From Metal Chips to Composite: Effect of Age-Hardening on Mechanical and Wear Properties of Al₂O₃ Reinforced AA7075 Composites

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Abstract:
Aluminum metal matrix composites were used in various fields such as aircraft, underwater, automobile, aerospace. Several fabrication techniques were used for the production of these composites. Among the various methods, the powder metallurgy route provides dimensional accuracy and high production rates. In this paper, metal matrix composites that include 7075 aluminum alloy chips and Al₂O₃ particles were produced by using a hot pressing. The effect of reinforcement amount and T6 heat treatment on the mechanical and wear properties of the composites was investigated. Al₂O₃ particles were added by weight of 5, 10 and 15 % and the composites were produced by hot pressing. T6 heat treatment consisted of a solution treatment at 480 °C for 2 h and artificially aging process at 120 °C for 24 h. Wear resistance of the composites was determined by a ball-on-disc dry sliding wear test.

Brinell hardness of the composites tended to increase with the increasing Al₂O₃ reinforcement up to 10 wt.% and with T6 treating. Particle reinforcement improved the wear resistance up to 10 wt.% Al₂O₃. In addition, volumetric material loss tended to decrease after the aging process.

Keywords: Metal matrix composites; AA7075; Aluminum; Chips.

1. Introduction
Aerospace or automobile industries need materials that have a combination of several features such as lightness, high strength, corrosion, and wear resistance [1, 2]. Aluminum metal matrix composites are widely used in these areas reinforced with hard particles such as silicon carbide, boron carbide, and aluminum oxide [3-5]. These particulates enhance mechanical properties such as hardness, young modulus, and yield strength [4, 5].

There are many techniques for solid-state producing of aluminum alloys such as equal channel angular pressing (ECAP), powder metallurgy, extrusion, and rolling [6-8]. The powder metallurgy technique provides high homogeneity, and dimensional accuracy, particle size control, and gas controlling [9].

It is quite hard to recycle the chips of aluminum alloys because of their shape and surface contamination such as oxides and machining oil. The powder metallurgy techniques, generally including pressing and sintering steps, are useful to downcycle the products from chips to a final product [10]. High-density composite parts can be produced by the hot
pressing method using the combination of pressure and temperature. Heat and uniaxial pressure are applied simultaneously during the hot pressing. The heat generates a high temperature for the pressed material and drags it to the plastic deformation regime [11, 12]. However, during the hot-pressing process, the thermal effects are experienced that exposes the heat treatable aluminum alloys to solution treatment conditions. As a result, hot-pressed heat treatable aluminum composite would exhibit lower mechanical properties than expected. Hence, T6 heat treatment is necessary after the hot pressing in case of using AA7075 chips for composite production.

Aging treatment is widely applied to AA7075 alloy to improve its mechanical properties. Aging, in the other words precipitation hardening, follows an order of supersaturated solid solution→GP zone(s)→metastable phase→\( \eta' (\text{MgZn}_2) \)→equilibrium phase→\( \eta \) [13-15]. The increment in strength and hardness is provided by the formation of metastable phase→\( \eta' (\text{MgZn}_2) \) [16].

The present study aimed to produce AA7075-Al\(_2\)O\(_3\) composites from AA7075 chips by the hot pressing method to investigate the functionality of the metal matrix composites produced. Also, the effect of Al\(_2\)O\(_3\) reinforcement and aging treatment on hardness and wear resistance of AA7075-Al\(_2\)O\(_3\) composites was investigated.

2. Materials and Experimental Procedures
2.1. Production of composites

In this study, AA7075 machining chips were used as a matrix material (Tab. I). The machining chips obtained by lathe turning is found to be curled, short chips of length 5–25 mm, width 3 mm, and thickness 0.5 mm (Fig. 1). Al\(_2\)O\(_3\) particles (Tab. II) have an average size of 1 µm. The densities of AA7075 and alumina powders were 2.81 and 3.97 g/cm\(^3\), respectively. The Al\(_2\)O\(_3\) particulates were dispersed in the matrix with 5, 10 and 15 % by weight. Mixed powders were loaded into a steel die and produced with a unidirectional hot press.

