A basic study on artificial aging in Mg-10Al$_{12}$Si+1Pb alloy

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Keywords: Magnesium, Artificial aging, In-situ casting, Composite, Hardness

Abstract

In this study, research has been made on the aging of metal matrix composite materials produced by the in situ casting system. Mg matrix composite material was produced by the in situ casting system. In this study, 90%Mg + 10% Al$_{12}$Si (wt) ingot casting was performed for alloy formation and 1% Pb was added as an alloying element to the melted structure. This study aims to examine the effect of the artificial aging (AA) process on hardness and microstructure after alloying and composite of Mg metal. The in situ casting system was used in the casting of Mg alloy under the Ar gas atmosphere. The material after required casting homogenization process; for the AA process, they were embedded in a powder graphite filled vessel and kept at 350 °C for 1 h and then quenched (with 25 °C water). Later; after quenching, the materials were kept at 150 °C for 2, 4, 12, 16 and 24 h and aged samples were obtained. Microstructure images were obtained from the samples by scanning electron microscope (SEM) and light optical microscope (LOM) and then the hardness values of the micro hardness device were measured. Grain structure because of AA heat treatment; showed changes according to un-aging material. The hardness value is directly proportional to the increasing aging time of the materials applied to the AA process; it was found that the levels increased approximately to 45% (86HV to 125HV) compared to the un-aging material and passed to the fixing phase.

1. Introduction

In this study, the production of a composite material and the artificial aging (AA) process are introduced to the literature with the experimental results. The term composite material is used primarily for new materials formed by using two or more materials together [1–3]. It is also used to indicate new types of materials with different properties than the materials in which they occur [4]. The high properties of composites make it preferred in many industrial applications [2, 5]. Composites are being used frequently in every field with developing technology and with different joining and production methods [6, 7]. Different geometric types of reinforcing materials are preferred to make the insufficient properties of the metal-based matrix material more acceptable [8]. Forms of silicon [9], carbon [10], boron [11] and other elements [12] such as fiber [10], particle [13], and platelets [14] are used as reinforcement material. The strength, abrasion resistance, corrosion and hardness properties of the new material obtained by combining the metal matrix and reinforcing material in this way are much better than the conventional materials [8]. Therefore, such materials can be used efficiently in the biomaterials [15], automotive [9], aerospace [16, 17] and nuclear industries [18]. The production methods of composites, which are used as the needs of many sectors, also include special applications [4]. In these applications, production methods such as casting [19], partial melting [20], hand lay-up [21] and layered-plate [22] are used. The casting method, which is one of the main production systems, is divided into subheadings [4, 19, 23]. In all casting methods, new chemical compounds and new phases are formed with the effect of alloying elements added to the structure [2]. The new phases, which are not present in the base metal and in the added materials, are formed by chemical reaction just before the casting process [24]. As a result, a metal-based reinforced composite material is produced.
Aging process in materials is divided into two basic groups. The aging process is known as natural aging (NA) [25, 26]. It is known that precipitate, particles, intermetallic, phase, etc structures within the metal matrix can be distributed homogeneously and heterogeneously because of temperature/time process [27]. Completion or continuation of this formation under ambient conditions without adding any natural process is called NA [28]. When the aging process is accelerated by increasing the temperature at certain rates, this process is called AA [29]. At the end of both processes, a new microstructure is formed in which residual stresses decrease and the microstructure changes [25]. Thus, a new material is obtained, which changes some properties. In addition; it greatly influences the morphology, size and density number of the main strengthening phases formed during AA [30]. GP zones, \( \beta' \), \( \beta'' \) phase, etc as well as their precipitation value contribution to the alloy [25]. As a result of the literature; Mg alloys such as Mg-RE [27], Mg–8Li–3Al–2Zn–0.5Y [28], ZK60 [31], Mg–3Sn–1Al [32], Mg–3Sn–2Zn–1Al [32] and Mg–Gd [26] were aged by AA method and aging/property correlation was investigated.

