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# Effect Of Ball Milling Time on Microstructural Properties of Mg/MgO

# Bilyalı Öğütme Süresinin Mg/MgO'nun Mikroyapısal Özelliklerine Etkisi

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## ABSTRACT

Magnesium and its alloys are desired for mechanical properties and biocompatibility in many research fields. The generation speed of MgO as oxide layers is very rapid, sometimes even ending with inflammation. This study focused on mechanical milling of Mg for different hours of time and its effects on mechanical properties as in density, compressive strength, and hardness values. The effect of milling times on Mg/MgO powder composite formation was investigated. Scanning electron microscope (SEM) images and X-ray spectroscopy (EDS) results were analyzed.

**Key Words**

Magnesium, mechanical milling, microstructural properties.

## ÖZ

Magnezyum ve alaşımları, düşük yoğunluk, büyük sönümleme kapasitesi, geri dönüştürülebilirlik ve boyutsal kararlılık açısından önemli araştırma konuları arasındadır. Magnezyumun oksijenle teması kolayca Magnezyum oksit oluşumuna neden olur. Bu çalışmada toz halinde satın alınan Magnezyum metali mekanik öğütme yöntemi ile çeşitli saatlerde öğütülmüştür. Öğütme sürelerinin Mg/MgO toz kompozit oluşumuna etkisi araştırıldı. Numunelerin taramalı elektron mikroskobu (SEM) görüntüleri incelendi ve Enerji dağılımlı X-ışını spektroskopisi (EDS) analizi yapıldı.

**Anahtar Kelimeler**

Magnezyum, mekanik öğütme, mikroyapısal özellikler.

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## INTRODUCTION

Magnesium (Mg) and magnesium alloys are well known as light weight construction materials and are used particularly in automotive applications, in computer, consumer and communication electronics [1-2]. Magnesium has the lowest density of all construction metals at 1.738g/cm$^3$. Besides, Magnesium has high specific strength (strength/density ratio), high damping properties and good weldability under inert gas [3]. The powder metallurgy (PM) method, which includes a field assisted sintering technique (FAST), has become an important interest in recent years. FAST is a low voltage, direct current (DC) pulsed current activated, pressure-assisted sintering, and synthesis technique, which has been widely applied for materials processing [4]. Mechanical milling (MM) is a process that involves the ball grinding of powders for a certain period. The severe plastic deformation, repeated flattening, cold welding and fracturing of powder particles during MM lead to the significant refinement of microstructure [5]. In addition, MM creates a fresh surface in powders. The sintering process affects the density, microstructural properties and mechanical properties of the final product. In powder metallurgy, sintering is generally desired at minimum temperature and time. The oxide layer formed on the surface of the Mg powder prevents the contact of the Mg powders. Therefore, higher temperature and time are required for sintering. To create fresh surfaces and shorten the sintering time, the effect of mechanical grinding for very long periods on the sintering process was investigated. The main purpose of this study is to investigate the effects of milling Mg with MM technique for various hours. The effects of long mechanical milling times on the size of Mg powders and MgO formation were observed. Also, the effects of different sintering temperatures on density and compressive strength were studied.

## MATERIALS and METHODS

Commercially available Mg powders (purity: ≥99.3%, size ≤220 µm, Kumas Manyezit Sanayi A.S., Turkey) were used as starting materials. Zinc Stearate ($C_{36}H_{70}O_4Zn$), (weighing 1% of magnesium powders) was used as a process control agent.

Mg powders were grinded by high energy ball milling (DECO-PBM-V-2L) under argon protective atmosphere for 18, 63 and 108 hours. The powders were planetary balls milled using 10 mm diameter stainless steel balls. The ball-to-powder ratio was 20:1 and ball milling was carried out at rotation speed of 160 rpm.

Graphite die with 10 mm inner diameter and height of 50 mm was used for sintering of samples. Mechanical milled powders were put into the graphite die and pressed under pressure of 45 MPa in argon atmosphere by using a FAST machine (Figure 1). Sintering temperature was 425 °C and the sintering time was 15 min. The densities of the samples were calculated by dividing the mass by the volume.

Density measurements of cylindrical samples were made according to Equation 1. The volumes of the samples were calculated according to Equation 2.

$$\rho = m / V \qquad \text{Equation 1}$$

$$V = \pi . \frac{d^2}{4} . h \qquad \text{Equation 2}$$

**Figure 1.** FAST machine.

*(ρ: Density of the sample (g/cm$^3$), m: Mass of the sample (g), V: Volume of sample (cm$^3$), d: Diameter of sample (cm), h: Sample height (cm))*

The EDS analysis and scanning electron microscope (SEM) images were obtained by using Thermo Scientific Apreo S Scanning Electron Microscope. Backscattered electrons were used to obtain SEM. MACRO brand hardness tester (Figure 2) was used as hardness tester. Tests were performed on the Brinell scale. In the measurements, a load of 62.5 kgf and a waiting time of 30 seconds were applied.