![Fig. 1. The morphology of AA7075 chips.](image)

The production steps of the AA7075/Al\(_2\)O\(_3\) composites followed the order of cold pressing at room temperature under 100 MPa for 30 s and hot pressing at 500 °C under 300 MPa for 1 h. The produced bulk composite was cut into 16x16x6 mm\(^3\) pieces. Then the second sintering process applied under 600 °C for 1 hour. The aging process is applied to a portion of the hot-pressed samples while maintaining only a portion of the hot-pressed state.
A portion of hot-pressed composites was solution treated at 480 °C for 2 h and then artificially aged at 120 °C for 24 h.

Then all specimens were mechanically ground using abrasive SiC papers of increasing grade and finished with 1200 grit. They were polished with 6 and 3 µ diamond suspension and then cleaned with ethanol.

Tab. I Chemical composition of AA7075 aluminum alloy.

| Element | Zn  | Mg  | Cu  | Cr  | Si | Fe  | Mn  | Zn + Ti | Other | Al  |
|---------|-----|-----|-----|-----|----|-----|-----|---------|-------|-----|
| wt.%    | 5.1–6.1 | 2.1–2.9 | 1.2–2.0 | 0.18–0.28 | 0.4 max | 0.5 max | 0.3 max | 0.25 max | 0.15 | Balance |

Tab. II Chemical composition of Al₂O₃ powder.

| Compounds | wt. % |
|-----------|-------|
| α alumina | 93    |
| Fe₂O₃     | 0.7   |
| TiO₂      | 1.7   |
| CaO       | 1.2   |
| Other     | 3.4   |

2.2. Characterization: Density and Microstructure

The density of the composites was measured according to the Archimedes principle. Microstructural investigations were fulfilled by Nikon Eclipse LV 150 optical microscope using Clemex Software. Keller etchant reagent was used for optical investigations. The surface morphologies of the samples were investigated by scanning electron microscope with energy dispersive spectroscopy (SEM-EDS, Carl Zeiss 300VP).

2.3. Hardness Test

Hardness measurements were carried out steel ball has 2.5 mm diameter under 6.25 kg by using Duravision 2000 EMCO Test Brinell hardness tester.

2.4. Wear Test

CSM instruments ball-on-disc wear-test unit was employed for the tribological analysis of coatings against alumina ball (Ø6 mm) under dry-sliding conditions. Wear test was carried out at room temperature with a sliding speed of 10 cm/s under 2 N load. The sliding distance and radius are 200 m and 3.52 mm, respectively. Worn surfaces were examined under a scanning electron microscope and stereomicroscope.

3. Results and Discussion

3.1. Microstructure and Density

The theoretical densities of the samples were calculated and actual density measurements were fulfilled. Samples reached the density values shown in Fig. 2. The density values reached were slightly below the theoretical values. The reason for this is the size and
shape of the chips used. In the powder metallurgy method, several negativities can be observed in the sintering of irregularly shaped powders. The nature of surface topography influences the frictional forces between the chips. Surface topography, size, and shapes of the chips also affect particle-to-particle contact during compaction and sintering steps [17]. When the powder metallurgy method is used for production, the addition of smaller particles may facilitate compressibility and may be reduced the amount of porosity. Therefore, the amount of porosity of the sample without reinforcement is higher than 5 and 10 % Al₂O₃ reinforced samples. Powder particles having a small size settled in the cavities between the chips and increased sinterability in 5 and 10 wt.% reinforced samples. However, this does not apply to a 15 wt.% reinforced sample. Al₂O₃ particles are ceramic-based hard reinforcing particles, although they have the appropriate size to fill gaps. The increase in the number of coordination within the sintering of hard particles causes the particles to break. Also, the sinterability of hard particles is much lower compared to aluminum. Thus, the excess increase in the number of hard particles caused a decrease in the compressibility and sinterability after a point and thus increased the amount of porosity [18]. Although the density values reached a little less than the theoretical values, almost the same density values were obtained when compared to other studies in the literature used with spherical dimensional and easily compressible powders. The density values and porosity ratios of the composite produced by using chips that support the recycling are thought to be superior to the other studies in this respect [10, 19].

![Image](image1.png)

**Fig. 2.** The change in density values of samples depending on Al₂O₃.

![Image](image2.png)

**Fig. 3.** Microstructure of composites from the surface: a) unreinforced, and reinforced with Al₂O₃ by weight b) 5% c) 10% d) 15%.
When microstructures in Fig. 3 are examined, we observed that some AA7075 chips could provide properly merger with other nearby chips. However, there has not been a complete merger between some chips and this has caused some porosity among the metal chips. This may be derived from that the essentialness of the grains should be as close as possible to each other before the sintering process in the powder metallurgy technique [20]. This requirement could not be achieved in some area because the chips especially that have curly formed were resistant to compressing.

