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COMPARISON OF IMPACT PROPERTIES OF RECYCLED AND COMMERCIALLY AVAILABLE PM TITANIUM ALLOYS

Mustafa USTUNDAG*, **Remzi VAROL**

Suleyman Demirel University, Engineering Faculty, Dept. of Mechanical Engineering, Isparta, Türkiye

Keywords

Ti-6Al-4V,
Sintering,
Recycled,
Impact properties,
Compaction.

Abstract

Titanium alloys are used in many specific applicants such as biomedical, military, sporting goods and aerospace industry due to their high strength properties and low aspect ratio. Even though their outstanding properties, titanium alloys have low utilization rate and relatively low recycling ratio during manufacturing of Titanium parts. In order to determine its mechanical and impact properties for low-cost Ti-based alloy applications, powder size distribution and compacting pressure strongly affect the microstructural properties of the sintered Ti-6Al-4V alloy. This paper presents how the narrow particle size distribution affect the impact properties of both recycled and commercially available Ti-6Al-4V alloys. Below 40 µm particle size distribution range of Ti-6Al-4V alloys were conducted on 3-point bending, Charpy impact test and microstructural evaluation. The results indicate that samples produced with commercial powder, with a particle size of -40 µm reach higher strength values.

GERİ DÖNÜŞTÜRÜLMÜŞ VE TİCARİ OLARAK TEMİN EDİLEN TM TİTANYUM ALAŞIMLARININ DARBE DAYANIMININ İNCELENMESİ

Anahtar Kelimeler

Ti-6Al-4V,
Sinterleme,
Geri dönüşüm,
Darbe dayanımı,
Sıkıştırma.

Özet

Titanyum alaşımları yüksek kütle – dayanım oranı gibi özellikleri sayesinde, biyomedikal, askeri, spor ve havacılık gibi birçok endüstriyel alanda kullanılmaktadır. Birçok endüstriyel alanda tercih edilmesine rağmen, yüksek maliyeti ve düşük geri dönüşüm oranı titanyum alaşımı malzemelerin kullanım alanlarını kısıtlamaktadır. Bu çalışma kapsamında partikül boyut dağılımının, ticari ve geri dönüştürülmüş toz metal Ti-6Al-4V alaşımlarının darbe dayanımları üzerindeki etkileri araştırılmaya çalışılmıştır. Bu kapsamda -40 µm toz partikül boyutundaki TM numunelere Charpy darbe testi uygulanmış ve numunelerin mikroyapıları incelenmiştir. Sonuçlar -40 µm toz partikül boyutundaki numuneler için ticari toz ile üretilenlerin daha yüksek darbe dayanımı değerlerine ulaştığı göstermektedir.

Alıntı / Cite

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1. Introduction

Titanium (Ti), reactive metal, are known as expensive materials to produce due to its limitation in refining process. Titanium can be alloyed with many elements and resulted on high strength-to-weight ratio, toughness, low thermal expansion, good processability, excellent corrosion resistance and improving its biocompatible properties (Guitar et al., 2009). Ti-6Al-4V is a Ti alloy type which is produced to increase the mechanical properties of Ti and is the most commonly used Ti alloy in the biomedical

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[*] İlgili yazar / Corresponding author: mustafaustundag@sdu.edu.tr, +90-246 211-8098

industry as an implant material. Therefore, Ti demand is drastically increasing depending on the progress of technology and the fields of application are considerably higher even though the high expenses of Ti alloy product. However, due to the rapid increase in demand for titanium implants in the world and the high cost of titanium alloys, the need for recycled and cheap raw materials has come to forefront. For this reason, efficient recycling technologies and methods of using recycled titanium raw materials are rapidly gaining importance. Also the concept of environmentally benign recycling, which optimizes value and quality while reducing environmental impact, is gaining considerable attention (Aizawa et al., 2002).

