Tribological Behaviour of Aluminum Hybrid Nanocomposites reinforced with Alumina and Graphene Oxide

BY

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Dedication

This thesis is dedicated to my mother, beloved wife, Brothers, Sisters, and everyone who supported me.

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As a first step, I want to thank Almighty Allah for granting me good health, abilities, and perseverance to complete this research. In addition, I would like to thank my advisor and mentor, Dr. Mohammed Abdul Samad, and my committee members, Dr. Ihsan Ul-Haq Toor and Dr. Abbas Saeed Hakeem, for their encouragement and constant support throughout my thesis. I would like to acknowledge Tasnee Company for allowing me this opportunity and for providing me with the time I needed to complete my thesis. Thanks to Dr. Tahar Laoui for his assistance during the startup phase. I wish to express my sincere gratitude to my colleagues in Mechanical Engineering Department and IRC-HES for all their support and assistance throughout this research. I am truly thankful to my mother, who supported, encouraged and prayed for my success. My gratitude also extends to my wonderfully patient wife, who has been a strong supporter over the years. Also, my thanks go to all my family members for their encouragement. Finally, I would like to thank King Fahd University of Petroleum & Minerals (KFUPM) for accepting me into this program and providing me with all the facilities and equipment necessary to carry out my research. I am grateful for the financial support provided by the Deanship of Scientific Research (DSR) here at King Fahd University of Petroleum & Minerals (KFUPM) through Project No IN151009.

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LIST OF ABBREVIATIONS/NOMENCLATURE

Al	:	Aluminum
Al2O3	:	Aluminum oxide
GO	:	Graphene oxide
AHNC	:	Aluminum hybrid nanocomposite
SPS	:	Spark plasma sintering
Vol.%	:	Volume percent
T	:	Temperature.
T	:	time.
COF	:	Coefficient of friction.
GNP	:	Graphene nanoparticle.
SS	:	Stainless steel.
PCA	:	Process control agent.
Ar.	:	Argon gas.
GNS	:	Graphene nano sheet.
P/M	:	Powder metallurgy.
MMC	:	Metal matrix composite.
AMCs	:	Aluminum matrix composites.
SEM	:	Scanning electron microscope
MA	:	Mechanical alloying
XRD	:	X-ray diffraction
MPa	:	Mega pascal
Wt.%	:	Weight percent.
EDS	:	Energy dispersive spectroscopy.
Hr	:	Hour
Min	:	Minutes
Mm	:	Micrometer
Nm	:	Nanometer

HV	:	Vickers’ hardness
HRC	:	Rockwell hardness
PPM	:	Parts per million.

ABSTRACT

Name : Omar S. Al Jebreen

Title : Tribological Behavior of Aluminum Hybrid Nanocomposites Reinforced with Alumina and Graphene Oxide

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Aluminum (Al) has been the subject of a significant amount of research to improve its wear resistance by adding ceramic material such as Al_2O_3 and other fillers such as graphite, graphene, and graphene oxide. An extensive literature review shows a gap existing in the field of hybrid aluminum composite material development involving a variety of tribo-environments. Up to now, there is a lack of studies on the tribological properties of hybrid aluminum nanocomposite systems using graphene oxide reinforcements. Hence, the current study aims to explore the possibility of developing aluminum hybrid nanocomposites reinforced with alumina and graphene oxide to improve the tribological properties. In this research, spark plasma sintering technique was used to produce the hybrid nanocomposite. Wear and coefficient of friction tests were performed and analyzed using a profilometric technique to calculate the worn area and friction of the samples. Characterization techniques such as scanning electron microscopy (SEM) and EDS were used to characterize the morphology and the chemical composition of the samples and to evaluate the wear mechanisms. Samples were prepared by reinforcing aluminum with 10 vol% of alumina and varying amount (0.25, 0.5 and 1 wt%) of graphene oxide. This study revealed that Al - 10Vol%Al_2O_3 - 0.25wt% GO hybrid nanocomposite increased hardness by 48%, specific wear rate by 55%, and coefficient of friction (COF) by 5% compared with pure aluminum.

ملخص الرسالة

الاسم: عمر سعد الجبرين

عنوان الرسالة: الخصائص الترايبولوجية لهيجين الألومينيوم المدعوم بذرات اكسيد الألومنيوم متناهية الصغر وأكسيد الجرافين.

التخصص: الهندسة الميكانيكية

التاريخ: **/1443/03

أجرى كثرا من الباحثين دراسات وبحوث لتطوير خصائص التأكل لمادة الألومينوم بإضافة مواد سيراميكيه مثل بالألومينا وإضافة مواد اخرى مثل الجرافايت والجرافين واكسيد الجرافين. في هذا البحث قمنا بدراسة كثير من البحوث والدراسات ذات العلاقة ووجدنا فجوة في تطوير خصائص التأكل لماواد هيجين الألومنيوم المركب في بيئات مختلفة. حتى الأن يوجد ضعف كبير في الدراسات المعنية بتطوير خصائص التأكل لهيجين الألومينيوم المدعوم وأكسيد الجرافين.لذلك, الهدف من هذا البحث تطوير خصائص التأكل والإحتكاك لمادة هيجين الألومينيوم مدوعم بمركب الألومينا وأكسيد الجرافين. في هذا البحث تم استخدام طريقة ضغط المسحوق (التلبيد) وتقنية سبارك بلازما لصنع العينات المعنية لأجراء أختبارات التأكل. تم إختبار التأكل والإحتكاك بواسطة جهاز اختبار الـتأكل وتم حساب نسبة التأكل بواسطة تقنية البروفايلوميتر حيث يتم حساب مساحة منطقة التأكل ومن ثم يتم حساب التأكل بواسطة المعادلات الرياضية. بعد ذلك قمنا بتحليل العينات باستخدام اجهزه مخبريه ذات تقنية عالية لدراسة مورفولوجيا العينات المطورة كالماسح الضوئي التفاضلي والمتقدم،اشعة اكس وجهاز الطيف الضوئي. في هذا البحث قمنا بعمل العينات التالية, الألومينيوم, الألومينيوم المدعوم بمركب الألومينا (10%), الألومينيوم المدعوم بأكسيد الجرافين(0.25%), الألومينيوم المدعوم بمركب الألومينا (10%) وبنسب (0.25%, 0.5%, 1%) من أكسيد الجرافين. هذه الدراسة أضهرت أن مركب الألومينيوم المدعوم بمركب الألومينا (10%) وبنسب 0.25% من أكسيد الجرافين أعطت اكبر نسبة تطور في مستوى التآكل حيث قل التآكل بنسبة تفوق 55% وزادة نسبة الصلابة بنسبة تزيد عن 48% وقل الإحتكاك بنسبة 5% مقارنتا بمادة الألومينيوم.

CHAPTER 1

INTRODUCTION

Due to fast development in technology and industry, materials with properties such as higher stiffness, strength, thermal conductivity, wear resistance, and corrosion resistance coupled with lightweight are highly needed [1-5]. Research and development worldwide have started to use and develop composite materials to meet the target properties. Metal matrix nano composites (MMNCs) had the attention of scientists for the last 20 years where huge improvement in mechanical, thermal, tribological, and corrosion properties. MMNCs have found their way in automobiles and aerospace due to their excellent tribological properties. However, a few challenges such as in-homogeneity of reinforcement, porosity, and poor wettability remain. Aluminum is one of the most used materials in many fields because of its higher conductivity, lightweight, and high strength to weight ratio. In addition, many different materials can be added to aluminum (Al) to meet a wide range of applications, such as ceramic (Al_2O_3, SiC, and others additives) to increase its strength and others properties. Wear and friction are significant issues in industrial and aerospace applications leading to the damage to equipment and their failure [6]. A lot of research is being conducted on improving the wear resistance of Al, by adding fillers such as graphite, molybdenum disulfide, graphene, and graphene oxide. An extensive literature review shows a gap existing in the field of hybrid aluminum composite material development involving a variety of tribo-environments. There are only a few studies on tribological properties of hybrid aluminum composite systems using graphene oxide reinforcements. Hence, the current research explores the possibility of developing aluminum hybrid composites reinforced with alumina and graphene oxide for improved tribological properties.

1.1 Project objectives and approach

1.1.1 Objectives

This work aims to evaluate the tribological properties of aluminum hybrid nanocomposite (AHNC) for automobile and aerospace applications. The objectives of the present work are defined as the following tasks:

Task 1: Synthesize Al with 10 vol% Al_2O_3 nanoparticles and different wt% of (0.25wt%, 0.5wt%, and 1wt%) of graphene oxide, and Al with 0.25 wt% GO using spark plasma sintering technique (SPS) to obtain AHNC.

Task 2: Characterize the samples in terms of microstructure, porosity, and density.

Task 3: Sample preparation with mounting, grinding, and polishing.

Task 4: Conducting wear and friction tests

Task 5: Conducting profilometric characterization to calculate the worn area.

Task 6: Calculating the wear rate and specific wear rate of all samples.

Task 7: Conducting SEM and EDS to understand the wear mechanisms.

1.2 Research Methodology

Following is the methodology that has been adopted to achieve the objectives of the proposed research:

