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The Effect of Sintering Temperature and Time on Microstructure, Hardness and Wear Behaviors of Al 99.9/GNP Composites

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Abstract:

In this study, it was aimed to investigate the microstructure, hardness and wear behavior of graphene nanoplate (GNP) reinforced composites with Al 99.9 matrix produced by powder metallurgy. Different temperatures and times were applied in the sintering process. The hardness values of the composites increased as the sintering temperature and time increased. The hardness values decreased with the increase of GNP reinforcement ratio. The wear losses decreased depending on the increase in sintering temperature and time. With the increase in the GNP reinforcement ratio, reductions in wear losses were recorded. It has been concluded that the GNP reinforcement element in the composite structure reduces the friction coefficient and wear losses by having some lubricating effect. It was observed that the neck and bonding formation between Al 99.9 matrix grains improved with increasing sintering temperature and time. It was concluded that with the development of intergranular bonds, the porosity in the composite structure decreased and the mechanical properties increased.

Keywords*: Metal matrix composite; Al 99.9; Graphene nanoplatelet; Microstructure; Mechanical properties.*

1. Introduction

In recent years, metal matrix composites (MMC) have been used in engineering applications where high mechanical properties are primarily desired. Aluminum and its alloys are more preferred for metal matrix because of their numerous positive properties [1]. Aluminum and its alloys have low density, good conductivity and good machinability etc. Thanks to its properties, it is used in a wide variety of industrial areas.

However, pure aluminum is rarely used due to its rather poor tribological properties and low overall strength. Instead, it is preferred to use various Al alloys and aluminum-based composites (AMC). Al_2O_3, SiC, BN and B_4C etc. to improve the mechanical properties of AMCs. ceramic reinforcements are used. In recent years, a new class of reinforcing additives has emerged in the form of carbon nanomaterials (carbon nanotubes, fullerene and graphene,

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etc.). When these reinforcements are added to pure or alloyed aluminum, they both improve the physical and mechanical properties of the base metal and show self-lubricating properties [2].

Graphene, one of these next-generation materials, is considered the strongest material in the world and has some exceptional properties [3]. Graphene has recently attracted attention in both academic and important industrial fields due to material that improves the mechanical properties of metallic composites [4]. The addition of GNP in metals such as aluminum can increase the strength and wear resistance of the material and reduce the coefficient of thermal expansion. It is very difficult to achieve homogeneous distribution of GNP in the production of GNP reinforced AMC. The fact that the GNP has a huge specific surface area is the main reason for this difficulty [5]. In order to take advantage of the superior properties of the reinforcement in the metallic matrix, some technical difficulties are encountered during the production of MMCs reinforced with GNPs. The main ones are; Proper selection of reinforcement, distribution of reinforcement in the matrix, reactivity between reinforcement and matrix, interfacial decoupling and preferred reinforcement orientation. In particular, some of these difficulties may be related to the nature of these materials, while some may be related to the production technique [6]. For the reasons mentioned above, Powder Metallurgy (PM) method is more preferred in the production of nano-size garafen reinforced aluminum-based composites.

In recent years, nano-sized reinforcement materials have shown significant improvements in the properties of metal matrices compared to traditionally use macro-sized reinforcements. It can provide a significant increase in technical characteristics even with small volume reinforcement rates, while maintaining the ductility of the metal with which nano-sized materials are reinforced. Thus, MMCs; they can demonstrate high specific strength, improved hardness, improved thermal conductivity, good wear resistance and improved damping capacity [7]. Various studies are included in the literature on strengthening aluminum and its alloys with nano-graphene, carbon nanotubes nano-sized reinforcement materials and studying their various properties [8-19].

In the experimental studies in the literature, it has been observed that different aluminum alloys are generally used (2XXX, 6XXX, 7XXX etc.). Among these alloys, there are dominant alloying elements (Cu, Zn, Si, Mn Mg etc.) that improve the technical properties. In this study, the highest commercial purity 99.9% aluminum was used. Therefore, it is aimed to contribute to the literature by examining the technical properties of the nano graphene reinforcement element in pure aluminum. For this purpose, nano-composite materials were produced by powder metallurgy technique by reinforcing 0.00%, 0.25%, 0.50% and 1.00% by weight GNP into 99.9% commercially pure Al 99.9. Hardness measurements and wear tests were carried out on the all composites. The microstructures of the composite materials and their surfaces were examined by SEM microscope after the wear test.

2. Materials and Experimental Procedures

Technical properties of the matrix material aluminum Al 99.9 (EN AW-1090), the reinforcement element graphene nano-plate (GNP) obtained from the Nanograph Nanotechnology Company and used in the production of composite materials are given in Table I.

Firstly, the mixing ratios of Al 99.9+GNP were determined. In the mixing process of Al 99.9 and GNP, pre-mixing process was applied to GNP in Ethanol to prevent agglomeration of GNP. Then, Al 99.9+GNP test materials were mixed in a ball mill at 300 rpm for 120 min. At the last stage, the composite mixtures were compressed under 750 MPa pressure, and test samples with a diameter of 12 mm and a length of 20 mm were obtained. In the sintering process, two different sintering temperatures, two different fixed sintering times and two different temperature rise times were applied. The sintering process parameters applied according to the GNP reinforcement ratios are given in Table II, and the graphics of the sintering process are given in Fig. 2.

Tab. I Technical specification of test materials.

Al 99.9				**Graphene Nanoplatelet (GNP)**	
Cu	≤0.02 %	Intensity	2.71 g/cm^3	Pureness	99.5 %
Mg	≤0.01 %			Thickness	6 nm
Mn	≤0.01 %	Melting Degree	660°C	Diameter	18 μm
Fe	≤0.07 %			Colour	Grey
S	≤0.07 %	Hardness	18 – 20 HV	Thermal Conductivity	5300 W/mK
Zn	≤0.03 %			Specific Surface Area	170 m^2/g
Ga	≤0.03 %	Thermal Conductivity	220 W/mK	Thermal Expansion	-6×10^{-4}/K
V	≤0.05 %			Melting point	3000°C
Ti	≤0.01 %	Dimension	100-300 μm		
Al	99.9 %				

Tab. II Values of sintering process.

