

Effect of Particle Size on the Compressibility and Sintering of Titanium Powders

I. Kaseb, M. Moazami-Goudarzi* and A. R. Abbasi

Department of Materials Engineering, Science and Research Branch, Islamic Azad University, Tehran, Iran

ARTICLE INFO

Article history:

Received 12 July 2019

Revised 18 August 2019

Accepted 17 September 2019

Keywords:

Powder metallurgy

Titanium

Compressibility

Compaction equations

Sinterability

ABSTRACT

In this research, the effects of the powder particle size on the compaction and sintering of Hydride–Dehydride titanium powders are investigated. Commercially pure titanium powders with three different size ranges were utilized. Compaction was accomplished under applied pressures of 200 to 650 MPa. Sintering was carried out at 1100 to 1400 °C temperatures. The compressibility behavior of the differently-sized powders was studied by measuring the density of the green compacts. The microstructure of the produced compacts was studied using scanning electron microscopy. Results showed that the small powders have the least compressibility. The compressibility data was analyzed and studied by common compaction equations. The modified Heckel equation showed the best correspondence. In addition, measuring the density of the sintered compacts showed that the small powders had the highest sinterability. The highest amount of sintered density (98% theoretical) was attained for the small powders compacted under 650 MPa and sintered at 1400 °C. However, the sintering temperature of 1200 °C was recognized as the most appropriate temperature for the middle and large-sized titanium powders. The results of the hardness tests showed that the appropriate mechanical properties could be attained for commercially pure titanium powder compacts by vacuum sintering accomplished at the optimum sintering circumstances.

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1. Introduction

Titanium and its alloys are considered as advanced materials due to their multiple applications, especially in aerospace industries. It has high corrosion resistance and could be widely used in chemical industries, due to the passive oxide layer on its surface [1-4]. Nevertheless, titanium's high price, compared to other structural materials, limits these applications. Accordingly, using economical production methods, such as powder metallurgy (PM), can be useful for producing titanium parts. PM is the technology of pressing and consolidating metal powders with fine particles into specific shapes. It consists of two important steps, i.e. compaction and sintering [5]. Compaction, compressibility and powder consolidation

are essential issues in PM applications. Hadadzadeh et al. [6] reported that the relative density and compressibility of the green compacts of titanium are directly proportional to the applied pressure.

It is necessary to study the effects of the influential parameters of powder particles on compressibility. Hence many researchers have attempted to match their experimental data with compaction equations presented by Ge [7], Panelli–Filho [8], Heckel [9] to predict the compressibility behavior of the powders [10]. Machaka and Chikwanda [11] studied the correlation of compaction equations for commercially pure (CP) titanium powders Grade 2 with a particle size of 22 to 41 μm. They reported that the majority of the equations could describe the compaction behavior.

* Corresponding author

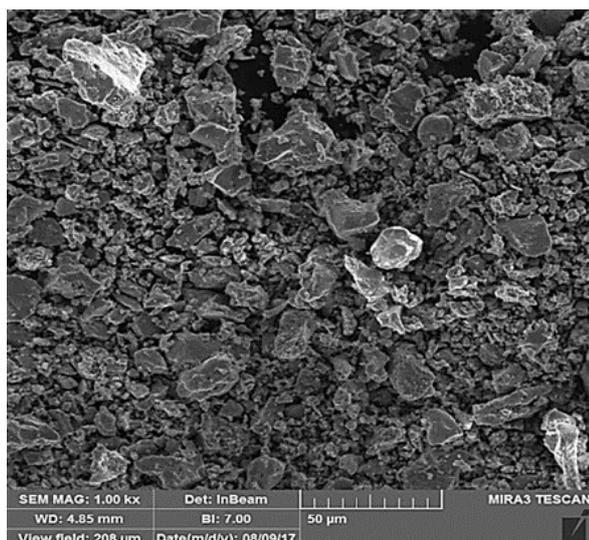
E-mail addresses: moazami@srbiau.ac.ir (M. Moazami-Goudarzi)

Sintering may be considered as the process by which an assembly of particles, compacted under pressure or simply confined in a container, chemically bond themselves into a coherent body under the influence of an elevated temperature. Sintering is one of the most critical steps of powder metallurgy [12]. Before sintering, the powder particles in green compacts have mechanical binding, which is not strong enough. Sintering makes this binding strong so that solid and hard compacts can be made [13]. It is well known that if the relative density of green compacts is elevated, it will result in more sinterability. The parameters that affect sinterability are the temperature, particle size of the powder, green density and sintering atmosphere [14, 15]. Bolzoni et al. [16] studied the compressibility and sinterability of different titanium alloy powders and reported that the relative density of the sintered compacts increased by increasing the sintering temperature. According to Quian [17], by studying the influence of titanium powder particle size variation on the relative sintered density for Hydride–Dehydride (HDH) titanium powder, it can be concluded that if the titanium powder size decreases, the relative sintered density will increase.