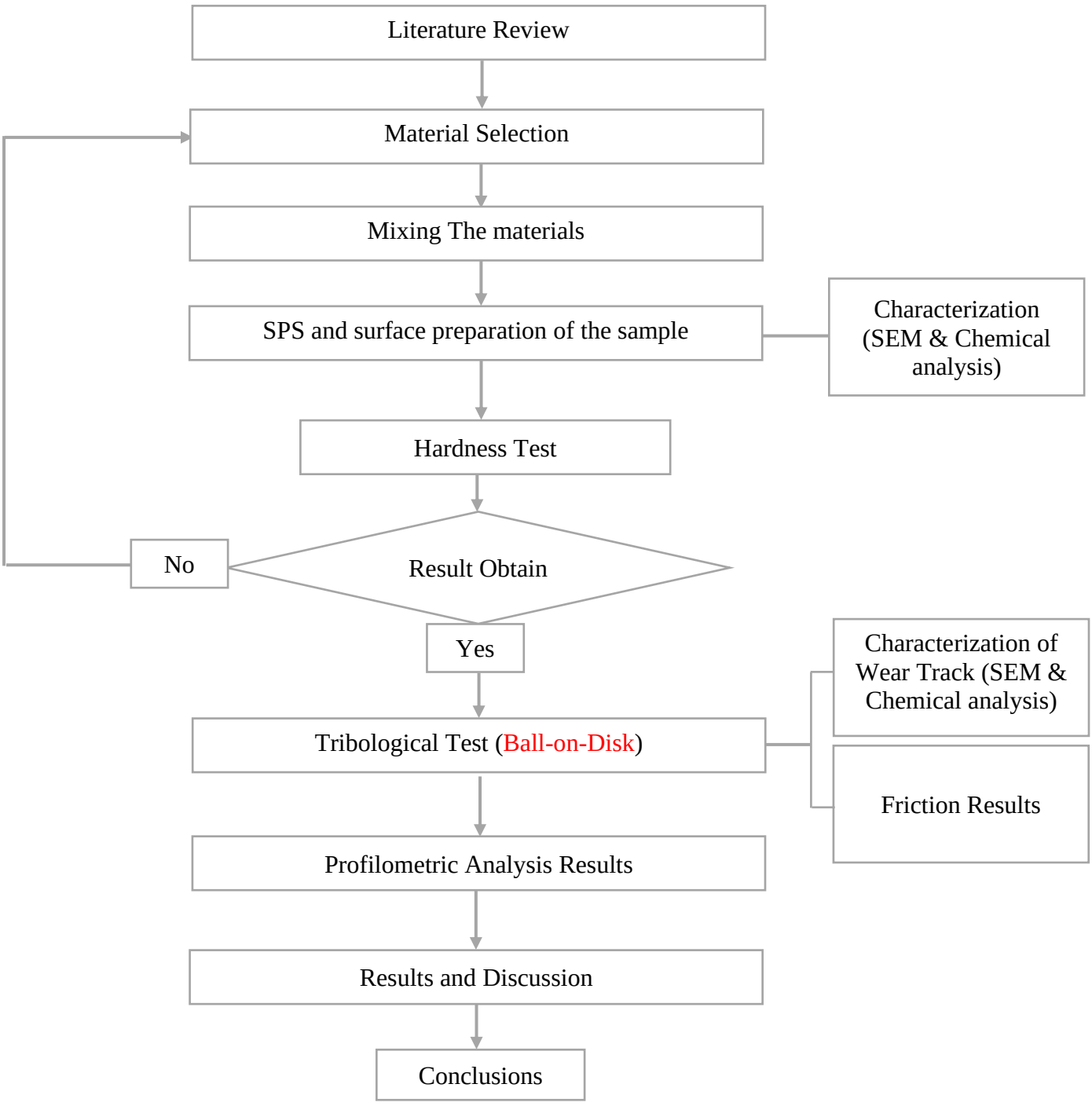

Figure 1: Flow chart showing the adopted Research Methodology.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Aluminum has been well studied over the years and is known to have excellent thermal conductivity, lightweight, and high strength to weight ratio, which can further be improved by the dispersion of nano-fillers [7]. One of the limitations of aluminum is its tendency to wear under various contact conditions, which limits its usage in demanding tribological applications such as in sliding components of machines and bearings. However, several researchers over the years have focused their efforts on improving the tribological properties of aluminum by introducing one or more additives. Adding two or more fillers often is referred to as a hybrid composite. It has been shown that in most cases, hybrid composites showed much better properties as compared to composite material. Tribological tests of hybrid composite resulted in highly improved properties, such as low coefficient of friction (COF) and low wear rate.

2.2 Importance of aluminum matrix composites:

Aluminum matrix composites (AMCs) are potential materials for different applications because of their high mechanical properties. It has been observed that introducing fillers as reinforcements into the aluminum matrix improves the hardness, strength, wear rate, and fraction coefficient compared to conventional materials. Ajit S. Dange et al. evaluated the mechanical and wear properties of aluminum–Al_2O_3 composite material. They incorporated different percentages (10, 15, and 20%) of Al_2O_3 into Al–6082 matrix. Tribological tests using the stick-on-circle method were conducted under different loads of 20, 40, and 60N. **Table 1** is the summary of the wear tests,

wherein it can be seen that increasing the alumina fraction decreased the wear rate, and increasing the load, increased the wear rate of the composite [8].

Sr.no	Material	Load N	Wear rate(μm)
1	Aluminium 6082	20	62
2		40	89
3		60	199
4	Al6082 with10% Al_2O_3	20	45
5		40	73
6		60	151
7	Al6082with15% Al_2O_3	20	35
8		40	54
9		60	110
10	Al6082with20% Al_2O_3	20	30
11		40	32
12		60	93

Table 1: Wear rate of Al 6082 reinforced with (0, 10, 15 and 20%) Al_2O_3

Abdulrahman Abed Najim et al. studied the effect of Al_2O_3 on the aluminum matrix. Al_2O_3 (5wt%, 10wt%, and 15wt%) was added to the aluminum matrix and researchers concluded that an increase in the content of Al_2O_3 up to 10wt% significantly decreased the wear rate and increased the hardness of the nanocomposites. However, exceeding 10wt%, more porosity was initiated, and it reached a point wherein further increase reduced the strength of the produced composites, as shown in Figure 2 [9].

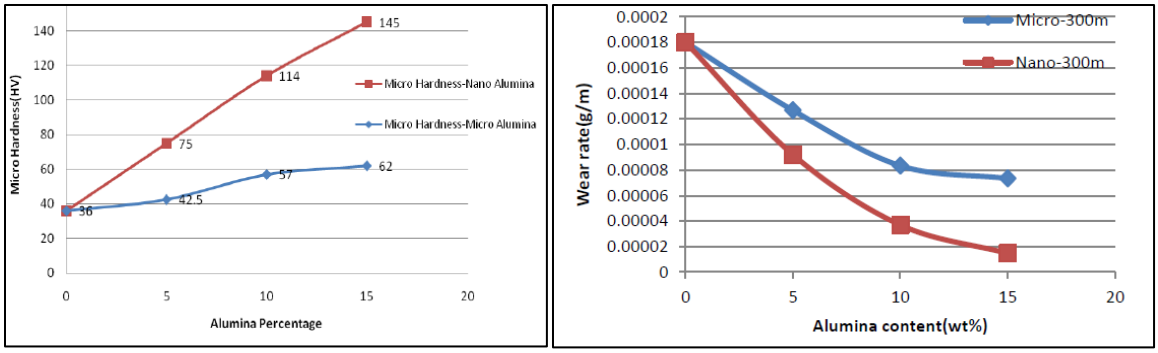

Figure 2: Hardness and Wear Rate of different Al_2O_3 content [9]

Bharath et al. prepared 6061Al composites with different fractions of alumina (0, 6, 9, and 12%) by stir casting. A pin-on-disc tribometer was used with the following parameters:

- The sliding speed is **1.256 m/s**.
- Diameter of track **80 mm**.
- The acting load is **19.62N**.
- Time is **40 minutes**.
- At **room temperature**.

They concluded that increasing the fraction of alumina led to an increase in the resistance to wear as compared to pure aluminum, wherein, 12% of alumina gives the minimum wear rate of 0.020 mg as compared to pure aluminum of 0.035 mg. Figure 3 shows the reduction in the wear rate of the composites [10].

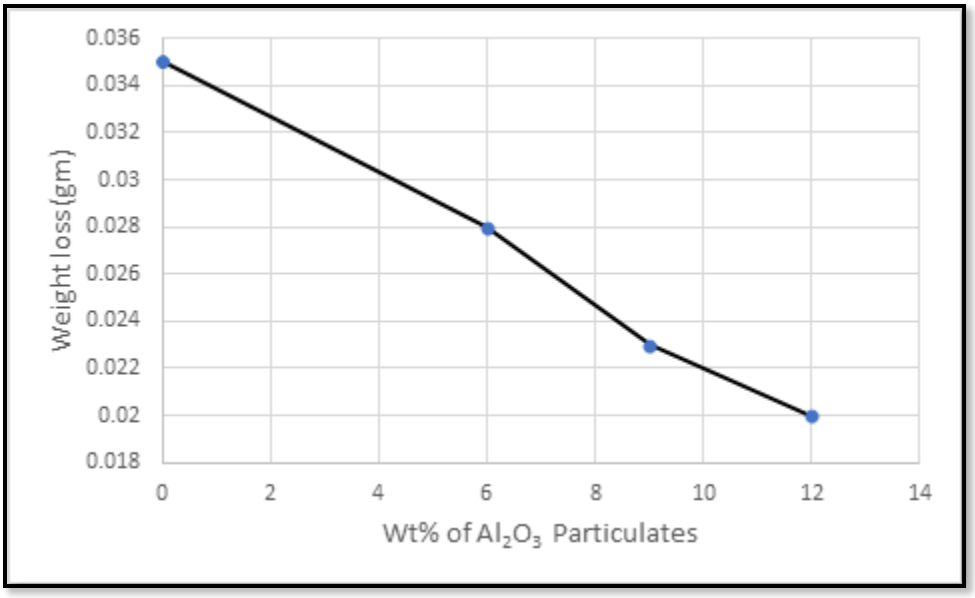

Figure 3: Wear rate of different alumina fraction into Al matrix [10].

2.3 Role of fillers

There are many types of nano-fillers that have been studied and experimented with by scientists and researchers. They can be classified into metal, ceramic, polymer, and carbon-based/noncarbon-based fillers. Throughout the following sections, we will discuss how the alumina and graphene-oxide fillers led to our choice of these materials as reinforcements.

2.3.1 Al_2O_3 (alumina) filler as reinforcement:

Aluminum matrix composites (AMC) have become one of the main composites used in the last few decades. Different processes such as ultrasonic casting, stir casting, powder metallurgy, and others motheds, have been used to develop AMC, for better properties such as higher hardness, higher corrosion resistance, low thermal expansion, higher stiffness, low coefficient of friction, and low wear rate. Al_2O_3 reinforced AMC has been highly used in the aerospace and automotive field because it has a higher strength-to-weight ratio and better tribological properties as compared to the other alloys [11]. It has been observed that the high hardness of Al_2O_3 has a major influence

to improve the wear rate [12 -13]. Rahimian et al. developed and tested Al/ Al_2O_3 composites and found that incorporating 10 wt% of alumina reduced the wear rate by 32% as the hardness increased from 46 to 62 HB with a value of 4.7 $x10^{-3}$ mm^3/m to 6.2 $x10^{-3}$ mm^3/m. [14]. Al-Qutub et al. [15] studied the effect of Al_2O_3 filler (0 to 30%) into 6061 aluminum on dry wear properties using pin on disk technique, wherein 10 Vol% Al_2O_3 reduced the wear rate by 45%. In comparison, 20 Vol% and 30 Vol% alumina reduced the wear rate by113% and 145%, respectively. Finally, he concluded that as hardness increased, wear rate decreased significantly. Similar studies were conducted by N. Hosseini K et al. [16]. S. Raju F et al. [17]. N. Radhika et al. studied the effect of incorporating different percentages of alumina of 3, 6, 9% to Al-Si10Mg - 3% graphite matrix to evaluate the wear and frictional properties [18]. Pin-on-disc test experiments used loads of 20, 30, and 30 N and different speeds of 1.5, 2.5, and 3.5 m/s showed that increasing the alumina content increased the wear resistance. This was attributed to an increase in hardness, which in turn decreases the contact area of alumina with the steel counterface, resulting in a reduced wear rate [19]. Straffelini et al. studied and showed that the hardness has a high influence on the wear behavior of Al-Al_2O_3 composites. Adding alumina to the aluminum matrix decreases the grain size and increases the dislocation [20]. Huda A. Al-Salihiet et al. studied the effect of introducing Al_2O_3 nanoparticles on the tribological behavior and mechanical properties of Al where they concluded that increasing the percentage of Al_2O_3 increased the mechanical properties of the matrix such as hardness, strength, and elongation resulting in reducing the wear rate by half as compared to pure Al [21]. However, it is to be noted that an increase in hardness will not always result in a reduced wear rate [22, 23]. Rahimian et al. observed that increasing the alumina fraction to 20 wt%, resulted in an increase in the hardness from 62 to 74 HB, but it resulted in increasing the wear rate [14]. Some researchers attributed the increase in wear rate to the increase in the porosity of the

aluminum/alumina composite [22, 24, 25]. Another explanation is that an increase in the alumina concentration leads to agglomeration of the particles, which detach from the matrix resulting in a higher wear rate of the composite. Hence, the key point is to have a good bonding with homogenous dispersion in the aluminum matrix, which will lead to better wear properties.

The size of alumina particles also influences the tribological properties of the composite. N. Hosseini K et al. [16] and Zhang et al. [26] concluded from their studies that, as the size of the alumina particles increased, the wear rate increased in 6061 aluminum alloy with 1 to 3 Vol% alumina nanocomposites. However, Radhika et al. concluded that as the particle size increased, the wear rate decreased [18]. A similar result reported was by M. Kök [27] and CAPUS [28]. The impact of Alumina Particle Size and Weight Fraction on Wear Behavior of Aluminum Metal Matrix Composites was studied and found that as the partical size decrease the wear resistance increase accordingly, Raghavendra N et al. study the effect of different size particals of alumina (0, 100 mesh, 140 mesh and 200 mesh) using A pin on disc wear test method and results confirm that the lowest particle size produces the highest wear rate [50]. Vijayakumar.S et al. studied the effect of ifferent partical size of silicon carbide, zircon and garnet. They revealved that as the partical size decrease the wear rate and coefficient of frition decrease accordingly as in figure 4.

