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The Effect of Aging Processes on Tribo-Metallurgy Properties of Al Based Ternary Alloys Product by P/M Technique

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Abstract:

In this study, Al matrix composites consisting of different amounts of Zn, Cu, and Mg were produced using the powder metallurgy technique. In the alloying, powders were ball milled for 120 min via mechanical alloying. After alloying process, the powders were pressed at 800 MPa at room temperature. Sintering (2 h at 600 °C in argon atmosphere), solution treatment (2 h at 480 °C), and aging processes (3, 6, 9, and 12 h, respectively, at 120 °C) were applied to the samples. The hardness and tribo-metallurgy properties of the products were investigated. After sintering, the hardness values of the three alloys were close to each other; however, the solution treatment after sintering caused the hardness values to increase significantly. It was determined that the Al-2Zn-5Cu-4Mg alloy had the highest hardness increase with the 6-h aging heat treatment. The wear resistance of the materials in all the compositions had increased. The highest wear resistance and lowest friction coefficient were obtained for the Al-2Zn-5Cu-4Mg alloy with the 6-h aging heat treatment.

***Keywords**: Wear; Ternary Alloys, Tribology; Aging; Sintering.*

1. Introduction

Since the 19th century, aluminum (Al) and its alloys have been preferred in many industrial fields such as the aviation, manufacturing, and automotive industries because of their economical and easy manufacturability as well as for their superior physical and mechanical properties. However, the main reason for the preference of Al and its alloys is their low density and high corrosion resistance [1-3].

Powder metallurgy (PM) has recently attracted the attention of researchers because this process improves the mechanical and physical properties of Al and its alloys [4-7]. PM technology makes possible the near net shape production of components and minimizes machining, thus reducing costs. Therefore, PM may be preferred over conventional casting and other manufacturing methods. In addition, PM technology may be used to modify the chemical composition of the materials by using different alloying elements, which often improves the mechanical and corrosion properties [8-11].

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Copper (Cu), magnesium (Mg), manganese (Mn), silicon (Si), lithium (Li), and zinc (Zn) are the main elements used in Al-based alloys [12-15]. Solution heat treatment and aging processes provide increased strength and hardness, but decrease elongation in Al-Cu alloys. Although the aging properties of binary Al-Cu alloys have been studied in more detail than other systems, there are actually very few commercial binary Al-Cu alloys available. Most commercial alloys also contain other alloying elements [8]. Al-Zn alloys have been known for many years, but stress corrosion cracking during the casting and forging of the materials restricts their desirability. Al-Zn alloys offer high tensile properties in combination with other elements. The addition of Mg to Al-Zn alloys in the range of 3-7.5 % Zn improves the strength properties. The addition of Cu to the Al-Zn-Mg system with a small amount of Cr and Mn ensures the highest commercial-strength Al-based alloys [8, 16-17].

After mechanical alloying (MA), Al-Cu-Mg-Zn alloys are expected to form Al-rich interfaces during 2 h sintering at 600 °C in argon atmosphere. As a result of cooling in the air, θ (Al_2Cu) and/or *s* (Al_2CuMg) phases may precipitate at the grain boundaries. However, compared to Al, the melting point of Mg is low, so the diffusion of small Mg atoms in the Al matrix is quite rapid, and significant amounts of Mg migrate primarily towards the grain boundaries. Sintering at 600 °C leads to gaps, and the Cu atoms migrate to these gaps [18-21]. The proposed alloy system created in this study should be able to exhibit much more active behavior than an ordinary Al-Cu binary alloy system. Thus, it can be a more active candidate in the highly specific strength family of Al-Cu alloys [20-22]. The demand for weight reduction in structural components requires a further increase in the strength of commercial structural alloys.

In this context, especially in the production of wear-resistant materials, this technique can be used in industrial applications to increase the aging response and cold deformation (such as MA technique) before aging. The effects of different alloying elements were investigated to improve both hardness and wear resistance of an age-harden able Al alloy. For this purpose, Al matrix ternary alloys containing Zn, Cu, and Mg were produced via the PM method and the effects of the alloying elements on hardness, microstructure, and wear behaviors were investigated.

2. Materials and Experimental Procedures

2.1 Production of samples and microstructure analysis

In the study, high purity Al, Cu, Mg and Zn powders were alloyed by the MA method and three different compositions with an Al matrix were produced. The compositions of the samples were obtained by developing findings from a previous study [23]. The composition of the samples is given in Table I. Average grain sizes of the high purity powders used were 50, 70, 40, and 80 µm, respectively. The alloying process was carried out in an alcohol environment with a ball grinder at a ratio of 1:10. Thus, oxidation problems with the powders were eliminated. After alloying process, the powders were pressed at room temperature under 800 MPa to the size of Ø10 × 20 mm.

