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# Effect of pure titanium particle size on density, hardness, wear resistance and microstructure properties

Tugba MUTUK[1,*] and Mevlüt GÜRBÜZ[2]

[1] *Department of Metallurgical and Materials Engineering, Ondokuz Mayıs University, Samsun, 55200, Turkey*
[2] *Department of Mechanical Engineering, Ondokuz Mayıs University, Samsun, 55200, Turkey*

[*]**Corresponding author e-mail:** tugba.isitan@omu.edu.tr

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**Abstract**

In this study, pure titanium particle samples with different particle sizes were produced by powder metallurgy method. In the sample production, pure titanium particles having a different particle size of ≤30µm, ≤43µm, ≤150µm were used. Titanium (Ti) samples were sintered at 1100°C for 120 min. Density, hardness, wear resistance and microstructure analyzes were performed on the Titanium samples. According to the results, the best mechanical properties values were obtained with ≤30 µm particle size of Titanium. The best density and hardness results are 4.28 $g \cdot cm^{-3}$ and 419.8 HV, respectively. The results of the wear rate were found under 30 N load of $8.21\times10^{-5}$ $mm^3 \cdot Nm$. Mass loss results were measured as 5.2 mg (under load of 30 N). SEM analyses showed that a good bonding and strong neck formation between the particles were observed for particle size of ≤30 µm.

## 1. Introduction

In today's world, the need for materials with superior mechanical properties is increasing along with the developing technology. Titanium (Ti) and titanium alloys have good mechanical properties such as superior corrosion resistance, low density and high strength. For this reason, titanium has become an attractive material. For example, it provides high performance and fuel savings in the aviation and automotive industries, while in the biomedical industry it shows biocompatibility in the human body. However, pure titanium exhibits poor wear resistance with limiting application areas. For this reason, titanium matrix composites are being developed for applications where the mechanical properties of pure titanium are insufficient [1-3].

Pure titanium has a close-packed structure at room temperature, and at high temperatures it becomes an allotropic material with body centered cubic crystal lattice structure. Roughly 888°C is required in order to convert the α-phase in the close-packed structure to the β-phase in the body-centered cubic. This temperature is called the β change temperature. The β change temperature changes according to the amount and type of alloy metals contained in pure titanium. Also, β change temperature increases with the effect of atoms such as nitrogen and oxygen that stabilize the α phase [4,5]. Pure titanium microstructure has two types of α grains: needles and coaxials. Coaxial α grains have high ductility and strength, and exhibit high resistance to plastic deformation and stress corrosion cracking. The fracture toughness and creep resistance are very high in the needle α [6].

The matrix material in the production of metal matrix composites is important in terms of particle size mechanical and microstructure properties. While titanium metal matrix composite produced, titanium particle size affected the material in terms of mechanical and microstructure. In the study carried out by Kim et al. [7], $B_4C$ reinforced titanium matrix composite materials were fabricated. The particle size of Ti is approximately 50µm. These materials have been tested for hardness, wear resistance and friction. The hardness value of composite material with 3.76 wt.% added $B_4C$ has doubled compared to pure Ti. Wear resistance has improved at all reinforcement rates and has increased four times by 3.76 wt.% compared to pure Ti. The creep coefficient decreased of 30% compared to pure Ti by 3.76 wt.% $B_4C$ reinforcement. Alman et al. [8], produced composite material by using ≤43 µm particle size of titanium together with 0, 2.5, 5, 10, 20, 40 vol.% of TiC, $TiB_2$ and $Si_3N_4$ reinforcements. The hardness and wear behavior of the composite materials were investigated. According to the results, the best hardness values were 413 HV in 40 vol.% of reinforced TiC, 610 HV in 20 vol.% reinforced $TiB_2$ and 1199 HV in 20 vol.% reinforced $Si_3N_4$. The result of wear test showed that the average mass loss and wear coefficient increased as the volume rate of reinforcement phase increased. Gürbüz and Mutuk [9], the production of graphene nano-platelets reinforced titanium matrix composite of 0.15 wt.%, 0.30 wt.%, 0.45 wt.%, 0.60 wt.% was performed. The change of hardness values of the composite was investigated with varying temperature and time. Sintering temperatures 1000°C, 1050°C and 1100°C and time 60 and 120 min. were used. The best result was obtained as 566 HV in composite material with 0.15wt.% graphite reinforced which was sintered for 120 min. at 1100°C. In the work of Zhang and Liang [10], graphene reinforced titanium matrix composites were produced by powder metallurgy method with pure Ti of ≤ 45 µm size of