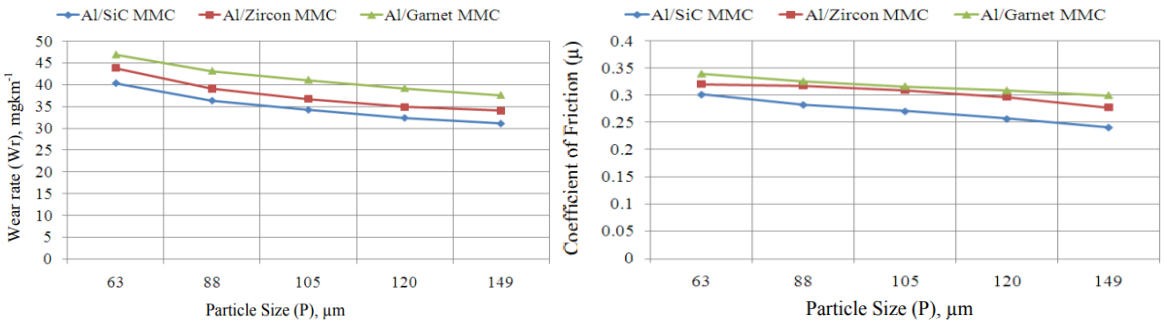

Figure 4: Effect of particle size on coefficient of friction

2.3.2 Graphene-oxide as reinforcement

Traditionally produced using the Hummer's method or its modifications [29,30], graphene and graphene oxide and reduced graphene oxide as shown in Figure 4 are known to possess exceptional properties such as high fracture toughness, mechanical properties exemplified by the high values of Young's modulus of 1 TPa and 130 GPa tensile strength, high thermal and electrical conductivities [31,33]. The wear phenomena in graphene are to break the plane bonds between carbon atoms and shearing at the interface of graphene layers. The friction on the graphene surface is proposed to be due to Van der Waals interactions. The flat carbon atoms in graphene, which can be seen in Figure 5, can flex relatively without breaking apart, which in turn gives it a high stiffness. Taylor et al. stated that increasing the oxide agents (O-) and (OH-) will lead to an increase in the bond length at each hexagonal lattice [51].

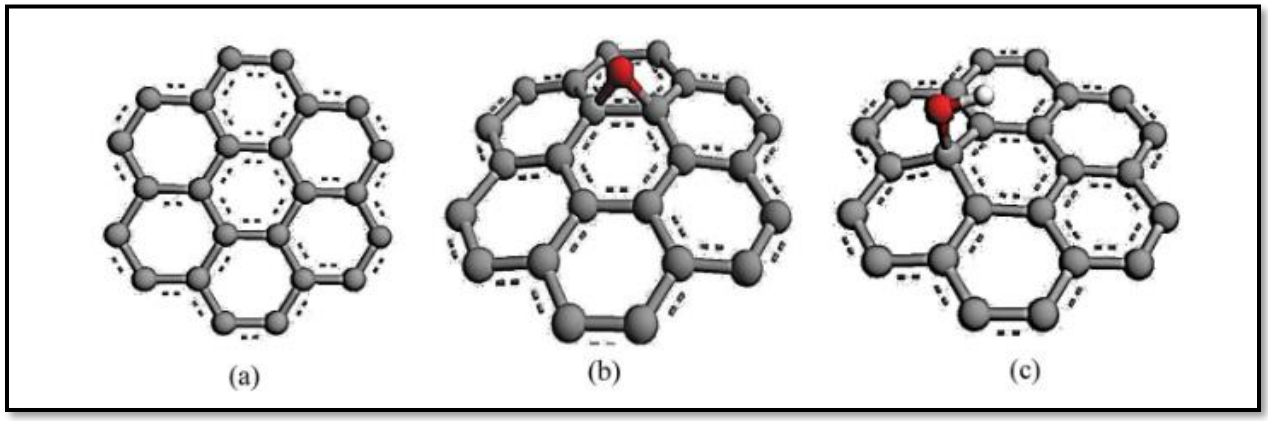

Figure 5: Schematic representation of the graphene sheet: (a) pristine graphene; (b) graphene with the oxide agent (–O) and (c) graphene with the oxideagent (–OH) [51].

Aswin. S et al. study the effect of rGO with different friction (0.05wt%, 0.5wt%, 1wt%, 2wt%, 5wt%) on the wear rate. Wear rates decline up to a maximum concentration of 0.5 %, then increase sharply with every subsequent concentration, as shown in Figure 6. This may be caused by the agglomeration of rGO in the material, causing insufficient strength. [34].

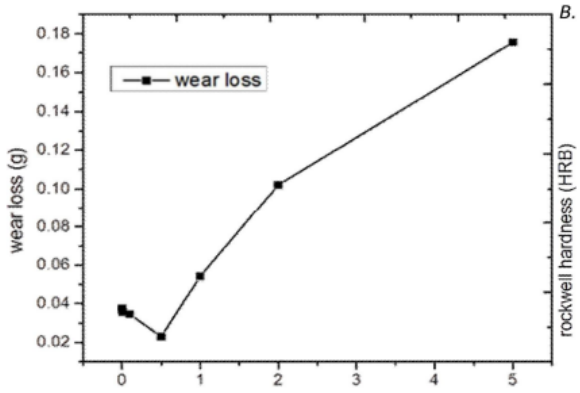

Figure 6: Wear loss plot against rGO concentration for the selected sample. [34].

A. M. Sadouna et al. studied the effect of introducing graphene into the cupper/ Al_2O_3 composite wherein they claim that the hardness of hybrid nanocomposites containing 1.2% of graphene nanoplatelets (GNPs) increased by 30 % as compared to pure copper and the wear rate significantly decreased. The coefficient of friction (COF) decreased with increasing graphene filler because of the lubrication effect provided by graphene [35]. Zeng X [36] studied the effect of incorporating 0.3 to 0.7 wt% of reduced graphene oxide (rGO) into Al-Si matrix with 10 wt% silicon carbide, fabricated using the PM method. Wear tests conducted using the ball-on-desk method, showed that incorporating 0.5 wt% rGO reduced the wear rate by 65%. El-Ghazaly A et al. [37], investigated the addition of 3 to 5 wt% of graphene into aluminum (AA2124) and observed an improvement in the hardness by 133 and 102%, a reduction in the wear rate by 34 and 25% and COF reduced by 25 and 9%, respectively.

2.3.3 Al_2O_3 (alumina)-Graphene Oxide hybrid:

Many hybrid materials based on aluminum/alumina have been investigated for mechanical and tribological properties. Radhika et al. [38] reported that graphite as a second reinforcement to aluminum/alumina matrix improved the wear resistance and lowered the friction. Of the many reinforcements, graphite is a popular material since it acts as a solid lubricant and reduces the wear of the composite. Radhika et al. [39] analyzed the same hybrid and reported some positive results in terms of tribological properties. The analysis concluded that adding alumina and graphite to aluminum, increased the wear resistance and decreased the coefficient of friction. Seah et al. [40] stated that adding graphite from 1% up to 3% into the AMC decreased the wear rate. Dharmalingam et al. [41] reported that introducing molybdenum disulfide as a solid lubricant contributed to decreasing the wear rate. However, the effect of introducing graphene oxide into the aluminum/alumina matrix is still not clear, which is the main focus of the current study.

Fathy et al. [42] studied the effect of adding different volume fractions (0 wt%, 0.2 wt %, 0.6 wt % and 1.4 wt%) of GNPs. Compared to pure aluminum, nanocomposite Al-10%Al_2O_3/1.4%GNPs achieves 2.45-fold hardness improvement and 19.2-fold reduction in wear rate.

2.3.4 Summary

Aluminum is one of the most used materials due to its mechanical properties and low strength to weight ratio. Tribological properties of aluminum are limited, which motivated researchers to develop and improve its properties by incorporating ceramic materials such as alumina and others, or/and carbon-based materials such as graphene and graphene oxide or adding both to make hybrid composites. Researchers examined the effects of adding different fractions of alumina to aluminum matrix under varying conditions and evaluated wear and friction properties, concluding that the addition of certain fraction alumina to aluminum matrix will improve the mechanical and tribological properties. Furthermore, researchers have performed several studies and experiments with hybrid nanocomposites such as Al/Al_2O_3/graphene or graphite will provide significantly better properties than a composite where graphene and graphite act as solid lubricants and improve the wear properties. Lastly, no research has been conducted on the tribological properties of aluminum hybrid nanocomposite using alumina and graphene oxide which presents us with a unique opportunity to explore this hybrid nanocomposite further.

2.3.5 Significance of the study

From the extensive literature review presented above, it was observed that limited studies were carried out for evaluating the tribological properties of aluminum hybrid nanocomposites. Up to now, there are just a few studies conducted for aluminum hybrid using aluminum and graphene oxide. Therefore, in this study, we focus on the development of aluminum hybrid nanocomposite reinforced with alumina/graphene oxide and evaluating its tribological properties. Several wear and friction tests were conducted to evaluate the tribological performance of the hybrid material.

CHAPTER 3

EXPERIMENT PROCEDURES

3.1 Material

Aluminum powder from Alpha chemical company, Dartmouth, Canada, with a purity of 99.5% and an average particle size of 30 μm used as the matrix material. Aluminum oxide (Al_2O_3) with a purity of 99.88%, 300 nm particle size, and surface area of 85 - 115 m^2/g from union carbide corporation for Buehler Ltd, USA were used as reinforcement. Graphene oxide from AD Nano Company, Shimoga, India with 99% and surface area of 250 m^2/g. Table.2 showed the XRF analysis of the materials used.

Table 2: Aluminum, Alumina and Graphene oxide powder specification

Material	**Content & wt%**	**Particle size**
Aluminum Powder	Aluminum >99.5	30 microns
	Silicon<0.25	
	Iron<0.15	
	Titanium <0.25	
Alumina	Alumina 99.88	300 nanometers
	Silica 0.034	
	Phosphorus oxide 0.0085	
	Sulfur 0.026	
	Potassium oxide 0.027	
	Titanium dioxide 0.0022	
	Vanadium pentoxide 0.0041	
	Manganese oxide 0.0016	
Graphene oxide	Carbon 77	1-2 layers nano
	Oxygen 22	
	Others 1	

3.2 Fabrication process of aluminum nanocomposite and aluminum hybrid nanocomposite

3.2.1 Ultrasonication and ball milling

Sonication was carried out using a probe sonicator (Sonics VCX 750, Newtown, CT, USA), as shown in Figure 7. To prevent agglomeration, 10% of alumina and GO were sonicated in ethanol for 1 hour separately, at room temperature, with cycles (on: 20 sec/Off: 5 sec). Afterward, the sonicated and dried additives were blended with aluminum powder in the vials for milling.

Figure 7: Sonication process device from Sonics VCX 750.

Time(hr)	Amplitude (%)	Temperature(ºC)	On: Off cycles (Sec)
1	35	Room Temperature	20: 5

Table 3: Process parameters of Sonication

Stainless steel ball milling vial was used (high energy ball mix, model: HD/HDDM/01, Union process, Inc. USA) as shown in figure 8.

Figure 8: High energy ball mix from Union process

To obtain homogeneous mixing, zirconium oxide balls (5 mm) with a ratio of (10:1) were used in conjunction with Al+ 10 vol%Al2O3, the composite was mixed for 24 hours at 200 rpm at room temperature under argon gas to prevent oxidation. The composite was then dried for 12 hours at 80 °C. Graphene oxide was added to the composite in different concentrations of 0.25, 0.5, and 1 wt% and mixed for 24 hours at the same parameter. The mixture was dried for 12 hr. at 80 °C. Table 4 lists the parameters used in the process.

