Graphene Nano platelets reinforced a composite fabricated through Ultra-High frequency induction sintering

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ABSTRACT: In this work, Graphene NanoPlatelets (GNP)/Aluminum (Al) composites reinforced from 0 wt% to 2.0 wt% GNP were studied. All different composition powders were stirred for 2 h at a speed of 35 rpm in a V-type mixer to obtain a homogeneous dispersion. Then the compositions were synthesized by ultra-high frequency induction heated sintering (UHFIHS) at processing conditions of 620 °C for 5 min and 40 MPa pressure under vacuum environment. The density, surface roughness, weight loss and Vickers hardness of the nanocomposites were evaluated. SEM, EDX and XRD analyses were performed and the obtained results were examined. The effect of the Graphene addition in an aluminium was evaluated and the optimum contribution of 0.8 percentage by weight GNP was determined.

KEYWORDS: Aluminium metal matrix composites (MMCs); Graphene NanoPlatelets (GNP); Mechanical properties; Sintering; UHFIHS

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RESUMEN: Nano plaquetas de grafeno reforzado con un compuesto fabricado mediante sinterización por inducción de alta ultra frecuencia. En este trabajo, se estudiaron compuestos de nanoplaquetas de grafeno (GNP) como refuerzo de una aleación de aluminio, estudiándose refuerzos con adiciones variables de hasta un 2,0% en peso de GNP. Los polvos, de diferentes composiciones, se agitaron durante 2 h a una velocidad de 35 rpm en un mezclador tipo V para obtener una dispersión homogénea. Posteriormente, se sintetizaron mediante un tratamiento térmico a 620 °C, en un horno de inducción de ultra alta frecuencia (UHFIHS) durante 5 min y 40 MPa de presión en un entorno de vacío. Se evaluó la densidad, rugosidad superficial, pérdida de masa ocurrida durante el proceso y la dureza Vickers de los nanocomposites obtenidos en cada caso. Se realizaron análisis SEM, EDX y XRD y se examinaron los resultados obtenidos. Se evaluó el efecto de la adición de grafeno en el aluminio y se determinó que las mejores propiedades se producían para una adición de un 0,8% en peso de GNP.

PALABRAS CLAVE: Compuestos de matriz metálica de aluminio (MMC); Nanoplaquetas de grafeno (GNP); Propiedades mecánicas; Sinterización; UHFIHS

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1. INTRODUCTION

The need for advanced engineering materials is increasing in the aeroplane, aerospace, energy, defence, and transportation industries, because a single material often cannot meet the requirements (Ozden *et al.*, 2007; Topcu *et al.*, 2009; Varol *et al.*, 2017). Powder metallurgy (Çavdar *et al.*, 2014a; Çavdar *et al.*, 2014b; Çavdar *et al.*, 2014c; Çavdar and Atik, 2014a), nanotechnology (Altıntas *et al.*, 2016; Cavdar *et al.*, 2020; Kusoglu *et al.*, 2020) applications and their heat treatments (Çavdar and Atik, 2014b; Çavdar *et al.*, 2015; Matik, 2016; Perianyagam *et al.*, 2016; Altıntas *et al.*, 2019; Yazıcı and Çavdar, 2017) are helping to meet needs.

Different nano and micro powder compositions are initially mixed and pressed, after that sintered in the powder metallurgy (PM) applications. Sintering is the most important parts of PM by reason of it causes an extraordinary increase in sample strength. Contrary to what is used in this study, conventional sintering applications are generally performed by using a furnace. This sintering method is the cheapest and easiest heating method in PM however, this can increase the properties of the material to a certain extent. Aluminium based nano or micron powders are commonly using for PM applications. In this work, the optimum contribution of the Graphene in the Al matrix was investigated for induction sintering applications. Cavdar and Akurt (2018), Rashad et al. (2015) and Khorshid et al. (2016) fabricated Al-GNP composites by using nano powders by using conventional sintering method. They investigated the microstructural, mechanical (Çavdar and Akkurt, 2018), electrochemical properties (Rashad et al., 2015) and tribological behaviours (Khorshid et al., 2016) of Aluminium composites reinforced with Graphene NanoPlatelets.