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particle. The hardness value of 3 wt.% Graphene added matrix increased 197 HV from 150 HV. Liu et al. used ≤ 45 µm size of titanium particle to produce graphene reinforced titanium matrix composite materials by powder metallurgy [11]. Hardness, porosity, yield strength and compressive strength tests were made with respect to pure Ti at different temperatures of 25°C, 200°C, 400°C, 600°C, 800°C. When we look at the results, it was observed that as temperature increased so porosity, while other mechanical behaviors decreased and mechanical properties of pure titanium and graphene reinforced titanium matrix composites converge together.

In this study, pure titanium samples were prepared using powder metallurgy method and mechanical, microstructural analysis were performed. For production, ≤30 µm, ≤43 µm, ≤150 µm size of pure Ti particles were used. Density, hardness, wear resistance and microstructure tests were performed on titanium samples. This study will examine mechanical and microstructure properties of different particle sizes.

## 2. Experimental

### 2.1 Materials

In this study, three different particle size of pure titanium powder (Nanography, ≤ 30 µm), (Alpha Aesar, ≤ 43 µm), (Nanography, ≤150 µm) were used. The purpose of this work, is to study the effect of different particle size of pure Titanium on hardness, wear resistance and microstructure properties. These pure titanium particles were encoded in Table1. Figure 1 (a-f) gives the particle size analyses and SEM micrograph of the powders. As given SEM and particle size graphs confirm the size of the particles. 30Ti, 43Ti and 150Ti has spherical, and edged microstructure, respectively.

**Table 1.** Sample codes of Titanium particles.

| Particle size | Sample code |
|---|---|
| ≤30 µm | 30Ti |
| ≤43 µm | 43Ti |
| ≤150 µm | 150Ti |

### 2.2 Method

Different sizes of titanium particles were separately mixed with ethanol. The powders mixed with the ethanol medium were subjected to ultrasonic homogenizator for 15 minutes and then milled for 18 hours in the ball mill. Filtration and drying were carried out to remove ethanol from titanium powders. Compression was performed using molds of different shapes to produce the test sample. Then, the mixed powders were shaped in a 10 mm diameter stainless steel mold at 800 MPa. After shaping (samples of 10mm radius and 3 mm thickness), the samples were sintered in the high-temperature furnace under vacuum. Titanium is a material that can be oxidized very quickly, so that test samples were sintered at 1100°C for 120 minutes in high vacuum (under $10^{-5}$ Pa vacuum). The flow chart of the production is shown in Figure 2.