Sample	Medium	Mixer speed (rpm)	Time (Sec)	Atmosphere
Al	ethanol	200	24	Argon
Al+ Al_2O_3	ethanol	200	24	Argon
Al+0.25%GO	ethanol	200	24	Argon
Al+ Al_2O_3+0.25%GO	ethanol	200	48	Argon
Al+ Al_2O_3+0.5%GO	ethanol	200	48	Argon
Al+ Al_2O_3+1%GO	ethanol	200	48	Argon

Table 4: Summary of milling process parameters

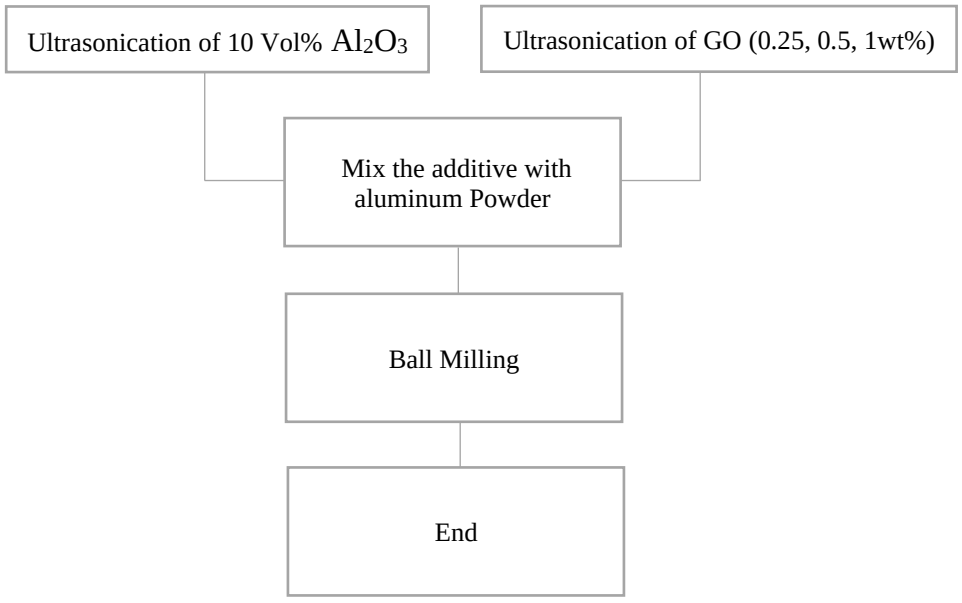

Figure 9: Powder mixing and ball milling plan

3.2.2 Spark plasma sintering (SPS)

The powder samples (Al, Al+ x vol% Al2O3 + x wt% GO) were sintered using a spark plasma sintering machine (FCT group, SPS GmBH Germany system), as shown in figure 10. Samples were filled into a graphite die with a 20 mm diameter and a 0.30 mm graphite sheet was placed between the powder and die to allow easy removal. Sample powders were pressed into the graphite mold at 550 °C under the pressure of 50 Mpa for 10 minutes with a heating rate of 200 °C per minute. The summary of parameters is outlined in Table 5.

Figure 10: Spark plasma Sintering machine from FCT group

Die Material	graphite
Die diameter	20mm
temperature	550 ºC
Heating rate	200 ºC/minutes
Holding time (t)	10 minutes
Pressure (Pr.)	50 Mpa

Table 5: Process parameter of Spark plasma sintering

3.2.3 Density Measurement

The density of sintered samples can be measured by immersing the sample in deionized water according to Archimedes' principles and the rule of mixture. The density of solid ρ can be calculated as follows:

$$\rho = \frac{W\ air}{Wair - W\ liquid}(\rho 0 - \rho L) + \rho L$$

Where ρ is the Density of the sample, W_{air} is the weight of the sample in air, W_{liquid} is the weight of the sample in the auxiliary liquid, ρ_0 is the density of the auxiliary liquid and ρ_L is the density of air. (kern ABT weighing scale, 320g capacity, UK) was used as shown in figure 11.

Figure 11: Kern ABT weighing scale, 320g capacity, UK

3.2.4 Mounting

The SPS samples were mounted in a 25-millimeter diameter mold using a hot mounting press (Qualitest, hot mounting press, USA). Mounting produces specimens of uniform size so that they can be handled in automated holders for further preparation steps (figure 12).

Figure 12: Hot mounting press from Qualitest .

3.2.5 Grinding and ultrasonic cleaning

The SPS samples were ground using progressively finer grit papers (240, 320, 400, 600, 800, 1200) and then polished with 0.3 μm alumina paste, as shown in figure 13.

Figure 13: Grinding and polishing machine.

The samples were soaked in ethanol for 15 minutes after polishing to remove the debris remaining on the surface after grinding and polishing.

3.3 Hardness test

Hardness was measured with hardness tester (Zwick Roell, Germany) as in figure 14, where 500 gf were used with diamond indents of 136 degrees between opposite faces in the form of a right pyramid with a square base, with an average of five test spots for each sample, and the Vickers number calculated using the following equation:

$$HV = 1.854(P/A^2)$$

Where HV is Vickers hardness, P is the load acting in kilograms, and A is the indentation area in millimeters.

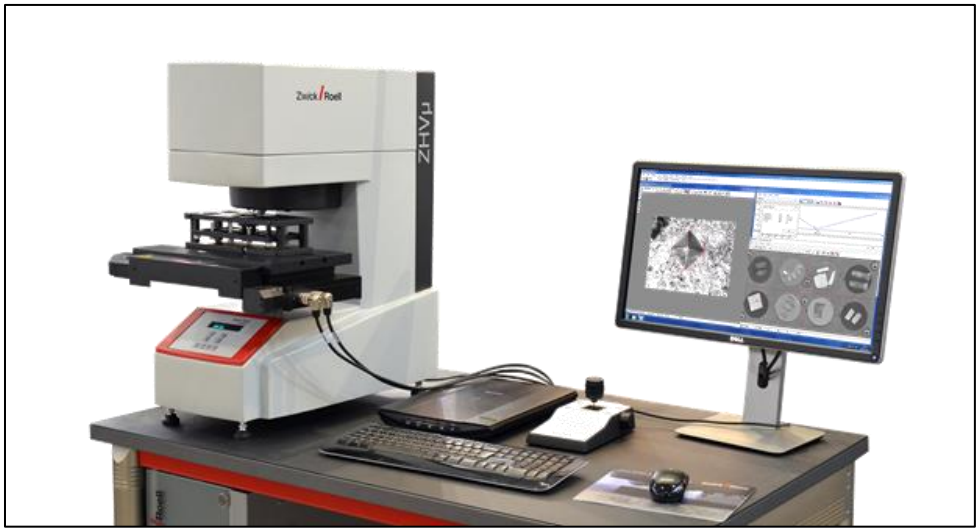

Figure 14: Zwick hardness tester.

3.4 Tribological test

Using ball-on-disk equipment from Bruker (UMT Tribolab), Germany as shown in figure 15, dry sliding wear tests were performed on six different samples. The samples (disk) with 20 mm in diameter and 7 mm in height. A ball (counter face) composed of 440C stainless steel with 6.3 mm diameter and 63 HRC hardness was used to slide against the samples. Friction coefficients were measured for each sample.

Figure 15: Tribotest equipment from Bruker (UMT Tribolab).

3.5 Profilometry analysis

To analyze and measure specific wear rates, we used an instrument from Bruker, Germany (ContourGT) to determine the volume loss of the sample, shown in Figure 16. We measured the average areas of 4 different spots over each sample to calculate the wear volume loss:

$$V = 3\pi(A)$$

Where V is the volume loss and A is the track area calculated by the profilometer apparatus. To determine the wear rate, the volume loss was divided by the sliding distance using the following equation:

$$w = V/d$$

Where w is the wear rate, V is the volume loss and d is the sliding distance of the tribotests. Specific wear rate is then calculated using the following equation:

$$Ws = w/P$$

Where Ws is the specific wear rate, w is the wear rate and P is the load acting.

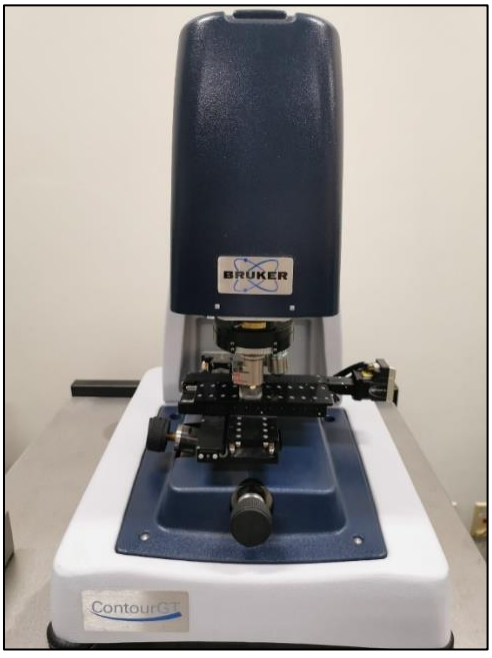

Figure 16: Profilometer equipment from Bruker, Germany (ContourGT).

3.6 SEM and EDS analysis

With a Quanta FEG 250 scanning electron microscope from Thermo Fisher, USA, and with a Quanta FEG 250 energy dispersive X-ray spectroscopy detector, SEM imaging was performed on the received powders, hybrid composite powders, and the tested SPS samples. SEM is an essential technique for analyzing the morphology, topography, and composition of objects. The characterization results were obtained from the generation of high-energy focus electrons of secondary and backscattered electron. All samples were placed in a vacuum at a distance of 6 millimeters and using a voltage of 20 kilovolts. EDX spectroscopy detector was used to characterize the elemental composites. Figure 17 shows the SEM used.

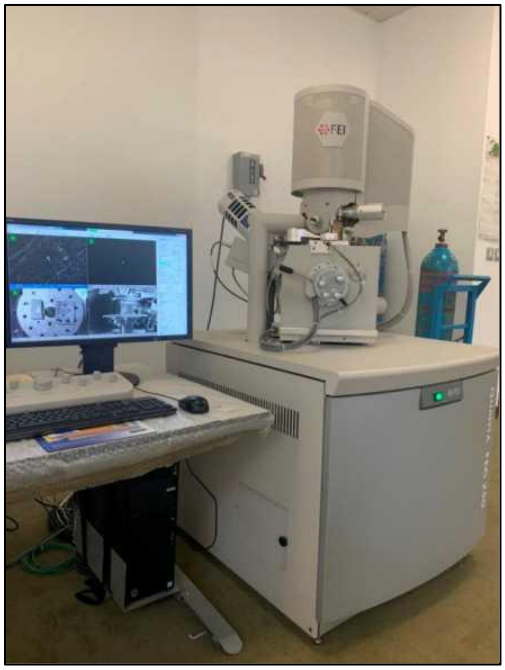

Figure 17: SEM used to characterize the samples from Quanta FEG 250, Thermo Fisher Scientific, USA.

3.7 X-ray diffractometer

X-ray diffractometer is an important tool for analyzing and identifying the phases of crystalline materials. For our study, we used a Rigaku Miniflex X-ray diffractometer as in figure 18. Cupper Kα radiation was used (ʎ = 1.5406 Å) with 2θ range at a scanning speed of 2˚/min and acceleration voltage of 30kV. X-rays are high energy light with a periodic wavelength ʎ, and produce constructive interference when conditions are fulfilled by Bragg's Law:

$$2d \sin \theta = n\lambda$$

where d is the spacing between diffracting planes, θ is the incident angle, n is an integer, and λ is the beam wavelength.

Figure 18: Rigaku miniflex X-ray diffractometer.