Graphene has attracted great attention in lots of studies, owing to its unique electrical mechanical (Shah and Batra, 2014; Vijayaraghavan *et al.*, 2014; Wang *et al.*, 2014; Zhang *et al.*, 2011), electrical (Zaminpayma and Nayebi, 2015; Shid *et al.*, 2014) and thermal features (Tian *et al.*, 2014; Fan *et al.*, 2014; Gan *et al.*, 2015). GNPs build-up of several layers of Graphene sheets have a high specific surface area, and because of it is grorious properties, is immense for fabricating materials (Rashad *et al.*, 2015). The dispersion of GNPs has an excellent effect on the performance of the aluminium matrix.

Saboori *et al.* (2017) investigated the compaction and the sinterability of PM Al-GNPs samples. They reported that at early stages of consolidation the rearrangement of particles is dominant, increasing with pressure, and based upon the effect of GNPs, the powder densification rate decreases. The highest hardness of the nanocomposites observed was 67 HV₅ and high thermal conductivity was only observed in the low GNP contents. Rashad *et al.* (2015) investigated electrochemical, mechanical and microstructural properties of Al-GNP materials before and after extrusion. They reported that the corrosion rate increased by the contents of GNPs and the Graphene acts as an effective cathode by accelerating the corrosion.

The aim of this work is to add Graphene nano plates into the aluminium though the induction sintering method to obtain a uniform dispersion of Graphene nano plates in the matrix. The optimum GNP contribution is determined by Vickers hardness', densities and tribological tests and from micro structural images of the aluminium based compacts.

2. MATERIALS AND METHODS

In this study, the particle size of 40-50 μ m, purity of 99.5% alumina nanoparticles (44931 Alfa Aesar Aluminum oxide NanoDur) and sub-micron particles, S.A. 500 m²·g⁻¹ Graphene (47132 Alfa Aesar, Graphene nanoplatelets aggregates,) were used. 0, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.8, 1 and 2 wt.% of GNP was added to aluminium nanoparticles and coded as in Table 1 according to the various GNP addition.

TABLE 1.	Aluminum based sample codes for used GNP
	additions

Sample codes	Compositions			
	Aluminum (wt.%)	GNP (wt.%)		
S 0	100	0		
S 1	99.9	0.1		
S2	99.8	0.2		
S 3	99.7	0.3		
S4	99.6	0.4		
S 5	99.5	0.5		
S 6	99.4	0.6		
S 8	99.2	0.8		
S 10	99	1		
S20	98	2		

The aluminium powders and GNPs were blended by ball milling in a planetary ball mill machine at rotation rate of 200 rpm for 5 h. The mixtures of Al-GNP obtained were placed in the graphite die (outside diameter 45 mm; inside diameter 20 mm; height 18 mm). The hot-pressed induction sintering process was carried out using a 2,8 kW power supply at a 900 kHz ultra-high frequency. Ten different Al-GNP compositions were sintered by ultra-high frequency induction heated sintering (UHFIHS) system at processing conditions of 620 °C for 5 min dwell time and 40 MPa pressure under vacuum environment. All induction sintered compacts were cooled by naturally. The dimensions of GNP/Al composites were 20 mm in diameter and 2.5 mm in height. The flow chart for the whole induction sintering processes is given in Table 2.

The relative densities of the sintered composites were measured by the Archimedes Method. The Radwag as 220/C/2 Archimedes Scale was used for this test. The surface roughness values of the samples were measured with the Mitutoyo Surf Test SJ-301 optic profilometer. Measurements were made at three different points of each sample. In this test, the parameters for Ra, Ry and Rz were measured. Vickers hardness value was measured with the FM 700 micro hardness Tester by applying under a load of 1 kg with a dwell time of 15 s. For each sample, five hardness measurements were recorded and the average of hardness values was considered.

The composite dimension was processed in CNC according to international standard ASTM E8/ E8M-16a (2016) with a gauge length of nearly 5 times the diameter. The tensile test of Al-GNP composites was conducted by using Autograph Shimadzu G-IS 100 kN universal test machine (Shimadzu Corporation, Kyoto, Japan). The specimen for tension test was mounted and tested with a strain rate of 0.005 s⁻¹ at the ambient temperature.

To investigate the tribological behaviour of the samples, pin-on disk tests under dry conditions were conducted and measured with the CMS Tribometer. Cylindrical pins with dimension of 6 mm in diameter and 8 mm in height were utilized from the compacted samples in the tests. The pin-on disk experiment was conducted with 15 N normal loads and sliding speeds of 150 rpm at a constant sliding distance of 1.13 km.

