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Research Paper / Makale

Effect of Different Sintering Temperatures on Microstructure and Mechanical Properties for Pure Al Material Produced by Powder Metallurgy

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Abstract: Addressing the need for light materials in today's industry, the present study aims to analyze the effects of sintering parameters on pure Al material manufactured using powder metallurgy technique. 99% pure and commercially obtained Al powder at a dimension of 180 µm was subjected to pre-heating and placed in a specially designed die. Al powder was compacted into a die using an upper punch with 500 MPa pressure and later removed from conical die for the sintering process. In order to find the optimal sintering temperature and duration, the compacted samples were sintered separately at 500^{o}C, 550^{o}C and 600^{o}C for 45, 60 and 75 minutes for each temperature value. Microstructural analysis, microhardness test and density test of each sample obtained from the sintering process were performed to determine the most optimal sintering durations at a given temperature, and changes caused during these processes were observed. As a result of examinations, it was determined that the optimal sintering temperature and time was 500 ^{o}C/60min, 550 ^{o}C/45min, 600 ^{o}C/45min. Finally, the samples obtained in different sintering durations for three different temperatures were subjected to a tensile test. According to the tensile test result s, it was determined that the most suitable sintering parameter for pure Al was 600 ^{o}C/45min.

Keywords: Powder metallurgy; Sintering; Pure Al; Microstructure

Toz Metalurjisi Yöntemi ile Üretilen Saf Al Malzemenin Farklı Sinterleme Sıcaklıklarının Mikro Yapı ve Mekanik Özelliklerine Etkisi

Öz: Bu çalışmada,günümüzde hafif malzemelere olan ihtiyacın artması nedeniyle toz metalürjisi yöntemi ile Al üretimi yapılarak, sinterleme parametrelerinin üretilen malzeme üzerindeki etkileri araştırılmıştır. Bu araştırma sırasında ticari olarak elde edilen %99 saflıkta ve 180 µm boyutlarındaki Al tozu ön ısıtma işleminden geçirilerek özel olarak hazırlanmış kalıp içerisine yerleştirilmiştir. Üst zımba vasıtasıyla kalıp içerisinde 500 MPa basınç ile sıkıştırılan Al tozu iç içe geçmiş konik kalıptan çıkartılarak sinterleme işlemi için hazır hale getirilmiştir. Doğru sinterleme sıcaklığı ve zamanının belirlenebilmesi için sıkıştırılmış numuneler sırasıyla 500, 550 ve 600 oC sıcaklıklarda ve her bir sıcaklık değeri için 45, 60 ve 75 dk sürelerde ayrı ayrı sinterlenmiştir. Sinterleme işlemi sonrasında elde edilen her bir numunenin mikro yapı incelemeleri, mikro sertlik testleri ve yoğunluk deneyleri yapılarak belirlenen sıcaklık değerlerindeki en iyi sinterleme süreleri ve bu sürelerin numunelerde meydana getirdiği değişimler tespit edilmiştir. Yapılan incelemeler sonucunda en iyi sinterleme sıcaklık ve sürelerinin 500 oC/60dk, 550 oC/45dk, 600 oC/45dk olduğu belirlenmiştir. Üç ayrı sıcaklık değeri için tespit edilen sinterleme sürelerinde hazırlanan numuneler, çekme testine tabi tutularak elde edilen sonuçların tamamı bir araya getirilip saf Al için en iyi sinterleme parametresinin 600 oC/45dk olduğu belirlenmiştir.

Anahtar Kelimeler: Toz metalürjisi; Sinterleme; Saf Al; Mikroyapı

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1. Introduction

Aluminum is widely used in a number of applications in automotive, electricity, electronics and construction technology thanks to its characterization of weight/strength ratio, high corrosion resistance, low specific gravity, low cost, environment-friendly nature and easy manufacturing [1,2]. The studies on aluminum have gained momentum in recent years due to the increasing need for light and high-performance materials [3,5].

Various methods are used to manufacture aluminum and its alloys. However, when several factors such as production cost, energy, time and occupational health and safety factors are taken into account, it has been observed that powder metallurgy (P/M) offers greater advantages compared to other conventional manufacturing methods [6,7]. P/M is the technique of manufacturing materials difficult to be manufactured using materials in powder form with a minimum level of tolerance. It is based on the principle that metal powder is compacted into a die and sintered below its melting temperature [8,9].