Sintering temperature, 530°C				**Sintering temperature, 600°C**			
Sample No.	GNP ratio, wt. %	Temp. rise time, min	Constant sintering time, min	Sample No	GNP ratio, wt. %	Temp. rise time, min	Constant sintering time, min
1	0.25	30	90	17	0.25	30	90
2	0.50			18	0.50		
3	1.00			19	1.00		
4	0.00			20	0.00		
5	0.25	60		21	0.25	60	
6	0.50			22	0.50		
7	1.00			23	1.00		
8	0.00			24	0.00		
9	0.25	30	180	25	0.25	30	180
10	0.50			26	0.50		
11	1.00			27	1.00		
12	0.00			28	0.00		
13	0.25	60		29	0.25	60	
14	0.50			30	0.50		
15	1.00			31	1.00		
16	0.00			32	0.00		

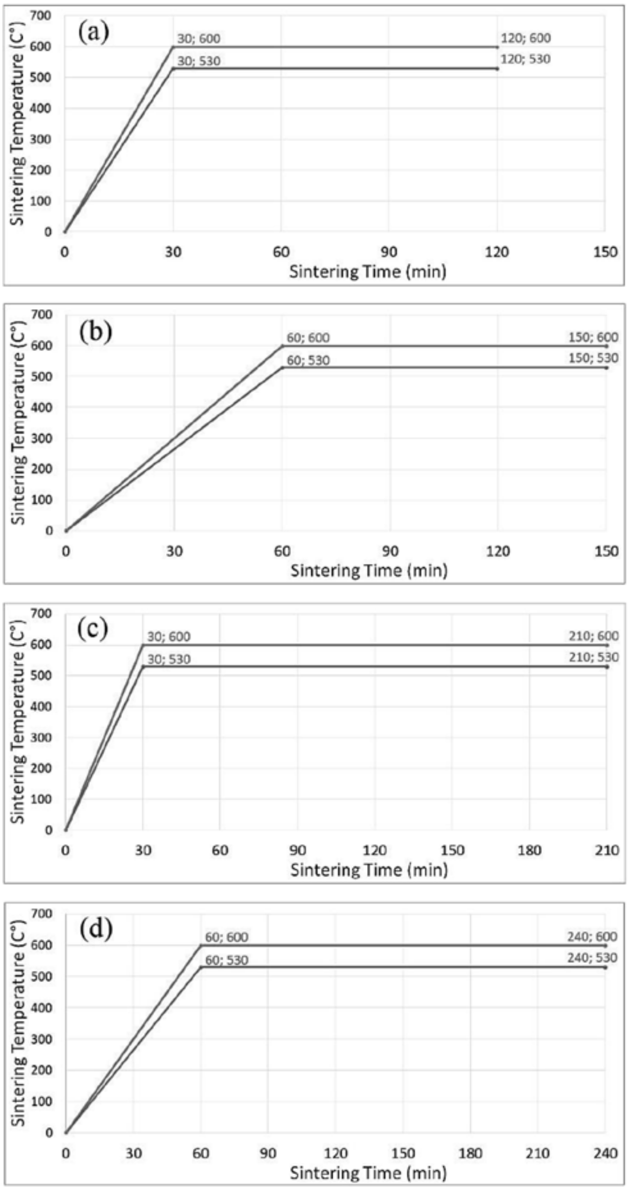

Fig. 1. Sintering processes at different temperatures and times, (a) 30 min temperature rise time/90 min sintering time, (b) 60 min temperature rise time/90 min sintering time, (c) 30 min temperature rise time/180 min sintering time, (d) 60 min temperature rise time/90 min sintering time.

The images were taken using (*QUANTA FEG 450* and *JEOL JSM 5600 LV)* SEM in order to examine the microstructure of the composite experimental specimens after sintering processes were completed. Then, the hardness measurements were made with the Vickers method (with the *Qness Q10 A+*) from three different regions of the 12 mm diameter surface of each composite sample and then taking the arithmetic averages. The wear tests were carried out on a steel disc with a hardness of 207 HV, applying a constant test load of 30 N, a speed of 1.1 ms^{-1} and a wear distance of 400 m to carry out with the pin-on disc wear device. The wear test specimens were weighed on an electronic balance with a sensitivity of 0.0001 g before starting the test. After the test, the same samples were weighed again and the wear loss was recorded as in grams. In Fig. 2, photographs taken during the tests and examinations carried out in this experimental study are given.

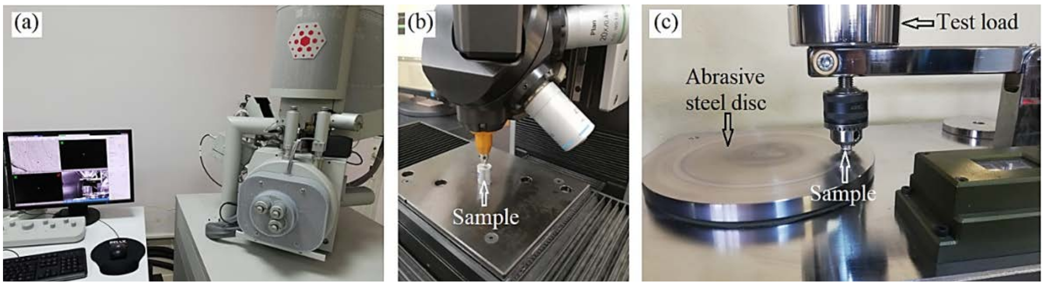

Fig. 2. (a) SEM imaging, (b) Vickers hardness measurement, and (c) Pin-on disc wear test.

3. Results and Discussion
3.1. Evaluation of microstructures

The microstructure of Al 99.9 composite materials produced by powder metallurgy method at 0.00%, 0.25%, 0.50% and 1.00% GNP reinforcement ratios were investigated in two stages. For this purpose, firstly, SEM images of the composite materials with surface polishing were taken (Figs 3 and 4). In the second stage, fracture surface images were taken with SEM to better examine the intergranular bond formation (Fig. 5). The images given in Figs 3 and 4 were selected from the samples with the minimum and maximum GNP reinforcement ratio and sintering time in order to better understand the difference between the microstructures of composite materials.