**Figure 2.** Hardness tester.

## RESULTS and DISCUSSION

### 1. SEM and EDS Analysis of Powders

Scanning Electron Microscope (SEM) images and Energy-dispersive X-ray spectroscopy (EDS) analysis of Mg powders that were not applied MM, various hours MM applied are shown in Figure 3 (a-d).

A significant decrease in Mg powder sizes was observed in the first 18 hours with the effect of mechanical milling. Existing oxide films were broken, and new, fresh surfaces were formed on the powders. After 63 hours of grinding, the powder size was further reduced and the amount of fresh surface increased, the least amount of oxide was observed at this stage. The amount of oxide formed with the 108th hour milling is much higher than the other milling hours.

### 2. Mechanical Properties of FAST'ed Samples

Density, compressive strength, and hardness values of sintered samples obtained from powders unmilled and milled for 18, 63 and 108 hours are given in Figure 4.

The highest amount of oxide is in the unmilled powders. With the milling process, this amount decreased for a certain period of time, while an increase was observed in longer periods. When the densities of the powders were examined after applying the FAST process, it was observed that the samples obtained from the powders that did not undergo mechanical milling were less compacted than the others. Mechanical milling, cleaned the oxide layers on the surface of powders between 0 to 63 hours. After 63 hours powders started to oxidize again with the effect of residual heat on the powders.

A positive effect of mechanical milling on the compressive strength results was observed. However, this effect decreases over certain grinding hours. It was observed that the hardness value of 18 mechanically milled Mg decreased compared to its unmilled state and increased as it was grinded more.