C HAPTER 4
RESULTS AND DISCUSSIONS

4.1 Formulation:

The aluminum hybrid nanocomposite has been optimized previously [49], in that Al_2O_3 content has been enhanced in the Al matrix by means of density and hardness. In contrast, the GO content has been enhanced by means of mechanical and thermal properties. The objective of this project is to determine its tribological properties. Table 6 shows the formulation developed.

Formulation No.	**Formulation**
Formulation 1	Al
Formulation 2	Al + 10 Vol% Al_2O_3
Formulation 3	Al + 10 Vol% Al_2O_3+ 0.25 Wt% GO
Formulation 4	Al + 10 Vol% Al_2O_3+ 0.5 Wt% GO
Formulation 5	Al + 10 Vol% Al_2O_3+ 1 Wt% GO
Formulation 6	Al + 0.25 Wt% GO

Table 6: Formulation of the aluminum hybrid.

4.2 Characterization

4.2.1 Scanning Electron Microscopy and XRD of received samples

The morphology of pure aluminum, Al_2O_3, and GO were characterized by high magnification images using scanning electron microscopy. As shown in figure 19(a) pure aluminum with a spherical particle shape with about 30 μm average size. It can be observed from figure 19(b) alumina particles with an average size of 300 nm. Whereas Figure 19(c) shows, graphene oxide is in flake form, with single-layered morphology in which the layers are stacked upon each other. XRD characterization was conducted using a Rigaku Miniflex X-ray diffractometer. Cupper Kα radiation was used (ʎ = 1.5406 Å) with 2θ range at a scanning speed of 2°/min. As shown in figure 19(d) aluminum diffraction pattern as received powder and in figure 19(e) alumina and graphene oxide as in figure 19(f).

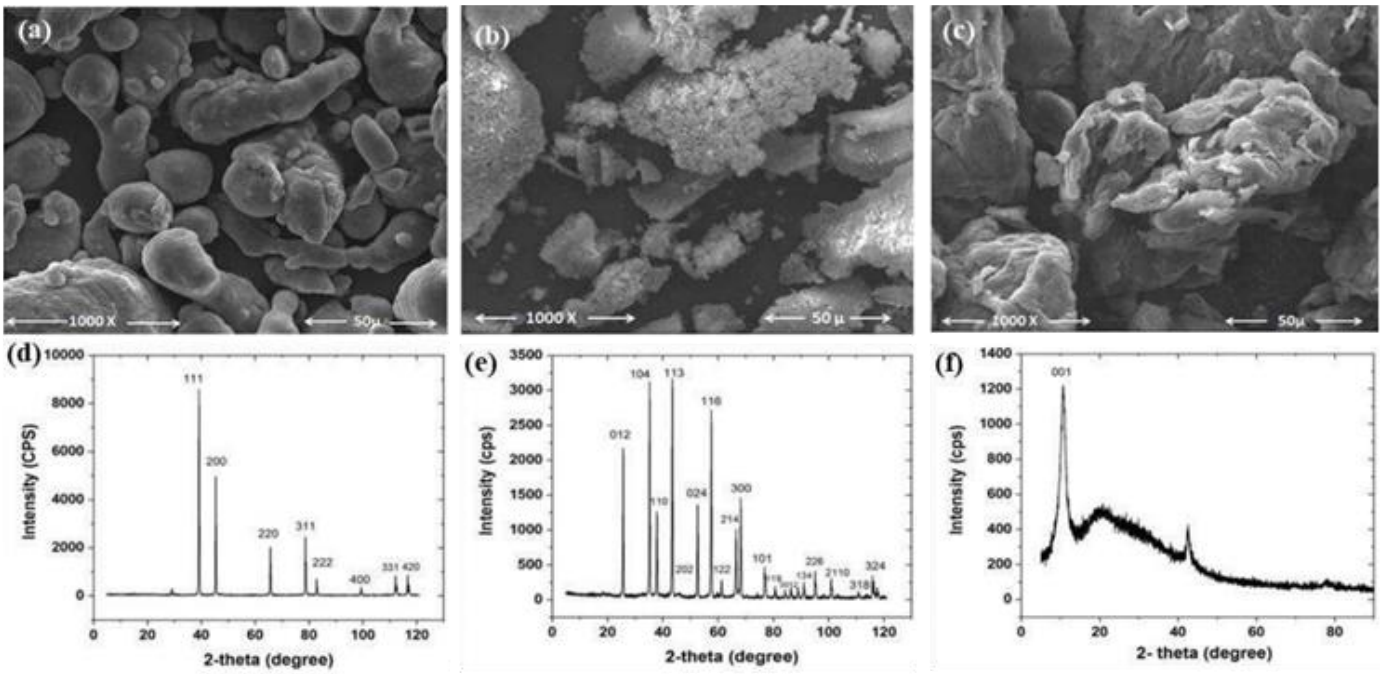

Figure 19: SEM image of Al (a), Al_2O_3(b), graphene oxide (c). XRD (d) Al, (e) Al_2O_3(f), graphene oxide.

4.2.2 Scanning electron microscopy and XRD of nanocomposite samples

SEM and XRD were conducted for aluminum hybrid nanocomposites (Al-10Vol% Al_2O_3 +0.25wt%, Al-10Vol% Al_2O_3 +0.5wt%, and Al-10Vol% Al_2O_3 + 1wt%), where a clear image of the nanocomposite morphology. SEM images of Al-10Vol% Al_2O_3 +0.25wt% as in figure 20(a) show the greater distribution of graphene oxide along the grain boundaries, where alumina is distributed in the grains. The same distribution of GO and alumina are in Al+10Vol% Al_2O_3 +0.5wt%, and Al+10Vol% Al_2O_3 + 1wt% as in figure 20(b, c). XRD has been conducted for all hybrid nanocomposite SPS samples as in figure 20(d) where Al+ Al_2O_3 has a higher intensity for Al and Al_2O_3 as compared to the other hybrid materials. one reason is the ball milling time will affect the distribution of Almina and graphene oxide into the aluminum matrix and homogeneity of the nanocomposite wherein Al+ Al_2O_3 nanocomposite were ball milled for 24 hours whereas 48 h ball milling time of the hybrid nanocomposite. Due to the low fraction percentage of graphene oxide, the GO phase cannot be observed. Moreover, it was observed that graphene oxide and alumina have no chemical reaction as there is no new phase.

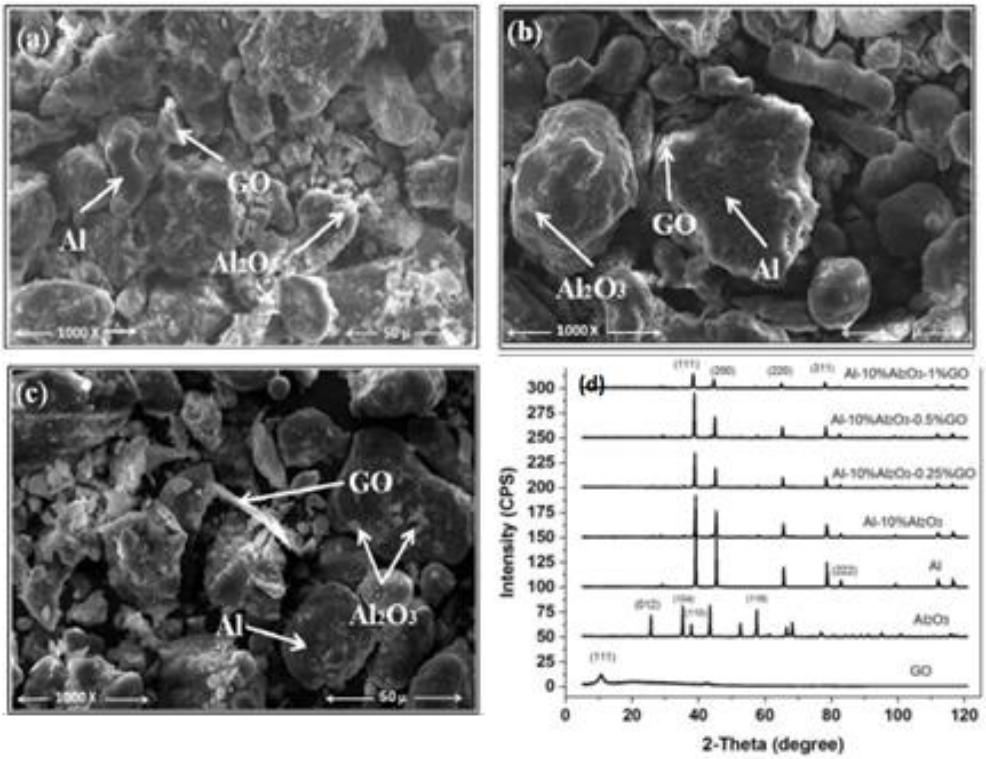

Figure 20: SEM image of (a) Al+10Vol% Al_2O_3 +0.25wt%, (b) Al+10Vol% Al_2O_3 +0.5wt%, (c) Al+10Vol% Al_2O_3 + 1wt%, (d) XRD of All Samples.

4.3 Densification:

Densification measurements of received samples (Al, Al+ Al_2O_3, Al+0.25%GO, Al+ 10 Vol% Al_2O_3 + 0.25/0.5/1wt%GO) from sintering were carried out using the Archimedes' principle. Table 7 showed that pure aluminum has higher densification of about 99.8 %, whereas incorporating a 10-Vol percentage of alumina into aluminum reduced the density to 99.5%. Introducing the graphene oxide with a fraction of 0.25, 0.5, and 1 wt% decreased the density to 98.9, 98.6, and 97.43%, respectively. That reduction in the relative density attributed to graphene oxide tends to allocate along the grain boundaries, as shown in the SEM image figure 20(b), which obstructs the densification process and results in bigger porosity with a higher content of graphene oxide.

No.	Sample	Densification			Average Densification
		T1	T2	T3	
1	Al	99.79	99.81	99.81	99.8
2	Al+ Al_2O_3	99.48	99.50	99.52	99.5
3	Al+ Al_2O_3+0.25%GO	98.96	98.97	98.975	98.97
4	Al+ Al_2O_3+0.5%GO	98.59	98.63	98.63	98.62
5	Al+ Al_2O_3+1%GO	97.40	97.44	97.46	97.43
6	Al+0.25%GO	99.20	99.22	99.23	99.22

Table 7: Densification result of all samples.