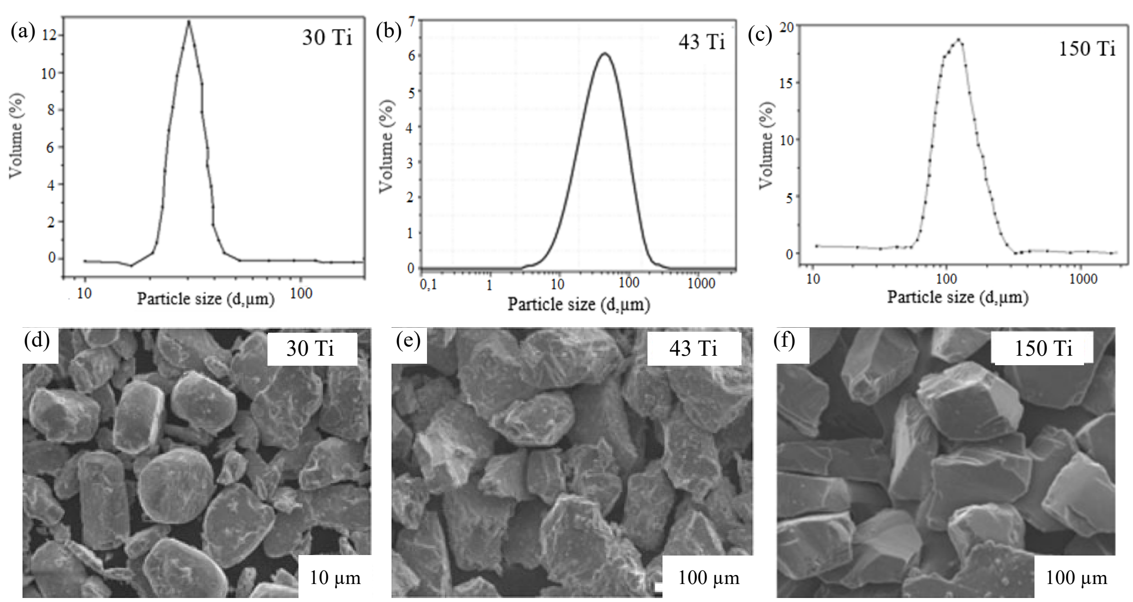


**Figure 1.** Particle size distributions and SEM analyzes of the 30Ti, 43Ti and 150Ti powders.

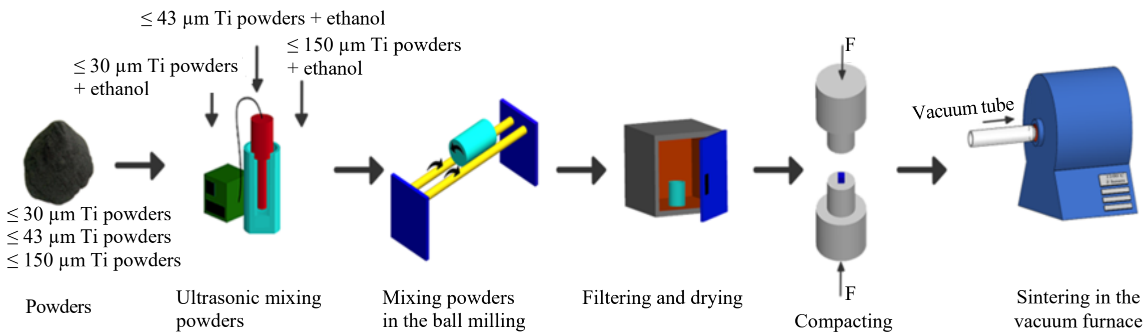


**Figure 2.** Flow chart production of different particle size of Titanium for mechanical analyzes.

## 2.3 Mechanical characterization

The values for temperature and time in this study resulted from our previous study about sintering [9]. The experimental densities ($\rho_D$) of the samples after sintering were calculated in Eq. (1) according to the Archimedes principle [9],

$$\rho_D = [M_S/(M_D\text{-}M_A)]\ \rho_{su} \qquad (1)$$

where $M_S$ is the dry mass of the sample, $M_D$ is the water-saturated mass of the sample and $M_A$ is the mass of the suspended body measured in water. The density measurement was performed 6 times according to the Archimedes principle.

Surfaces of the samples were polished before the micro vickers hardness test. Hardness values were measured with digital micro Vickers hardness tester (HV1000B) under a load of 500 g (HV 0.5) and waiting time of 15 s. The vickers hardness measurements were performed six times different parts on the surfaces of the sample.

The dry friction coefficient between the different particle sizes (≤30 µm, ≤43 µm, ≤150 µm) and the stainless steel disc were determined the wear rate. Dry wear tests were carried out under three different load values of 10 N, 20 N and 30 N in the wear test setup, at a rotation speed of 200 rpm and for 20 minutes.

# 3. Results and discussion

## 3.1 Density

The result of experimental density is shown in Figure 3. The theoretical density of titanium is 4.51 g·cm$^{-3}$ and is indicated by Ti coding. The highest density result is 4.28 g·cm$^{-3}$ with a particle size of ≤ 30 µm. The bulk density before sintering is around 76%. But after the sintering process the bulk density reaches approximately 95%. The densities of the particles with sizes of ≤43 µm and ≤150 µm decreased to 4.19 g·cm$^{-3}$ and 4.09 g·cm$^{-3}$, respectively. The results show that while the particle size decreased, the density increased. This increase in density supports that, if sample has a small particle size, then good bonding between particles during sintering occurs.

