

# Mechanical Alloying and Sintering of Advanced Ternary Alloys

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**Abstract**—In this study, Ti-5Al-2.5Fe alloy was prepared by powder metallurgy. The elemental titanium, aluminum, and iron powders were mechanically alloyed for 10 h in a vacuum atmosphere. A stainless steel jar and stainless steel balls were used for mechanical alloying. The alloyed powders were then sintered by vacuum hot pressing at 950 °C for a soaking time of 30 minutes. Pure titanium was also sintered at the same conditions for comparison of mechanical properties and microstructural behavior. The samples were investigated by scanning electron microscopy, XRD analysis, and optical microscopy. Results showed that, after mechanical alloying, a homogeneous distribution of the elements was obtained, and desired  $\alpha$ - $\beta$  structure was determined. Ti-5Al-2.5Fe alloy was successfully produced, and the alloy showed enhanced mechanical properties compared to the commercial pure titanium.

**Keywords**—Ti5Al2.5Fe, mechanical alloying, hot pressing, sintering.

## I. INTRODUCTION

TITANIUM is one of the most important material especially for automotive and aerospace industry. This is because of its excellent combination of specific mechanical properties (properties normalized by density) and outstanding corrosion behavior [1]. Titanium and its alloys are also preferred for orthopedic and dental implants. The most common titanium based implant material is Ti-6Al-4V alloy due to its chemical stability, corrosion resistance, mechanical properties, and easy control of its microstructure [2]. Because of toxic effect of vanadium, many researchers are trying to develop safer titanium based biomaterials which are free of vanadium. For this reason, Ti-5Al-2.5Fe alloy which has almost similar properties to those of Ti-6Al-4V alloy is applicable for replacing implant applications.

In the current study, powder metallurgy was used for the production of Ti-5Al-2.5Fe alloy from elemental powders. The main restrictive cause of using titanium alloys in industrial applications is high production cost due to the high affinity of Ti alloy for oxygen and nitrogen in the air during casting process [3]. Powder metallurgy provides a near net shape for the final product with basic production parameters [4]. Powder metallurgical techniques provide using of 90% of raw material compared with casting. In powder metallurgical

techniques, new materials can be developed and produced in a wide range of field [5], [6].

Two distinct methods are used in Ti powder metallurgical production, the pre-alloyed (PA), and blended elemental (BE) methods. The blended elemental method is one of the most low-cost titanium production processes providing high degree of freedom in the selection of alloying elements, which is not possible for other production techniques [7].

To produce Ti-5Al-2.5Fe alloy from elemental powders, we used mechanical alloying (MA) technique. MA was developed by John Benjamin and his colleagues around 1966 [8]. In the MA, mixtures of powders are milled together. Material transfer is provided in this process to obtain a homogeneous alloy. Therefore, MA is a solid-state processing technique consisting of repeated welding, fracturing, and rewelding of particles in a high energy ball mill. At the beginning, MA was designed for oxide dispersion strengthened nickel and iron based superalloys. However, recently, a number of different equilibrium and non-equilibrium alloy systems have been used from elemental or pre-alloyed powders by using MA technique [8]. As mentioned before, mechanical alloying of Ti and its alloys are interesting because it is possible to obtain new alloy systems without requiring a melting process [9].

Mechanically alloyed powders were consolidated by vacuum hot pressing. As a pressure assisted sintering, hot pressing is helpful for obtaining higher density materials. Applying a pressure to a powder compact during sintering causes a direct increase in the driving force of densification and an increase in densification kinetics. External pressure enhances the densification rate, with this effect the sintering temperature as well as sintering time can be reduced resulting in suppressing of further grain growth [10]-[12]. In hot pressing, loose powders are placed into a die, which is then placed between two punches and heated under pressure. This operation may be conducted under protective atmosphere. For uniaxial hot pressing, graphite dies and punches are commonly preferred for their high conductivity and self-lubrication properties. The graphite setup is suitable for induction and resistance heating [13].

In this research, elemental powders were prepared by mechanical alloying and sintered by hot pressing. The mechanical and microstructural properties are discussed.