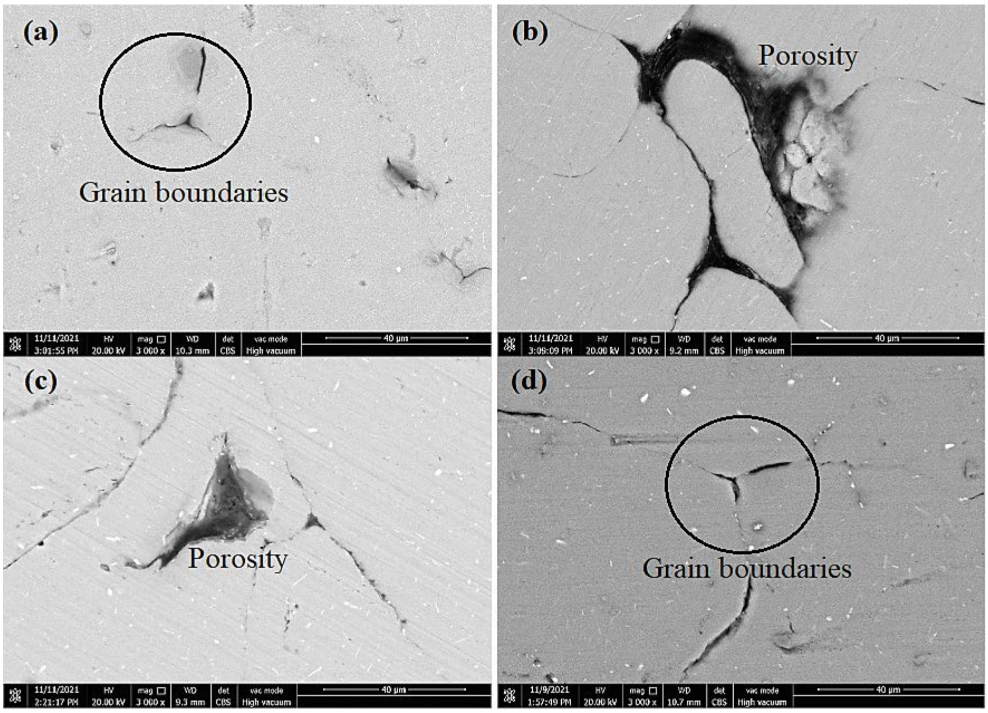

Fig. 3. Microstructures of GNP reinforced Al 99.9 composites sintered at 530°C and polished; (a) 0.25% GNP-30 min temperature rise time/90 min sintering time, (b) 1.0% GNP-30 min temperature rise time/90 min sintering time, (c) % 0.25 GNP-30 min temperature rise time/180 min sintering time, (d) 1.0% GNP-30 min temperature rise time/180 min sintering time.

The difference in microstructure between 530°C sintering and 600°C sintering can be seen very clearly by SEM images in Fig. 4. It can be said that the bond formation of Al 99.9 matrix material particles after sintering is weaker in composites sintered at 530°C compared to 600°C. This result shows that due to the large difference in melting points, a low bond is formed between graphene and Al, and atomic diffusion facilitated at high temperatures [20].

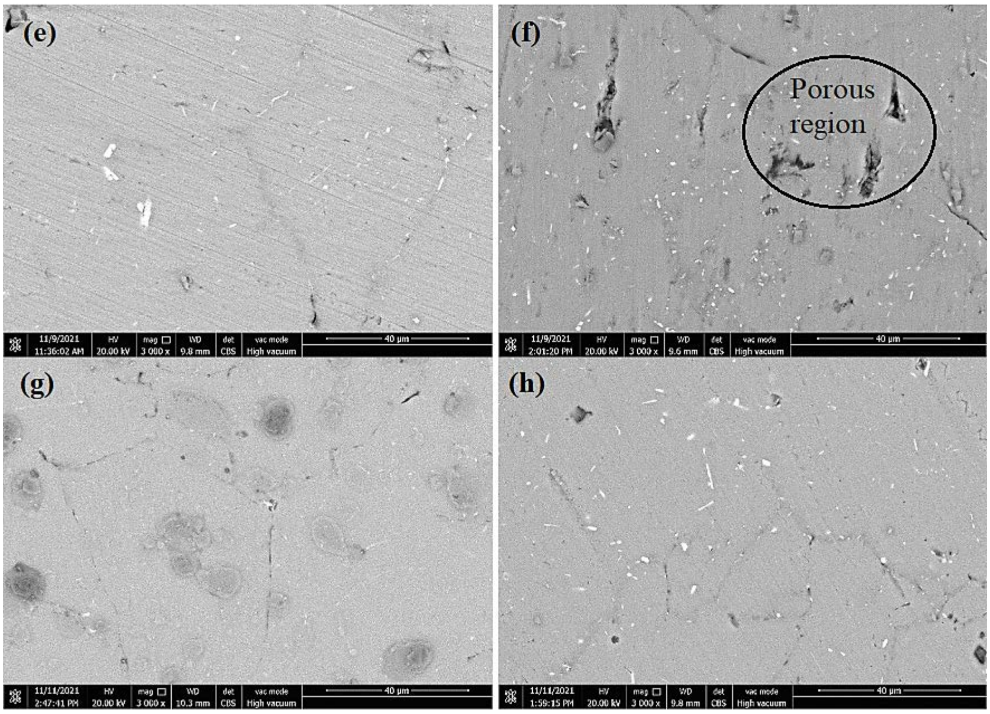

Fig. 4. Microstructures of GNP reinforced Al 99.9 composites sintered at 600°C and polished; (e) 0.25% GNP-30 min temperature rise time /90 min sintering time, (f) 1%, 0 GNP-30 min temperature rise time/90 min sintering time, (g) 0.25% GNP-30 min temperature rise time/180 min sintering time, (h) 1.0% GNP-30 min temperature rise time/180 min sintering time

The spaces between the Al 99.9 grains can be clearly seen in Fig. 4. And also, it is understood that this weak bond formation affects the amount of porosity in the composite structure (Fig. 4 b and c). In accordance with the conclusion that the GNP reinforcement ratio affects the microstructure, in this study, it was concluded that both intergranular dec and porosity in the structure were negatively affected by the increase in the amount of GNP from 0.25 to 1.00%. At the same time, it can be said that the density of the composite structure decreases with the increase in the amount of GNP. A similar result was reported in a study in the literature [21]. It is thought that the GNP particles, which settle between the Al 99.9 particles of the matrix material and can agglomerate in places, have a negative effect on the microstructure. In a study in the literature, a significant agglomeration effect of graphene added to Al is mentioned [22]. In another study, it was concluded that agglomeration of graphene adversely affected the mechanical properties [23]. In order to better examine this situation, more detailed evaluations were made on the SEM images in Fig. 4 taken from the fractured surfaces of the composites. Finally, in the evaluation of the microstructures in Fig. 4, the effect of sintering time on microstructures can be mentioned. In order to make a clear evaluation of the sintering time, SEM images of the samples that were sintered at the lowest and highest times were selected (30 min, temperature rise time /90 min, sintering time at constant temperature and 30 min, temperature rise time /180 min, sintering time at constant temperature). It can be stated that as the sintering time at constant temperature increased from

90 to 180 minutes, the bonding between the grains increased, and the Al 99.9 grains came together to form larger grains. This difference can be understood when the microstructure images in Fig. 4 a-c and b-d are carefully examined.