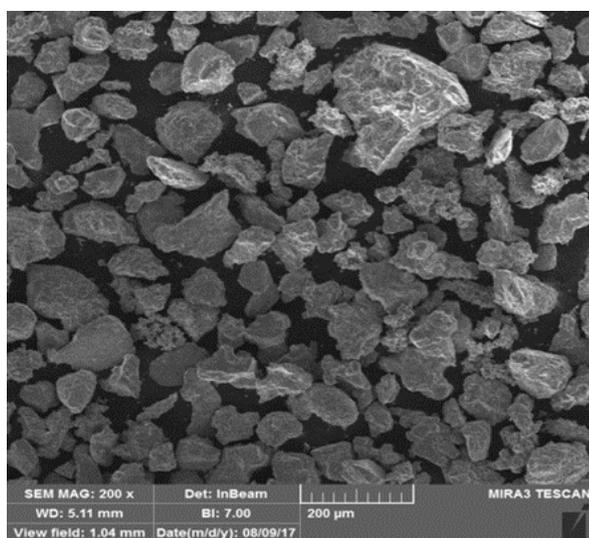
Compressibility of powder particles is important in the effective and economical production of PM parts. As a result, improvement in compressibility is a significant industrial objective. Compressibility data for various metal powders such as iron, aluminum and copper particles has been widely provided in the literature by different researches. However, the compressibility of differently sized titanium powders and its relation with their sintering behavior have been rarely reported. In the present work, the effects of the titanium powder particle size, compression pressure, and sintering temperature on the density, microstructure and hardness of the produced compacts are investigated. Before sintering, the correlation between the relative density of the compacts and the uniaxial compaction pressure is experimentally evaluated and compared with that predicted by some commonly used compaction equations.

2. Experimental Procedure

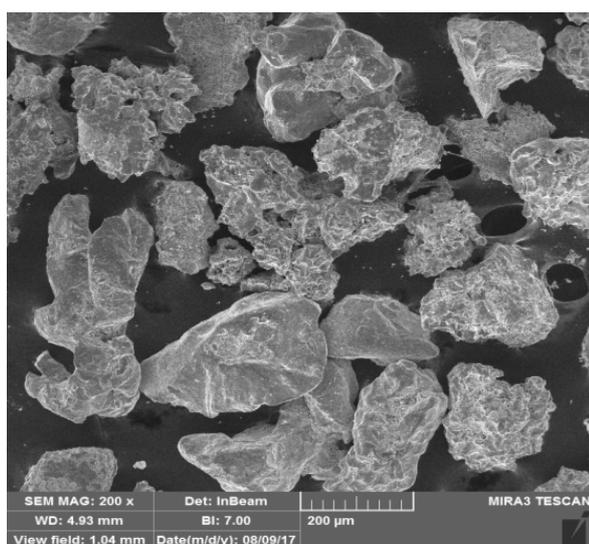
The HDH CP titanium powders were classified into three different particle sizes: small (less than 25 μm), middle (38 to 100 μm), and large (150 to 250 μm). The morphology of the different powders was examined by MIRA3 TE-SCAN scanning electron microscope (SEM). Typical SEM images of different particle size ranges of CP titanium powders are shown in (Figs. 1a–c). All powders are blocky and angular. A FRITSCHE ANALYSETTE 22 laser particle size analyzer was utilized to determine the average particle size and size distribution of the classified powders. The apparent density and flowability of the powders were determined by using the Carney funnel according to ASTM B 213 and ASTM B 417 standards, respectively. All three classified titanium powders were cold pressed in a cylindrical steel die (15 mm inner diameter) on a single acting hydraulic press, while zinc stearate was used as the die lubricant. The compacting pressures were 200, 350, 500, and 650 MPa. The densities of the green compacts were determined by measuring their mass and volume [11]. The values of the relative densities were used for evaluating the compressibility of different compacts. The green compacts were sintered at 1100, 1200, 1300, and 1400°C for 90 min in a vacuum furnace up to 10^{-2} Pa with the heating and cooling rates of 5°C/min [16]. The same method, that was used for measuring the density and relative density of the green compacts, was also applied for sintered compacts. For all of the tests, the average value of 3 measurements was considered. The microstructure of the sintered compacts was studied by scanning electron microscopy. Hardness measurements were conducted using an ESE WAY DVRBM Vickers hardness tester with a load of 98 N applied for 15 seconds.



(a)



(b)



(c)

Fig. 1. Typical SEM micrographs of titanium powders with different particle sizes: (a) small (<25 μm), (b) middle (38–100 μm) and (c) large (150–250 μm).