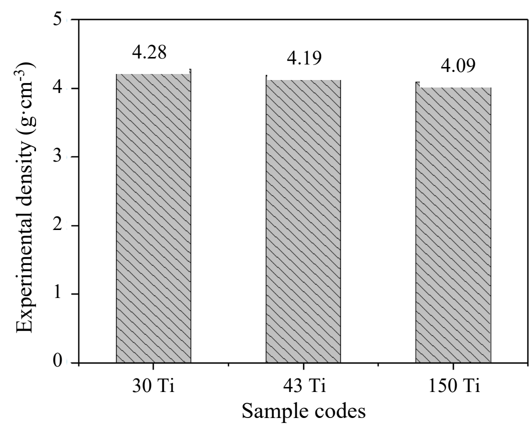


**Figure 3.** Experimental density changes of different particle size of Ti samples

## 3.2 Micro vickers hardness test

Figure 4 shows the result of the vickers hardness test. As seen in Figure 4 the hardness values decreased when the particle size increased. The highest vickers hardness was obtained for sample of 30Ti (420 HV). Hardness values of 43Ti (398 HV) and 150Ti (233 HV) samples were compared to 30Ti hardness value. There has been a noticeable decline. The Ti particle shapes are very different for each powder. It creates the porosity with increasing size of the particles which reduces the density. On the other hand, the more changing of hardness can be observed due to the dislocation density which depends on the grain size of the samples. Therefore, the hardness of the 30Ti higher than the 150Ti due to the higher dislocation density for 30Ti.

Particle size and average particle diameter have a significant effect on the mechanical properties of multi-crystalline metals. Grains with different crystal orientations act naturally as a barrier to dislocation movements. Because of the different orientation of these grains, the dislocation movement must change its direction while moving between grain boundaries. And as this orientation increases, the dislocation movement becomes more difficult. If the dislocation movement coincides with a large angle boundary, it cannot pass and is stacked at the grain boundaries. This causes internal stresses in the material.

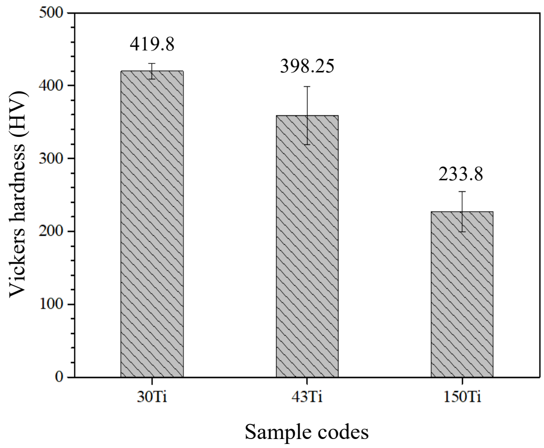


**Figure 4.** Vickers hardness of Ti samples.

When materials have small particle sizes, they get more grain boundaries after sintering process. More grain boundaries prevent the dislocation movement better than the coarse particle size of materials. This means that materials with small particle sizes have better strength than coarse grained ones. The Hall-Petch equation (Eq. (2)) also shows the relationship between the yield strength and particle size of the material [12].

$$\sigma_{ak} = \sigma_0 + k_y\sqrt{d} \qquad (2)$$

where, $\sigma_0$ shows the yield strength, $k_y$ are material-specific constants and $\sigma_{ak}$ shows the mean grain diameter. As indicated in Eq. (2), it indicates the relationship between yield strength and grain diameter. Shrinking particle size increases the strength as well as the toughness of the material [12]. In this study, the hardness value of the material increased with the decrease in the size of the pure titanium particles. This shows that the results are supported by the Hall-Petch equation.