Fig. 4 shows that the particles are homogeneously distributed in the matrix when the general structure observed. However, in some regions, shown in Fig. 5, Al$_2$O$_3$ particles tend to generate agglomeration in the porosity between the grain boundaries and the chips. It is thought that in this case some of the mechanical properties may affect negatively. Because the mixing quality of powder and chips are insufficient as scaled, inhomogeneities and nonlinearities were observed in microstructural examinations. Fig. 5 shows the EDS results of 5, and 15 wt.% reinforced and aged samples.

![Fig. 4. SEM image of 10 wt. % reinforced the unaged sample.](image)

![Fig. 5. SEM images and EDS results of a) 5 wt.% reinforced and aged, b) 15 wt.% reinforced and aged.](image)
3.2. Hardness and Wear Test

The graphical analysis of hardness in AA7075 chips-Al2O3 composites is shown in Fig. 6. When the measured Brinell hardness values for aged samples were compared, there was an increase in the hardness of the reinforced samples with 5 and 10 wt.% compared to the composite sample with the reinforcement sample. The reason for this increase is the presence of hard particles added to the matrix. The fact that the hardness of the 10 wt.% reinforced sample is higher than the 15 wt.% reinforced sample. Due to the agglomeration of the hard particles, the compressibility and sinterability capability will decrease and the porosities are increased in this case [17, 18]. As observed in the density and microstructural analysis, the sample which was reinforced with 15 wt.% were observed to have some decrease in hardness due to insufficient compressing and sintering.

If we examine the effect of aging on the hardness values of composite samples, it is seen that it was improved the hardness of composites. This may be due to aging kinetics in reinforced composites. When aging is applied, the phase which begins to precipitate around hard particles in particular causes an increase in mechanical properties [13-16].

It was observed that the effect of aging was higher than the others in the 5 wt.% Al2O3 reinforced sample. This may be attributed to the reinforced particles form a deformation zone around itself. This deformation zone causes an increase in the hardness of the structure. However, when the fortified particles begin to be located too close to each other, they create an obstacle for recrystallization, which reduces the effect of aging [21]. Ferry and Munroe [22], were produced Al2O3 particle reinforced AA2014 metal matrix composites in their study. They showed that the ceramic particles subjected the matrix to additional stress hardening during deformation. The reason for this is the hot deformation has higher activation energy and the flow stress of the composites was higher than the alloys at the deformation conditions. This behavior in the composite can be attributed to the localized increase in both tension and strain rate in particulates near the matrix regions.

Wear tracks of unaged-unreinforced AA7075 alloy and unaged-10 wt.% Al2O3 reinforced AA7075 alloy are shown in Fig. 7. Al2O3 reinforced composite samples exhibited discontinuous and shallow wear traces. Unreinforced samples have deeper and more continuous wear traces. When the effect of the amount of reinforcement on the wear pattern characteristic of the unaged samples was examined (Fig. 7) unaged and unreinforced AA7075
wear track was found to be wider than Al$_2$O$_3$ reinforced composite. The increase in hard particle reinforcement reduced the effect on wear.

Fig. 7. SEM images of unaged a) unreinforced, b) 10 wt.% Al$_2$O$_3$ reinforced samples wear tracks.

Fig. 8. SEM images of aged reinforced with Al$_2$O$_3$ by weight percent a) 5, b) 10, and c) 15 samples wear tracks.

Examination of wear marks of samples where aging is applied, there is the wavy structure at the time of wear may indicate to us that it is worn with stick-slip mechanism (clearly seen in Fig. 8). In the wear types where this mechanism is effective, the particles that are detached from the surface with the effect of abrasion were transported with the aid of abrasive balls, and they adhere to the other region with the effect of the force acting on the wear zone. As the amount of reinforcement in aged samples increases in Fig. 8b and 8c, this mechanism was observed and wear loss was reduced.