Particularly during the machining operations, low utilization rate and high amounts of energy are consumed during the recycling of these raw materials by conventional methods. In this case, the necessity of using more efficient methods such as cold heart melting (CHM) and vacuum arc re-melting (VAR) for the recycling of these chips is on the foreground. But those methods are expensive and gives inhomogeneous composition at the end product (Oh et al.,2014). In particular, solid-state recycling is attractive as it avoids the energy-intensive melting processes. Techniques based on hot extrusion have been attempted to recycle magnesium, aluminum, and iron machining chips, although lack of full density and inferior technical properties are often reported (Chino et al., 2006; Gronostajski et al., 2000). The high cost associated with titanium melting makes it a perfect candidate for solid-state recycling

The effect of recycling Ti alloy powder on mechanical properties of bulk parts is not clear. Some studies described that certain powders can be recycled many times without affecting their mechanical properties of its end product (Hill, 2001; Silva et al., 2005)]. Tang et al (Tang et al., 2015) found that after 21 times recycled of Ti alloy powder shows higher ultimate tensile and yield strength. Because of number of recycled increase, powders are exposed to air and powder particle size distribution become narrower up to 80μm. During clean-up process, reduced moisture in the recycled powder at high temperature (>550 °C) enables to minimize residual stresses. However they did not find a significant change in powder morphology or particle size distribution for recycled powder products and also no research describe on mechanical properties of specific particle size distributed recycled Ti alloy parts. Another study from McDonald et. al investigated mechanical properties of Ti-6Al-4V recycled and commercial alloys in terms of conventional mill-annealing. Due to presence of oxygen content on recycled Ti-6Al-4V alloys, annealing results on oxygen diffusing away from the dissolving oxide which hindered local grain growth and makes fine dimpled structure similar to commercial mill-annealed material (McDonald et al., 2014).

In this study, recycled and commercially available Ti-6Al-4V powders were selected to determine their mechanical, impact properties and microstructural evaluation in terms of particle size distribution. Those two types of powder were prepared by conventional P/M techniques and sintered for final product. Mechanical properties of those two groups were evaluated using three-point bending and impact fracture toughness was conducted by Charpy test. Recycled powder properties, such as particle size distribution and microstructure are evaluated and compared against that of the commercial available Ti-6Al-4V powder.

2. Literature Review

Chikosha et al. (2014) examined the effects of the morphologies and dimensions of metal powders obtained from the alloys of CP-Ti and Ti-6Al-4V on the compressibility of the powders. Metal powders of 4 different morphologies were used in the studies. To compare compare the characteristics of the powder groups more easily, powders were divided into three groups: -45 μm, +45-180 μm, +180 μm. The experiments were carried out on 12 different powder groups. As a result of their experimental studies, it has been suggested that metal powders having small particle size have higher compressibility.

Wen-bin et al. (2011), with the help of ball mills, produced a molar 50% Ti - 50% alloy composite metal powder and examined the surface morphologies, powder forms and powder contents of the metal composite powders they produced. The grinding operations were carried out in stainless steel containers at a speed of 400 rpm using 9 to 10 mm balls for 9 hours and 99.99% purity in a protective Ar atmosphere. They examined the metal powders produced after grinding by SEM, TEM and HREM (high resolution electron microscopy). As a result of the experimental studies and analyzes, they suggest that the metal powders produced are thin long structures with rounded edges and heterogeneous distribution of Al-Ti grains.

Bolzoni et al. (2017) investigated the porosity of the samples produced by powder metallurgy from Ti-6Al-7Nb metal powders and the effect of these pores on mechanical properties. They produced samples from Ti-6Al-7Nb metal powder mixture formed from elemental powders by using powder metallurgy method and examined the porosity of the samples they produced by making changes in the sintering parameters of these samples. Sintering processes in tube furnaces; 1250-1300-1350 ^{o}C, under high vacuum (about 10^{-5} mbar) and on zircon balls. Zircon balls used to prevent the diffusion of the material in contact with the samples. The sintering process took 120 minutes. Samples; It was heated to 5 °C / min and cooled at the same rate. The sintering process was improved by temperature increase and SEM and EDS analysis. They showed that the increase in rising sintering temperature decreased sample porosity (from 7% to 4%) and that the hardness of materials increased.