3. Results and Discussion

3.1. Powder characteristics

It is well known that the characteristics of the powders such as morphology, microstructure, apparent density, flowability, size, and size distribution influence the compaction behavior and consolidation process of PM products. Most of these characteristics are influenced by the powders' material [10]. The results of the particle size analysis as well as the measured apparent density and flow rate of the classified powders have been presented in Table 1. Large powders (150–250 μm) had the highest amount of apparent density, and flowability was higher when the particle size increased. According to Table 1, small powders (< 25 μm) had a high amount of friction between their particles due to their high amount of specific surface area [10, 18]. As a result, these powders did not move through the Carney funnel's aperture and their flowability and apparent density could not be measured by this method. It can be seen that the large powders (150–250 μm) exhibited the highest amount of the apparent density. This was because of the less bridging of the particles when the particles' size increased. Larger particles had lower surface friction due to their lower specific surface area. Generally, the flowability becomes better when the particle size increases [10].

Table 1. Characteristics of as received HDH titanium powders

Particle size range/ μm	Designation	Oxygen content/wt %	D ₁₀ / μm	D ₅₀ / μm	D ₉₀ / μm	Apparent Density / $\text{g}\cdot\text{cm}^{-3}$	Flowability / $\text{s}\cdot(50\text{ g})^{-1}$
< 25	Fine	0.78	3	11	26	-	-
38 to 100	Middle-sized	0.62	15	67	103	1.68	10.5
150 to 250	Coarse	0.51	155	180	258	1.75	9.2

3.2. Compressibility of powders

The density of green compacts was measured and the relative density was calculated for each compact. The relative density variation as a function of compacting pressure is shown in (Fig. 2). Increasing the applied pressure elevated the relative density of all of the powders. The small or fine powders showed the

least compressibility, and the large or coarse powders had the highest amount of compressibility. According to (Fig. 2), as expected, increasing the applied pressure increased the relative density of all of the powders. It is observed that the small or fine powders have the least compressibility. This is due to their higher specific surface area; and consequently, higher friction between them [18]. Furthermore, small particles get work-hardened faster, due to their high specific surface area, and gain high strength so that they resist more toward the applied pressure and present less plastic deformation. Therefore, they exhibit less compressibility. By increasing the powder particle size, the relative density, and as a result, the compressibility increased. This is due to the lesser specific surface area and better flowability. The large powders showed the highest relative density and the best compressibility. These results indicate that the relative density of green compacts is directly proportional to the applied pressure and powder particles' size. In the highest compacting pressure (i.e. 650 MPa), the achieved green density was 73%, 77% and 79% of the theoretical value for fine, middle-sized and coarse CP Ti powders, respectively.

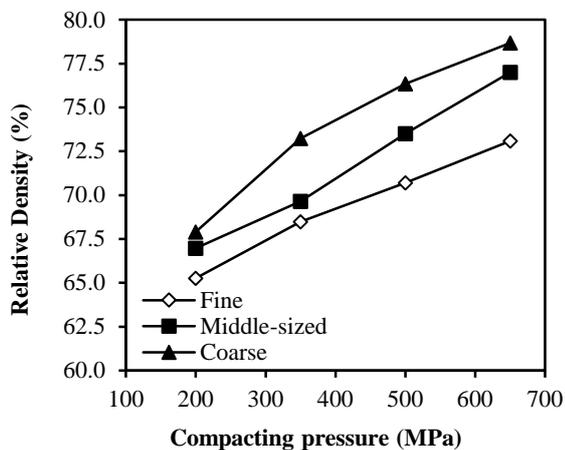


Fig. 2. Relative density variation as a function of compacting pressure for differently sized Ti powders.

3.3. Correlation of compacts' density and compaction equations

It is essential to predict the powders' behavior when different compaction parameters affect the green and relative densities of the compacts. Several compaction equations and models were presented for

this purpose, but not all of them were accurate enough. In this research, the correlations of equations such as Ge, Panelli-Filho, Heckel, and modified Heckel were studied. In order to match the experimental data with these models and evaluate their correlations, parameters like R^2 (correlation coefficient), D_0 (initial porosity, or density when the applied pressure is zero), ν (Poisson's ratio), and σ_0 (yield stress in the modified Heckel equation), obtained from experimental data, must be compared to those obtained from equations. One equation that is accurate enough for modeling powder compaction behavior is Ge [7] equation:

$$\log\left(\ln\frac{1}{1-D}\right) = K_G \log P + B_G \quad (1)$$

where D is the relative density of the green compact, P is the applied pressure, and K_G and B_G are the constants of the equation. Another useful equation is presented by Panelli-Filho [8]:

$$\ln\frac{1}{1-D} = K_P \sqrt{P} + B_P \quad (2)$$

where K_P and B_P are constant and are related to the characteristics of the powder. Heckel [9] presented the compaction behavior of the powders as a first-degree equation:

$$\ln\left(\frac{1}{1-D}\right) = K_H P + B_H \quad (3)$$

where K_H and B_H are constants. In the Heckel equation, the yield stress is considered as a constant. If the yield stress is considered as a function of the applied pressure, the results will be more accurate. Therefore, the modified Heckel equation is obtained [10]:

$$\ln\frac{1}{1-D} = \ln\frac{1}{1-D_0} + \frac{1}{3K_1} \ln\left(1 + \left(\frac{K_1 P}{\sigma_0}\right)\right) \quad (4)$$

where σ_0 is the yield stress at $P=0$ and K_1 is the constant. Denny [19] proposed an equation relating the term k_1 to the Poisson's ratio (ν):

$$K_1 = \frac{2\nu^2}{1-2\nu} \quad (5)$$

In all of these equations, Parameters B_G , B_P , and B_H can be used to determine the relative density at $P=0$ (D_0), which can be compared to the powders' relative apparent density.

The experimental compaction data of all powders were fitted by the Eqs. (1), (2), (3), (4) using the least squares analysis. Fitting constants and correlation constants obtained from the Ge, Panelli-Filho, Heckel, and modified Heckel equations are presented in Table 2. High R^2 values indicate that the

experimental data of the present investigation fit well by all of the equations. Presented data for the Modified Heckel equation were calculated by the Problem Solver in Microsoft Excel [20]. The relatively high R^2 values presented in Table 2 indicate that the experimental data of the present investigation fit well by all of the equations. Modified Heckel equation showed the highest R^2 values among other equations, indicating its higher capability to predict the compaction behavior of titanium powders. The Poisson's ratio values that are calculated by this equation correspond well to titanium's Poisson's ratio (0.34 to 0.4) reported in the literature [21]. The yield stress (σ_0) values that are calculated by the modified Heckel equation fit well with those of CP titanium grade 2 yield stress reported in the ASTM B 348 standard (275 MPa).

3.4. Sinterability

Figures 3a–c show the variation of the relative sintered densities with sintering temperatures for differently sized titanium powders compacted under different applied pressures. As it is observed, the highest amount of the sintered density was obtained for the small powders pressed under 650 MPa and sintered at 1400 °C. The middle and large-sized titanium powders were sintered more appropriately at 1200 °C.

As it is observed in (Fig. 3), for the fine powders (Fig. 3a), the relative density increased when the sintering temperature exceeded from 1200 °C, regardless of the compacting pressure. The relative density of ~99% was attained for the fine titanium powders compacted at the 650 MPa applied pressure which is about 30% higher than that obtained for the same compact sintered at 1200 °C. In fact, the sintering temperature of 1100–1200 °C is not enough for the fine titanium powders and it is necessary to increase the sintering temperature to 1400 °C in order to produce dense compacts. The increased density at higher sintering temperatures could be attributed to the enhanced diffusion of titanium atoms during sintering. In small particles with larger specific surface areas, the neck radius and diffusion paths' size are small resulting in an increased diffusion rate [14, 15]. Similar trends for -325 mesh CP Ti powders were reported by other authors [16, 17].

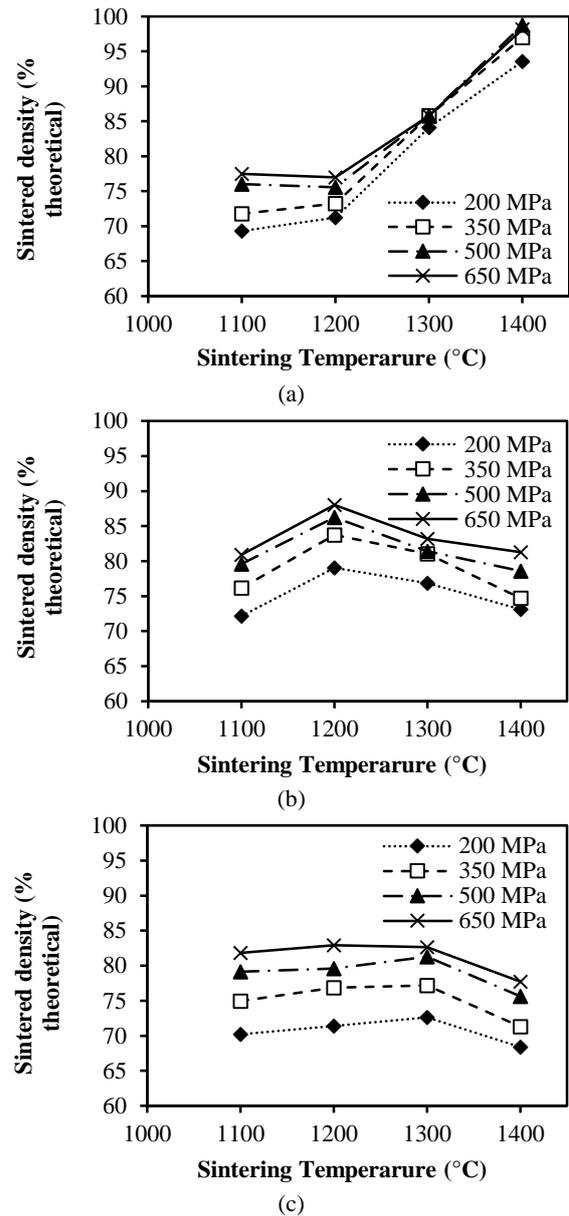


Fig. 3. The variation of the relative sintered density as a function of sintering temperature at different compaction pressures for each titanium powder: (a) <25 μm , (b) 38–100 μm , (c) 150–250 μm .