In a study conducted in the literature, it was stated that the 0.1% GNP reinforced Al/GNP composites bond between the particles and good neck formation were observed during sintering at 600°C for 180 minutes from SEM analyses of [24]. Similarly it is understood from the SEM images that the two different temperature rise times applied in the sintering process did not cause a significant change on the microstructure of the composites. This judgment was reached as a result of the necessary preliminary evaluations by taking the microstructure images of all composite samples produced in this study. Therefore, no additional comments were made regarding the rise time to the sintering temperature. In order to examine in more detail how the sintering heat treatment was successful and how it affected the grain structure, SEM images at different magnifications were taken from the fractured surfaces of the composite samples. SEM images created from samples of composites with the lowest and highest GNP reinforcement, sintered at 530°C and 600°C, and subjected to the sintering process in the minimum and maximum time, are given in Figs 5-6.

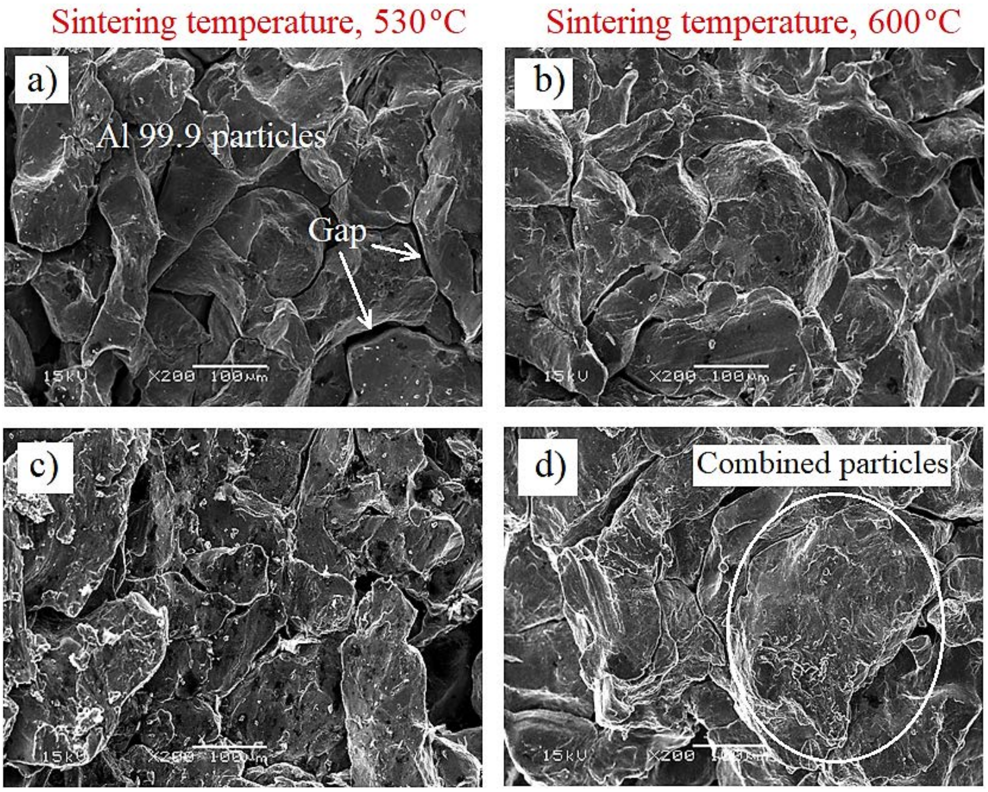

Fig. 5. Fractured surface SEM images of 0.25% GNP reinforced composites sintered at 530°C and 600°C; a) 30 min temperature rise time/90 min sintering time-530°C, b) 30 min temperature rise time/90 min sintering time-600°C, c) 30 min temperature rise time/180 min sintering time-530°C, d) 30 min temperature rise time/180 min sintering time-600°C.

When the microscope images in Fig. 5 are examined in detail, it dec understood that there are more gaps between the Al 99.9 particles in the sample sintered at 530°C (Fig. 5, a). Especially, this structural difference emerges more clearly between Fig. 5 a and b. Likewise, it is seen that the grain structure varies somewhat between samples sintered for 30/90 min and samples sintered for 30/180 min. It is seen that with increasing sintering time, Al 99.9 grains bind more and form larger grains (Fig. 5, d). Due to the non-spherical shape of the Al 99.9 powders used in the production of composites, it is understood that the grains that come together during sintering form a mixed-shaped grain structure. It is known that the 530°C and 600°C process temperatures applied in sintering are a suitable range for Aluminum materials. As can be seen from the SEM images taken after polishing the surfaces of composite materials, it can be said that the sintering process was successful (Fig. 4). However, in the images in Figs 5 and 6 taken from the fractured surfaces of the composite materials, it has been observed that a different structure was emerged. During the separation of the Al 99.9 matrix material from each other, it was revealed that the bonds between the grains were not strong enough. It is thought that this situation is caused by the GNP reinforcement element in the composite structure. More detailed evaluations on this issue are made in the following sections. SEM images of 1.00% GNP reinforced composites are given in Fig. 6.