## 3.3 Mass loss and wear rate

As a result of the wear test in this study, mass loss graph and wear rate were formed in Figure 5. To determine the wear rate, the weight difference method was used. The mass loss and wear rates of the samples were calculated in Eq. (3-5). The mass loss ($\Delta$m) parameter is also used to compare the amount of wear on the material during the wear test. In order to calculate the mass loss parameter Eq. (3) can be used,

$$\Delta m = m_i - m_s \qquad (3)$$

where $m_s$ is the final weight and $m_i$ is the initial weight of the samples [13]. An important parameter in the wear test is the sliding distance. This parameter; the radius (r) of the abrasive disc is calculated in Eq. (4),

$$L = 2\pi r \times n \times t \qquad (4)$$

where, n is the the rotational speed and t is the wear test time of the disc [13]. Another parameter used to compare the wear rates in the material is the wear rate. This parameter is calculated by taking into account the change in volume and the shear distance as seen in Eq. (5) [13]:

$$W = \Delta V/(P\times L) \qquad (5)$$

The results are observed in the wear rate (W) graph also support the results of mass loss. With the increase in the load amount, the wear rate increased in all samples. The minimum wear rate was obtained in 30Ti (W= $1.89\times10^{-5}$ $mm^3\cdot N^{-1}m^{-1}$ under the load of 10N). When compared with 150Ti wear rate ($1.2\times10\times10^{-4}$ $mm^3\cdot N^{-1}m^{-1}$ under the load of 10N), it is seen that the difference is quite high. These results show that the lower particle size titanium has very good results in terms of mechanical properties

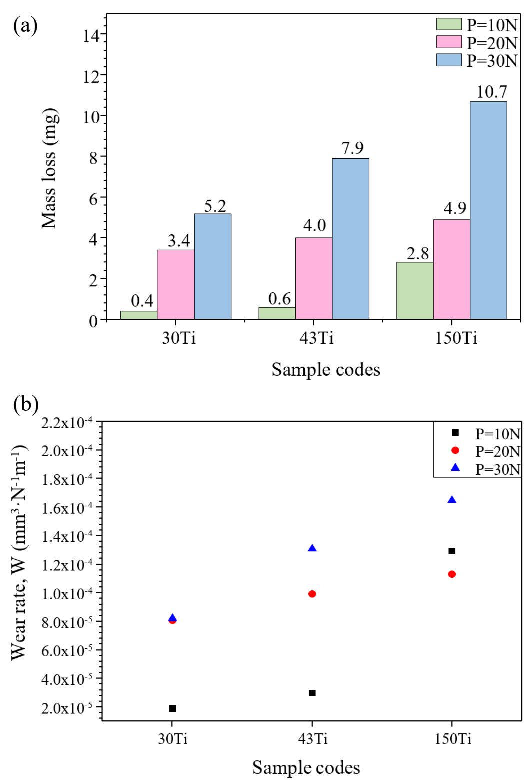


**Figure 5.** (a) Mass loss (b) wear rate of Ti samples.

## 3.4 Microstructural characterization

Scanning electron microscope (SEM) analysis were done of Ti samples. SEM images of low and high magnification of 150Ti and 30Ti samples are shown in Figure 6. The porosity of 30Ti samples is significantly reduced compared to 150Ti samples (Figure 6 (a) and (c)). The stereo images from polished surface confirm the SEM images. It is clearly shows the less porosity in 30Ti samples. Also, strong particle bonding and necking between Ti grains are observed in Figure 6 (d). From the SEM images, particle size has a great importance to fabricate denser microstructure because small particle size cause of lots of small grain size. It makes enhancement of dense structure.

