



## EXPERIMENTAL INVESTIGATION OF P/M PARTS MANUFACTURING CONDITIONS USING AA2014 AND ELEMENTAL AL AND CU POWDERS

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### Keywords

*Powder metallurgy,  
Elemental Aluminum powder,  
Pre-alloyed AA2014,  
Sintering,  
Microstructure,  
Mechanical properties.*

### Abstract

The purpose of the work is by comparing the mechanical tests results, fracture surface analysis and metallographical studies of the samples obtained from two different groups of powders (prealloyed, elementally formed AA2014), trying to determine whether the values obtained by the use of elemental powder can have sufficient mechanical properties as design material. Samples were produced by using pre-alloyed (AA2014), elemental aluminum and copper powders by classical powder metallurgy (PM) method. As the same proportion that AA2014 alloy contains, copper was added elementally to the commercial pure Aluminum powder and cold compressing was performed in the cast. In the similar conditions, blending and mixing operations were carried out to obtain similar composition from pre-alloyed (AA2014) powders and elemental powders. In the sintering conditions, three different temperatures (600°C, 610°C, 620°C) were used and the sintering time kept constant (30 mins) . At this stage, it is expected that the aluminum matrix will dissolve Copper atoms to form solid solution. Metallographic studies carried out to examine the realization of this aspect. As a result, aluminum and copper powders can be used in elemental form instead of pre-alloyed AA2014 for a long die life.

## AA2014 VE ELEMENTEL Al VE Cu TOZLARI KULLANILARAK T/M PARÇA İMALATI ŞARTLARININ DENEYSEL ARAŞTIRILMASI

### Anahtar Kelimeler

*Toz Metalurjisi,  
Elemental Alüminyum Tozu,  
Ön-alaşımli 2014,  
Sinterleme,  
Mekanik Özellikler.*

### Öz

Bu çalışmanın amacı, iki farklı AA2014 toz grubundan (önalaşımli, elementel formda) elde edilen numunelerin mekanik test sonuçları, kırık yüzey analizi ve metalografik çalışmalarını karşılaştırarak, elementel tozun kullanımıyla elde edilen değerlerin tasarım malzemesi olarak yeterli mekanik özelliklere sahip olup olmadığını belirlemeye çalışmaktır. Ön alaşımli (AA2014), elemental alüminyum ve bakır tozları kullanılarak klasik toz metalurjisi (TM) yöntemiyle numuneler üretilmiştir. AA2014 alaşımının içerdiği oran kadar, ticari saf Alüminyum tozuna bakır eklenmiş ve dökümde soğuk presleme yapılmıştır. Benzer koşullarda, ön alaşımli (AA2014) tozlar ve element tozlarından benzer bir bileşim elde etmek için harmanlama ve karıştırma işlemleri gerçekleştirilmiştir. Sinterleme koşulları olarak, üç farklı sıcaklık (600°C, 610°C, 620°C) kullanılmış ve sinterleme süresi sabit tutulmuştur (30 dakika). Bu aşamada, alüminyum matriksinin katı çözeltiler oluşturmak için Bakır atomlarını çözmesi beklenir. Bu durumun gerçekleşmesini incelemek için metalografik çalışmalar yapılmıştır. Sonuç olarak, alüminyum ve bakır tozu, uzun bir kalıp ömrü için alaşımli AA2014 yerine elemental olarak kullanılabilir.

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## 1. Introduction

The three master reasons for using P/M are economic, uniqueness, and captive applications. With the developing technology the new materials developed along, attract the attention of the automotive industry. Especially aluminum alloys are a good candidate material for producing vehicles with lower weight (Hirsch, 2004; Anonymous, 2003; Edwards, 2004; Waldman, 1986). Powder metal aluminum alloys are widely preferred in the production of several parts such as camshaft bearings (Dowson and Whittaker, 2008). Along with some other disadvantages of prealloyed such as lower compact density, using prealloyed powders accelerates the wear of the molds due to their high hardness compared to the elemental powder blends, which in turn reduces the life expectancy of the molds which are costly. Some researchers have shown that it is possible to produce high strength aluminium alloys via P/M method starting from elemental powders (Gökçe, 2013). In this study, it was aimed to form 2014 aluminum alloy with exactly chemical composition amount by elemental powders; we probe the sintering behavior of a Pre-alloyed and elemental aluminum 2014 alloy including the effect of compaction pressure, sintering temperature and time.