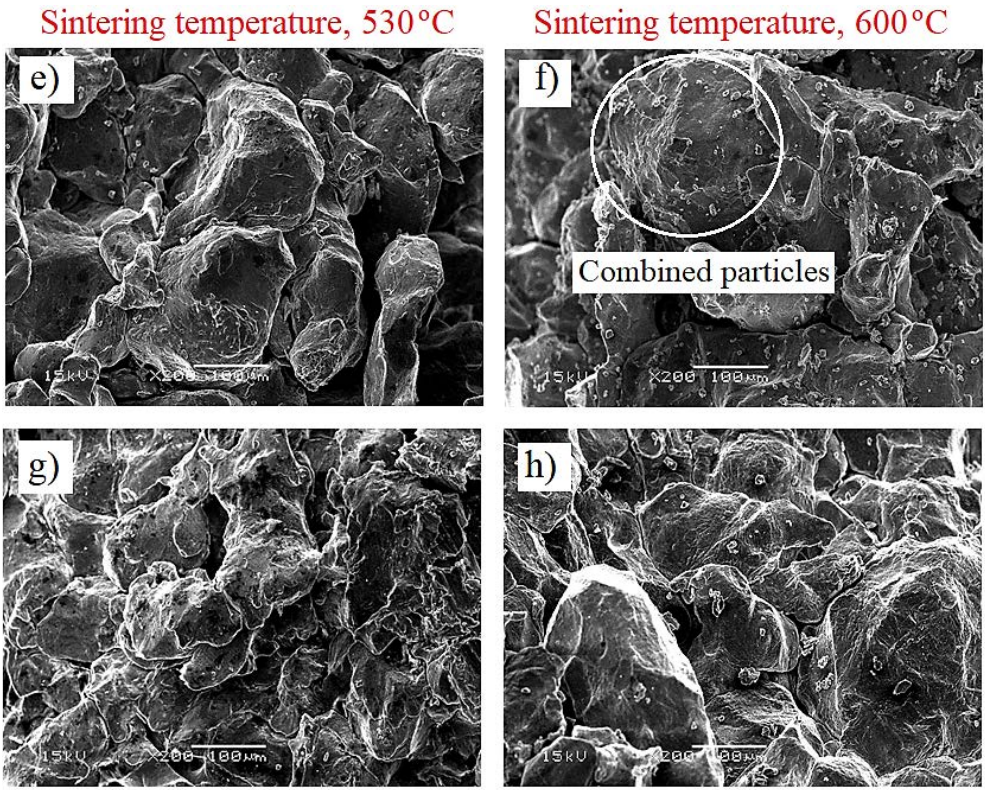

Fig. 6. Fractured surface SEM images of 1.00% GNP reinforced composites sintered at 530°C and 600°C; e) 30 min temperature rise time/90 min sintering time min-530°C, f) 30 min temperature rise time/90 min sintering time min-600°C, g) 30 min temperature rise time/180 min sintering time min-530°C, h) 30 min temperature rise time/180 min sintering time min-600°C.

Looking at the SEM images in Fig. 6, it can be stated that they are similar to the microstructures in Figure 5. Also it can be said that as the sintering temperature, time increase and the bonding between the grains increases and it turns into larger Al 99.9 grains. However, it was also understood that the sintering process becomes a little more difficult with the increase of the GNP reinforcement ratio from 0.25% to 1.00%. It is thought that the very different thermal properties between GNP and Al 99.9 cause this situation. Because the thermal conductivity of Al 99.9 material is 220 W/mK, GNP is 5300 W/mK. In addition, GNP has a very high specific surface area of 170 m^2/g. Although the GNP reinforcement element in the composite structure is doping at a rate of 1.00% by weight, it is considered that it absorbs some of the sintering temperature thanks to its superior thermal conductivity properties. Particularly, it is thought that GNP particles, which enter between Al 99.9 particles and can form larger surface areas by clumping from place to place, act as a kind of thermal barrier. In a study, it was stated that graphene could not be distributed homogeneously along the Al grain boundaries. It has been stated that the agglomeration of graphene and the non-uniform size and morphology of the Al grains cause this situation [20]. In another study, it was emphasized that with the increase of GNP doping ratio, the tendency to agglomerate also increased, and the agglomerated graphene weakened the contact area between the matrix particles [24]. The magnifications of the SEM images in Figs 4, 5 and 6, where the microstructures were examined, were not sufficient to distinguish the nano-sized graphene plates in the composite structure. For this reason, SEM images in Fig. 7 are given to show the reinforcing element GNP particles in the structure.

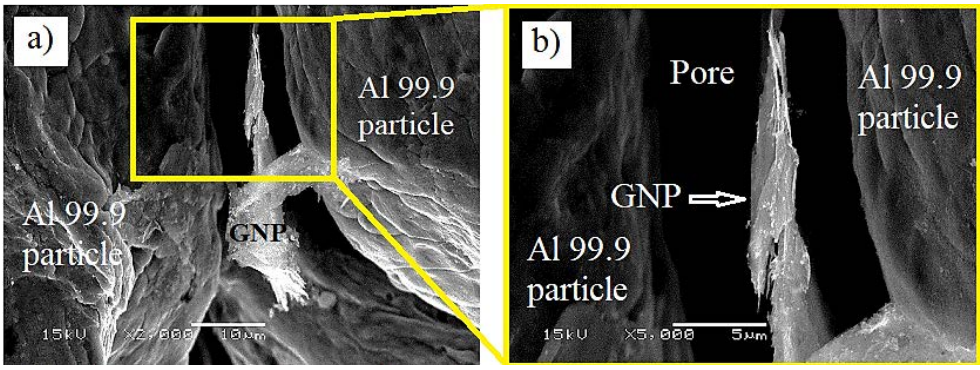

Fig. 7. GNP image located between Al 99.9 grains.

In Fig. 7, the GNP particle can be easily seen at ×2000 and ×5000 magnifications. As stated above, it was understood that GNP settles between the Al 99.9 matrix particles, preventing the heat transfer during sintering to some extent and causing porosity. In addition, it has been reported in a study that GNP with high specific surface area and wrinkled morphology reveal multiple interfaces in composites and cause defects in grain aggregation [11]. One of the important problems encountered in the production of such metal matrix composites is the wetting problem between the matrix and reinforcement. In the literature, it is stated that the mechanical strength of GNP reinforcements depends on the reinforcement distribution in the matrix, the volume ratio, the interfacial wettability and the direction of the reinforcement [23]. It was also noted in the literature that wetting nano-sized materials by the matrix element was more difficult than micron-sized ceramic particles. It was stated that this problem manifests itself at the highest level, especially in materials with lubricating properties such as graphene [25]. In addition, during the mixing process applied in the preparation of composite mixtures, graphene plates can adhere to the Al matrix. During the subsequent heating process, aluminum and graphene can react with aluminum carbide phase to form. It is thought that the aluminum carbide formed in the composite structure reduces the mechanical properties by causing brittle areas. Similar results were reported in a study in the literature [26].

3.2. Evaluation of Mechanical Properties

Mechanical properties of Al 99.9 matrix composites produced at 0.25%, 0.50% and 1.00% GNP reinforcement ratios by powder metallurgy method were examined with hardness and wear behavior. The hardness measurement results of composite samples sintered at 530°C and 600°C and for different periods using the Vickers method are given in Fig. 8.

