foams are shown in Figure 6. The results indicate that the samples processed by this method have three phases: Ti, C and Si_3Ti_5. In fact, SiC nanoparticles have a reaction with titanium matrix during sintering and Si_3Ti_5 particles are the product of the reaction [9]. Dimensions of the particles are dependent on the SiC particles.

Figure 7 shows the effects of contents of TiH_2 and SiC nanoparticles on porosity. As shown in the figure, for each series of samples with increase in weight percent of TiH_2 in the constant foaming temperature, the percentage of porosity is increased due to more gas production caused by decomposition of more TiH_2 particles. Besides, by increasing TiH_2 weight percent the porosities are more adjoined due to more entrapped gas between the powders [3,7,21]. With increasing SiC weight percent in the constant TiH_2 content and foaming temperature, the percentage of porosity is decreased. This is because of increasing the relative density and strength of the samples. In this case, more gas pressure is required to create porosity.



(**a**) (**b**)

Figure 4. SEM images of Ti matrix foams sintered at 1050 °C: (**a**) with 1.5 wt % SiC; and (**b**) with 0.5 wt % SiC.

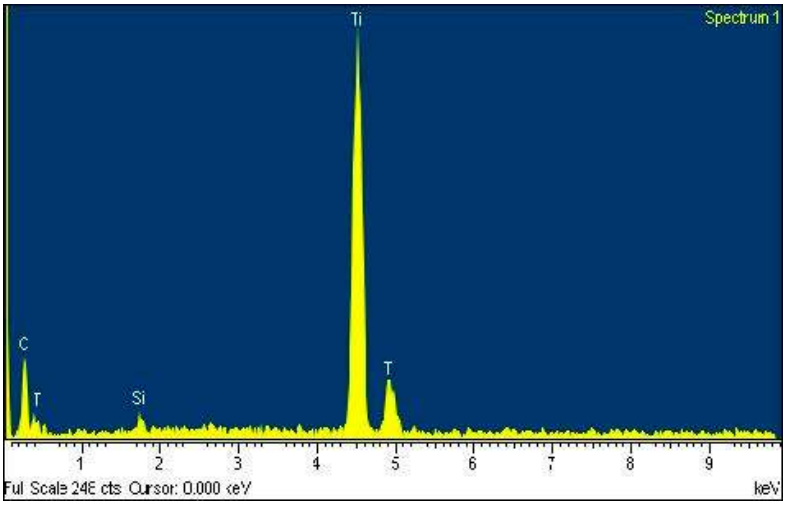


Figure 5. EDS (Energy Dispersive Spectroscopy) spectra of Ti matrix foam.

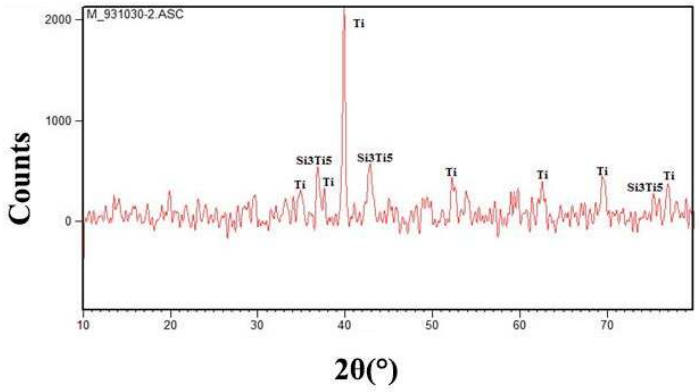


Figure 6. Typical XRD (X-ray diffraction) pattern of Ti-matrix foam.