Mg metal and alloys used in this study is preferred by increasing day by day in applications where lightness is essential [12]. Mg metal; with its low-density property, is used actively from the automotive industry [24] to aerospace applications [16]. Today, reducing the kg/fuel ratio in the automotive industry has increased environmental protection [24]. In this words; Mg, which can show the same level of strength as steel, reduces the fixed weight level of vehicles and reduces the appearance of carbon footprint [12, 24, 33]. There is an increasing trend in the use of Mg in the industry every year [24]. This rate increases by 12%–15% every year since the 2000s [12]. However, due to the low advantages of pure Mg metal such as low resilience and tensile strength, the use of this metal as a composite has become more important [4, 34–36].

Finally, as it is known; the type, size and ratio of reinforcement added to the structure in metal matrix composite materials affect all the properties of the composite material [4]. For example, it is known that mechanical values increase when ceramic-based reinforcing products are homogeneously present in the matrix [37]. In Mg metal, Al and/or Si reinforcement generally forms Mg\(_2\)Si and Mg\(_{17}\)Al\(_{12}\) intermetallic in the structure of the composite material [12, 38–40]. These compounds give the whole structure different properties [12, 38]. Intermetallic compounds (specially Mg\(_2\)Si) are preferred in phase type due to their high melting temperature [41], low density [42], high hardness [40] and low thermal expansion coefficient [43].

It is found in the literature that other elements added to Mg improve corrosion resistance and hardness values [11, 12, 34, 38, 40, 44–51]. However, the limited use of Pb addition is remarkable during the studies are examined. In our previous studies, it was found that different amounts of Pb (0.2%, 0.5%, and 1%) were added to Mg metal and phase ratios in the structure changed [12, 34, 38]. In addition, as is known, the use of heavy metals such as Pb has been limited by many international health organizations [52–54]. The amount we used in our study was determined based on these limitations. The aim of this study is to observe the basic changes of aging (\( \%90 \) Mg–(\( \%10 \) Al\(_{12}\)Si) + (\%1) Pb (short name Mg10AS1Pb) alloy produced by in situ casting system in addition to our studies in literature [12, 34, 38]. In our previous studies [12, 38], Mg10AS1Pb alloy, which gave the best results corrosive (%1 Pb), was examined in terms of AA process. In this study, the quantities of the phases in the structure of the composite material produced are discussed. With these effects, phase changes occurred in the structure of the composite material applied AA and an increase for hardness was determined.

2. Experimental sections

2.1. Materials preparations
In this study, 90% Mg (A) metal and 10% Al\(_{12}\)Si (B) ingot alloy (wt) were used as starting point material for composite production. The A + B structure melted in a special Ar atmosphere controlled furnace (figure 1) at approximately 750 \( ^\circ\)C was transferred to the mold by in situ casting method. It produced stainless steel material by CNC machining method and it about 300 \( ^\circ\)C at the time of casting (figure 2). The basic casting errors that may occur by heating the mold are prevented. At this stage, obtaining a particle reinforced composite material is completed. Pb was re-melted in 1% ratio to Mg–Al–Si master alloy produced with A and B structures. The final products were subjected to homogenization heat treatment at 400 \( ^\circ\)C for 4 h under atmospheric control. The samples were freely cooled to room temperature without being removed from the furnace.

2.2. Artificial aging process
After the casting process and homogenization heat treatment, AA heat treatment was applied to the materials. The steps of the AA process as a special process and the temperature flow regime are given in figure 3. The AA treatment, aging temperatures, and aging times were determined from similar studies [26–28, 31, 32]. In this study, Mg10AS1Pb material that is produced as a special alloy has been used to analyze the effects of aging. Samples prepared in certain dimensions (5 mm \( \times \) 5 mm \( \times \) 15 mm) were kept in steel box and powder graphite (in order to stop contact with oxygen and 100 micron particle size) at 350 \( ^\circ\)C for 1 h and the first step of
AA process was completed. Mg is a very high oxygen affinity element [36]. Here, protection methods are used to reduce the effect of oxidation on Mg. In the AA process carried out in an atmosphere-controlled furnace, Ar gas was used as a preservative and in addition the samples taken into powder graphite. Powder graphite was used in this study as a separate protection security in order to avoid any negative effects despite Ar gas control. Powder graphite is already used as a simple oxidation inhibitor in homogenizing processes after casting [55]. The AA application graph and Mg-Al-Si triple phase diagram [43] shown in figure 3(b) show that the temperature level of 350 °C remains below the solidification temperature of the alloy. In the second step of the AA process, the samples were allowed to cool down in water at a temperature of 25 °C, thus passing the quenching step. Then, in the third step, the samples were aged again in steel box and powder graphite at 150 °C for 5 different time periods between 0–24 h and then air cooled (Aging 1; 150 °C–2 h, Aging 2; 150 °C–4 h, Aging 3; 150 °C–12 h, Aging 4; 150 °C–16 h, Aging 5; 150 °C–24 h). As a result, AA process was completed according to the graph given in figure 3(b). Hardness monitoring was made at each stage of aging and since the hardness increase continued, after 4 h, it was directly switched to 12 h.