Tab. I Compositions of sample produced by PM (wt.%).

Alloy	Al (%)	Zn (%)	Cu (%)	Mg (%)
Al-10Zn-2Cu-2Mg	86	10	2	2
Al-6Zn-3Cu-3Mg	88	6	3	3
Al-2Zn-5Cu-4Mg	89	2	5	4

After pressing, the sintering process was carried out in a protective gas atmosphere (high purity argon) at 600 °C for 120 min. After sintering, the solution treatment was applied to all samples at 480 °C for 2 h. Following the solution treatment, the samples were aged 3, 6, 9, and 12 h at 130 °C. Schematic diagram of experimental setup was given in Fig. 1.

The sintered and worn surfaces were investigated using field emission scanning electron microscopy (FESEM) and element dispersions in the microstructure were determined by element dispersion spectroscopy (EDS). In addition, the worn surfaces were investigated using the MAPPING technique for determining the elemental distribution in detail.

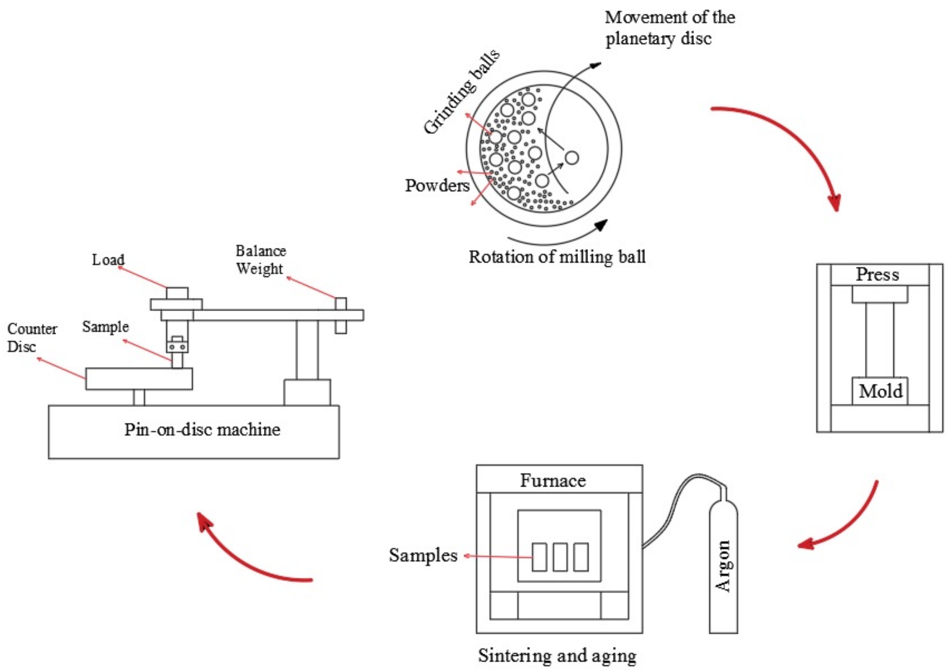

Fig. 1. Schematic diagram of experimental design.

2.2 Microhardness and wear experiments

The microhardness of the samples was determined by the Vickers testing method using a 136° pyramidal diamond indenter. The applied load was 200 g with 10 s dwell time using the Hardway DV-1AT-4.3 hardness tester.

All wear tests were carried out according to ASTM standard G99 using a pin-on-disk apparatus under dry test conditions. Wear results in the study were given as volume loss and coefficient of friction. The Ø10 × 20 mm Al matrix specimens were used with an abrasive disk (Ø60 × 12 mm) of AISI 52100 steel. The hardness value of the disk material was determined as 60 HRC. The wear tests were carried out at a distance of 3600 m at a speed of 2 m/s under a 15-N load.

3. Results and Discussion
3.1 Microstructure analysis

The FESEM images of the Al-2Zn-5Cu-4Mg alloy system after sintering (Fig. 2a) and after aging periods of 3 h (Fig. 2b), 9 h (Fig. 2c), and 12 h (Fig. 2d) are given at different magnifications. In the FESEM images, it can be seen that the powders deformed by MA process formed a lamellar microstructure [24-26], and new grains were formed as a result of recrystallization after sintering at 600 ºC [27]. These new grains can be seen in Fig. 2a. The accruing recrystallization provided a microstructure which had a more homogeneous distribution and improved mechanical properties [28]. In addition, the solution treatment and aging processes applied after sintering led to much more homogeneous distribution and high hardness values due to the finely dispersed and internally formed grains [29].