Cao et al. (2017) studied the mechanical properties of Ti-6Al-4V alloy materials produced by powder metallurgy. In their study, Ti materials were generally sintered in high vacuum and 1200 °C and the materials produced as a result of these sintering conditions had high β phase grain density. They suggested that high β-phase grain density resulted in collateral lamellas, which resulted in low flow and fracture strength. They used TiH_2 as matrix metal powder to improve mechanical properties and obtained TI-6Al-4V alloy by HDH method at low temperatures after reaching sintering temperature. In their work, they went outside the general and made sintering in H_2 atmosphere and under high vacuum. İn the end of the study relative density values reached up to 100%.

Leguey et al. (2002), Ti-6Al-4V in α + β phase and Ti-5Al-2,5Sn alloy samples in phase α were subjected to 3 point bending and tensile test and microstructure analyzes on the fractured surfaces of the samples. They performed sintering process in 1200 °C and 10^{-3} mbar vacuum environment. Mechanical experiments were carried out at temperatures ranging from room temperature to 475 ° C. As a result of their studies, Ti-6Al-4V and α-β-phase Ti-5Al-2,5Sn samples in the α + β phase showed similar brittle mechanical behaviors.

3. Material and Experimental Procedure

The Ti scraps used in this study were residual machining chips of Ti-6Al-4V bars. The process of recycling Ti alloy scrap began with cleaning, raw scrap, milling and sieving process respectively. Milling was operated by Retsch Vibratory Disc Mill RS 200 and sieving was done on Retsch Vibratory Sieve Shaker AS 200.

After milling the raw scrap at 700 rpm for 1 min, raw scrap were crushed to obtain fine chips suitable for milling pod. Afterwards, for getting finest powder distribution, powders were re-milled at 800, 900 and 1000 rpm for 10 min. Each milling conditions were collected and sieved as -100, 100-200, +200 μm size range.

Particle size distribution of classified raw powders and commercially available Ti-6Al-4V powders were performed with Malvern Masterseizer 2000 particle size analyser for confirming their sieve analysis. After particle size analysis, all powders were re-sieved to classify as follows; -40, 40-105, 105-150, 150-212 μm particle size range.

Before compaction test, for each particle size distribution at above, green density was calculated according to Hall-Petch relation. Compaction test was performed at various pressure ranging from 600 to 1500 MPa. Sintering was taken place in ALD high vacuum sintering furnace. Sintering parameters for all samples were fixed at 1200°C for 2 hours under 10^{-6} mbar vacuum pressure. Mechanical tests were conducted for evaluating the as-sintered recycled and commercially available Ti-6Al-4V alloy. The microstructure was observed by Olympus-B51 optical microscope. Particle shape and elemental analysis were characterized by Quanta FEG 250 scanning electron microscopy (SEM). A computer controlled Shimadzu/ AG-IC and ACT /AIT-300EN instruments are used for the three-point bending and impact test, respectively.

4. Results and Discussions

4.1. Powder Morphology

Figure 1a. shows the SEM images of -40 μm sized and sieved powders and commercially available Ti-6Al-4V powder distribution before compaction. It is clearly seen that the dust grains are in irregular, angular and inhomogeneous shapes due to HDH grinding method applied on metal powders. Another reason for the irregular shape and size distribution observed in the commercially available metal powders is accumulation. Depending on the milling time and temperatures, grains were agglomerated or mechanically clamping by secondary binding forces.