## 2. Literature Review

Durmuş at al. 2005, they were investigated the age hardening behavior of PM AA2014 alloy. The specimens were made from powder basic materials by pressing (at 600 MPa) and sintering ( $600 \pm 5$  C,  $610 \pm 5$  C,  $620 \pm 5$  C). Solution treatment ( $510 \pm 5$  C), quenching (water) and aging (20 C,  $150 \pm 5$  C,  $200 \pm 5$  C) were the steps that occurred in age-hardening. The hardness test results were showed that the sintering temperature, suitable solution treatment and aging operations has great influence on hardness characteristics of the PM alloys.

Rudianto at al. 2011, were focused on the evaluation of the sintering and mechanical properties of Al-14Si-2.5Cu-0.5Mg powder alloy by using conventional powder metallurgy techniques. The sintering of the alloy was made up to the temperature of  $570$  °C under nitrogen atmosphere. Sintered specimens were later T6 heat treated to improve mechanical properties. They concluded that the Sintered density could reach up to 97% of true density in the optimum sintering condition, and after T6 treatment, the alloy showed the UTS of 280 MPa and the hardness of 80 HRB.

Chua vd. 2014, they were investigated the effects of compaction method (uni-axial die compaction and cold isostatic pressing) on two aluminium powder metallurgy alloys. Both systems (PM2324, PM7075) were mixtures of elemental and master alloy powders. Mechanical and physical properties of prepared samples were compared by using both methods. Results indicated that sintered products of a largely comparable quality could be realised for both alloys regardless of the compaction approach employed.

Gökçe vd. 2016, they were investigated the Sintering and aging behaviours of Al-Cu-mg powder metallurgy (P/M) alloy produced from elemental powders. After thermal analysis, tests were carried out on Al-4Cu alloys with magnesium content 0.5%, 1 and 2 by weight. Results indicated that by adding magnesium to the Al-Cu system, grain size reduction was realized in the range of 14-45%, and the addition of 1 mg by weight was the most effective to increase the tensile strength of Al-Cu P/M alloys after both sintered and heat treatment conditions.

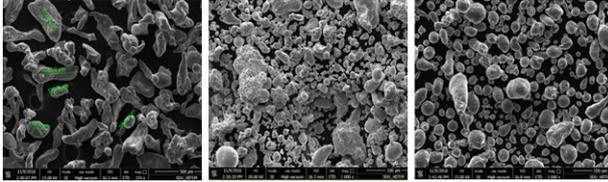
Gökçe at al. 2013, they were aimed to develop an alternative way to produce high strength aluminium alloys using Powder Metallurgy (PM) technique. 4 different types of Al-Cu based alloys were produced (i.e., Al4Cu, Al4Cu0.5Mg, Al4Cu1Mg and Al4Cu2Mg) by adding micro (< 0,5 wt.%) level additions of elemental powders into the developed alloys and heat treatments were applied subsequently. The specimens where compacted at 400 Mpa. The sintering was conducted for 2 hours under nitrogen atmosphere, Sintering temperatures and expansion-shrinkage behaviour of the alloys were determined by using DSC and dilatometry. As a result, it was shown that, it is possible to produce high strength aluminium alloys via PM method starting from elemental powders.

## 3. Material and Methods

The raw powder used was the commercial powder (Al-4Cu-2.26Mg, density= $2.67$  g/cm<sup>3</sup>). Zn-stearate used as a lubricant for all used powders. Water atomized (96% Al) and (4% Cu) and also adding powders are mixed in a double sided conical mixer at a speed of 22 rpm for 20 minutes. Pre-alloyed AA2014 are mixed with lubricant in the same manner. The properties of the used materials are shown in Table 1. The characteristics and morphology of the powders used in the work were inspected using SEM and representative particle morphologies are shown in Figure 1.