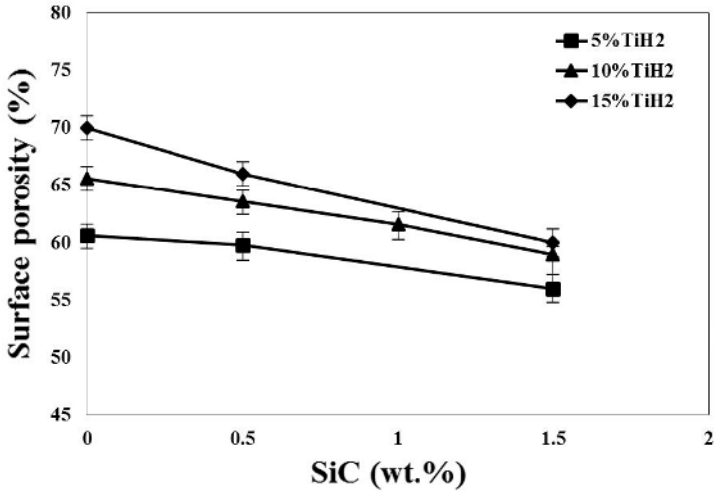


Figure 7. Effect of TiH_2 weight percentage on porosity.

3.3. Compressive Properties and Energy Absorption of Produced Foams

Figures 8 and 9 show the compressive behavior of titanium-matrix foams with 0.5 wt % and 1.5 wt % SiC. Due to the low weight percent of SiC, titanium-matrix foams exhibit no brittle performance

and have homogenous behavior during compression test; therefore, no sudden break of samples is seen. As the weight percent of SiC particles increases, the compressive strength of titanium-matrix foams increases. However, increasing weight percentage of TiH_2 causes reduction in the compressive strength of samples because of the presence of more porosities and reduction in the relative density of samples. Moreover, comparing with the pure titanium foam (Figure 10), the titanium-matrix foams show higher compressive strength due to the higher strength of SiC particles and more relative density of the foams. Similar results on the mechanical properties of titanium foams reinforced by different ceramic particles have been reported in the literature [2,9,16,21].

Table 1 lists compressive strength, energy absorption and other properties of titanium-matrix foams. From the results, it is known that the energy absorption of foams is dependent on the relative density and the compressive behavior of samples. With increasing weight percentage of SiC particles, the relative density and energy absorption of titanium-matrix foams increase. However, with increasing percentage of TiH_2, the relative density and energy absorption decrease. In contrast to Sample 8, although the sample with 1.5 wt % SiC and 15 wt % TiH_2 has more porosity and less relative density, it shows better ductility, so high energy absorption is obtained. Table 1 shows that the sample with 1.5 wt % SiC and 5 wt % TiH_2, in comparison with the other samples, has the highest energy absorption because of its more strength and ductility.

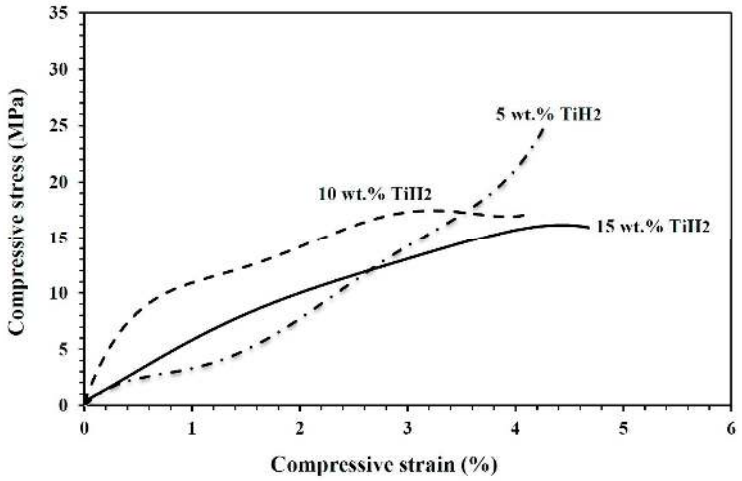


Figure 8. Compressive behavior of Ti matrix syntactic foam with SiC powder weight fraction of 0.5%.