As a result of the wear test, the volumetric material loss was found to show better wear resistance of composites containing 10 wt.% Al$_2$O$_3$ (Fig. 9). In the measurements made according to the standard DIN 4776 [23], the accuracy of the measurement results cannot be mentioned if the wear trace depth is greater than 10 µm according to the standard JIS B 0601-
2001 [24] at 2.5 mm sampling length. Since the depths of the samples without reinforcement are above the values in the standard, the samples are not included in the graph. The loss of wear for unsupervised samples is calculated by taking into account the calculations based on this maximum value, and we can say that it is greater than $150 \times 10^{-3}$ mm$^3$. The increase in the amount of the reinforcement effected positively on the wear resistance. But, there is an increase in material loss in 15 wt.% $\text{Al}_2\text{O}_3$ reinforced samples. This may be due to the amount of reinforcing particles is increased by accumulating at the grain boundaries and in the absence of a bond between the chips, the abrasion resistance may be reduced. When the effect of aging on abrasion resistance is examined, it is observed that material losses are decreased with the aid of precipitated phases. The effect of aging on the 10 wt.% $\text{Al}_2\text{O}_3$ reinforced sample, which is thought to be distributed more homogeneously, was observed more sharply. This may be due to precipitation phases begin to precipitate around the reinforced particles.

![Fig. 9. Volumetric material loss of the reinforced samples.](image)

4. Conclusion

The production of metallic composites was carried out by hot pressing using AA7075 chips to investigate the functionality of the composites produced. T6 treatment was applied to the composites produced with different reinforcement quantities and the effect of the amount of reinforcement and aging on the mechanical properties and the wear resistance were investigated. The findings of the experimental studies are summarized as follows:

1. Microstructural images shown that grains are equiaxed. Samples have micro-sized pores existed within the matrix phase. This can be attributed to the low compressibility of the chips matrix phase which resulted in the insufficient bond between the soft phase particles at this level of pressure. Increasing the process pressure, the liquid phase may flow better and fill the voids.

2. As the amount of reinforcement increased, hardness increased and the highest hardness values were observed in 10 wt.% $\text{Al}_2\text{O}_3$ reinforced composites. Excessive increase in the amount of reinforcement decreased compressibility, caused agglomeration and adversely affected the hardness. In unaged samples up to 26.3% and in T6 treated samples up to 24.5% were observed with the amount of
reinforcement. The highest hardness value was reached in the aged and 10 wt.% Al₂O₃ reinforced sample, and the hardness of this sample increased by 28% compared to the untreated sample without reinforcement.

In unreinforced and unaged sample, deep cavities and continuous wear traces were formed, but in reinforced and aged samples discontinuous and shallow wear traces were observed. The highest wear resistance was observed in 10 wt.% Al₂O₃ reinforced composite sample. The loss of volumetric material was reduced by 51% at 10 wt.% Al₂O₃ reinforced-aged composite compared to the 5 wt.% Al₂O₃ reinforced-unaged sample. As a result of the wear tests, the loss of the materials decreased by 36% in 10 wt.% Al₂O₃ reinforced sample compared to the unreinforced sample.