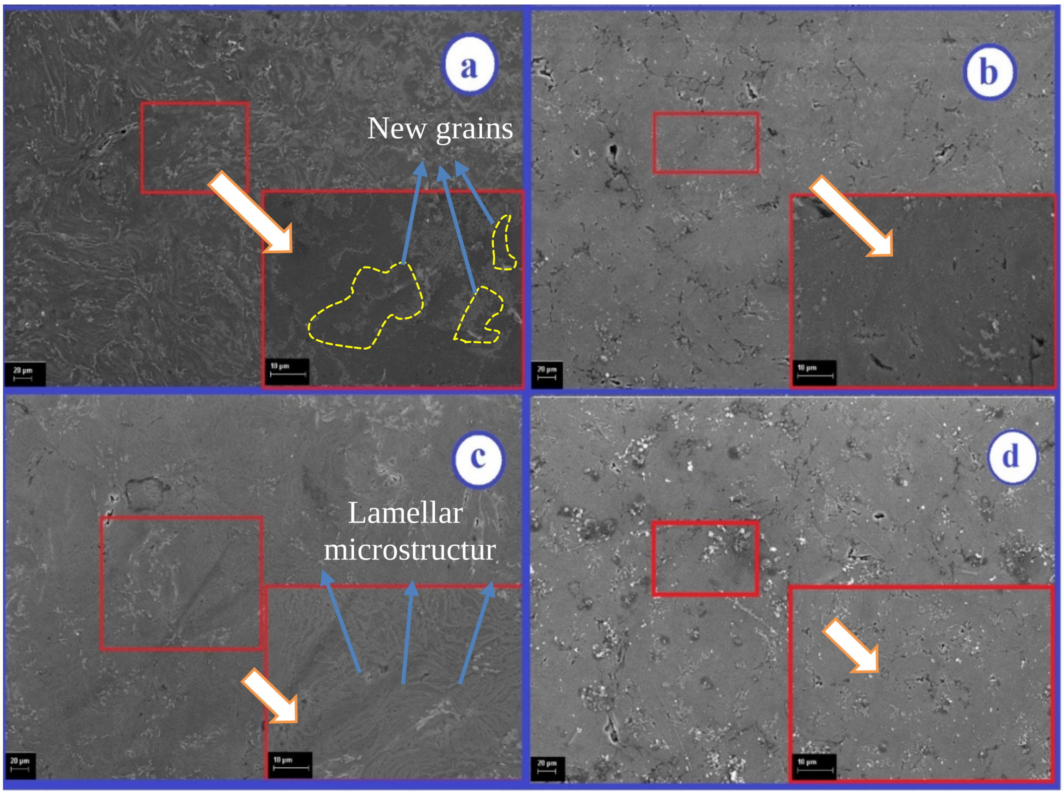

Fig. 2. Microstructure of Al-2Zn-5Cu-4Mg alloy: a) sintered; b) 3-h aged; c) 9-h aged; d) 12-h aged.

3.2 Microhardness and wear test results

The microhardness results for the Al alloy with three different compositions produced by PM are given in Fig. 3. It shows that the hardness values of the three alloys after sintering were close to each other; however, the solution treatment after sintering caused the hardness values to increase significantly. In addition, while the hardness values of alloy-1 and alloy-2 were close to each other after the solution treatment at approximately 130 HV, the hardness value of alloy-3 had increased to 146 HV. It was thought that the Cu played an active role in the grain retention mechanism and this caused higher hardness in alloy-3 compared to alloys 1 and 2 [30]. The reason that the hardness value increases of alloys 1 and 2 were not as high as that of alloy-3 (increased to 157 HV) was due to the effect of the solution and aging heat treatments on the alloy elements. Savaskan and Hekimoglu also reported that increased addition of Cu the microstructure directly affected hardness [31].