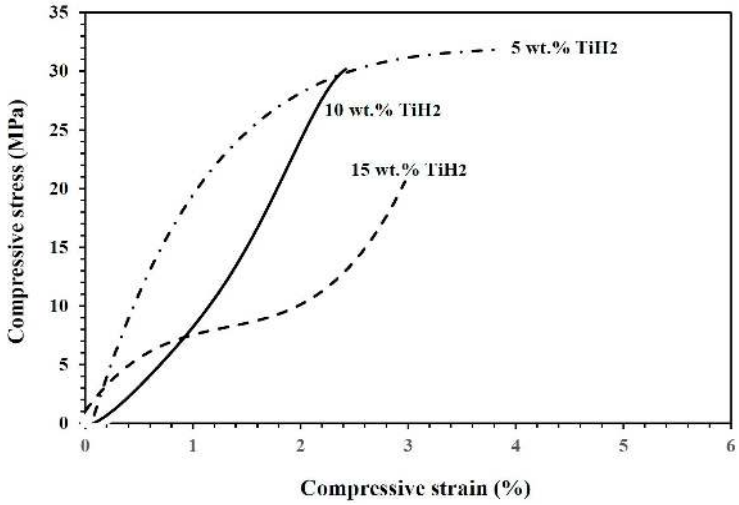


Figure 9. Compressive behavior of Ti matrix syntactic foam with SiC powder weight fraction of 1.5%.

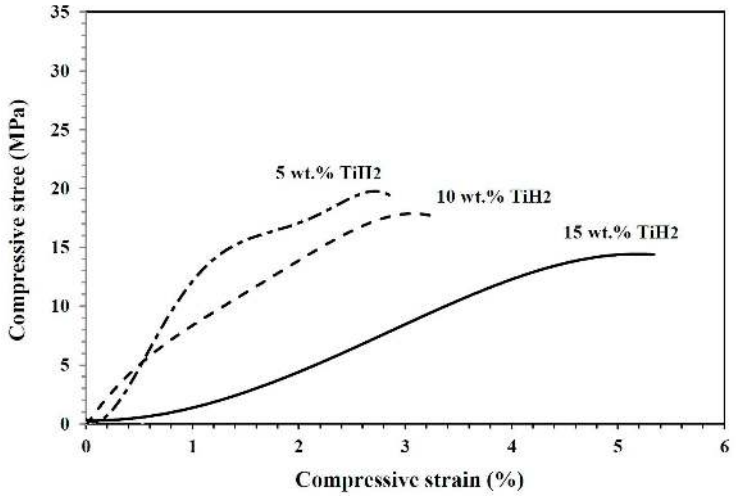


Figure 10. Compressive behavior of pure Ti foam.

Table 1. Porosity, relative density, compressive strength and energy absorption of Ti matrix foams.

Sample	SiC (wt %)	TiH_2 (wt %)	Relative Density	Porosity (vol %)	Compressive Strength (MPa)	Energy Absorption (MJ/m^3)
1	0	5	0.39	60.6	19.83	11.33
2	0	10	0.34	65.6	18.00	11.22
3	0	15	0.30	70.0	14.46	7.10
4	0.5	5	0.40	59.8	24.31	21.63
5	0.5	10	0.36	63.6	17.77	8.47
6	0.5	15	0.34	66.0	16.51	7.72
7	1.5	5	0.43	57.2	32.30	33.09
8	1.5	10	0.41	59.0	30.40	5.74
9	1.5	15	0.40	60.0	20.37	8.97

4. Conclusions

In the present study, titanium-matrix foams with SiC nanoparticles were produced through powder metallurgy technique and employing TiH_2 powder as foaming agent. The following results were achieved:

1. Using this procedure, foams with 57 vol % to 70 vol % porosity were obtained.
2. The pore size was in the range of 20–150 μm and the structure was closed cell.
3. Thermal analysis results of TiH_2 powders indicated that the decomposition of TiH_2 starts at 450 °C and finishes at 630 °C. Therefore, the best temperature for foaming was determined as 630 °C.
4. It was found that the compressive strength of samples was in the range of 14.4–32.3 MPa.
5. With increasing SiC weight percent, relative density was increased to 0.43 in the sample with 1.5 wt % SiC.
6. The energy absorption of samples was increased with increasing SiC weight percent up to 33.09 MJ/m^3.

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Author Contributions: Seyed Abdolkarim Sajjadi and Abolfazl Babakhani conceived and designed the experiments; Ensieh Edalati performed the experiments; Ensieh Edalati, Seyed Abdolkarim Sajjadi and Abolfazl Babakhani analyzed the data and contributed reagents/materials/analysis tools; Seyed Abdolkarim Sajjadi wrote the paper.

Conflicts of Interest: The authors declare no conflict of interest.

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