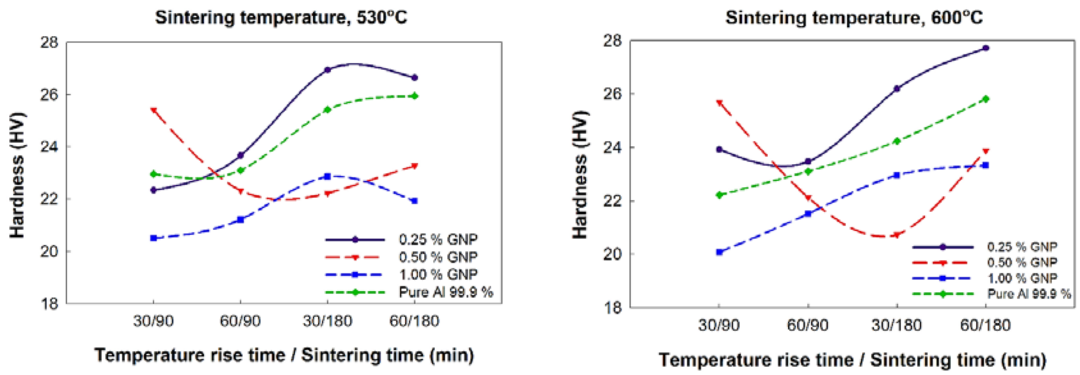

Fig. 8. Hardness values of GNP reinforced composites with Al 99.9 matrix sintered at different temperatures and times.

Looking at the graphics in Figure 8, the first thing that stands out is that the hardness values of the composite samples sintered at 600°C are slightly higher than at 530°C. Increasing the sintering temperature had an effect on the mechanical properties of the composites. A similar result was obtained with the conclusion of P.D. Srivyas et al. that an increase in the sintering temperature increases the hardness value of the material [27].

The most important reason for this is that with increasing temperature, the bond formation between Al 99.9 grains gets stronger and the porosity in the structure decreases. With the increase of the bonding between Al 99.9 grains, the porous regions in the structure gradually decreased and during the hardness measurement, the tip of the measuring probe encountered less porous regions. Another important point that stands out in the graphics is that the hardness values have increased due to the increase in the sintering time in general. The reason for this is that as the sintering temperature increases, the neck and bond formation between Al 99.9 grains gradually increases with the increase of the time, and the composite structure becomes more rigid. A sudden increase in hardness values occurred as the sintering time at constant temperature increased from 90 minutes to 180 minutes. Therefore, if high hardness is desired in such aluminum matrix composites, it was considered that it would be more appropriate to have a sintering time of 180 minutes. It can be said that the rise time of the heat treatment furnace to the sintering temperature during the sintering process has little effect on the hardness values. However, this effect is more stable on samples sintered at 600°C. As the temperature rise time increased from 30 minutes to 60 minutes, the hardness values increased. In samples sintered at 530°C, this situation appears as partly an increase in hardness and partly a decrease in hardness. Due to the very inhomogeneous microstructures in such composite materials, unstable results can sometimes occur in the mechanical properties and their measured values. It is known that the most important reason for this is the low wetting caused by the matrix-reinforcement mismatch and the resulting porosity. In addition, the fact that the reinforcing elements in the particle-reinforced metallic composites condense in certain areas and cause agglomeration is another important negative situation. Due to the reasons mentioned above, sometimes extraordinary results can occur in mechanical tests such as hardness measurement, tensile, bending and abrasion. It is known that the matrix material Al 99.9 used in this study and the reinforcement element nano graphene plate are two very different structures from each other in terms of both physical and thermal properties. Therefore, it can be considered normal for the composite structure in which these two materials are combined to exhibit some unstable mechanical values.

If an evaluation is to be made according to the GNP reinforcement ratio in the composite structure, the first thing that stands out is that the lowest hardness values are obtained in 1.00% GNP reinforced composite and the highest hardness values are obtained from a 0.25% GNP reinforced sample at two sintering temperatures of 530°C and 600°C and at different sintering times. In a similar study, it was stated that the highest hardness value was obtained from the material containing 0.25% GNP reinforcing ratio. It was reported that this value is 98% higher than the hardness value of pure aluminum. In another study in the literature, it was stated that Al matrix and 0.3% GNPs reinforced composite exhibited 11.8% higher Vickers hardness compared to monolithic aluminum [28]. In another study, composites were produced by reinforcing 0.25%, 0.50% and 1.00% GNP to Al using powder metallurgy method. In the hardness measurements, it is stated that the highest value was measured in 0.25% GNP reinforced composite material [29]. With the increase of GNP reinforcement ratio, the amount of porosity in the composite structure increased. In addition, it was evaluated that GNP nano-plates, which are placed between the Al 99.9 particles or partially cover the particles, acted as a thermal barrier and reduced the required bonding during sintering. Therefore, the hardness values were measured less in the 1.00% GNP reinforced samples containing the highest amount of GNP. SEM images supporting this view are given in Figures 4,5,6 and more detailed explanations are given in item 3.1 where microstructures are evaluated.

The graphs created according to the values obtained from the wear tests carried out to examine the mechanical properties of Al 99.9/GNP composite samples produced by powder metallurgy method and sintered at 530°C and 600°C and at different times are given in Fig. 9.

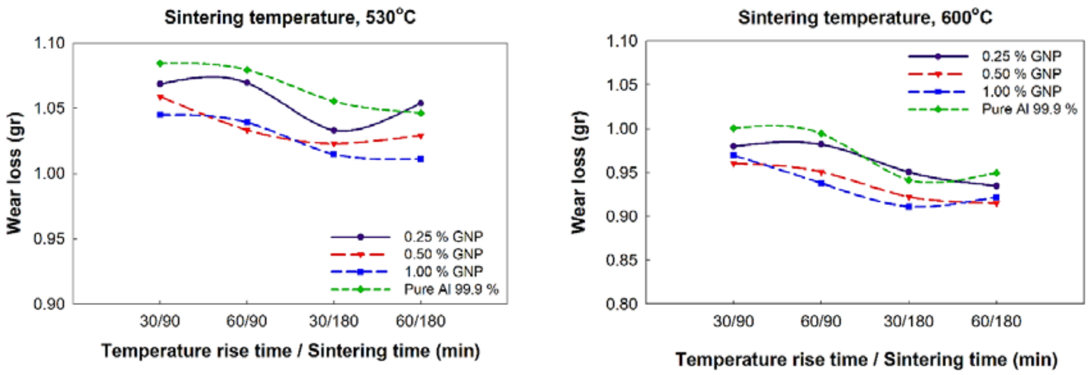

Fig. 9. Wear loss values of GNP reinforced composites with Al 99.9 matrix sintered at different temperatures and times.