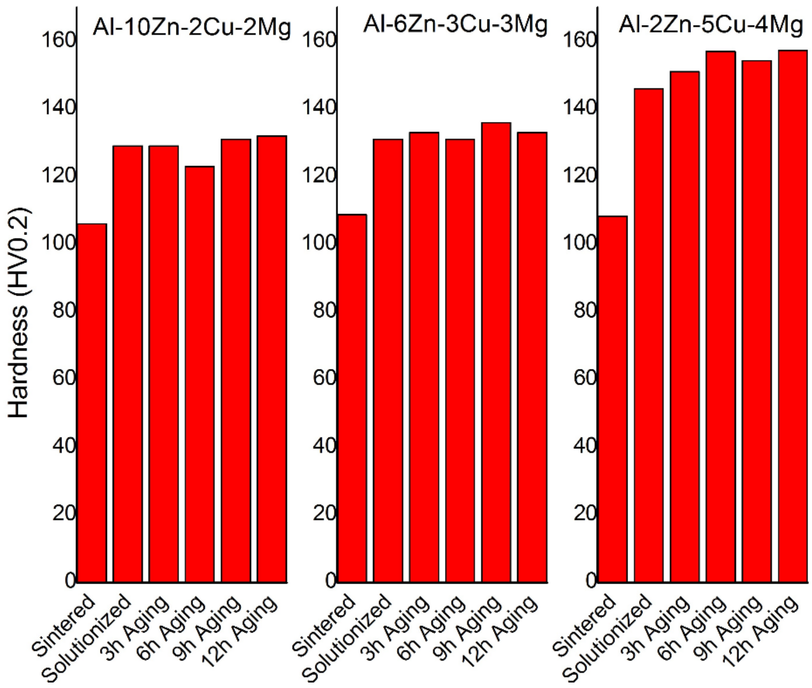

Fig. 3. Hardness variation of ternary alloys under various heat treatments.

The Al-10Zn-2Cu-2Mg alloy contained 10 % Zn. Al-Zn alloys with amounts of up to 15 % Zn have a single-phase (α) structure. Since the Zn contained in the microstructure of Al-Zn alloys is dissolved during the α phase, a distortion in this lattice structure was expected. Distortion of the lattice structure prevents the progression of dislocations, resulting in an increase in the hardness and strength of the alloy [32-34]. However, as shown in Fig. 3, although the addition of Zn and Mg played a vital role in the hardness, in the aging process, the hardness increased more in alloy-3, which had a Cu ratio exceeding 3 %, than with the other two alloys. This result shows that the secondary phases (Al_3Cu, Al_2Cu, Al_2CuMg) formed by precipitation hardening had a significant effect on hardness [35]. It was known that adding copper at less than 2 % does not have an important effect on the microstructure. It has been reported that when the Cu content of Zn-Al-based alloys exceeds 2 %, the ε phase occurs in the interdendritic regions [31]. In addition, Savaşkan and Murphy found that the wear properties could be improved positively by increasing the amount of Cu added to more than 2 % [36, 37].

In Fig. 4, volume loss is given depending on the different aging times for each of the three alloys. The figure clearly shows that the content ratios in the microstructure directly affected the volume loss. The volume loss decreased with the increase of the aging time, and there was a significant decrease in the volume loss with the increase of the amount of Cu in the microstructure.

The lowest volume loss was obtained in the alloy having 5 % Cu, which indicated that the internally formed phases in the microstructure were directly involved in the hardness increases [35-37].

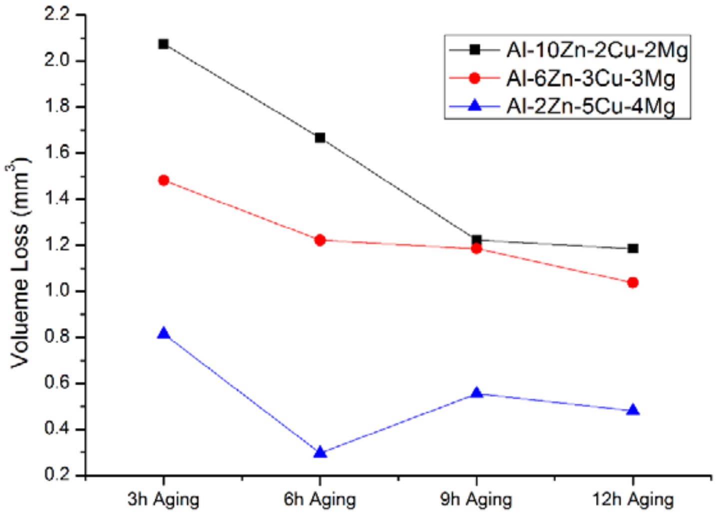

Fig. 4. Volume loss of ternary alloys.