For the Middle-sized powder compacts (Fig. 3b) pressed at a given applied pressure, the highest density was achieved for the compact sintered at 1200 °C. The sample compacted in 650 MPa and sintered at 1200 °C showed the highest relative density about 88% theoretical. In fact, for this category powders, the relative density increased by elevating the temperature up to 1200 °C and then decreased by a further increase of temperature up to 1400 °C.

A relatively similar trend was found for the coarse powders (Fig. 3c). As observed, the sintering

temperature of 1200–1300 °C is suggested to obtain samples with a higher density from sintering coarse titanium powders. However, the density of this category of sintered powder compacts shows the least dependence on the sintering temperature and is mostly controlled by the applied pressure during the compaction stage. For both middle-sized and coarse powders, reduction of the relative density at higher temperatures can be due to the excessive grain growth and undesirable growth of β phase after 1300 °C [17].

In order to remove the effect of the green density on the sintered density, the densification parameter (Ψ) was calculated using the following equation:

$$\Psi = \frac{\rho_s - \rho_g}{\rho_{th} - \rho_g} \quad (6)$$

where ρ_s , ρ_g , and ρ_{th} are the sintered density, green density and theoretical density, respectively. As a result, the densification parameter is representative of the amount of densification gained through sintering. (Fig. 4) shows the variation of the densification parameter with sintering temperature for different sintered compacts. For the fine powders (Fig. 4a), large Ψ values (near 1) at 1400 °C indicate the great sinterability of the compacts. This is due to the significant shrinkage of the green compact consisting of fine titanium powders during sintering. (Fig. 4b) shows that the highest densification parameter value for middle-sized titanium powders, i.e. ~ 0.5 , was obtained when their green compacts were sintered at 1200 °C. In fact, in both fine and middle-sized titanium powders, the variation of densification parameter with sintering temperature is similar to that of the relative sintered density. However, the lower densification parameter values obtained for middle-sized powders indicate a reduction in the sinterability of powders by increasing their size. In the case of coarse titanium powders (Fig. 4c), the highest densification, $\Psi \approx 0.2$ was obtained by sintering at 1200 or 1300 °C for powders pressed at different pressures. In addition, the negative value obtained for coarse powders sintered at 1400 °C indicates their expansion during sintering. These results clearly indicate that the sinterability of titanium powder compacts decreases by increasing the powder size.