Figure 1b shows similar agglomeration and forging effect on commercially available Ti-6Al-4V metal powders. Main differences between those two metal powder is that -40 μm sized sieved raw powders have much more rounded corners since the frictional effect that both the powder particles in the pulverized disk grinder are in contact with each other and the powder particles are rubbed against the pot walls and discs in the vibratory disc mill. Apparent density decreased with getting smaller the powder size range. However their apparent density at -40 μm particle size show no flow rate because of higher surface area in contact with each powders. The high surface area results in increased friction between the powder granules and a heavier accumulation of large sized powders.

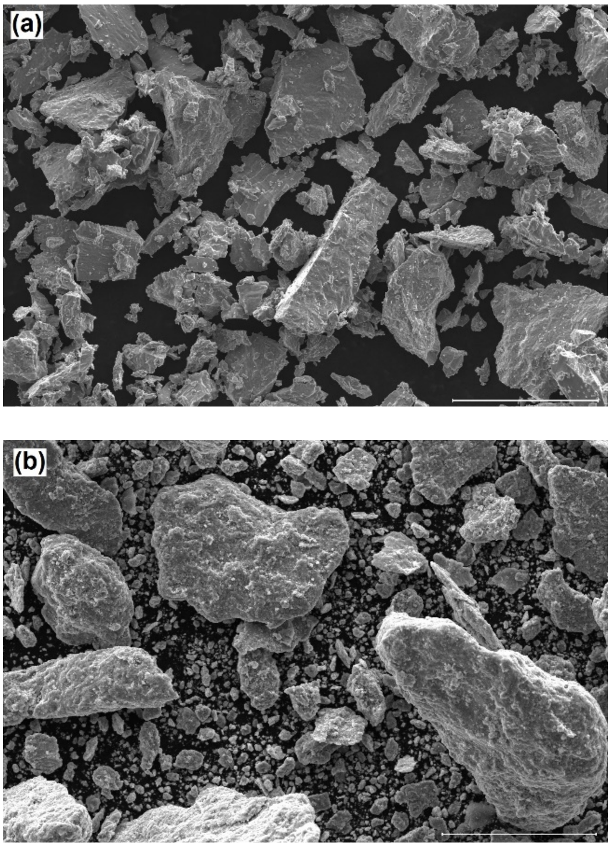

Figure 1. SEM images of (a) commercially available -40 µm sized sieved Ti-6Al-4V powder, (b) recycled -40 µm sized sieved powders before compaction. The SEM scale bar for samples set as 100 µm.

4.2. Pre &Post-sintered Density of Specimens

According to the literature survey, it has been determined that the %85 theoretical density is an ideal pre-sintering density value. In order to get %85 theoretical density, pre-sintered density measurements were made at different pressure. For below 40 µm raw powder, zinc stearate used as lubricant for easily removing from the mold. By starting from 600 MPa to 1500 MPa pressure, 85% pre-sintering density value reached at 1150 MPa and its relative density is 3,66 g/cm^3. Experimental studies have shown that even though higher relative density values are reached at pressures higher than 1150 MPa, these pressure values damage the mold and do not significantly increase the relative density value. All samples to be used in the compression tests were made with reference to the pressure value of 1150 MPa. After compacting all samples at 1150 MPa, all samples were being sintered together at 1200°C for 2 hours under 10^{-6} mbar vacuum pressure. 1% mass loss of Zinc stearate was observed in each sample due to evaporation during sintering. Sintered samples reached to %99-99,5 relative density.

4.3. Three-point Bending Tests

Flexural strengths of fractured samples were measured by three-point bending test. As shown in Figure 2, samples of recycled Ti-6Al-4V has 468,58 MPa average flexural strength while commercially available Ti-6Al-4V has 665,83 MPa. Depending on their particle shape, the more angular and irregular structure of powder in contact with each other; resulting in increased contact surface quantities and, consequently, increased diffusion during sintering.