## II. EXPERIMENTAL

In this study, commercial pure Ti (Alfa Aesar), Al (SentasBIR A.Ş.) and Fe (Baymet Dış Tic. Ltd. Şti.) were used as starting materials. Fig. 1 shows the SEM images of elemental powders. Difference between the shapes and sizes of the

powders comes from powder production method. 5Al (wt.%) prevent the reaction of the powders from the air because of and 2.5Fe (wt.%) were added into 92.5 Ti (wt.%) to create Ti- high affinity of Ti against oxygen and nitrogen. 8 mm stainless 5Al-2.5Fe alloy then mechanical alloying was carried out in steel balls were used during mechanical alloying. stainless steel jar under  $10^{-2}$  mbar vacuum atmosphere to

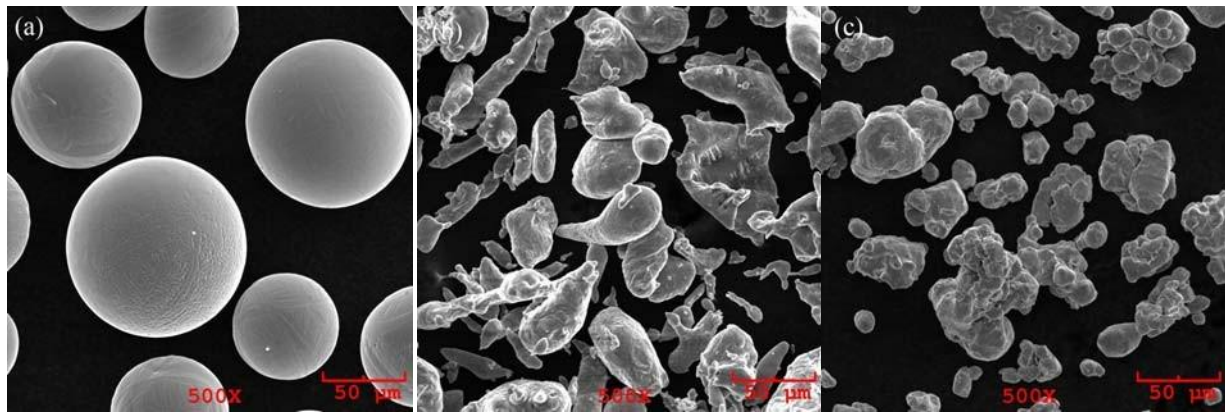


Fig. 1 SEM images of powders used in this study, (a) titanium, (b) aluminum and (c) iron powders

Sintering process were carried out by uniaxial vacuum hot pressing (DIEX Corp.). Mechanically alloyed powders were poured into a graphite die as seen in Fig. 2. Hot pressing process was carried out at 950 °C for 30 min holding time under  $10^{-4}$  mbar vacuum atmosphere. Pressure during sintering process was kept constant as 50 MPa. Dimensions of sintered samples were measured as 20 mm in diameter and 4 mm thickness.

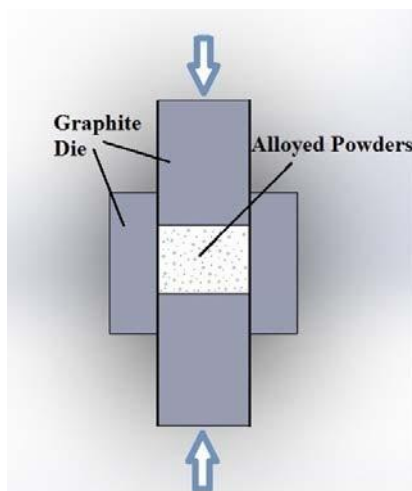


Fig. 2 Schematic image of die setup for hot pressing

To prevent the melting and leakage of Al from the graphite mold under pressure, during heating process, different temperature and holding steps were applied as seen in Fig. 3. In first step, 5 min at 550 °C, in second step 5 min at 650 °C, and in third step 5 min at 750 °C were waited for the dissolution of each elements in titanium. After these steps, samples were heated to exact sintering temperature for consolidation and final dissolution (950 °C). Sintered samples were prepared with metallographic operations by grinding with 600, 1000, and 2000 SiC papers. For polishing, 9, 6, 3, and 1 μm diamond solutions were used. Density measurements have been examined by Archimedes' method.