5. References

1. A.J. Knowles, X. Jiang, M. Galano, and F. Audebert, Microstructure and mechanical properties of 6061 Al alloy based composites with SiC nanoparticles, J. Alloys Compd. 615 (2014) 401-405.
2. M. Laska and J. Kazior, Influence of various process parameters on the density of sintered aluminium alloys, ActaPolytech. 52, 4 (2012) 93-95.
3. A. Egelja, J. Majstorović, N. Vuković, M. Stanković, D. Bučevac, Synthesis of Highly Porous Al₂O₃-YAG Composite Ceramics, Science of Sintering 48 (2016) 303-315.
4. Y. Wu and G.Y. Kim, Compaction behavior of Al6061 and SiC binary powder mixture in the mushy state, J. Mater. Process. Technol. 216 (2015) 484-491.
5. S. Soltani, R. AzariKhosroshahi, R. TaherzadehMousavian, Z.Y. Jiang, A. FadaviBoostani, and D. Brabazon, Stir casting process for manufacture of Al–SiC composites, Rare Met. 36, 7 (2017) 581-590.
6. R. Chiba and M. Yoshimura, Solid-state recycling of aluminium alloy swarf into c-channel by hot extrusion, J. Manuf. Process. 17 (2015) 1-8.
7. N. Saheb and M. Shahzeb Khan, Compressive Strength and Thermal Properties of Spark Plasma Sintered Al-Al₂O₃Nanocomposite, Science of Sintering 50, 1 (2018) 1-14.
8. J. Gronostajski, H. Marciniak, and A. Matuszak, New methods of aluminium and aluminium-alloy chips recycling, J. Mater. Process. Technol. 106, 1-3 (2000), 34-39.
9. T.M.K. Tabonah, M. Akkaş, and S. Islak, Microstructure, Wear and Corrosion Properties of NiB-TiC Composite Materials Produced By Powder Metallurgy Method, Science of Sintering 51, 3 (2019).
10. J.B. Fogagnolo, E.M. Ruiz-Navas, M.A. Simón, and M.A. Martinez, Recycling of aluminium alloy and aluminium matrix composite chips by pressing and hot extrusion, J. Mater. Process. Technol. 143-144, 1 (2003) 792-795.
11. L.F. Francis, Materials Processing: A Unified Approach to Processing of Metals, Ceramics and Polymers, Ch 5, Powder Process, Academic Press, 2016.
12. J.M. Torralb, Comprehensive materials processing, S. Hashmi, Ed., Improvement of Mechanical and Physical Properties in Powder Metallurgy, Elsevier, Vol.3, 2014.
13. G. Özer and A. Karaaslan, Properties of AA7075 aluminum alloy in aging and retrogression and reaging process, Trans. Nonferrous Met. Soc. China27, 11 (2017) 2357-2362.
14. C.Y. Sheu and S.J. Lin, Ageing behaviour of SiCp-reinforced AA 7075 composites, J. Mater. Sci. 32, 7 (1997) 1741-1747.
15. J.T. Staley, Aging Kinetics of Aluminum Alloy 7050, Metall. Trans. 5, 4 (1974) 929-932.
16. P.V. Kumar, G.M. Reddy, and K.S. Rao, Microstructure, mechanical and corrosion behavior of high strength AA7075 aluminium alloy friction stir welds – Effect of post weld heat treatment, Def. Technol. 11, 4 (2015) 362-369.
17. G.S. Upadhyaya, Powder Metallurgy Technology, Ch3, Metal Powder Characteristics, Cambridge International Science Publishing, 2002. (in Kanpur)

18. N.M. Rahaman, Ceramic Processing, Ch.2, Synthesis and Preparation of Powders, 2nd ed., CRC Press, 2017. (in Boca Raton)

19. V. V. Shanbhag, N.N. Yalamoori, S. Karthikeyan, R. Ramanujam, and K. Venkatesan, Fabrication, surface morphology and corrosion investigation of Al 7075-Al2O3 matrix composite in sea water and industrial environment, Procedia Eng. 97 (2014) 607-613.

20. Pietsch, Agglomeration in Industry: Occurrence and Applications, Ch.8, Applications in Environmental Control, Wiley VCH, Vol.2, 2005. (in Florida)

21. M. Ferry and P.R. Munroe, Recrystallized grain size prediction in a particulate reinforced metal matrix composite, J. Mater. Sci. 38, 9 (2003) 1925-1930.

22. M. Ferry and P.R. Munroe, Hot Working behaviour of Al-Al2O3 particulate reinforced metal matrix composite, Mater. Sci. Technol. 11, 7 (1995) 633-641.

23. DIN4776, Standard for Measurement of Surface Roughness, 1990 Edition, May 1990.

24. JIS B 0601-2001, Surface Roughness, Geometrical Product Specifications (GPS) - Surface texture: Profile method.

** Boothak:** Алуминијумски метални композити се користе у разним областима као што су летелице, подводни уређаји, аутомобили. Неколико различитих техника је коришћено за добијање ових композита. Међу многим методама, метод металургије праха пружа прецизност у димензијама и висок принос. У овом раду је представљено добијање композита који се састоје од 7075 алуминијумских листића и Al2O3 честица топлим пресовањем. Испитиван је утицај побољшања и топлотног третмана T6 на механичка својства композита. Честице Al2O3 су додате у тежинским процентима од 5, 10 и 15 % и композити су добијени топлим пресовањем. Температурски третман T6 се састоји из грејања раствора на 480 °C 2 h и процеса старења на 120 °C 24 h. Отпорност на хабање композита одређена је тестирањем клизања узорка по сувом диску. Тврдоћа композита по Бринелу има тенденцију раста са порастом Al2O3 до 10 wt.% и са T6 третманом. До 10 wt.% Al2O3 су побољшили отпорност на хабање. Такође, запремински губитак материјала се смањује након процеса старења.

**Кључне речи:** метални композити, AA7075, алуминијум, листићи.