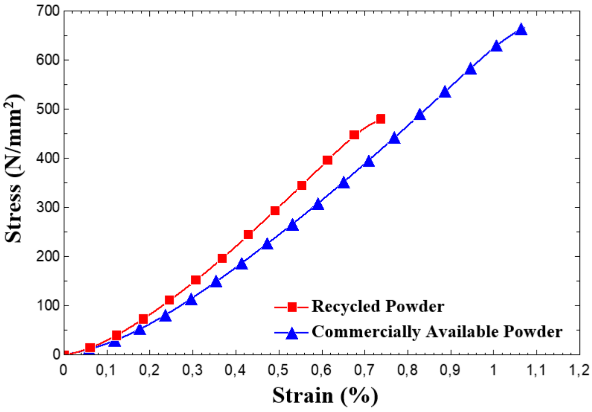

Figure 2. Flexural strength of -40 µm sieved recycled and commercially available Ti-6Al-4V powders.

Apart from their flexural strength, elongation modulus of recycled powder shows higher value than commercial one. Since neck formation against recycled powders change form and become more elongated. This could be a consequence of additional particle movement during the sintering process.

4.4. Impact Tests

Charpy impact toughness test results were averaged and the results are illustrated in Figure 3. The impact properties of commercially available alloys show higher values than recycled Ti-6Al-4V. This differentiation is coming from high intra-granular fracture energy of commercially available Ti alloy makes sample more ductile and inhibit crack prolongation. Moreover, impact toughness is related to sample relative density. As the relative density increases, the impact toughness increases as well.

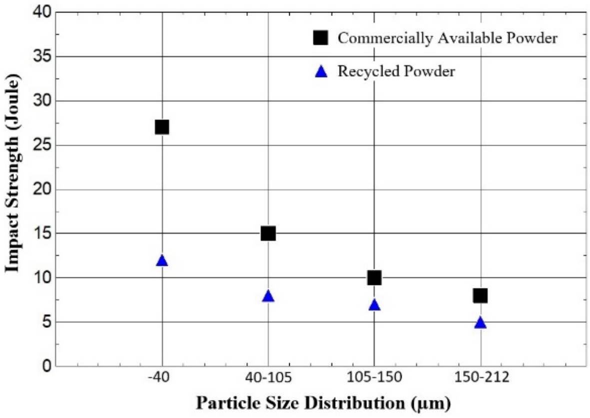

Figure 3. Comparison of impact strength against particle size distribution on recycled and commercially available Ti-6Al-4V alloys.

4.5. Analysis of Microstructure

Fractured samples were investigated by scanning electron microscopy (SEM). By investigating both fractured commercially available and recycled Ti-6Al-4V samples, it appears that the type of fracture on the macroscale is brittle and there is a proportionally higher break in grain in commercially available samples (Fig. 4a). It can be said that the neck mechanisms that increase the strength are acting as the beginning of breaking in the grain and that this situation reduces the ductility. It can also be seen that the porosity is not formed in the samples produced from recycled alloys (Fig. 4b).