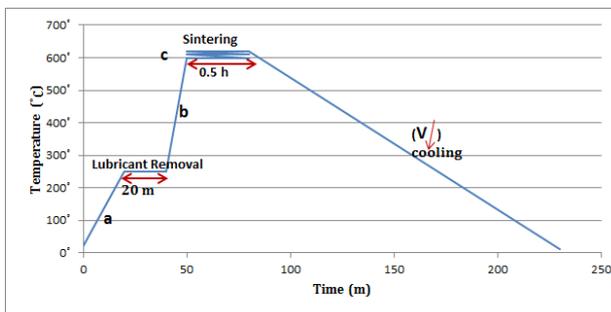
**Table 1.** The properties of the used materials

MATERIAL	PROPERTIES
Al	Composition: 99.9 % (purity) Particle size: -100 mesh between( $\pm 95 \mu\text{m}$ and $\pm 351 \mu\text{m}$ )
Cu	Composition: 99.5 % (purity) Particle size: differ between (1.781 $\mu\text{m}$ and 33.23 $\mu\text{m}$ )
AA2014	Composition: 99.5 % (purity) Particle size: differ between (2.487 $\mu\text{m}$ and 70.10 $\mu\text{m}$ )

**Figure 1.** Representative particle morphology of water atomized Al powder, Cu powder and pre-alloyed AA2014 powder from left to right; showing irregular Al articles and mostly spheroidal Cu and AA2014 particles

### 3.1. Compaction and Sintering

The mixtures were then compacted with a (400 mm.strok) uniaxial hydraulic press. For each compaction, a ( $\approx 14.168$  g) of powder was used and discharged into the die cavity. Rectangular specimens with the size of 55x10x10 mm (14.168 g of powder) were pressed at pressures in the range of (150-250) MPa for the mixed elemental powders and pre-alloyed powders were pressed in the range of (150-650) MPa in order to study the compaction properties and so to know the best compaction pressure which it can get high green density. The green density was calculated by measuring the dimensions by micrometer and the weight of the specimens by Precision Balance (type 0.001 g sensitive electronic balance). The sintering process was carried out under high purity (99.999%) atmosphere in the ceramic horizontal tube furnace at three different temperatures (600°C, 610°C, 620°C) for 30 m Figure 2. The sintered densities were calculated using Archimedes density measurement technique. Then, these rectangular bars are used for mechanical testing (Al-Obaidi, 2018).

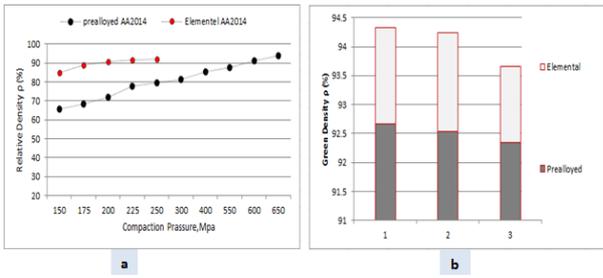
**Figure 2.** Example of temperature-time graph used in sintering processes (a and b = 5-7°C / min), (c = 610°C, 620°C)

### 3.2. Mechanical Testing and Metallography

After sintering some of the specimens are used for the three point bending test; to do this (400 mm.strok) uniaxial hydraulic press was employed, and the others were used for impact test by Charpy impact test device of capacity of 30 (kp.m.). Brinell hardness on the B scale was measured for the as-bended and as-impacted materials at a load of 62.5 kgf. Scanning electron microscope (SEM) was used for the microstructural examination of the broken surfaces after mechanical tests. The as-tested samples were prepared conventionally for metallographic test, and then they polished using diamond paste in spray form. Optical microscope was used to study the microstructure of the polished compacts.