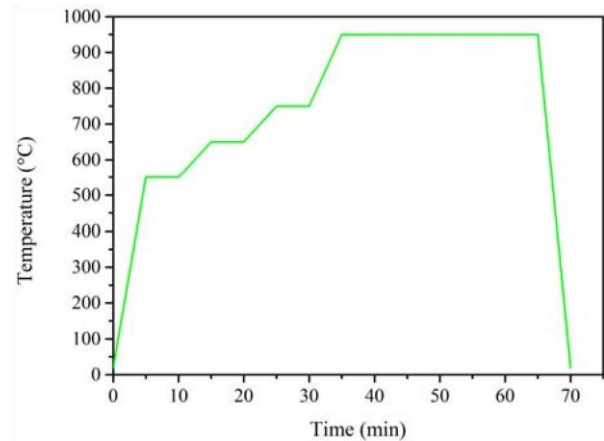


Fig. 3 Sintering cycle of mechanically alloyed powders by hot pressing

Polished samples were etched with Kroll's solution (3 HF+6HCl+91H<sub>2</sub>O wt.%) about 2 secs, then samples were investigated by optical microscopy (Olympus BX41M-LED) and scanning electron microscopy (JEOL JSM-6060) equipped with EDX for microstructural analysis. Hardness tests were carried out in Vickers scale, using 10 kgf and 10 s duration time. For bending test, samples have been cut in 4x4x20 dimensions by precise cutting, then three-point bending test was carried out with 0.5 mm/min cross-head speed. XRD analysis was carried out with an acceleration voltage of 40 kV, 20 mA current, and Cu-K radiation ( $\lambda=1.544 \text{ \AA}$ ) with the scanning angle ranged from 10° to 80° and the scan rate was 2°/min.

### III. RESULTS

Theoretical density of pure Ti is about 4.5 g/cm<sup>3</sup> and Ti5Al-2.5Fe is about 4.45 g/cm<sup>3</sup>. According to Archimedes results, pure Ti is about 4.40 g/cm<sup>3</sup>, and Ti-5Al-2.5Fe is about 4.45 g/cm<sup>3</sup>, which means that sintered samples have 98% relative density. Therefore, sintering process was carried out successfully, and

there is no need for higher sintering temperature or holding times. Siqueira et al. [7] in their research have used elemental Ti, Al, and Fe powders to create Ti-5Al-2.5Fe alloy. They sintered the alloy from 700 °C to 1400 °C for 2h by conventional powder metallurgical methods. Results showed that 96% density have been obtained for the samples sintered at maximum temperature of 1400 °C. These results show the advantages of hot pressing method for density compared to conventional sintering methods.

Optical microscopy images in Fig. 4 show that  $\alpha+\beta$  basketweave structures have been observed in sintered Ti-5Al-2.5Fe alloy. It can be seen that  $\alpha$  structures consist of dissolution of Al inside Ti as white contrast and  $\beta$  phase between  $\alpha$  phases with dark contrast. Shape of  $\alpha$  plates could be described as coarse, needle like, or lamellar.

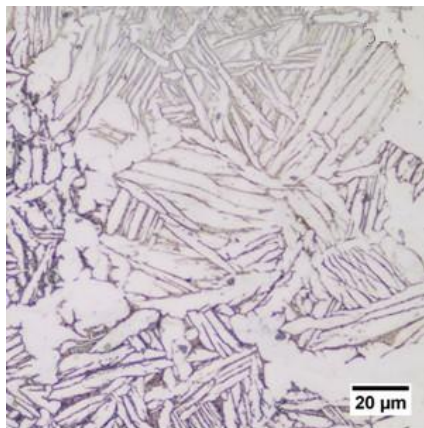


Fig. 4 Microstructure of Ti-5Al-2.5Fe alloy by optical microscopy

SEM images given in Fig. 5 also support these results. EDX analysis, taken from  $\alpha$  and  $\beta$  phases, shows that  $\alpha$  plates consist of Ti-Al solid solution, and  $\beta$  phases consist of Ti-Fe solid solution. Dissolved Al has been accumulated around  $\alpha+\beta$  phase and created  $Ti_xAl_y$  intermetallic phases. It means that a longer mechanical alloying time is necessary for a whole diffusion of Al into Ti matrix.