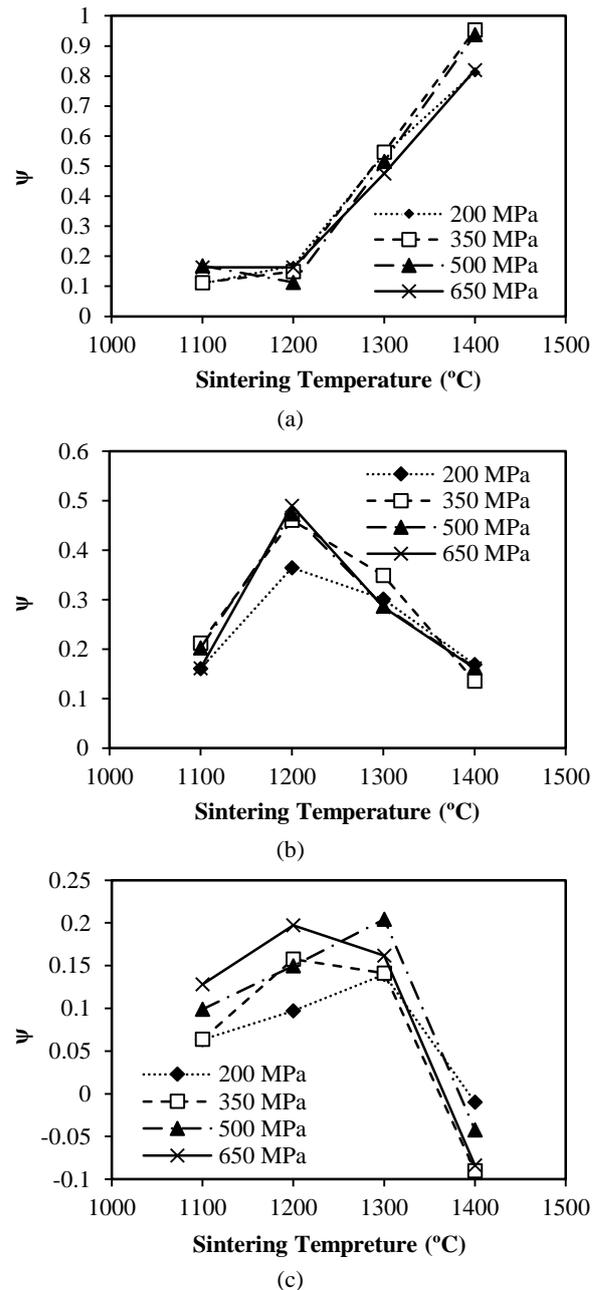


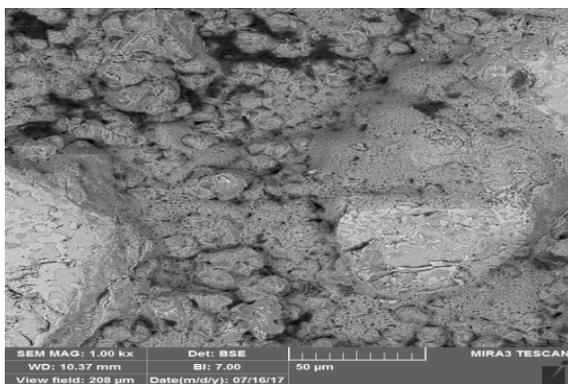
Fig. 4. Densification parameter as a function of sintering temperature at different compaction pressures for each titanium powder: (a) $< 25\mu\text{m}$, (b) $38\text{--}100\mu\text{m}$, (c) $150\text{--}250\mu\text{m}$.

3.5. Microstructure

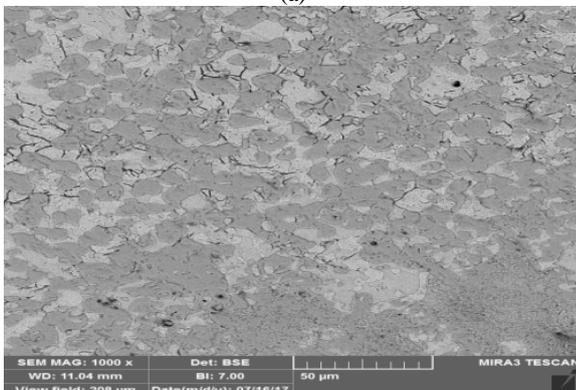
By using SEM Microscopy, the microstructures of fine and coarse titanium powder compacts, that were pressed under 650 MPa and sintered at 1200 and 1400 °C, are shown in (Figs. 5 and 6), respectively. These images show the complete consolidation of materials for compacts sintered at the optimum situations.

The microstructures of fine powder compacts pressed under 650 MPa and sintered at 1200 and 1400

°C are shown in (Fig. 5a and b), respectively. The microstructure of the compact sintered at 1200 includes some porosity which is probably due to the incomplete sintering process. As observed in (Fig. 5a) particle contacts are transformed into sintered bridges or necks in the form of microplates which are possibly due to high compacting pressure (650 MPa) before sintering [22]. Besides, a coherent network of pores is seen, indicating that the sintering has not entirely occurred. It is well known that a solid state sintering consists of three stages. In the first stage, the necks between particle contact points are formed and then continue to grow. In the second and third stages, the pore channels become cylindrical and isolated, respectively. The presence of interconnected pores within the microstructure of fine powder compacts sintered at 1200 °C (Fig. 5a) suggest that this temperature is not sufficient for fine pure titanium powders. When the sintering temperature was increased to 1400 °C, some closed pores were only remained in the compact, as observed in (Fig. 5b). This indicates the production of a relatively dense product.



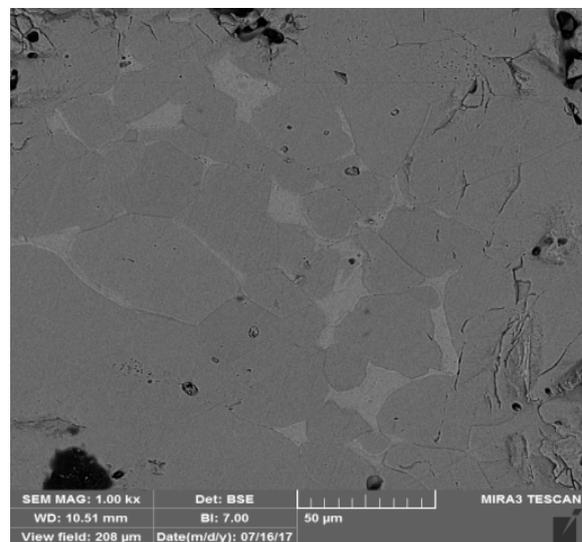
(a)