To obtain the distribution of GNP in the sintered aluminium matrix, as-sintered samples were observed by EDX analyse. To obtain the sintering behaviour of aluminium nanoparticles and the microstructural developments, SEM microstructure images were taken. The microstructure and EDX observation was conducted by ZEISS Evo 40 Scanning Electron Microscope (SEM).

3. RESULTS AND DISCUSSIONS

All densities, surface roughness and tribological results of the samples are given in Table 3. Micro hardness, Yield Strength (Y.S.), ultimate tensile strength (U.T.S.) and elongation results of the samples are given in Table 4. All tests results' error ranges (E.R.) are between ± 2 .

The theoretical density of aluminium and graphene is 2, 6989 g·cm⁻³ and 2 g·cm⁻³ respectively. The theoretical density of the compositions is given in Table 3 and were calculated according to the % weight values. Relative density results increased with GNP contribution.

The arithmetical mean roughness value (Ra), mean roughness depth (Rz) and maximum peak (Ry) values were measured from the surfaces of the sintered samples at three different points for each sample. The average roughness results are given in Table 3. According to the roughness results, Ra, Ry

Aluminum (Al) and Graphene NanoPlatelets (GNP) Powders	Pure Al	Mixing in the ball milling	UHFIHS at 620 °C for 5 minutes	Natural Cooling
	0,1 wt. % GNP + Al	Mixing in the ball milling	UHFIHS at 620 °C for 5 minutes	Natural Cooling
	0,2 wt. % GNP + Al	Mixing in the ball milling	UHFIHS at 620 °C for 5 minutes	Natural Cooling
	–0,3 wt. % GNP + Al	Mixing in the ball milling	UHFIHS at 620 °C for 5 minutes	Natural Cooling
	0,4 wt. % GNP + Al	Mixing in the ball milling	UHFIHS at 620 °C for 5 minutes	Natural Cooling
	0,5 wt. % GNP + Al	Mixing in the ball milling	UHFIHS at 620 °C for 5 minutes	Natural Cooling
	0,6 wt. % GNP + Al	Mixing in the ball milling	UHFIHS at 620 °C for 5 minutes	Natural Cooling
	0,8 wt. % GNP + Al	Mixing in the ball milling	UHFIHS at 620 °C for 5 minutes	Natural Cooling
	1 wt. % GNP + Al	Mixing in the ball milling	UHFIHS at 620 °C for 5 minutes	Natural Cooling
	2 wt. % GNP + Al	Mixing in the ball milling	UHFIHS at 620 °C for 5 minutes	Natural Cooling

 TABLE 2.
 Flow chart of all ultra-high frequency induction sintering processes

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Sample codes	Density results		Surface Roughness results			Tribological Results	
	Theoretical density (g·cm ⁻³)	Measured density (g·cm ⁻³)	Relative density (%)	R _a (µm)	R _y (μm)	R _z (μm)	Weight loss (g)
S0	2.6989	2.2751	84.3	0.47	6.91	4.82	0.0597
S1	2.6982	2.2962	85.1	0.64	7.07	6.65	0.0423
S2	2.6975	2.4143	89.5	0.73	7.61	7.16	0.0411
S 3	2.6968	2.4325	90.2	0.84	7.79	7.38	0.0363
S4	2.6961	2.4561	91.1	1.23	9.72	7.49	0.0344
S 5	2.6954	2.5552	94.8	1.36	14.87	8.80	0.0301
S 6	2.6947	2.5923	96.2	1.53	14.95	9.06	0.0267
S 8	2.6933	2.6636	98.9	1.57	14.96	9.83	0.0235
S 10	2.6919	2.6703	99.2	1.60	15.52	10.06	0.0237
S20	2.6849	2.6795	99.8	1.74	17.41	12.70	0.0241

TABLE 3. Average test results of sintered samples (E.R.= ± 2)

TABLE 4. The micro hardness, tensile strength (TS), elongation of GNP/Al composites average test results (E.R.= \pm 2)

Sample	Hardness Results	UTS	Elongation
codes	(HV)	(MPa)	(%)
S0	39	135	13.2
S 1	46	138	7.1
S2	51	151	6.3
S 3	55	168	5.9
S4	59	174	5.7
S5	63	184	5.3
S 6	66	193	4.9
S 8	70	208	4.3
S10	70	162	3.9
S20	67	146	3.3

and Rz results increased with GNP contribution. The reason for this increase is that more GNP particles are found on the surface horizontally and vertically with increasing GNP content.