2.3. Experiments and characteristics
Grinding with different grade silicon carbide (SiC) grit papers and polishing process with 3 μm and 1 μm diamond suspensions were applied for microstructure analysis. Firstly light optical microscope (LOM) (with
Leica) and secondly Carl Zeiss Scanning Electron Microscope (SEM) equipped with EDX-MAP was used to examine the microstructure of alloy composite. In addition, the hardness values of the specimens were measured by performing five successful experiments from each sample under 1 kg load on Vickers micro hardness device (Qness). Then, the approximate phase amount was calculated by basic area/percentage measurement method in order to determine the phases in the structure over the obtained microstructure images. While applying this method, microstructure was transferred to Image-J program, different structures were marked, and a simple area/percentage calculation was made. This calculation method was applied to at least 3 microstructure pictures taken with LOM and an average phase amounts were determined.

3. Results and discussions

Samples produced by in situ casting system were subjected to hardness measurement and microstructure analysis after AA stages. As a result of these experiments, microstructure photographs shown in figure 4 were first taken with LOM for microstructure investigations. Phases, α-Mg, Mg17Al12 and Mg2Si, which are likely to occur in triple alloys consisting of Mg-Al-Si elements, have been determined by considering the auxiliary studies and shown on the images [12, 38, 40]. With increasing aging time, changes occurred in percentages of the phases in the structure and these changes can be observed through microstructures. For the un-aging sample, the image shown in figure 4(a) shows the as-casting microstructure of the material. Mg-Al binary alloys [12, 38] Mg17Al12 intermetallic phase in this image is partially visible in the grain boundaries. The rate of increase of this phase in post-aging processes was followed during this study. As reported by Chen et al gradual temperature changes trigger phase changes within the structure and dispersion changes [43]. In another study, Wanyu et al proved that phase ratios of a similar alloy changed as a result of AA [25]. Based on references and images, the ratios/types of phases changed with increasing time because of AA in this study.

In addition to the images obtained by LOM, SEM images were taken from the samples and EDX-MAP analyzes were performed. SEM images are detailed in appendix A. It is seen in the SEM images that; because of AA, phase ratios and structures have been changed. It is clearly observed that the ratio of α-Mg structure decreases and the ratio of Mg17Al12 intermetallic phase increases in the structures shown in appendix A. Mg2Si particles showed slight increase in microstructure and their geometric shape changed. The EDX analyzes performed to see the chemical composition of the phases in the structure are given in figure 5. Figure 5 shows the
elemental ratios of the respective phases. The Mg, Al and Si element ratios of Mg$_2$Si, $\alpha$-Mg and Mg$_{17}$Al$_{12}$ phases in the structure showed a consistent result. In addition, EDX analysis shows the presence of Pb element. In each point analysis, appendix A can be examined for the presence of Pb and in this examination, it can be seen that the element Pb is distributed throughout the structure.

In order to support the microstructure images given in figure 4 and appendix A, the approximate percentage values calculated with the Image-J program are graphed in figure 6. Among these phases, the ratio of $\alpha$-Mg phase, known as matrix phase, decreased after AA due to increasing aging time. The ratio of Mg$_{17}$Al$_{12}$ phase, which is an intermetallic phase, which gives rise to a fragmented distribution at the grain boundaries, has increased. In the Mg$_2$Si phase, which included the structure in the composite material group, particle size changes occurred due to the increasing aging time, but a slight increase in the rate of presence occurred [25, 30, 43].