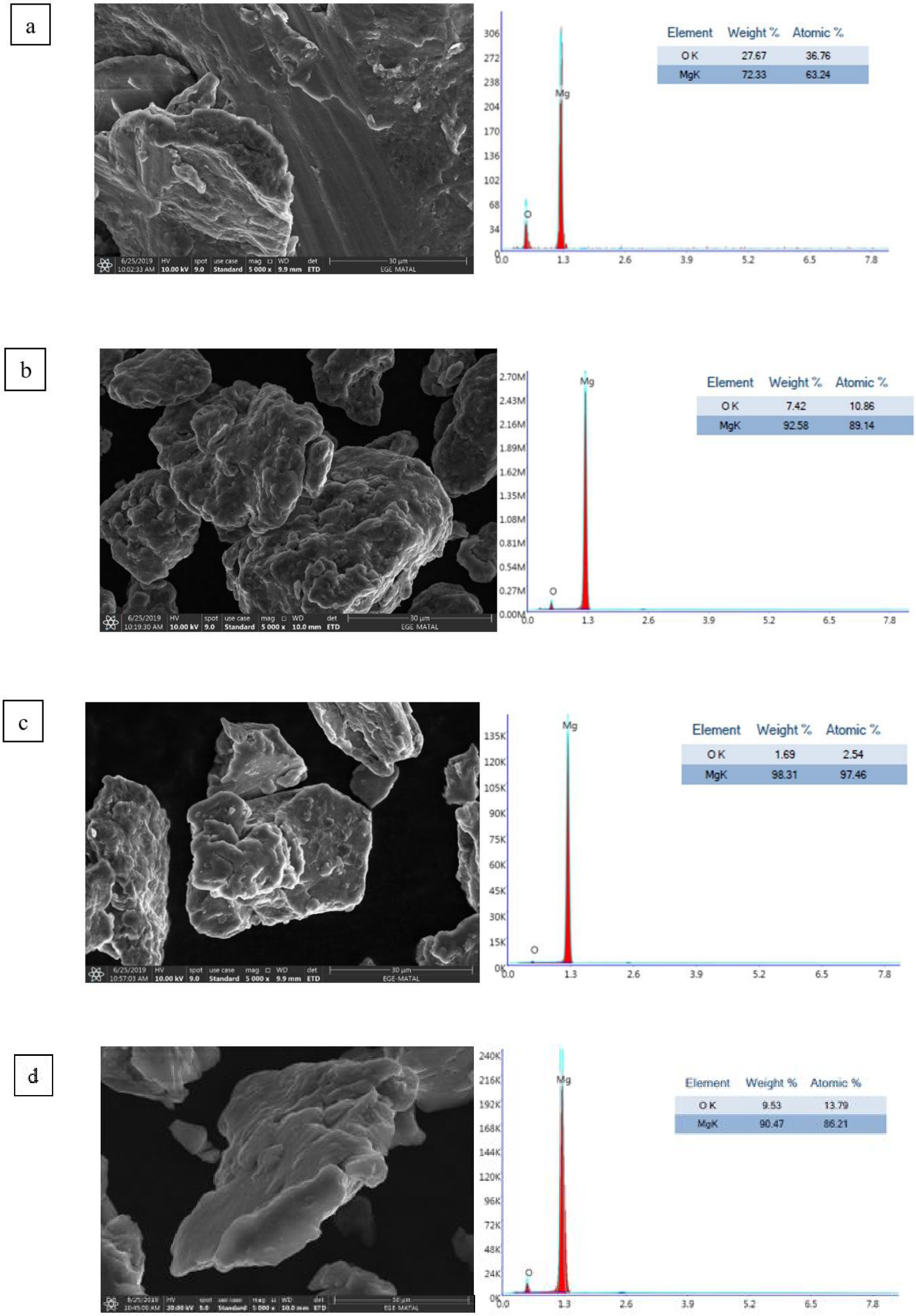


**Figure 3.** Scanning Electron Microscope (SEM) images and EDS analysis of Mg powders; a) Unmilled, b) 18 h milled, c) 63 h milled, d) 108 h milled.

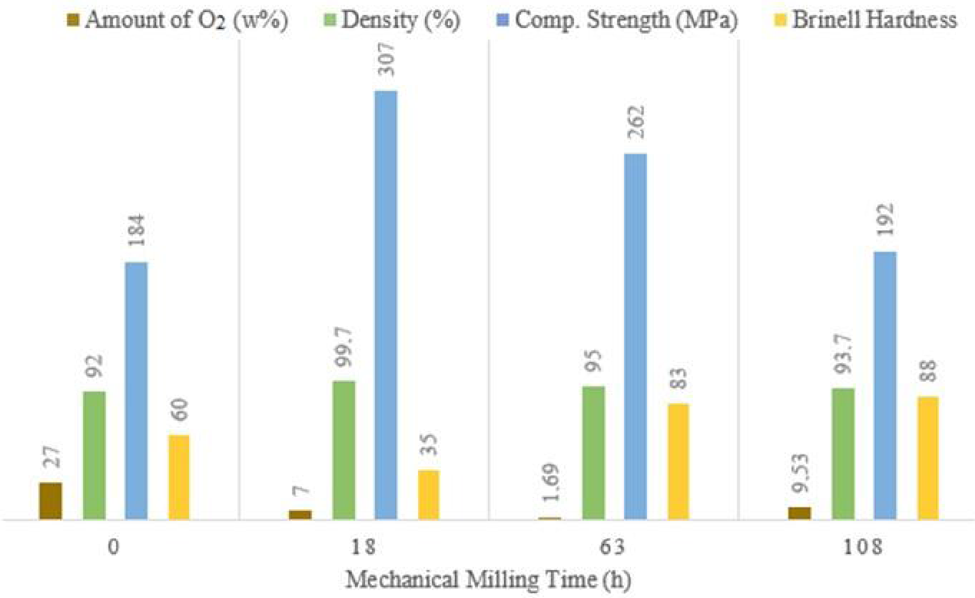


**Figure 4.** Density, compressive strength, and hardness values of sintered samples.

## CONCLUSION

While mechanical grinding for a certain period of time may increase the mechanical values thanks to fresh surfaces, excessive grinding may decrease these values as it causes an excessive increase in the oxide level. If powders are mechanically grinded for a long time, the powder sizes will decrease rapidly at first and then to a lesser extent. Oxide formation was observed depending on the powder size, increasing surface amount, and increasing surface energy.

When the densities of the samples are examined, it is thought that the decrease in their compressibility is the resistance created by the oxides at the contact points of the powders. It is thought that the compressive strength increases as the fresh surfaces formed by milling can establish better contact with each other. If the grinding time is increased further, it is thought that since the oxide films on the powder surface will increase, the contact between the powders decreases and therefore a decrease in the compressive strength is observed. However, with the powders grinded 108 hours, even with compressive strength is decreased but hardness increased. Figure 4 shows that the desired property output can be manipulated with grinding time. If we expect more toughness, grinding time should be around 18 hours, but if hardness is the desired property the grinding time must be increased.

Mg/MgO metal matrix composite has the ability of changing the matrix to reinforcement ratio with the grinding time. Thus, the grinding time has a direct impact on material properties and this phenomenon will be further investigated.

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