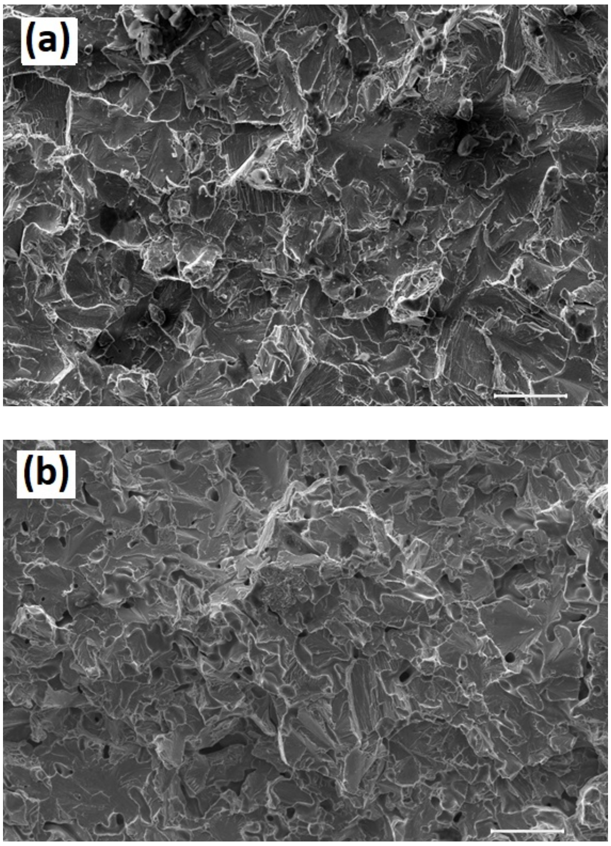

Figure 4. High magnification SEM image of (a) commercially available and (b) recycled samples deformed at Charpy test. The SEM scale bar for all samples set as 100 µm.

As seen in Figure 5, investigation of pore size distribution, distribution of α / β phases, grain boundary and grain irregularity were investigated by optical microscope for all samples. α (hexagonal close packed- HCP) phases are shown in region A (bright surface) and β phases are seen in regions indicated by B (dark surface). The β (BCC) phase has a much more ductile structure than α phase. The β phase conversion is low due to the fact that the cooling rate is kept at very low levels during the sintering process. The high rate of α phase in the microstructure confirms that the samples behave brittle.

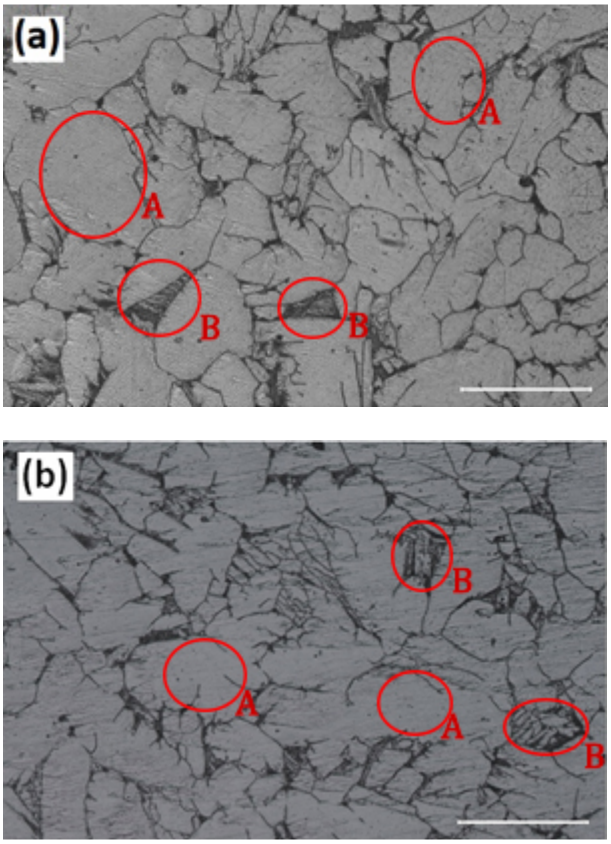

Figure 5. Optical microscope image of (a) commercially available and (b) recycled Ti-6Al-4V alloys after sintering. Bright area represents as α-phase while dark area shows β phase. The optical microscope scale bar is set as 100µm.

5. Conclusions

The following conclusions can be made from this study.

- The powder morphology of the recycled Ti-6Al-4V powder is more rounded than commercially available powder Ti-6Al-4V powder due to powders behaviour during milling process.
- Spherical and rounded shape powders lower the impact fracture toughness which makes commercially available alloys more ductile and inhibits intra-granular crack prolongation during fracture.
- Small change in mechanical properties of recycled and commercially available Ti-6Al-4V alloys was observed.
- It is also observed that the temperature conditions and durations during sintering process affect the material microstructures and phase distribution.

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Conflict of Interest

No conflict of interest was declared by the authors.

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