It is known that the most effective way to improve the performance of materials manufactured using this technique is to increase its density, which can be achieved by using sintering method (9). It is possible to reach theoretical density of a reference material by 99% when optimal sintering parameters are used [11]. Eksi, compacted Al powder with 600 MPa pressure and applied sintering to the material at a temperature of 600^{o}C for 20 minutes, yielding a sample with a theoretical density of 99.3%. [12]. Sinha and Farhat reported that when cold isostatic pressing was applied to pure aluminum, the density was calculated as 2.51 gr/cm^3 and that it reached 2.62 gr/cm^3 in a sintering duration of 20 minutes at 560^{o}C [13]. Herzallah et al. compacted Al material with 560 MPa pressure and applied sintering to it at 600^{o}C for three hours in a vacuum furnace, and the hardness of the manufactured material was calculated as 30 HV [14].

Pure Al manufacturing using P/M technique is seldom studied in the existing literature due to a lack of binding alloys and declining popularity of pure materials. In this respect, the present study focuses on the effects of sintering process on pure Al manufacturing, which is one of the most important steps of P/M. Thus, it will shed light on future studies and offer comparative values for alloyed Al and Al matrix composite manufacturing using P/M technique.

2. Material and Method

In the present study, 99% pure Al powder at a dimension of 180 µm were commercially obtained. Inner walls of the conical die specially designed for P/M applications were lubricated using zinc strearate, which helps reduce die-metal powder friction during the compacting of metal powder with high pressure and facilitates the removal of the manufactured sample from the die following the compacting process [15]. In order to eliminate lubricant removal process prior to the sintering process, zinc strearate was not mixed with Al powder. Pre-heating was applied at 300^{o}C to powders after die preparation process to reduce its moisture and eliminate oxide layers. Following the pre-heating process, metal powder was stirred using a steel wire while it was being poured into the die to prevent the formation of spaces in the die. Upper surface temperature of metal powder was measured as 70 ^{o}C using a thermocouple. Compacting was performed using UTEST press with a capacity of 2000 kN at a loading speed of 0.6MPa/s. All samples were compacted with 500 MPa. As shown in Figure 1b-d, the die was opened and the aluminum sample with a height of 90 mm and a diameter of 20 mm was removed from the die.

The compacted samples were cut into pieces of 20 mm in order to determine optimal sintering parameters. Later, they were subjected to sintering process at 500, 550 and 600^{o}C for 45, 60 and 75 minutes for each temperature value in a furnace without atmosphere control. Finally, they were cooled up to room temperature following the sintering process.

Figure 1. a) Pressing, b-d) Removal of samples following the pressing process

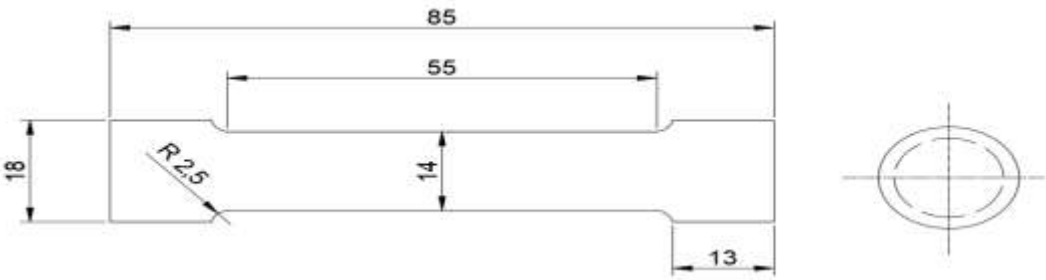

Figure 2. Tensile strength size (mm)

The samples obtained in each step were subjected to grinding and polishing processes, and etched in Keller chemical (1 ml HF, 1.5 ml HCl, 2.5ml HNO_3, and 95 ml H_2O). Afterwards, a Nikon optical microscope was used to analyze the microstructure of the samples and a Shimadzu GMV-20 device was used to measure the microhardness of the samples with a load of 100 grams. Experimental density of the samples was calculated using Archimedes' principle and analyzed in tables. Tensile test samples were prepared as shown in Figure 2 (non- standard sample) and tested using a ZwickZ100 device at a speed of 1 mm/min.