Elemental distribution analysis of Ti-5Al-2.5Fe is given in Fig. 6. It can be seen that distribution of alloying elements is homogenous in the matrix.

Bending results can be seen in Fig. 7. When compared with reference sample (pure Ti), alloying elements increased the bending strength. 720 MPa bending strength was determined for pure Ti sample, while with the addition of Al and Fe elements, it increased sharply to 1200 MPa.

Vickers hardness results are shown in Fig. 8. When compared with the reference sample (pure Ti), hardness increased in Ti-5Al-2.5Fe alloy from 225 HV to 268 HV, which describes that alloying elements in Ti matrix enhanced hardness properties. These results can also be attributed to new hard intermetallic phases as  $Ti_3Al$  and  $Ti_2Al_5$ .

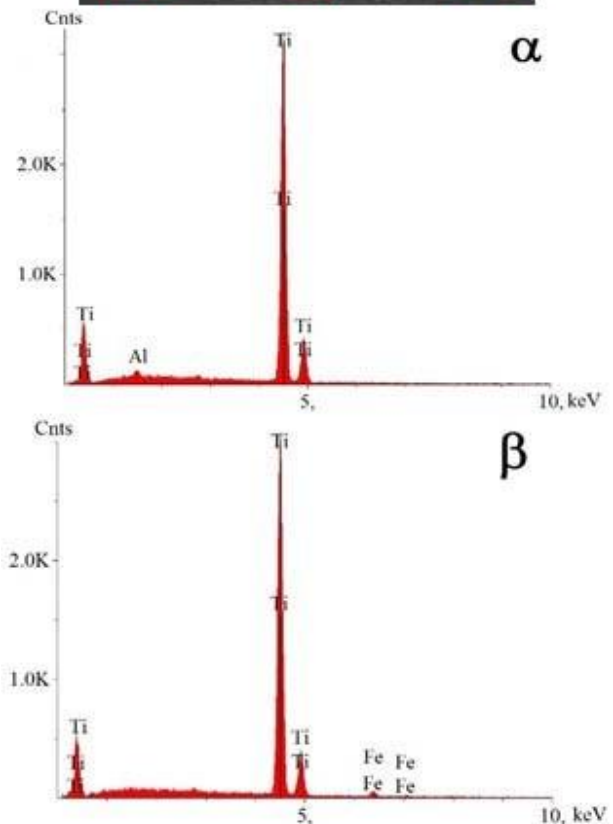
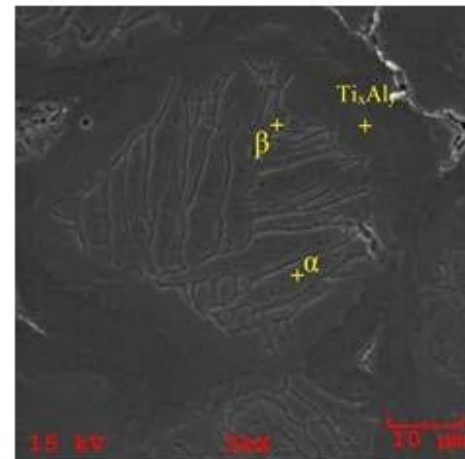


Fig. 5 SEM image of Ti-5Al-2.5Fe alloy with EDS analysis

Phase identification research has showed that new phases occurred in the main alloy. According to XRD results shown in Fig. 9, pure Ti consists of  $\alpha$  and  $Ti_xO_y$  phases. Addition of Al and Fe creates new phases in the structure. With dissolution of Fe within Ti matrix,  $\beta$  phase was obtained. With dissolution of Al,  $\alpha$  phase and new intermetallic phases occurred in the alloy as  $Ti_3Al$  and  $Ti_2Al_5$ . These new intermetallic phases play an important role on the mechanical properties of the main alloy.

#### IV. CONCLUSION

In the current study, we successfully produced  $\alpha-\beta$  type titanium alloy by mechanical alloying and sintering with

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