## 4. Results and Discussions

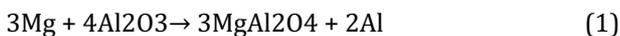
After mixing process the elemental and pre-alloyed powders were compacted at different pressures to study their compressibility. The final product (sample) of the desired size and shape is obtained on the basis of the largest relative density value that can be reached. The compaction pressure-relative density curve for each elemental and pre-alloyed 2014 Al alloy presented graphically in (Fig. 3, a). Pressing pressures in the range of (150-650) MPa for the pre-alloyed AA2014 were tested and it was observed that there was no significant change in the density of the powders after pressing pressure of 625 MPa in accordance with the results obtained (Fig. 3, a). For this reason, the pressing pressure was selected as 650 MPa. However, the pressing pressures were tested for elemental (150-250) MPa, and after the 250 MPa pressing pressure, 92% theoretical density was obtained, and 250 MPa was selected as the pressing pressure since there was no change thereafter. The reason of difference in compression pressure values applied on two different Aluminum alloys is the specific hardness of the metal or alloy involved the particle shape, the internal porosity, and the particle size distribution, the addition of alloying elements or solid lubricants. So the raw density of powders with coarse grain size distribution (elemental alloy) is higher. In addition to Cu, in the pre-alloyed powders may be magnesium or the like alloy elements. For this reason, dislocation movements become more difficult, resulting in higher hardness and strength values. As the yield strength value also increases, more force is required to start the plastic deformation. However, pure or elemental powders do not contain such alloying elements. Compression pressures are therefore found to be lower than for pre-alloyed powders. These reasons are the same reasons of being the green density of elemental compacts higher than the green density of pre-alloyed one (Fig.3, b).



**Figure 3.** Mechanical properties of pre-alloyed AA2014 versus elemental AA2014 alloys (a)Compaction pressure versus relative density, (b)Green density (%)

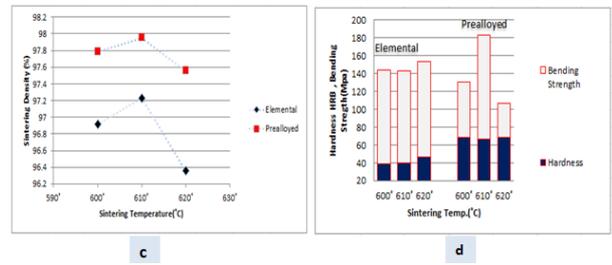
The densification behaviour during sintering of selected (pre-alloyed and elemental Al alloy) green compacts is illustrated in Figure 4.c which represents difference in sintered density at each sintering temperature and for the two alloys as compared. It is noticed that the rate of change of sintered density is higher for the compacts of lower green density (pre-alloyed); while its value is lower for the compacts of higher green density (elemental). The reason behind this is related with the driving force required for sintering. In the earlier stages of sintering, the driving force is produced by the surface energy, which is related with the internal surface area of the particles. For sintering, a decreasing green density indicates an increase in the amount of internal surface area and, as a result, a greater driving force (Kang, 2005).

According to the Al-Cu phase diagram, aluminum and copper makes eutectic phase at 548 °C between 6% and 53% copper content. In this work, AA2014 alloy (i.e., 4 wt.% Cu) was sintered at (600 °C,610°C,620°C) for 30 min. At these temperatures though, there are α-liquid phase formed as seen on the diagram (Fig. 5). Liquid eutectic fills pores and causes densification by capillary action. The forming of a liquid phase is also significant to obtain a high sintering density (Kang, 2005). As shown in Fig.4. c, the density was increased with increasing sintering temperature since a large amount of liquid was produced at the high sintering temperature. The highest sintered density, a relative density of ~98% for pre-alloyed and ~97% for elemental both were obtained at 610°C. However, the density rapidly decreased at the sintering temperature of 620°C because the liquid phase accumulated to one side and eventually large gaps (Fig.6,a) and grooves (channels) were formed and grown as shown in scanning electron micrograph images (Fig.6, b). (Figure 6. a, b) shows formation of blisters (Al<sub>2</sub>O<sub>3</sub>) on the surface of compacts because the N<sub>2</sub> medium does not have sufficient clean sintering furnace environment. This case only seen in pre-alloyed because of presence of Mg, Mg element in the aluminum matrix is very reactive and has the capability to react with the surface oxide layer of the Al powder and possible reaction is,