3. Results and Discussion

3.1. Microstructural Properties

Microstructural image of the pure aluminum sample manufactured using P/M technique prior to sintering process is shown in Figure 3. When microstructure image was examined some gaps in black color were observed after pressing process. These gaps are called porosity, and sintering process usually reduces the number of such porosities [16]. Another process which is known to reduce porosity effectively is compacting pressure limit [17]. Pure Al powders compacted under 500 MPa pressure but ıt was observed in preliminary steps of the present study that pressure limits higher or lower than this point caused breaks in samples while they were being removed from the die. Therefore, it was assumed that 500 MPa pressure was the optimal limit for the present study.

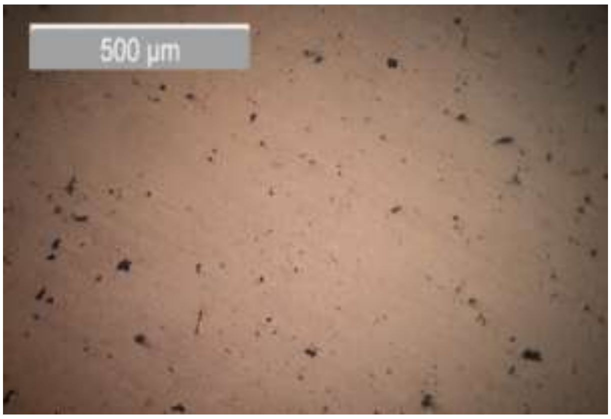

Figure 3. Microstructural images of the aluminum sample manufactured using P/M prior to sintering process

In Figure 4, microstructural images of the pure aluminum sample manufactured using P/M technique at different sintering temperatures and for different durations are shown. In addition, in Figure 4a-c, microstructural images obtained following a sintering process at 500^{o}C for 45, 60 and 75 minutes are shown. When the porosity structure prior to and following the sintering process at the temperature given above for 45 minutes was compared, it can be seen that the porosity distribution is simplified but the porosity structure is enlarged. After a sintering process of 60 minutes, it was observed that porosity structure gradually shrank and spread, which can be considered as an indicator of an advance towards a spherical porosity along with a tendency to isolate porosities due to the increasing sintering duration [18]. 75 minutes sintering process resulted in a mixed version of the structures observed in previous durations, which can be attributed to changes in grain size due to the increasing sintering duration.

It is shown in Figure 4d-f that porosity increased with increasing sintering duration during sintering processes at 550^{o}C for 45, 60 and 75 minutes, respectively. Finally, microstructural porosities occurring as a result of increasing sintering duration in 45, 60 and 75-minute sintering processes at 600^{o}C are shown in Figure 4 g-i, respectively.

The microstructural analysis obtained from sintering processes at different temperatures and for different durations in the present study indicated that the most optimal sintering durations were 60 minutes at 500°C, 45 minutes at 550°C and 45 minutes at 600°C. Because the structure was 99% pure aluminum, compound formations were ignored and only porosities were assessed.

Figure 4. Microstructural images of the pure aluminum sample manufactured using P/M technique at different sintering temperatures and for different durations a) 500°C / 45 min., b) 500°C / 60 min., c) 500°C / 75 min., d) 550°C / 45 min., e) 550°C / 60 min., f) 550°C / 75 min., g) 600°C / 45 min., h) 600°C / 60 min., i) 600°C / 75 min.

3.2. Microhardness Properties

In order to support experimental results obtained from the microstructural analysis, a microhardness test was applied to the samples manufactured at different sintering temperatures and for different durations. Microhardness values are shown in graphs in Figure 5. It can be seen that the highest microhardness value, 58 HV, was obtained at 600°C with a sintering process of 45 minutes. It was also observed that sintering duration was inversely proportional to microhardness values. In sintering process at 550°C, it was found that the highest microhardness value was obtained as 53.62 HV for 45 minutes. Similar to the sintering process at 600°C, sintering duration was inversely proportional to microhardness value. It can be suggested that this resulted from grain growth occurring as a result of a longer sintering duration [19].

Finally, In sintering process at 500°C, it was determined that the highest microhardness value was obtained as 52.6 HV for 60 minutes. It was also observed that microhardness values decreased when sintering process was longer or shorter than this duration. In addition, a sintering process at this temperature for 45 minutes resulted in a decreasing microhardness value, indicating that sintering processes must not be performed at temperatures lower than 500^{o}C. Thus, it can be concluded that the experiment in the present study was conducted at an accurate temperature.

A higher microharndess value in P/M processes does not necessarily mean finding accurate sintering parameters. Although the microhardness value of the pure Al sample obtained from the compacting process was 67.8 HV, the most optimal microhardness value was calculated as 58 HV following a sintering process at 600oC for 45 minutes. It was thought that the reason for the occurrence of this situation is due to the process of removing the stresses on the powders by heat during sintering (11).