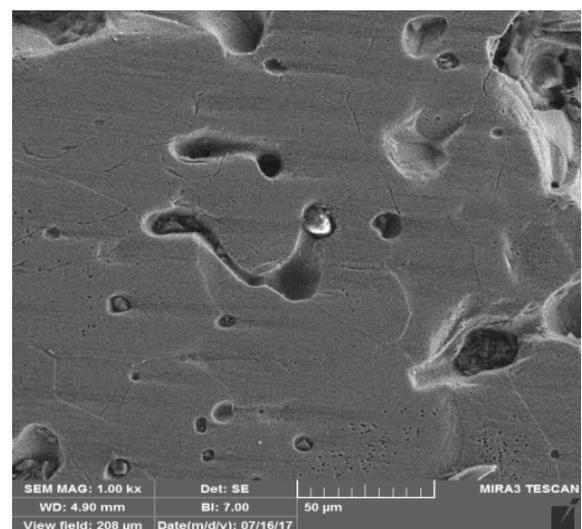
(b)

Fig. 5. SEM images of fine titanium powder compacts pressed under 650 MPa and sintered at (a) 1200 °C and (b) 1400 °C.

The microstructures of coarse titanium powder compacts pressed under 650 MPa and sintered at 1200 and 1400 °C are presented in (Fig. 6a and b), respectively. The microstructure of the samples sintered at 1200 °C (Fig. 6a) contains some small and large but isolated pores indicating that some degree of densification has occurred. However, the compact sintered at 1400 °C (Fig. 6b) includes some interconnected pores which can be attributed to excessive grain growth. The formation of such pores resulted in an expansion of the compact and negative densification parameter is caused as previously shown in (Fig. 4c).



(a)



(b)

Fig. 6. SEM images of coarse titanium powder compacts pressed under 650 MPa and sintered at (a) 1200 °C and (b) 1400 °C.

3.6. Hardness

Figure 7 shows the variation of hardness with sintering temperature for differently sized powders compacted under various applied pressures. High hardness values were obtained for fine titanium powders sintered at 1400 °C. It is evident in this image that the hardness of sintered compacts of fine titanium powders was mainly affected by the sintering temperature. Increasing the sintering temperature from 1200 to 1400 °C for fine titanium powders pressed under 200, and 650 MPa increased their hardness by 104 and 113%, respectively. This considerable hardness increase could be mainly attributed to the improved sintering and enhanced densification, as previously shown in (Fig. 4a). Vickers hardness testing via indentation is known to be affected by the density, porosity, and distribution of the sintered body. It is believed that the hardness value increases by decreasing mean particle size because the specific surface area is large and the sintering temperature is high, which leads to a higher content of dissolved interstitial elements [23]. Furthermore, fine powders have more oxygen content due to their large specific surface area; as a result, the hardness of the sintered body increases as the oxygen content increases. The higher oxygen content can also justify the necessity of utilizing a higher sintering temperature for fine

powders as previously shown in (Fig. 3). In fact, to overcome the diffusional obstacle resulting from the higher oxygen content, the sintering temperature of fine Ti powders should be increased from 1200 to 1400 °C. However, it is evident in Fig. 7 that the hardness of Middle-sized titanium powders is more influenced by the applied compacting pressure during the cold press stage rather than the sintering temperature. For example, the hardness value for the middle-sized titanium powder compacts sintered at 1200 and 1400 °C increased by 46 and 41% when the compacting pressure increased from 200 to 650 MPa. The pronounced effect of the compacting pressure in the final hardness of the middle-sized titanium powder compacts is due to their higher compressibility and improved densification compared to those of fine powders (Fig. 2). For coarse titanium powders, neither sintering temperature nor compacting pressure showed a considerable influence on the hardness value. This could be mostly ascribed to the reduced sinterability and low volume shrinkage or even swelling during sintering as previously shown in (Fig. 4c). These results clearly signify the vital role of selecting the appropriate sintering temperature and compacting pressure in the consolidation of compacts consisting of differently sized titanium powders.