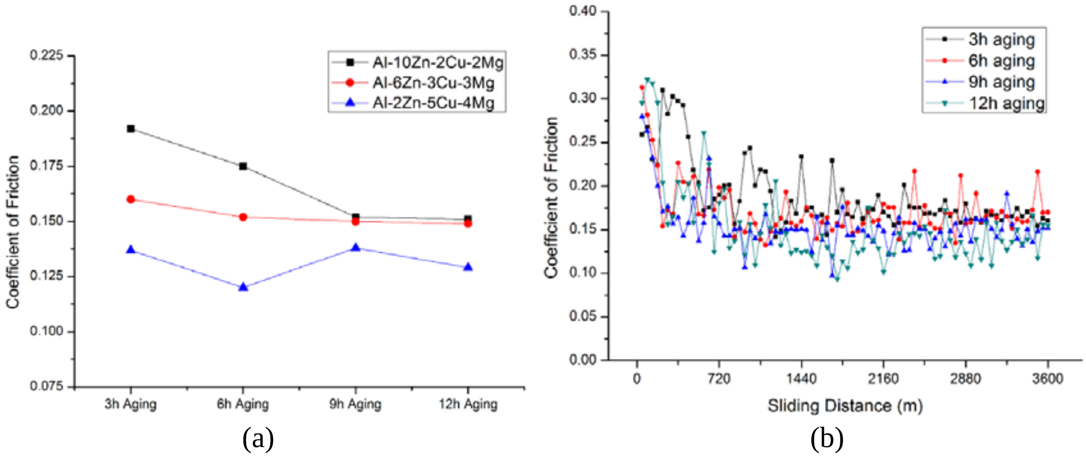

Fig. 5. (a) Average CoF of all compositions; (b) All CoF values of Al-10Zn-2Cu-2Mg alloy.

Another important parameter used in the characterization and investigation of wear behavior is the friction coefficient (CoF), which gives important information about tribological behavior during wear experiments [38]. The average CoFs of the three alloys depending on aging time as well as the CoF of Al-10Zn-2Cu-2Mg alloy depending on sliding distance are given in Fig. 5a and b, respectively.

In the wear experiments, the lowest CoF was obtained with the Al-2Zn-5Cu-4Mg alloy and the highest CoF with the Al-10Zn-2Cu-2Mg alloy. These findings are similar to the volume loss results. In other words, the alloy with the highest wear resistance was Al-2Zn-5Cu-4 Mg and the alloy with the lowest wear resistance was Al-10Zn-2Cu-2Mg. According to the aging processes, the lowest volume loss and lowest CoF were determined in the 6-h aged Al-2Zn-5Cu-4Mg alloy. The 9-h aging yielded the lowest CoF for the Al-10Zn-2Cu-2Mg alloy. The reason for this was that the grain growth occurring over the 9-h aging process caused a decrease in hardness [39-42]. In addition, the formation of oxide films may have been involved in the reduction in the CoF. Alloy elements such as Al and Cu cause oxide films to form on the surface, leading to a low CoF [43, 44].