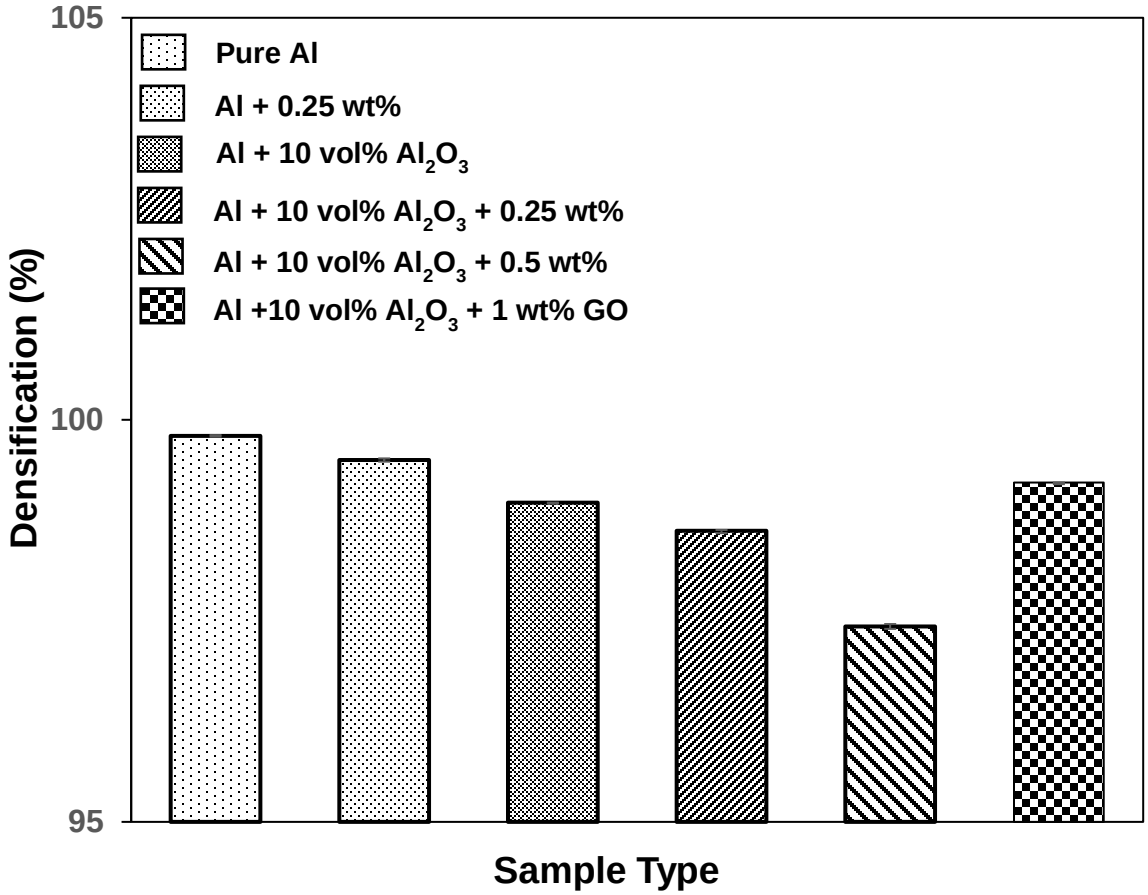

Figure 21: Densification result of all samples.

4.4 Hardness Measurement:

As shown in figure 22 and Table 8, the hardness of samples. Al hardness value was the lowest with a value of 32.8 HV. The addition of alumina with 10Vol% increased the hardness by 70%, with 55.8HV, which is due to the particles of alumina in the aluminum matrix impeding movement or dislocation during the hardness test. With 0.25 wt% GO added, the hardness increased because the two fillers dispersed uniformly in the aluminum matrix without agglomerating. Hardness value increased approximately two times over pure aluminum, with a high value of 63.56 HV. Additional evidence from SEM images has been obtained. Analysis of the microstructure and grain size of Al+10 Vol% Al_2O_3+0.25 wt% GO showed the grain size was very fine which led to an increase in the hardness. The increase in GO content decreased the hardness of the hybrid composites, which was attributed to the formation of agglomerates and the lower densification of the samples. Where adding more than 0.25wt% of GO reduces the hardness of the hybrid nanocomposite as in Al + 10 Vol% Al_2O_3+ 0.5 wt% GO and Al + 10 Vol% Al_2O_3 + 0.25 wt% GO with the value of 56.9 and 56 HV respectively. As a result of adding 0.25 wt% GO, a marginal increase in hardness was seen compared to Al_2O_3 nanoparticles. The resulting hardness improvement of the hybrid nanocomposite is mainly due to the Al_2O_3 reinforcement.

Sample	Test 1	Test 2	Test 3	Avg
Pure Al	35.4	31	32	32.8
Al + 10 Vol% Al_2O_3	54.4	57.1	56	55.83
Al + 10 Vol% Al_2O_3 + 0.25 wt% GO	67.7	64.2	58.8	63.56
Al + 10 Vol% Al_2O_3 + 0.5 wt% GO	60.2	58	52.7	56.96
Al + 10 Vol% Al_2O_3 + 1 wt% GO	59.2	52.8	56	56
Al+0.25wt%GO	37.8	35.8	33.7	35.76

Table 8: Hardness results of all samples

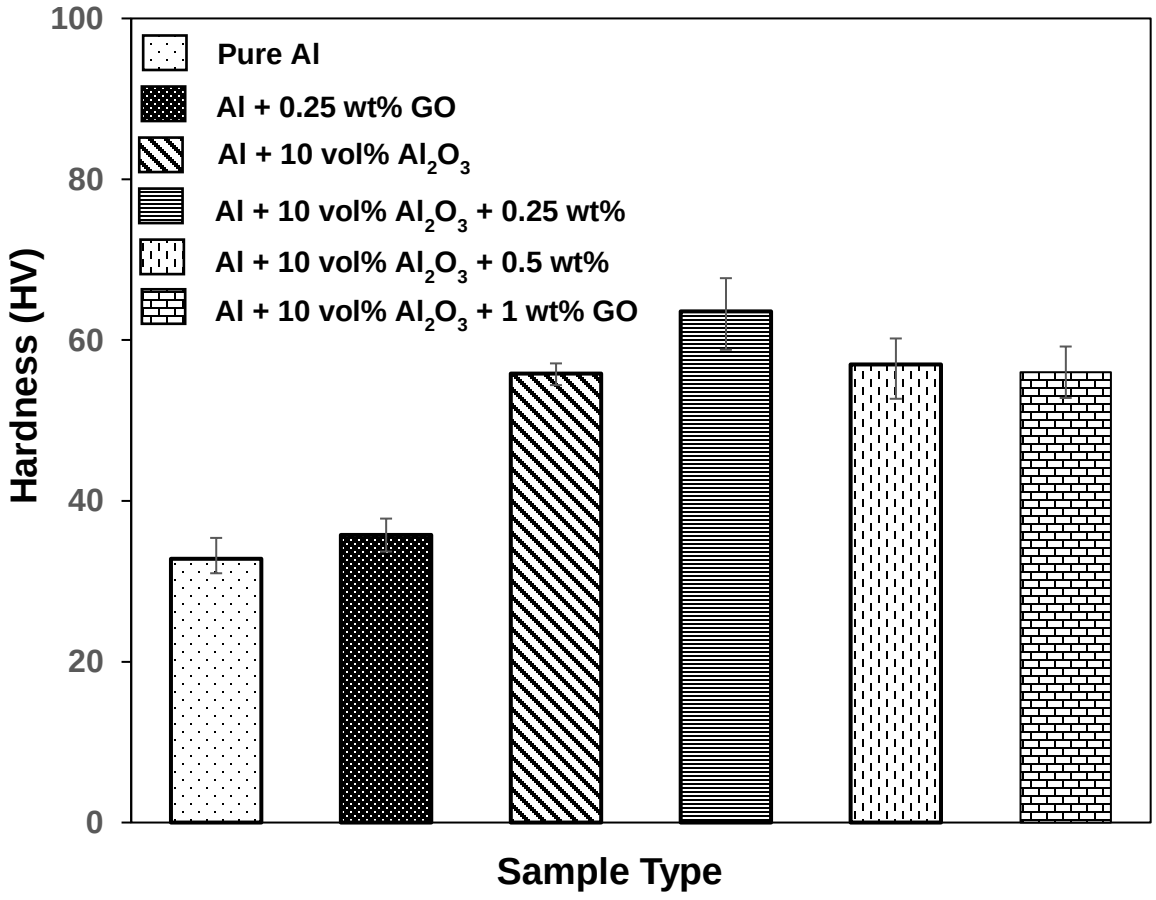

Figure 22: Hardness results of all samples

4.5 Tribological Experiments of received SPS Sample

4.5.1 Wear test

SPS polished samples were tested using a ball-in-desk method where the desk is the sample with 20 mm diameter and 7 mm length, and the counterface is the ball made from 440C stainless steel with a diameter of 6.3 mm and hardness of 62 RC. The tests were conducted with a speed of 0.1 m/s, load 3 N, 500 cycles, 3 mm wear track diameter and 100-meter distance. The wear volume V is calculated using the formula:

$$V = 3\pi * A \qquad mm^3$$

Where A is the area of the wear calculated using the profilometer. Then, the specific wear rate Ws was calculated for all samples using the formula

$$W_s = \frac{V}{D * L} \qquad mm^3/Nm$$

Where D is the sliding distance and L is the load applied to the sample. In Figure 22, the 3D optical profilometer is shown displaying several typical 2D profiles for each tested sample. Each sample's depth and width are depicted in Figure 24. In hybrid nanocomposite Al + 10 Vol% Al_2O_3+ 0.25 wt% GO, the wear track width and wear track depth were the lowest with results of 0.73 mm and 80 µm, respectively. The specific wear rate for aluminum composite materials (Al-0.25 wt% GO and Al+10 Vol% Al_2O_3) decreased upon comparison with pure aluminum, with Al+10Vol% Al_2O_3 achieving a higher reduction than Al+0.25 wt% GO. The reason is that, as shown in Figure 22, Al+10 Vol% Al_2O_3 composite has higher hardness than Al+0.25 Vol% GO composite. The specific wear rate of hybrid nanocomposites was reduced further when 0.25 wt% graphene oxide (GO) was added to Al+10 Vol% Al_2O_3, because of the improved hardness of the hybrid

nanocomposite (Figure 22), largely because of the hard alumina nanoparticles and the lubricious nature of GO. Hybrid composite (Al + 10% Al_2O_3 + 0.25 wt% GO) has a specific wear rate of 251 X 10^{-6} mm^3/Nm, which is a 55% reduction compared to pure aluminum. Also, a uniform distribution of both reinforcements within the aluminum matrix was observed in the hybrid nanocomposite Al-10 Vol% Al_2O_3+0.25 wt% GO, which increased wear resistance. The Specific wear rate of hybrid nanocomposites increased significantly as the content of GO increased (0.5 and 1wt%). This could be explained by the reduction in hardness of samples as in Figure 22, the decrease in density as Figure 21, and the agglomeration form between the reinforcements within the aluminum matrix.

Samples	Area (mm^2)	Wear Volume (mm^3)	Specific Wear rate (mm^3/Nm)
Pure Al	0.0180	0.169	566 x 10^{-6}
Al + 10 Vol% Al_2O_3	0.0096	0.091	303 x 10^{-6}
Al + 10 Vol% Al_2O_3+ 0.25 wt% GO	0.008	0.075	251 x 10^{-6}
Al + 10 Vol% Al_2O_3 + 0.5 wt% GO	0.027	0.251	837 x 10^{-6}
Al + 10 Vol% Al_2O_3 + 1 wt% GO	0.061	0.576	191 x 10^{-5}
Al +0.25wt%GO	0.014	0.132	439 x 10^{-6}