After the microstructure examination of the materials subjected to AA heat treatment, their hardness was taken in the micro hardness device and the values obtained are shown in figure 7. Due to the change for phases seen in microstructure analysis, the amount of hardness increased. According to the results of Image-J program in appendix A and figure 6, Mg$_{17}$Al$_{12}$ phase ratio increased in the structure. The rate of presence of $\alpha$-Mg phase, which

![Figure 4. Microstructures for different aging times. (a) Un-aging, (b) 2 h, (c) 4 h, (d) 12 h, (e) 16 h, (f) 24 h.](image)

![Figure 5. Un-aging specimen EDX analyses.](image)
is the main phase in the structure, decreased. Therefore, the proportional increase of Mg_{17}Al_{12} phase increased the bulking hardness of the samples. It has been found in similar studies that the increase of the intermetallic phase in the structure increases with the hardness value. Jiashi Miao et al reported that the majority of Mg_{17}Al_{12} precipitates in Mg-7Al-2Sn + Ag alloy increase the hardening as a result of aging [56]. In addition, Jung et al by the effect of Sn element added to AZ91 Mg alloy, formation of Mg_{17}Al_{12} phase increased and hardness value increased by 50% [57]. Su Mi Jo et al reported that the intermetallic phases of Mg_{17}Al_{12} and Mg_{2}Sn increase in the structure of Mg-8Al-2Sn-1Zn alloy and that hardness and yield strength can give higher values [58]. Based on similar studies, the numerical ratio of Mg_{17}Al_{12} phase increased by 40% (19.19 to 26.26) and so hardness value increased by 45% (86 to 125) because of AA in this study. In addition, the Mg_{2}Si phase, which acts as a particle in the structure, changed its distribution with increasing aging time and showing a slight increase in its ratio. The Mg_{2}Si phase has a high

Figure 6. Calculated (with Image-J program) phase rates for different aging.

Figure 7. Hardness graph and fixing-line.
hardness structure [59] and gives more hardness than many steel types [60]. It also has high melting temperature [41] and low density [42]. It has been found in the literature that it shows a high level of hardness of 700 HV [59] and 1050 HV [12]. The slight increase of this phase also resulted in an increase in bulking hardness (figure 8) in aged samples. If we look at the hardness results in another aspect; in the AA process, hardness value increase was realized as 2–3 units in 12 h, 16 h and 24 h experiments. Therefore, it can be said that an aging fixing line has been formed in the range of 12–24 h (at look; figure 7-Fixing line). Finally, an analysis covering all phases is shown in the Vickers indentation given in figure 8. Therefore, the increase in the intermetallic phases in the structure increased in terms of bulking hardness. As a general and basic result of the study, because of AA in Mg10AS1Pb alloy, phase ratios in the structure have changed and hence the hardness value has increased.

4. Conclusions

The effects of the artificial aging (AA) process in an alloy of Mg metal with commonly used elements were compiled in this study.

1. After AA treatment applied at 150 °C for 0–24 h (solution temperature 350 °C and quenching with 25 °C water); the proportions of Mg17Al12 and Mg5Si phases in Mg-Al-Si ternary alloys have increased.

2. Bulking hardness value of Mg alloy (occurred Mg-Al-Si + Pb) increased by about 45% with AA method applied under conditions determined by literature support (aging and solution temperatures, heating speed, holding times and aging method).

3. In this study all result determined; microstructure analysis (SEM and LOM), hardness measurement (HV), EDX-MAP elementel analysis (with SEM) and structure/phase ratio (with Image-J) results.

4. The main result of this study is to increase the hardness of this alloy by applying only AA heat treatment process.

Acknowledgments

As the authors, we would like to thank Karabuk University for providing financial support to KBU-BAP-C-11-Y-020 for the casting stages of this study. In addition, we would like to express our respect to Karabuk University, Iron and Steel Institute, Materials Research and Development Center (MARGEM), which provides support experimental studies and results.

Appendix A
Figure A1.

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