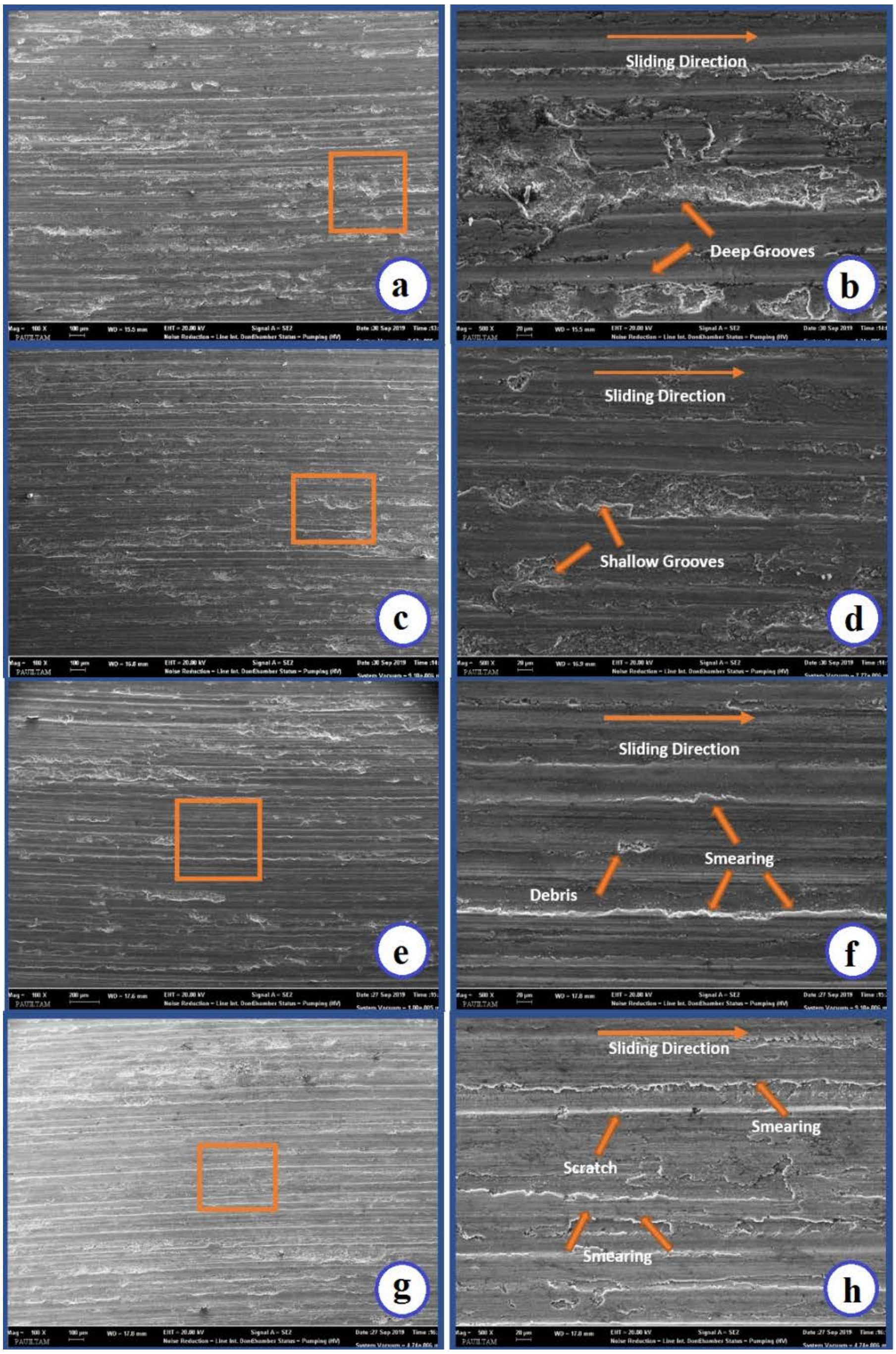

Fig. 6. Worn surface images of Al-10Zn-2Cu-2Mg alloy: (a)-(b) 3-h aged; (c)-(d) 6-h aged; (e)-(f) 9-h aged; (g)-(h) 12-h aged.

The FESEM images of worn surfaces presented in Fig. 6 a-h were used to characterize the effect of the aging heat treatments on wear behavior. Since the surface images of all alloys were similar, only the surface images of the Al-10Zn-2Cu-2Mg alloy are presented. The FESEM images show that all sample surfaces had deformations due to wear. In particular, the sample aged for 3 h had deep grooves on the surface. When the surface deformation depending on aging heat treatment was examined after wear experiments, the maximum surface deformation was seen in the 3-h aged sample, which was in parallel with the volume loss. The deformation decreased at 6 h and 9 h, while the samples that were aged 9 h and 12 h also had similar characteristics.

The decrease in the depth of the grooves contributed to increasing hardness, whereby aging heat treatment caused precipitation of the secondary phases [45]. In addition, deep grooves and wear debris were observed on the surface of the samples aged for 3 h and 6 h; whereas smearing was observed over the surface of the samples aged 9 h and 12 h. The abrasive wear mechanism in the Al alloys aged 3 h and 6 h, and the adhesive wear mechanism in the Al alloys aged 9 h and 12 h were determined using FESEM examinations [46, 47].