Table 9: Wear area, volume and specific wear rate of all samples

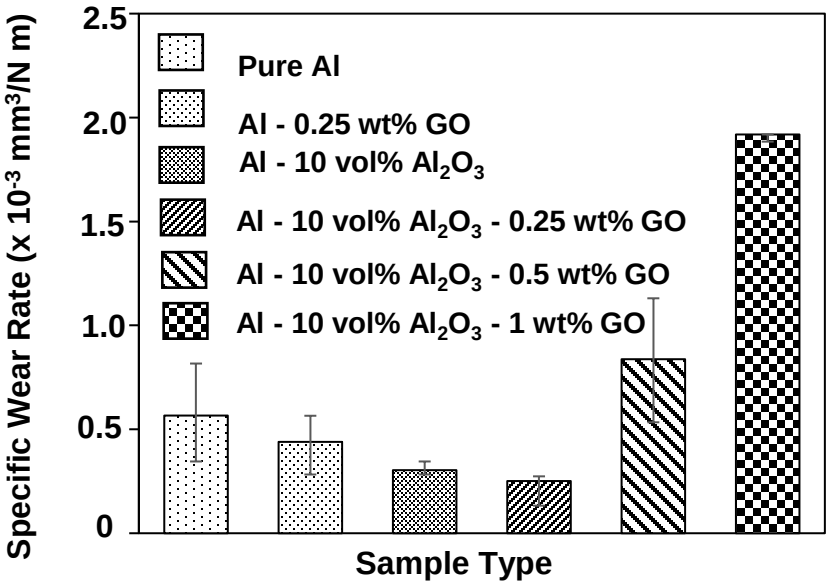

Figure 23: Specific wear rate of all samples

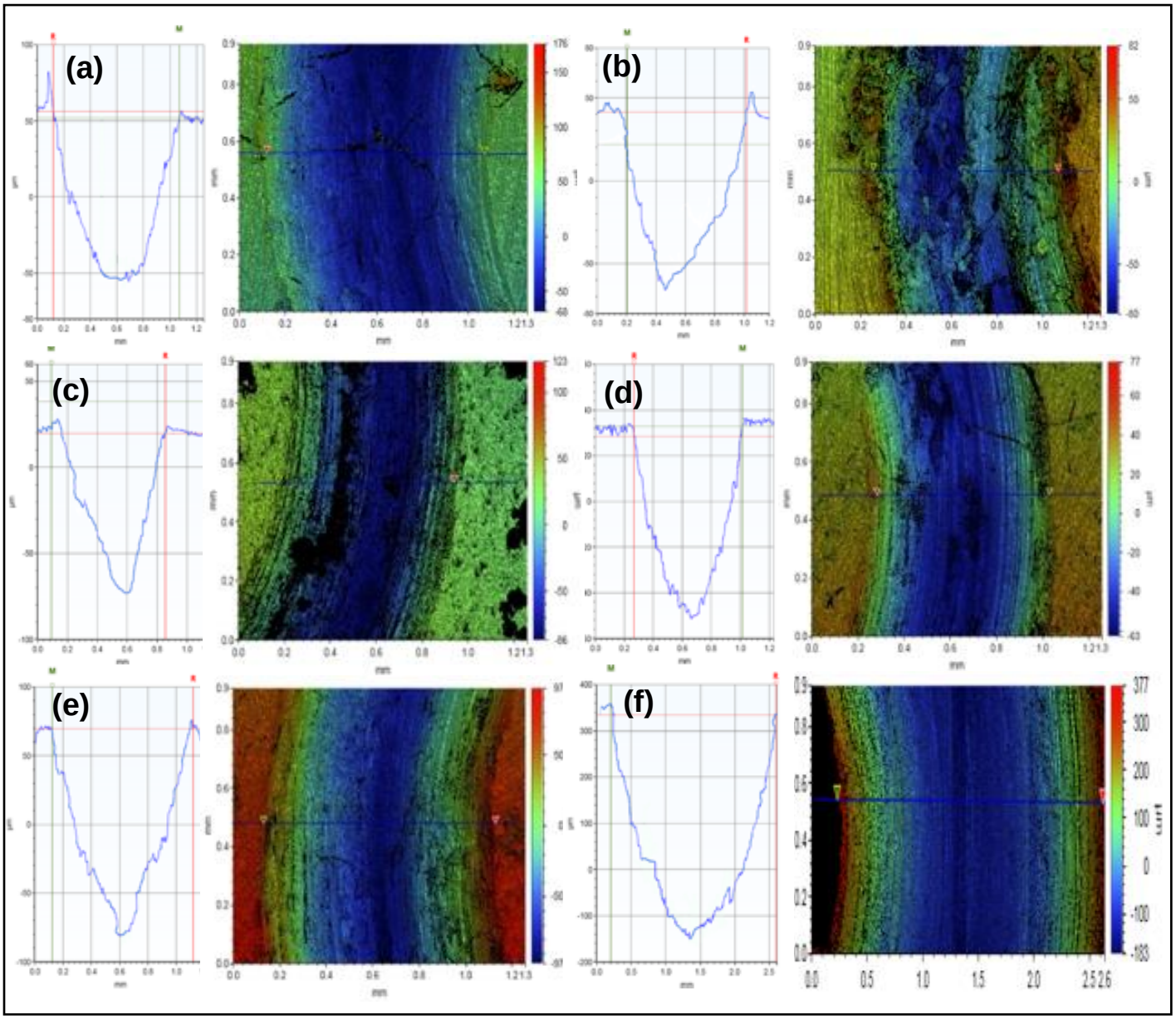

Figure 24: Profilometric analysis of a) Pure Al, b) Al+0.25wt%GO, c) Al + 10 Vol% Al_2O_3, d) Al + 10 Vol% Al_2O_3 + 0.25 wt% GO, e) Al + 10 Vol% Al_2O_3 + 0.5 wt% GO, f) Al + 10 Vol% Al_2O_3+ 1 wt% GO

4.5.2 Coefficient of Friction

As shown in Figures (25-26) and Table 10, typical frictional graphs demonstrate the wear test results and the average coefficient of friction (COF) of all samples tested under a 100 m sliding distance, with 3 N of load and 0.1 m/s of sliding speed. Figure (23-24) illustrates that the COF for the Al+10 Vol% Al_2O_3 nanocomposites was slightly higher than pure Al, owing to the hard alumina nanoparticles present. The friction value depends on the hardness and materials of the sample and counterface ball. Figure 25 shows that the COF was slightly higher for the Al+10 Vol% Al_2O_3 nanocomposites compared with pure Al, mainly because there were alumina particles present. The friction of pure aluminum is about 0.898, whereas Al+10 Vol% Al_2O_3 is 0.94. Additionally, due to GO's lubricous nature, the COF of Al+0.25 wt% GO was less than those of pure Al and Al+10 vol% Al_2O_3 nanocomposite. On the other hand, for a hybrid nanocomposite made with Al-10 Vol% Al_2O_3+0.25wt% GO, the COF increases slightly compared to Al+0.25wt% GO nanocomposites because hard alumina particles are added. Adding GO to the hybrid nanocomposite further led to a further decrease in COF. The primary purpose of adding GO was to reduce the COF, as demonstrated by these results. As in Figure 25 and Table 10, the COF of Hybrid nanocomposite decreased by more than 20 % compared to pure Al. An overview of all samples' coefficients of friction is presented in table 10.

Sample	Coefficient of friction
Pure Al	0.89
Al + 10 Vol% Al_2O_3	0.94
Al+ 0.25wt%GO	0.81
Al + 10 Vol% Al_2O_3 + 0.25 wt% GO	0.85
Al + 10 Vol% Al_2O_3 + 0.5 wt% GO	0.78
Al + 10 Vol% Al_2O_3 + 1 wt% GO	0.70

Table 10: Coefficient of friction of all samples

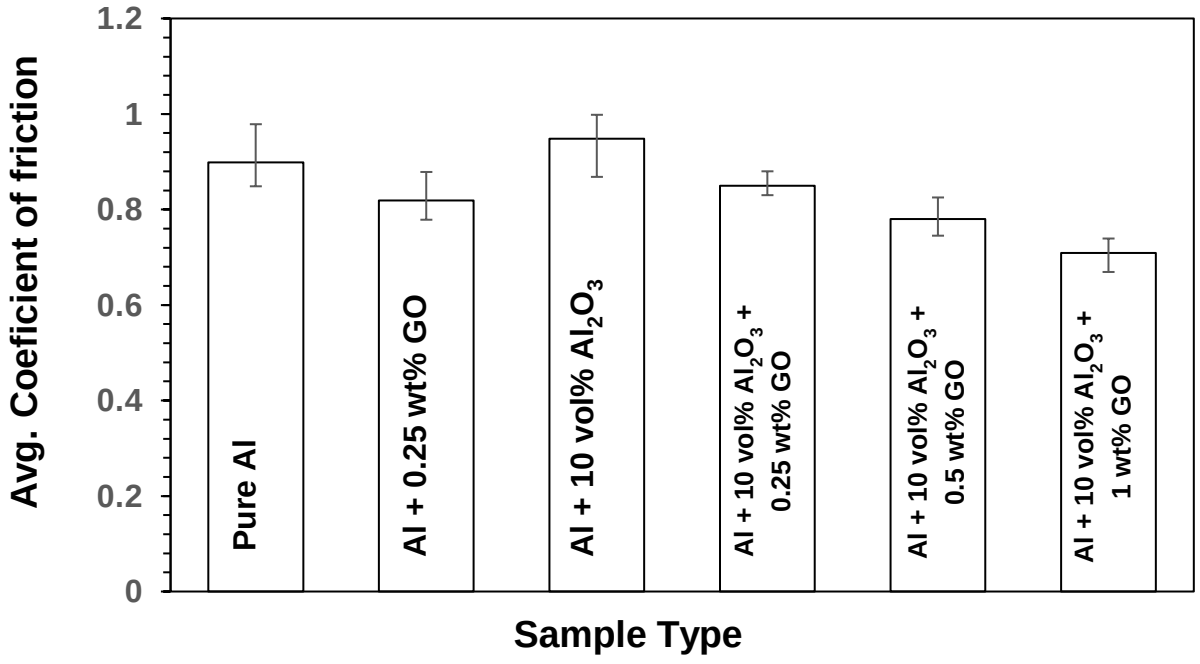

Figure 25: Coefficient of friction of all samples

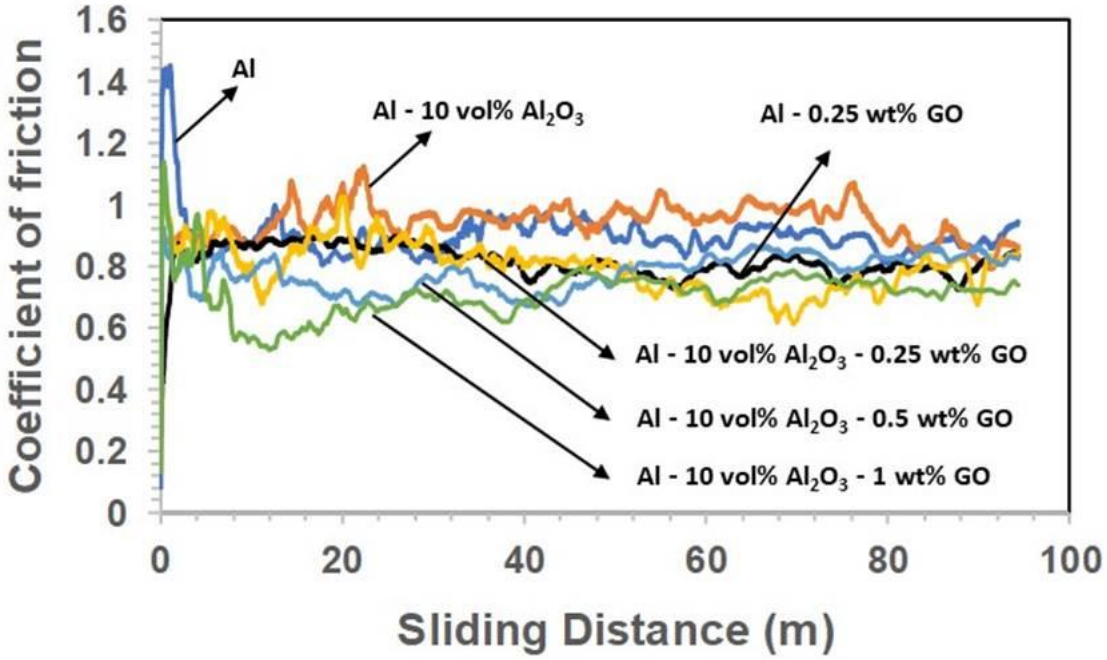

Figure 26: Typical Frictional graphs for the different samples at a normal load of 3 N and a rotational speed of 0.1 m/s