When the graphs given in Fig. 9 are examined, the first point that draws attention is that the wear losses in the composite samples sintered at 600C are less than those of the composite samples sintered at 530°C. This result can be explained in the simplest way by the fact that the hardness values of the composite samples sintered at 600°C are higher than those of 530°C. However, it is thought that this is not the only reason. It has been previously stated that there is less porosity in composites sintered at 600°C than at 530°C. In such particle-reinforced composite structures produced by powder metallurgy, insufficient wetting during sintering and weak bond formation at the matrix-reinforcement interface increase wear losses. In fact, the decrease in wear losses with the increase of sintering time supports this assessment. Increasing the sintering time increased the bonding between the Al 99.9 particles and the resistance against breakage and separation between the particles.

Another important point that stands out in the graphics is that the wear losses begin to decrease with the increase in the GNP reinforcement ratio. There are studies in the literature reporting similar results. Contrary to the increase in the GNP additive ratio, it was reported that the wear loss decreased [30]. In another study, it was stated that the wear loss decreased in contrast to the increase in the GNP additive [31]. This situation is thought to be caused by the lubricating property of GNP in the composite structure. It has been stated that the GNP reinforcement in the matrix material acts as a solid lubricant and provides lubrication under dry sliding conditions [27]. In this study, the lowest wear losses in GNP reinforced composite materials applied at 530°C and 600°C sintering temperatures were obtained from 1.00% GNP reinforced samples. Pure Al 99.9 material without GNP reinforcement was the sample with the highest wear losses. Also, it was concluded that wear losses increased in direct proportion to the decrease of GNP additives in the composite structure at both sintering temperatures. In fact, it can be thought that the porosity in the structure increased with the increase of the GNP reinforcement ratio, and for this reason, the composite structure can be eroded more easily. However, it can be stated that the spaces created by the porosity are partially filled by plastering Al 99.9 matrix material. Thus, it is considered that some material, which will wear away from the composite structure, is plastered on the surface of the wear sample. Therefore, this has had a reducing effect on wear losses. SEM images taken from the surfaces of the samples after the wear tests were given in Fig. 10 and Fig. 11. In order to make the difference more obvious, the lowest and highest GNP ratios and the lowest and highest sintering times were taken into account in the SEM images given.

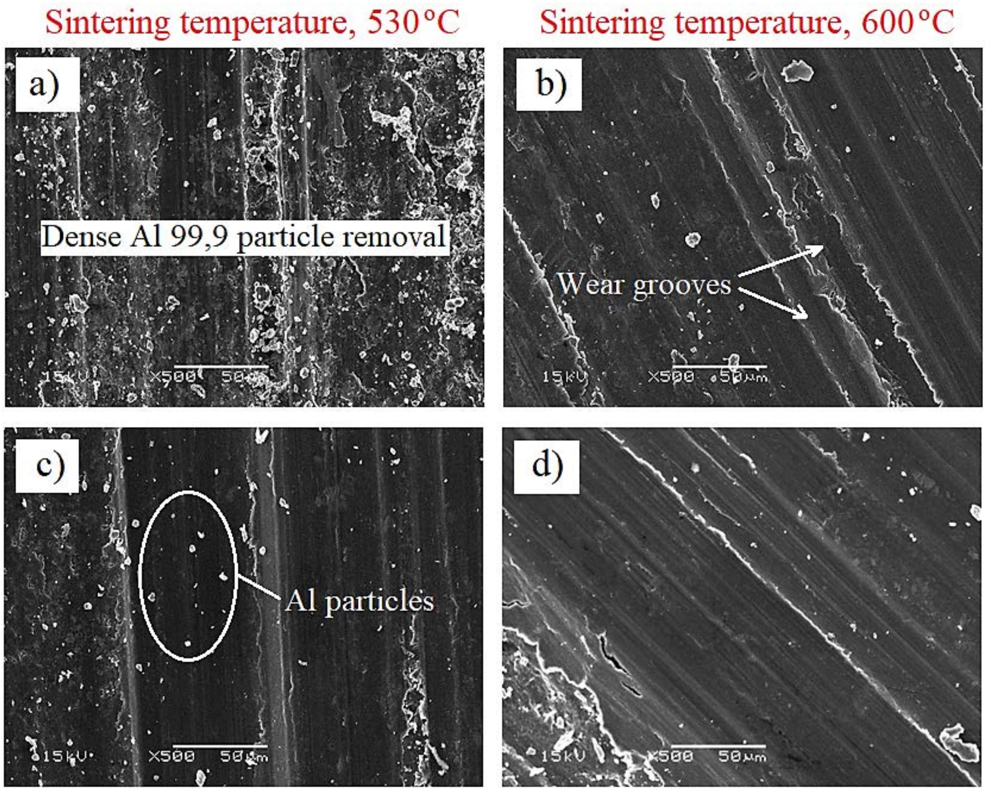

Fig. 10. SEM images of the worn surface of 0.25% GNP reinforced composites sintered at 530°C and 600°C; a) 30 min temperature rise time/90 min sintering time-530°C, b) 30 min temperature rise time/90 min sintering time-600°C, c) 30 min temperature rise time/180 min sintering time-530°C, d) 30 min temperature rise time/180 min sintering time-600°C.

When Fig. 10 was examined, it was seen that there was more particle rupture in samples sintered at 530°C than at 600°C. In addition, it can be said that the wear cavities are wider and deeper in samples sintered at 530°C, although not so much. As we have stated from the beginning, it is understand from the microstructure images that 530°C sintering is somewhat inadequate compared to 600°C. With sintering at 600°C, the composite structure formed a stronger bond. In the samples sintered at 530°C, it was easier for the Al 99.9 particles in the composite structure to separate from each other and move away from the structure due to the heat and pressure caused by the friction during the wear tests. When we look at Fig. 10 (a, c), this situation is clearly seen. It was stated in the evaluation of the graphics in Figure 9 that increasing the sintering time from 90 minutes to 180 minutes had an effect on reducing wear losses. When the worn surface images in Fig. 10 are examined carefully, it can be stated that the wear loss values are supported by the surface images. It is understand that with the increase of sintering time at both sintering temperatures, the broken Al 99.9 particles on the eroded surfaces decrease. In addition, it can be seen that there is a slight decrease in the width of the wear cavities with the increase of the sintering time. Worn surface images of 1.00% GNP reinforced composite samples subjected to abrasion tests under the same conditions are given in Fig. 11.