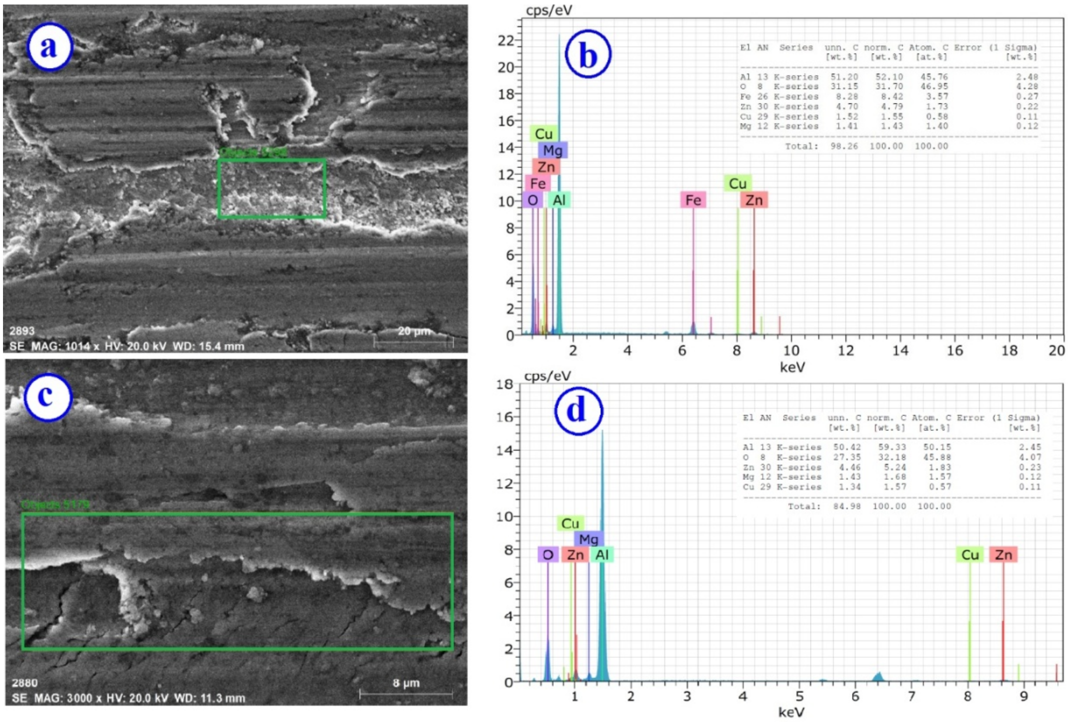

Fig. 7. Worn surface EDS analysis of Al-10Zn-2Cu-2Mg alloy: (a)-(b) 3-h aged; (c)-(d) 12-h aged.

The Fig. 7 shows the worn surface of 3-h (a) and 12-h (b) aged Al-10Zn-2Cu-2Mg alloy and the EDS analysis taken on the FESEM images. Figure 8 presents the elemental MAPPING distribution of the 3-h aged alloy-1 and the regional distribution of each element. When Fig. 8 (c)-(d) is examined in detail, it can be seen that there are alloy elements likely present in the microstructure and that the presence of O was also determined in the microstructure.

Increasing wear distance leads to thermal and thermomechanical effects on the material surface that cause the formation of thin oxide film layers. These hard layers provide a lubricating effect on the surface and hence, cause a low CoF and low wear resistance [48, 49]. In addition, Fig. 8 shows that in the brown (red + green) areas where oxidation starts, debris occurs on the surface. Moreover, Fe (yellow area) was determined on the material surface. It was thought that the Fe was smeared on the surface from the AISI 52100 steel disk used as the abrasive during wear experiments. The MAPPING analysis determined that after the wear tests, Zn, Mg and Cu were dispersed homogeneously on the surface. It was noted that the MAPPING results agreed with the EDS analysis given in Fig. 7.