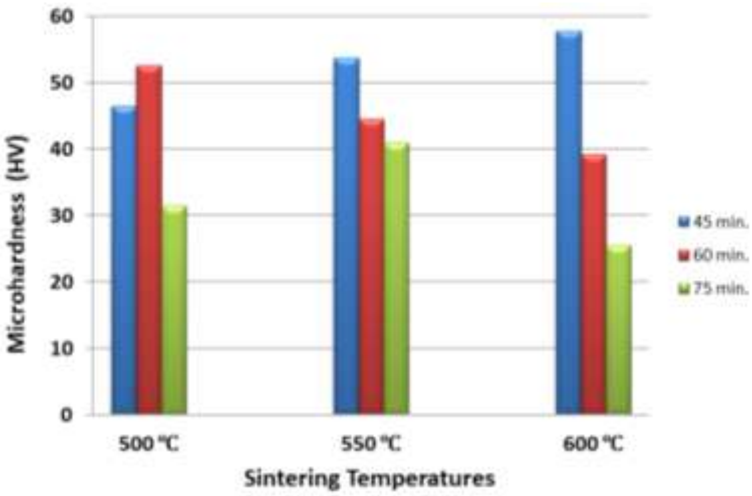

Figure 5. Microhardness values at different sintering temperatures and for different durations

3.3. Density Calculations

The other experiment required for the determination of accurate sintering parameters in manufacturing pure aluminum sample using P/M technique is density calculation. Archimedes' principle was used to analyze density properties of the manufactured materials. The density of a solid material dipped in a liquid whose density is known is calculated using the following equation.

$$Density\ of\ a\ Solid = \frac{w_{kr}}{(w_{kr} - w_s)} \cdot \rho_s \qquad (1)$$

Here, w_{kr} and w_s represent the weight of the dry sample in air and suspended in water, respectively. ρ_s is the density of the liquid used for calculation. Experimental density values of the manufactured samples using Equation 1.1 are given in Table 1.

Table 1. Experimental density values obtained in different sintering temperatures and durations

Sample	Density before sintering (gr/cm^3)	Sintering Temperature	Density after sintering duration 45 min (gr/cm^3)	Density after sintering duration 60 min (gr/cm^3)	Density after sintering duration 75 min (gr/cm^3)
99% Pure Al	2,50	500 ^{o}C	2,45	2,52	2,30
		550 ^{o}C	2,57	2,48	2,36
		600 ^{o}C	2,61	2,33	2,20

When the density values of the manufactured sample prior to and following the sintering process are compared, it can be observed that sintering temperatures and durations predicted during the microstructural analysis yielded the most optimal parameters. It was found that the most optimal density value was calculated as 2.61 gr/cm^3 obtained at 600^{o}C for 45 minutes. However, sintering durations longer than 45 minutes in this temperature value led to a decrease in density, which reveals the effect of thermal expansion on density and indicates that thermal expansion in materials occurring as a result of increasing sintering duration results in a decreasing experimental density value [20]. It was also found that the most optimal sintering duration for a sintering process at 550^{o}C was 45 minutes, while a decrease was observed in density when the sintering duration was longer. Finally, it was found that the most optimal sintering duration at 500^{o}C was 60 minutes, and the density decreased when this sintering duration was kept longer or shorter.

3.5. Tensile Test

It was demonstrated in the experiments in the present study that the most optimal sintering temperatures and durations were 500^{o}C/60 minutes, 550^{o}C/45 minutes and 600^{o}C/45 minutes. The samples manufactured using these three parameters were used for the tensile test. The preparation of the samples and tensile test results are shown in Figure 6. When the tensile test results are analyzed, it can be observed that a tensile strength value of 36 MPa was obtained following a sintering process at 600^{o}C for 45 minutes, which was the highest tensile strength among three samples. The second highest tensile strength value was calculated as 28 MPa following a sintering process at 550^{o}C at 45 minutes. The lowest tensile strength value, however, was calculated as 18 MPa following a sintering process at 500^{o}C for 60 minutes. The tensile test results also demonstrated that the highest elongation value was obtained as 1.5 mm in a sintering process at 550^{o}C for 45 minutes, followed by an elongation value of 1 mm at 600^{o}C for 45 minutes. The lowest elongation value was calculated as 0.3 mm in a sintering process at 500^{o}C for 60 minutes.