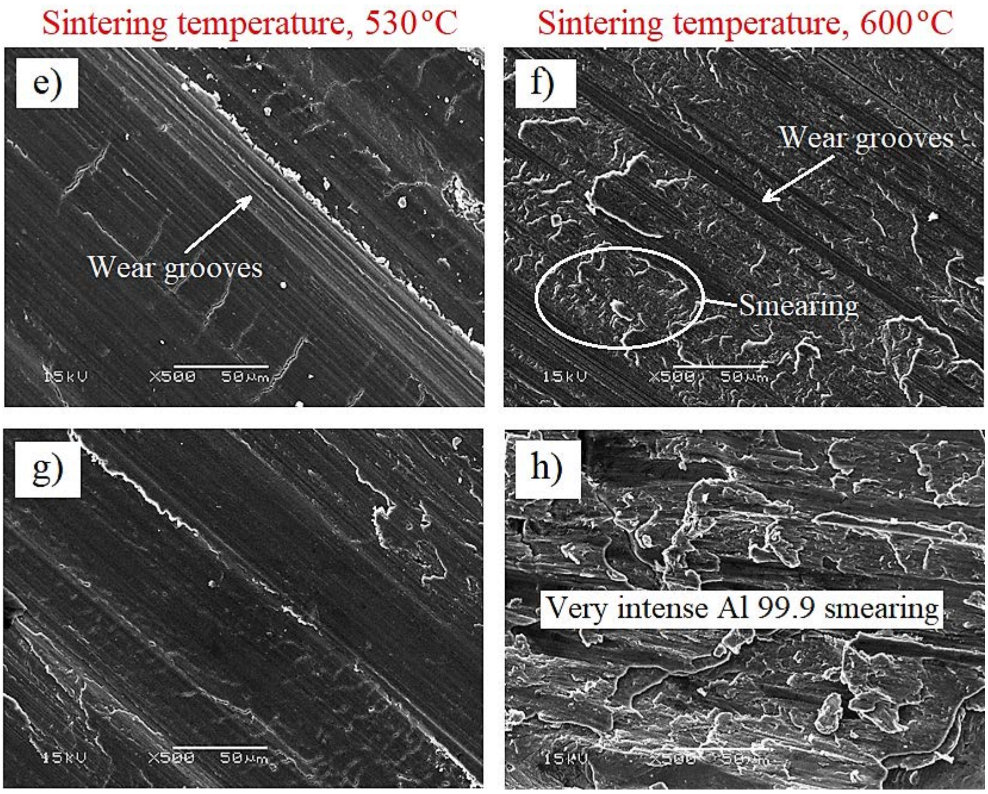

Fig. 11. SEM images of eroded surface of 1.00% GNP reinforced composites sintered at 530°C and 600°C; e) 30 min temperature rise time/90 min sintering time-530°C, f) 30 min temperature rise time/90 min sintering time-600°C, g) 30 min temperature rise time/180 min sintering time-530°C, h) 30 min temperature rise time/180 min sintering time-600°C.

Looking at the microstructure images in Fig. 11, similarities with the images in Fig. 10 are striking. However, when examined in more detail, it was understood that there were differences arising from the plastering of the Al 99.9 matrix material in the first place. Especially in the surface images in Fig. 11 (f, h), the excess of the plastered areas draws attention. In the evaluations made in the previous sections, it was stated that the porosity in the composite structure increased with the increase in the GNP reinforcement ratio. It was also mentioned that Al material could be filled by plastering the cavities created by the porosity and/or GNP agglomeration in the composite structure. The fact that these assessments could be correct was supported by the surface images in Fig. 11. A part of the material that could break away from the composite structure as a result of Al 99.9 plastering adhered to the sample surface again during the tests and had a reducing effect on the wear record values. The other point is that the wear cavities on the samples sintered at 600°C are slightly less wide than the samples sintered at 530°C. This difference can be seen when the surface images in Fig. 10 and Fig. 11 are examined mutually. Therefore, due to the reasons explained above, the wear losses in the samples sintered at 600C were less than in the composite samples sintered at 530°C.

4. Conclusion

As the sintering temperature increased from 530°C to 600°C, the mechanical strength of the composite structure increased. With the increase of sintering temperature, hardness values increased in all GNP reinforcement ratios, while wear losses decreased at the same time. Increasing the sintering time from 90 minutes to 180 minutes increased the hardness values and decreased the wear losses. Depending on the GNP reinforcement ratio in the composite structure, the hardness values first increased and then decreased. In general, the highest hardness values were obtained from 0.25% GNP reinforced composites at all sintering temperatures and times. The hardness values decreased as the GNP reinforcement ratio increased to 0.50% and 1.00%. It was understood from the microstructure images that the decrease in the hardness values with the increase of the GNP reinforcement ratio was mainly caused by the voids in the composite structure due to porosity. Depending on the GNP reinforcement ratio in the composite structure, the wear losses have decreased. The most important reason for this situation was considered to be the self-lubricating property of graphene. It has been evaluated that GNP, which enters between the Al 99.9 particles and adheres to its surface, acts as a thermal barrier and somewhat prevents the heat transfer between the Al 99.9 grains. According to the experimental parameters in this study, the sintering temperature of 600^{o}C and the sintering time of 180 min were considered appropriate. In addition, it was concluded that 0.25% GNP reinforcement in hardness and 1.00% GNP reinforcement in wear loss were optimum values.

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Сажетак: *У овом истраживању циљ је био да се испита микроструктура, тврдоћа и понашање приликом хабања композита добијеног од прахова металуршким процесом,* *а који има Al 99.9 као матрицу ојачану са графенским наноплочицама (GNP). Током синтеровања примењене су различите температуре и времена синтеровања. Уочено је да се тврдоћа композита повећавала са увећањем температуре и времена синтеровања. С друге стране, тврдоћа композита се смањивала са повећањем удела GNP-а. Губици настали услед хабања су се смањивали са увећањем температуре и времена синтеровања. Такође, са повећањем удела GNP-а забележено је смањење губитака услед хабања. Примећено је да додавање GNP-а у композитну структуру поред тога што доводи до смањења губитака услед хабања, доводи и до смањења коефицијента трења јер се ствара ефекат подмазивања. Примећено је да се формирање вратова и везивања између зрна у Al 99.9 матрици побољшава са увећањем температуре и времена синтеровања. Закључено је да са развојем интергрануларних веза порозност структуре композита се смањује при чему се вредности механичких својстава увећавају.*

***Кључне речи**: Композит са металном матрицом, Al 99.9, графенске наноплочице, микроструктура, механичка својства.*