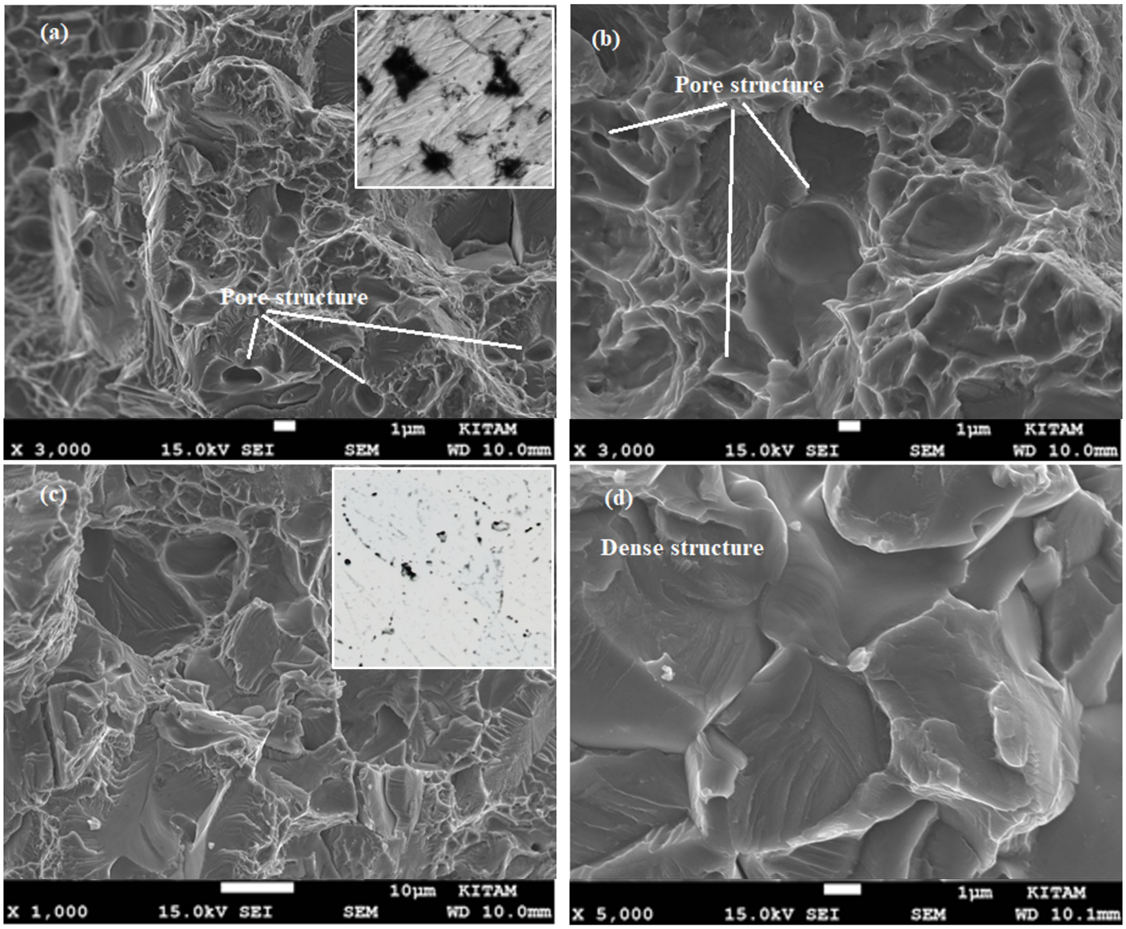


**Figure 6.** Stereo and SEM images of (a-b) high and low magnification of 150Ti (c-d) high and low magnification of 30Ti.

## 4. Conclusions

In this study, pure titanium samples were prepared by powder metallurgy method. In the first stage, powders having a particle size of ≤30, ≤43, ≤150 µm were pressed. It was then heat-treated at 1100°C for 120 minutes. Density, hardness, wear resistance and microstructure analyzes were performed on the samples. According to the results, the highest density value of 4.28 $g \cdot cm^{-3}$ with ≤30 µm particle size has emerged in the sample of pure titanium. The sample with ≤30 µm particle size gave the best results with 420 HV hardness, 0.4 mg mass loss and 10 N $1{,}896\times10^{-5}$ $mm^3 \cdot Nm^{-1}$, 20 N $8{,}060\times10^{-5}$ $mm^3 \cdot Nm^{-1}$, 30 N $8{,}218\times10^{-5}$ $mm^3 \cdot Nm^{-1}$ wear rate. Depending on the increased particle size, the number of grain boundaries decreased, the grains did not achieve good bond and interface interaction, and the porosity increased. Hence, the hardness and density values decreased and the mass loss and wear rate increased. As a result, the density and mechanical properties of the sample with a particle size of ≤30 µm were better than other samples. SEM analyses showed that a good bonding and strong neck formation between the particles were observed for particle size of ≤30 µm.

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