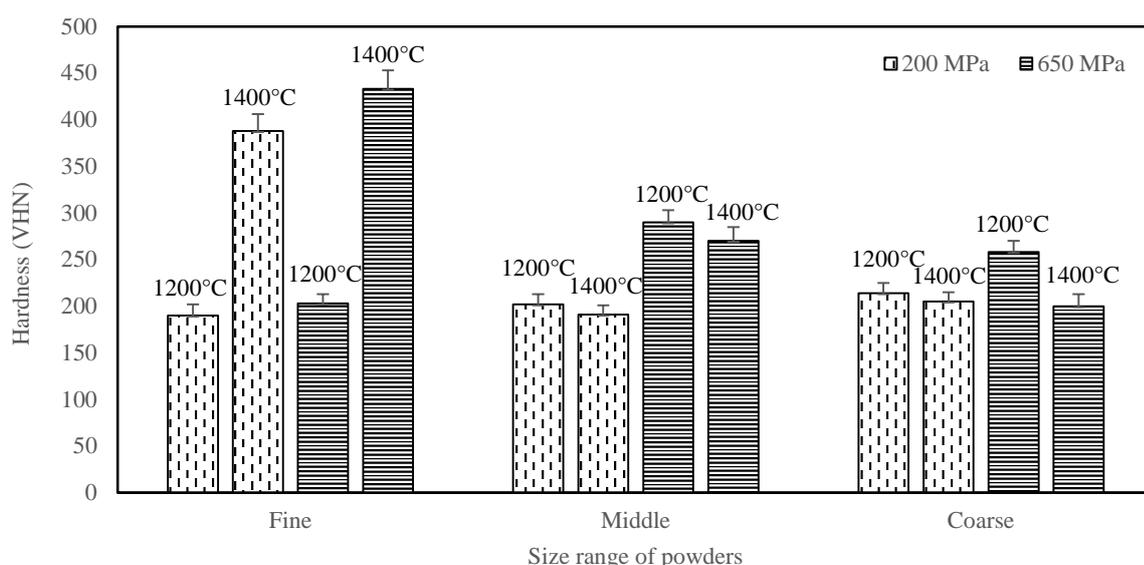


Fig. 7. Hardness of sintered compacts consisting of titanium powders with different sizes as a function of compaction pressure and sintering temperature.

4. Conclusion

- Coarse titanium powders showed the highest compressibility.
- The compaction data for all ranges of titanium powders were best fitted by the modified Heckel equation.
- The sinterability decreased by increasing the size of the powders. The highest density was attained for fine powders sintered at 1400 °C. However, the sintering temperature of 1200 to 1300 °C was found to be appropriate for middle-sized and coarse powders.
- Increasing the sintering temperature of fine titanium powder compacts changed the morphology of the internal pores from interconnected to the closed ones. This resulted in the production of nearly dense titanium compacts confirmed by microstructural studies.
- The hardness value of sintered fine titanium powders was mainly influenced by the sintering temperature. For middle-sized powders, the hardness value was controlled mostly by the compacting pressure applied in the cold compaction stage. However, for coarse titanium powders, neither sintering temperature nor compacting pressure showed a considerable influence on the hardness value.

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تأثیر اندازه ذرات بر فشردگی پذیری و تفجوشی پودرهای تیتانیوم

ایمان کاسب، محمد معظمی گودرزی و علیرضا عباسی

دانشکده فنی و مهندسی، واحد علوم و تحقیقات دانشگاه آزاد اسلامی تهران، تهران، ایران.

چکیده

در این تحقیق اثر اندازه ذرات پودر هیدرید-دی هیدرید تیتانیوم بر فشردگی پذیری و تفجوشی آن‌ها مطالعه شده است. به این منظور پودر تیتانیوم خالص تجاری با سه اندازه ذرات متفاوت به کار گرفته شد. فشردگی سازی تحت فشارهای اعمالی ۲۰۰ تا ۶۵۰ مگاپاسکال و تفجوشی در دماهای ۱۱۰۰ تا ۱۴۰۰ درجه سانتیگراد انجام شد. رفتار فشردگی پذیری پودرهای با اندازه متفاوت، با اندازه گیری چگالی خام نسبی پس از فشردگی سازی در فشارهای اعمالی مختلف مطالعه شد. ریزساختار خسته‌های تفجوشی شده نیز با استفاده از میکروسکوپ الکترونی روبشی ارزیابی شد. نتایج نشان داد که پودرهای ریز کمترین فشردگی پذیری را دارند. داده‌های فشردگی پذیری با استفاده از مدل‌های فشردگی متداول بررسی شدند که در نهایت معادله هکل اصلاح شده بهترین انطباق را نشان داد. علاوه بر این، بیشترین تفجوشی پذیری برای پودرهای ریز حاصل شد؛ به طوری که بالاترین چگالی نسبی (۹۸٪ تئوری) برای پودرهای ریز فشردگی شده در ۶۵۰ مگاپاسکال و تفجوشی شده در دمای ۱۴۰۰ درجه سانتیگراد به دست آمد. با این وجود، نتایج نشان داد که دمای ۱۲۰۰ درجه سانتی‌گراد برای تفجوشی پودرهای تیتانیومی با اندازه ذرات متوسط و درشت مناسب‌تر است. نتایج سختی سنجی نشان داد که تولید خسته‌های تیتانیوم خالص با خواص مکانیکی مناسب از طریق تفجوشی در دمای بهینه شده در خلأ امکان پذیر است.

واژه‌های کلیدی: متالورژی پودر، تیتانیوم، فشردگی پذیری، معادلات فشردگی، تفجوشی پذیری