The average HV hardness results are increased with the GNP content from 0.1 wt.% to 0.8 wt.% respectively as seen in relative densities and surface roughness results however above 0.8 wt.%, the GNP results did not increase, but showed no charge or a decrease in the hardness. The average hardness results of the samples together with the error ranges are given in Fig. 1. It was found that optimum hardness result of 0.8 wt.% GNP contribution. The fabricated Al-GNP nanocomposites obtained a high Vickers hardness of 68 HV5, which is in agreement with the Saboori *et al.* (2017) work and approximately 50% higher than monolithic aluminium.



According to the tribological test results given in Table 3, it was observed that the weight loss of the samples increasing with GNP contribution.

The variation of weight loss with 15 N normal load for pure aluminium and from 0.1 wt.% GNP-Al to 2 wt.% GNP - Al at a constant speed of 150 rpm is presented in Table 3. According to the tribological test results, the weight loss of the samples increased with GNP contribution from 0.1 wt.% to 0.8 wt.%. Contrary to the increase in GNP contribution, the wear rate (weight loss) of 1wt.% and 2 wt.% GNP-Al is more than 0.8 wt.% GNP-Al. As indicated with the hardness results in the same table, the pure aluminium has the lowest hardness, while 0.8 wt.% GNP-Al has the highest hardness. The hardness results change in direct proportion to the wearing results. The softer materials have higher wear rates compared to the harder materials (Rashad et al., 2015; Altintas et al., 2016; Tabandeh et al., 2016; Cavdar and Akkurt, 2018) in general. The wearing test results in the literature (Rashad et al., 2015; Altintas et al., 2016; Çavdar and Akkurt, 2018)

are in accordance with the presented wearing test results. It is illustrated that the 0.8 wt% GNP-Al has better coefficient of friction when compared to other samples.

Figure 2 (a-d) shows the SEM micrographs of the polished surfaces of the C20, C8, C4 and C2 coded samples at a magnification of 1000x respectively and Fig. 3 (a-d) shows 10.000x magnifications of the same samples. SEM images are well agreement with the density results of the Al-GNP nano composites. Relative densities are increasing and porosities are degreasing with GNP contribution.

EDX analysis was applied to the test sample S8, which had 0.8% by weight Graphene in the structure. The EDX analysis result is given in Fig. 4a, and the green point, Fig. 4b, indicates where the analysis is performed.

Figure 5 shows X-ray diffraction patterns from the S8 sample. Reflections attained to the aluminium element was presented. Al4C3 did not form during the process, according to the XRD patterns. It cannot be observed that the effect of chemical reaction between GNPs and Al matrix in the formation of a compound.





Hardness and tribological properties of conventional sintered Al/GNP composites were also investigated in our previous work (Çavdar and Akkurt, 2018). The composites containing same percentage of the GNP concentration in the matrix were sintered at 620 °C for 1 h under an argon environment and then cooled naturally. When the composites are compared with each other, it has been determined that the hardness values of the samples sintered by induction are approximately 3% harder and also more resistant to about 8% abrasion.

4. CONCLUSIONS

Pure Al and from 0.1 wt% to 2.0 wt% GNP/Al composites were sintered by 900 kHz ultra-high induction system. According to the microstructure observation and mechanical property characterization, the obtained results are given below;

• Horizontally and vertically GNP particles found on the surface of the Al composite. This is increased the surface roughness of the composite with GNP contribution.





FIGURE 2. SEM micrograph of: a) C20 (1000x), b) C8 (1000x), c) C4 (1000x), and d) C2 (1000x) samples.

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FIGURE 3. SEM micrograph of: a) C20 (10.000x), b) C8 (10.000x), c) C4 (10.000x), and d) C2 (10.000x) samples.



FIGURE 5. XRD diffraction of S8 composite.

FIGURE 4. EDX analysis: a) Result, and b) region of the S8 composite.

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- The HV hardness results of the composites increase with the GNP content to the 0.8 wt.%.
- The tribological test results are illustrated that the weight loss of the composites increasing with GNP contribution to the 0.8 wt.%. Contrary to the increase in GNP contribution, the weight loss is reduced. Consequently 0.8 wt.% GNP - Al composite recorded the lowest wear rate.
- All test results are shows that the optimum addition by weight of GNP is 0.8 in the aluminium composites for induction sintering process, compared to other composites each other.

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