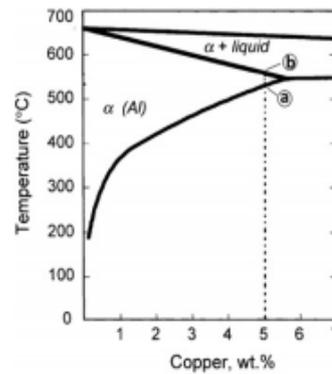


Pieckzonka et al. reported that Mg atoms in the Al particles immigrate to the surface oxide region and form a triple oxide Rudianto et al (2011). Surface aluminum oxide layer is possible to be broken by formation of ternary oxide. That was the possible reason to improve the sinterability of Al compacts.

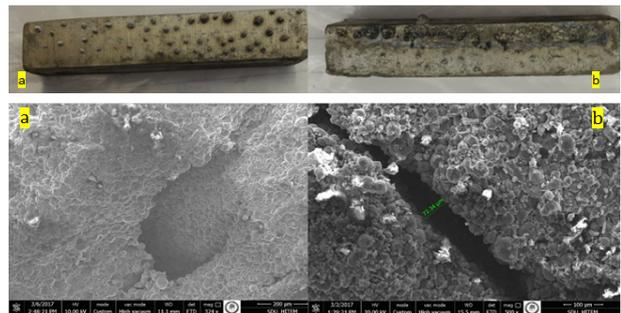
Figure 4.d shows that, generally in both alloys (elemental, pre-alloyed) strength values are higher than the hardness values. The hardness values and bending strength values are very close in 600°C and 610°C because of possible errors of ΔTsinter=10±3 during setting oven temperature it is thought that the temperature are close from each other, but increases in 620°C. In pre-alloyed one the hardness values are closes in three different sintering temperature but the strength values are change according to density were increase from 600°C to being higher in 610°C and go back to severe decreasing in 620°C (Al-Obaidi, 2018).



**Figure 4.** Mechanical properties of pre-alloyed AA2014 versus elemental AA2014 alloys (c)Sintering density versus sintering temperature (b)Hardness HRB and Bending strength versus sintering temperature