Although the above-mentioned findings suggest that the highest tensile strength was obtained in a sintering process at 600^{o}C for 45 minutes, the most optimal toughness value was obtained using in a sintering process at 550^{o}C for 45 minutes. However, a brittle fracture was observed in the sintering process at 500^{o}C for 60 minutes.

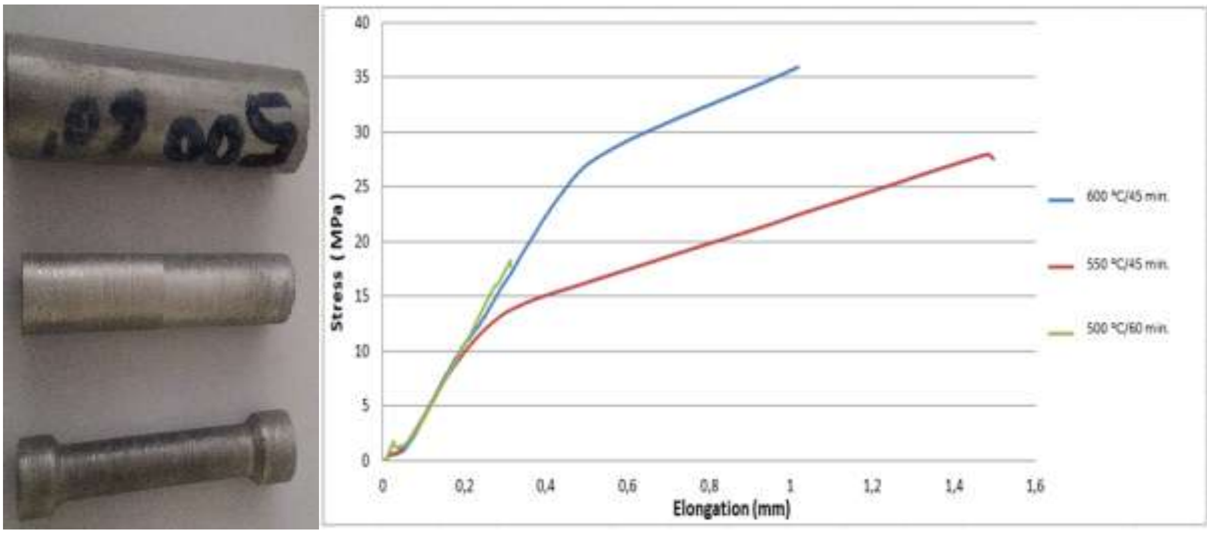

Figure 6. Preparation of the samples and tensile test results

4. Conclusion

The present study analyzed the effects of sintering process at 500^{o}C, 550^{o}C and 600^{o}C for 45, 60 and 75 minutes for each sintering temperature on the microstructural and mechanical properties of pure aluminum sample manufactured using powder metallurgy technique. The following conclusions were drawn in the experiments conducted in order to find optimal sintering parameters.

1. The microstructural analysis of the samples sintered at different temperatures for different durations demonstrated that porosities occurred in the manufactured samples. In addition, it was found that changing sintering temperatures and durations caused structural and proportional differences in these porosities. The experiments at different sintering temperatures for different durations demonstrated that the lowest structural gap was observed following a sintering process at 600^{o}C for 45 minutes.

2. Microhardness measurements performed following each sintering process indicated that the samples had different microhardness values. It was observed that except 500^{o}C, sintering durations were inversely proportional to microhardness values. The highest microhardness value for 500^{o}C temperature was obtained at 60 minutes, and it decreased in other sintering durations, indicating that the experiments in the present study started with an optimal sintering temperature and duration.

3. The most optimal density value was calculated as 2.61 gr/cm^{3} in a sintering process at 600^{o}C for 45 minutes. It was observed that Table 1 obtained results using density calculations was similar results to microhardness graph.

4. The samples manufactured in the optimal sintering durations corresponding to each sintering temperature were subjected to a tensile test. It was found the highest tensile strength value was obtained as 36 MPa in a sintering process at 600^{o}C for 45 minutes, while the highest toughness value was calculated in a sintering process at 550^{o}C for 45 minutes. However, a brittle fracture was observed in the sintering process at 500^{o}C for 60 minutes.

5. The present study sheds light on various mechanical properties of pure aluminum power manufactured using powder metallurgy technique. It also offers valuable information for future studies which will focus on aluminum alloys to be manufactured at different sintering temperatures.

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