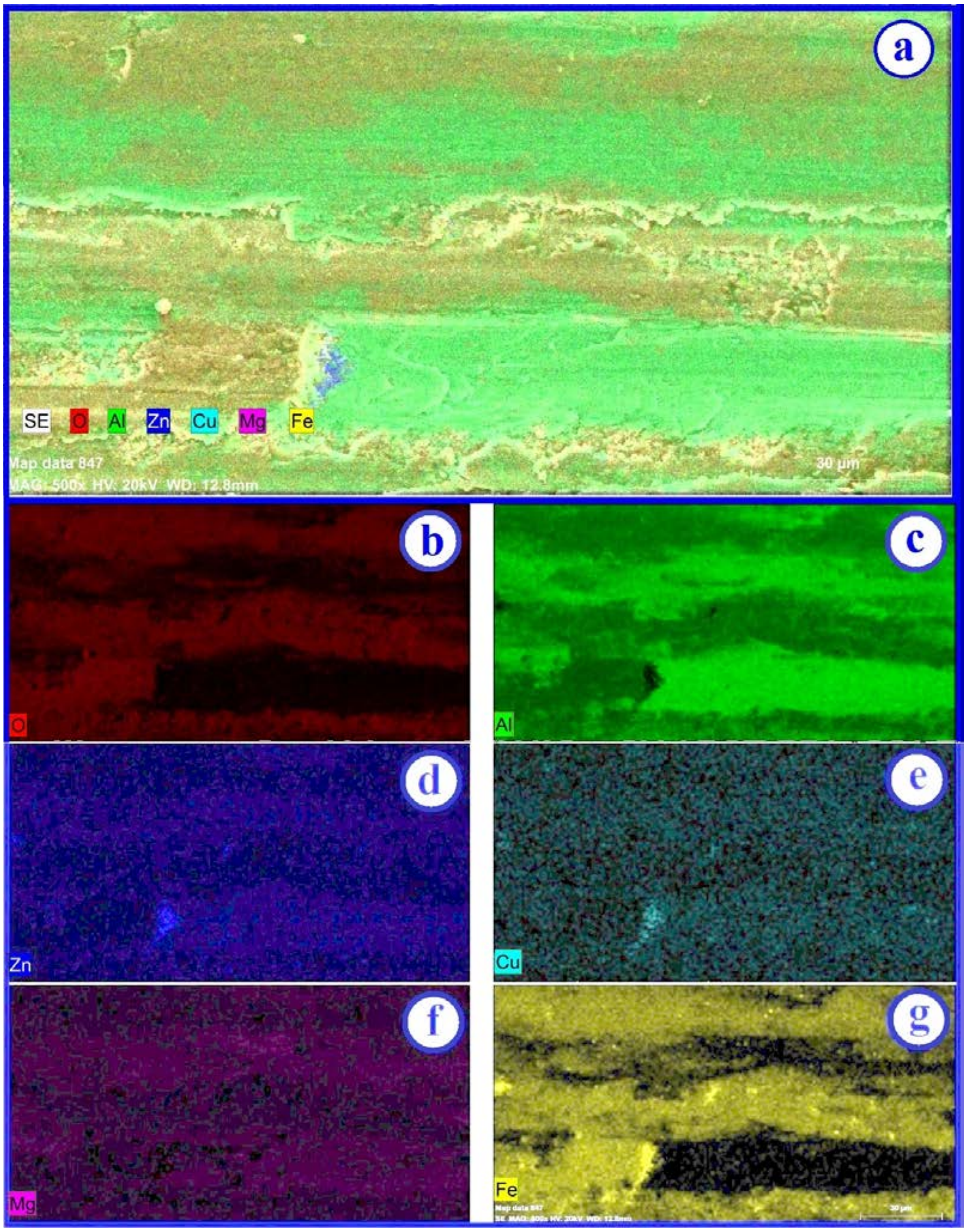

Fig. 8. MAPPING images of worn surface for 3-h aged Al-10Zn-2Cu-2Mg alloy: (a) general view; (b)-(g) elemental MAPPING.

4. Conclusion

In this study, Al matrix composite materials containing different ratios of Zn, Cu, and Mg were produced by the PM method. The following conclusions were reached as a result of the microstructure analysis and wear experiments.

- The highest hardness increase was obtained for the content of the Al-2Zn-5Cu-4Mg alloy. This hardness increase resulted from the secondary phases formed by the amount of Cu (exceeding 2 %).

- Parallel to the hardness values, the alloy with the highest wear resistance and lowest volume loss was Al-2Zn-5Cu-4Mg. The alloy with the lowest wear resistance was Al-10Zn-2Cu-2Mg.
- Depending on the aging time, despite the existence of both abrasive and adhesive wear mechanisms, abrasive wear was mainly determined in the microstructure of the worn surface.
- As a result of the experiments, it was determined that Cu played a vital role on hardness in the aging process. It played an active role in the formation of secondary phases in the structure.
- In the MAPPING examinations, O content was determined along with the other elements in the microstructure. Increasing wear distance led to thermal and thermomechanical effects on the material surface and those caused formation of thin oxide film layers. These hard layers provided a lubricating effect on the surface and hence, caused a low friction coefficient and low wear resistance

In general, the results can be interpreted to recommend that in Al-Zn-Mg-Cu alloy systems, Cu should be added to the structure in amounts of over 2 % in order to improve the wear behavior. This can provide high hardness and wear resistance, and a low friction coefficient and low volume loss.

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Сажетак: *У овом раду, добијени су Al матрикс композити који садрже различите количине Zn, Cu, и Mg техником металургије праха. Прахови су механички активирани 120 минута. Након млевења, пресовани су на собној температури под притиском од 800 MPa. Затим су примењени синтеровање (2 h на 600 °C у атмосфери аргона), третман раствором (2 h на 480 °C), и процес старења (3, 6, 9, и 12 h, истим редом, на 120 °C). Испитивана су трибо-металуршка својства и чврстоћа. Након синтеровања, вредности тврдоће за три легуре су биле блиске; ипак, третман раствором након синтеровања је условио значајан пораст тих вредности. Закључено је да је легура Al-2Zn-5Cu-4Mg имала највећи пораст чврстоће са шесточасовним третманом старења. Отпорност на хабање је такође порасла за све саставе. Највећу отпорност на хабање и најнижи коефицијент фрикције је постигнут код Al-2Zn-5Cu-4Mg легуре са 6-часовним третманом старења.*

Кључне речи*: хабање, тернарне легуре, трибологија, старење, синтеровање.*