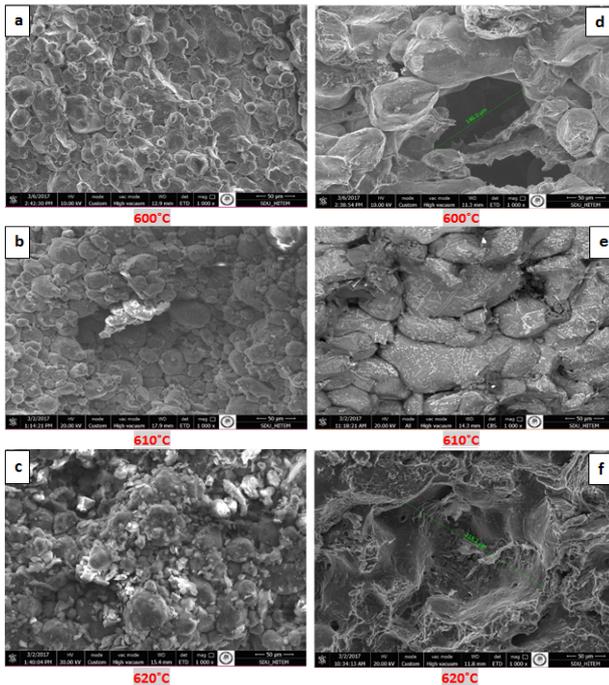


**Figure 5.** Al-Cu phase diagram



**Figure 6.** a and b. Surface and SEM images of prealloyed AA2014 sample sintered at 620°C

A presence of some large holes  $\approx (146\mu\text{m})$  was observed after SEM investigations for fracture surface of elemental alloy sintered at  $600^\circ\text{C}$  (Fig.7.d) vs. the pre-alloyed one shows few and very small pores at the same sintering temp. (Fig.7.a). Figure 7.e shows the grains and precipitates on the elemental alloy sintered at  $610^\circ\text{C}$ . Sintering has not been sufficiently achieved, the addition of mechanical locking between the powder particles has not resulted in complete diffusion. It looks like a crushed grain boundary break. A brittle fracture. It clearly shows insufficient sintering. A 97.9% (T.D) was achieved in this alloy after sintering. It was supposed that precipitation of intermetallic phases on the grain boundaries and inner sides of grains stops the motion of dislocations and thus intensively strengthens the material (have positive effect on the strength and micro-hardness of the alloy) Gökçe et al (2011). Fig.(7.f) shows the fractured surface appearance of the  $620^\circ\text{C}$  sintered elemental specimen. The figure shows the small dimple structure and broken or cracked particles. This indicates that the fracture occur ductile fracture in the Al matrix and also brittle fracture in the primary Cu particles.

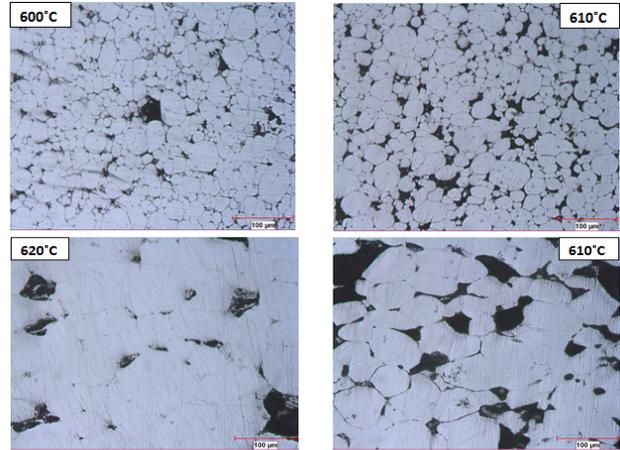


**Figure 7.** SEM views of fractured surfaces of (a,b,c) prealloyed, (d,e,f) elemental specimens

At  $600^\circ\text{C}$  and  $610^\circ\text{C}$  the microstructure consisted of a fully developed Al grains with a grain boundary phase rich in Cu. (Fig.8) shows the grains on the sintered pre-alloyed AA2014. A (97,9%) relative density was achieved in this alloy after sintering. A presence of some small size pores was observed. The dark places refer to the good diffusion of copper in the Al matrix.

The as-sintered microstructure of elemental P/M 2014 is shown in Figure 8. The presence of porosity in

the sintered alloy is shown in the micrograph by the black regions. (Fig.8) shows the microstructure of elemental alloy sintered at  $620^\circ\text{C}$ , the alloy seems very dense and the grain boundaries not clear. Although the alloy was very dense, it is known from the experiments that even a small amount of porosity can have negative effects (Al-Obaidi, 2018).



**Figure 8.** Microstructure images after sintering at temperatures of  $600^\circ\text{C}$  and  $610^\circ\text{C}$  of the prealloyed P/M 2014 and of elemental P/M 2014 after sintering at  $610^\circ\text{C}$  and  $620^\circ\text{C}$

## 5. Conclusions

The compression pressure of pre-alloyed Al powders is higher than that of elemental Al powders.

The green density value of P/M samples produced from elemental powder is partly higher (2%).

After sintering, average density values for both powder groups increased by 3-5%.

According to the results of the impact test, it was determined that the elemental powder samples were relatively ductile, with a generally brittle fracture observed.

When the hardness values were compared, it was determined that the pre-alloyed powder samples gave better hardness distribution and higher values.

The sintering time in elemental powders should be longer than the pre-alloyed powders. When the same durations were applied, insufficiency was found in the sintering process.

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## Conflict of Interest

No conflict of interest has been declared by the authors.

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