4.5.3 SEM and EDS of worn samples

The EDS and SEM of worn samples were performed to characterize the primary reasons for wear and wear mechanisms. In figure 28, the counterface ball optical images after sliding against the SPS samples are shown under 3 N of force, 0.1 m/s of sliding speed, and for 100 m of sliding distance. As shown in Figure 27, pure aluminum shows a high wear rate, as confirmed by SEM image as in Figure 27(a). In Figure 27(a), an extremely rough surface is depicted with deep grooves and debris particles as a result of ploughing during wear tests involving extreme abrasive wear mechanisms combined with adhesive wear (gouging). As shown in Figure 28(a), a large scar mark was also detected on the counterface ball sliding against the samples, which was attributed to the high deformation and adhesive components of the friction coefficient. Whereby the addition of 0.25 wt% GO to the Al matrix led to a reduction of 22% in the specific wear rate and a reduction of 9% in the COF. In figure 27(b), the wear track is substantially smoother than pure Al, with only a few adhesive wears along the edges. Furthermore, the hardness of the Al - 0.25 wt% GO was greatly increased when as shown in Figure 22, accounting for the superior tribological performance. A lubricating property of GO contributed to a reduction of COF compared with a pure Al Sample. Furthermore, the ball sliding against the Al-0.25 wt% GO sample also revealed a scar mark identical to seen with the Al sample. 10 Vol% of Al_2O_3 added to the Al matrix resulted in a significant improvement in hardness, as illustrated in Figure 23. The composite (Al+10 Vol% Al_2O_3) reduced the specific wear rate by 46 % and increased the coefficient of friction by 6% compared. In examining the wear track closely, it was confirmed that the wear mechanism mainly was abrasive, with clear ploughing marks in the sliding direction. Small traces of iron were also found in the wear track accoding to EDX analysis, which may have been transferred from the ball during the test Figure 27(c). It is also apparent that the scar mark on the counter face ball is considerably smaller as in Figure 28(c) than in the earlier cases (Al and Al+0.25 wt% GO). Adding

Al_2O_3 to the Al matrix primarily contributed to the improvement of the hardness, leading to a significant reduction in the specific wear rate. An interesting trend is observed when both reinforcements are incorporated simultaneously into the Al matrix as a hybrid nanocomposite. Adding 10 Vol% Al_2O_3 and 0.25 wt% GO to Al produces an increase of 48% in hardness and a reduction of 55% in specific wear rate as well as a reduction of 5% in COF. In addition, this hybrid nanocomposite has better wear resistance than Al-0.25 wt% GO and Al+10 Vol% Al_2O_3 composites Figure 23. According to SEM analysis of the wear track, the wear traces were primarily formed by plastic deformation on the smooth surface. Furthermore, the scar marks on the ball were considerably smaller than on the other samples, as in Figure 28(d). A major contribution to this significant performance of the Al+10 Vol% Al_2O_3+0.25 wt% GO hybrid nanocomposite by the hardness improvement due to the addition of both Al_2O_3 and GO particles. In contrast, the Al_2O_3 particles played a major role. As revealed by EDS analysis of the wear track as shown in Figure 27(d), the GO reduced the COF of the hybrid nanocomposites by forming a thin solid lubricant film over the wear track. Adding 0.5 and 1 wt% GO to Al+10 Vol% Al_2O_3 give a lower hardness, wear resistance, and Coefficient of Friction. These results were due to the formation of GO agglomerates, which diminished the hardness and density of the hybrids. In hybrid samples, GO agglomerates formed two phases of morphology, resulting in a loss of hardness, thereby lowering wear resistance as in Figure 23. According to SEM images, the wear track was examined after the wear test, which indicated that abrasive wear was the principal mechanism for the wear Figure 27(e, f). Furthermore, the scar marks on the balls sliding against the counter face were more pronounced than those on the other samples, as in Figure 28(e, f).

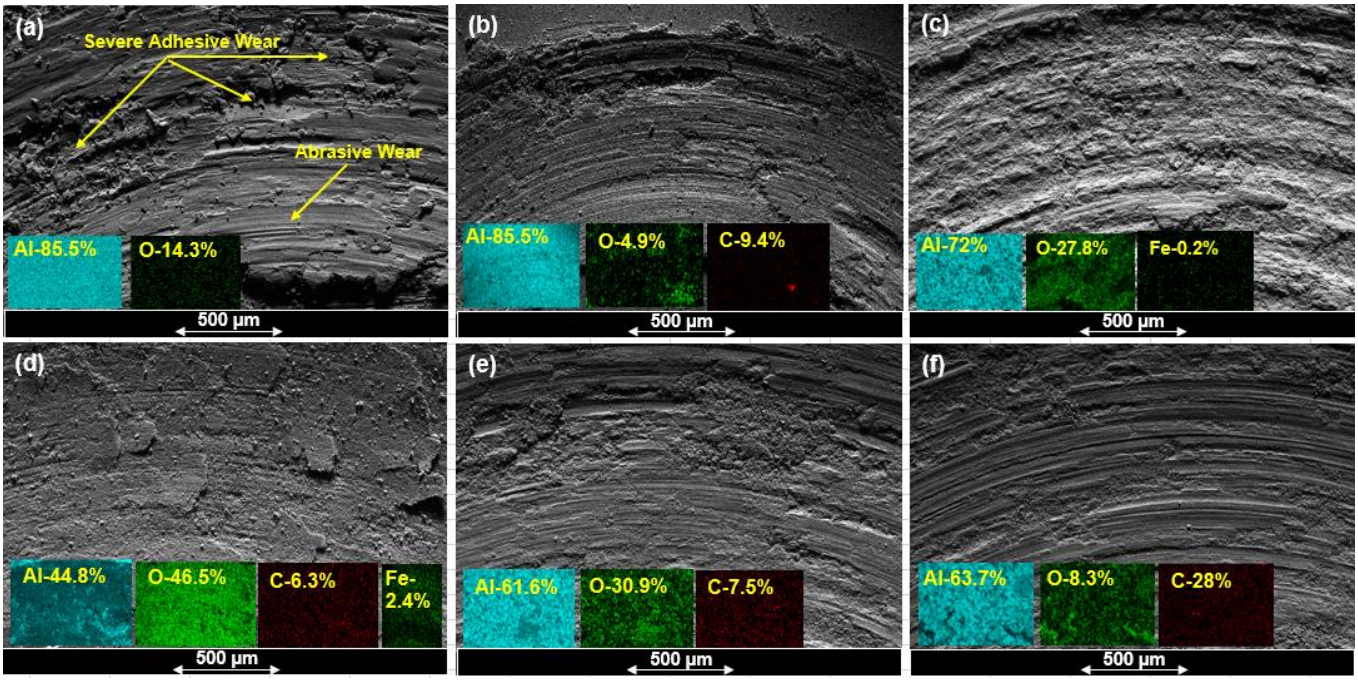

Figure 27. Typical SEM images and EDS mapping for the wear tracks of (a) Pure Al (b) Al+0.25% wt% GO (c) Al+10 Vol% Al_2O_3 (d) Al+10 Vol% Al_2O_3+0.25 wt% GO (e) Al+10 Vol% Al_2O_3+0.5 wt% GO (f) Al+10 Vol% Al_2O_3+1 wt% GO, after the wear at sliding distance of 100 m, a load of 3 N, and sliding speed of 0.1 m/s.

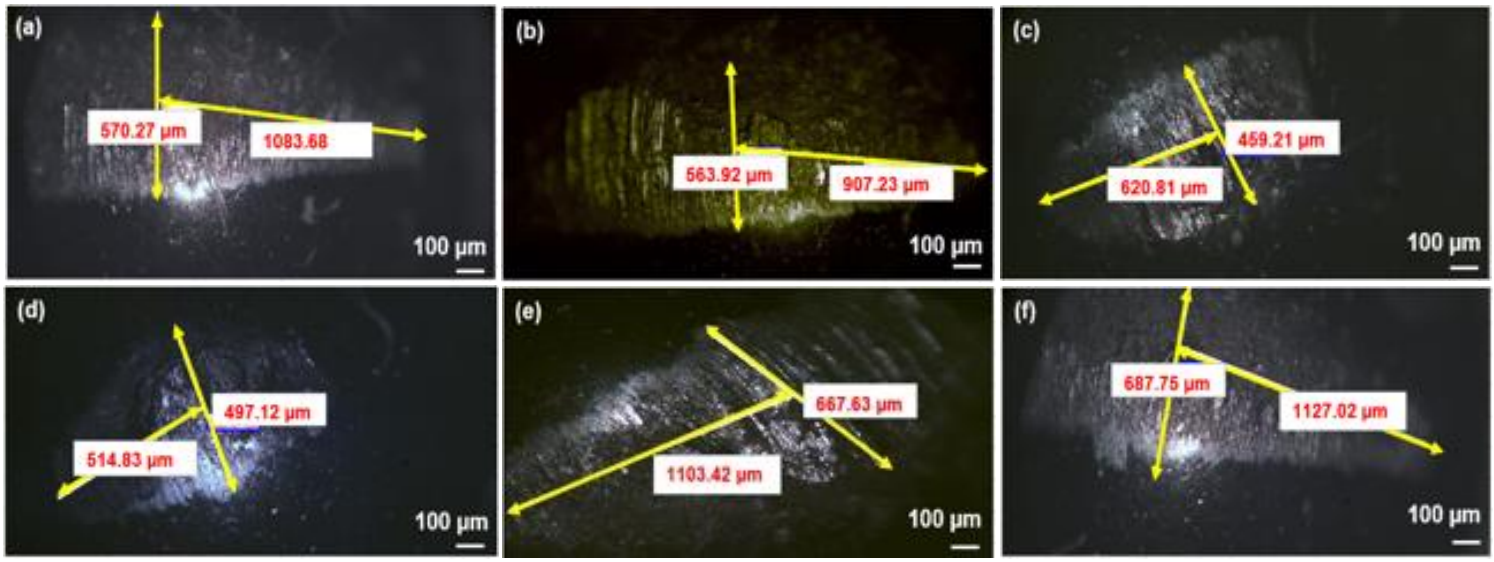

Figure 28. Optical counterface ball images (a) Pure Al (b) Al+0.25 wt% GO (c) Al+10 Vol% Al_2O_3 (d) Al+10 Vol% Al_2O_3+0.25 wt% GO (e) Al+10 Vol% Al_2O_3+0.5 wt% GO (f) Al+10 Vol% Al_2O_3+1 wt% GO, after the wear at sliding distance of 100 m, a load of 3 N, and sliding speed of 0.1 m/s.

4.5.4 Summary of the results

To summarize the results of this study, different hybrid nanocomposite was produced, tested, and characterized in terms of density, specific wear rate, hardness, and COF. Alumina with 10Vol% and a different fraction of graphene oxides were added to the aluminum matrix to improve the tribological properties. Alumina particles were the primary factor in increased hardness, whereas GO particles acted as solid lubricants and contributed little to hardness (Al+0.25wt%). We have achieved the highest specific wear rate (55% more than Pure Al) and hardness (two times pure Al) by adding both fillers with 10% alumina and 0.25wt% graphene oxide. Below is Table 11, which summarizes the results of all the samples.

Test	**Sample**				
	Al	Al + 10 Vol% Al_2O_3	Al + 10 Vol% Al_2O_3+ 0.25 wt% GO	Al + 10 Vol% Al_2O_3+ 0.5 wt% GO	Al + 10 Vol% Al_2O_3+1 wt% GO
Hardness (HV)	32.8	55.8	63.56	56.9	56
Relative density (%)	99.8	99.5	98.97	98.62	97.43
Specific wear rate (mm^3/Nm)	$566x10^{-6}$	$303.5x10^{-6}$	$251x10^{-6}$	$837x10^{-6}$	$192x10^{-5}$
Reduction of wear rate (%)	0	46.37	55.65%	-47.87%	-238.8%
Coefficient of friction	0.89	0.94	0.85	0.78	0.70

Table 11: Summary of tribological results of all samples.

Several studies to improve the tribological properties of aluminum composite/nanocomposite and aluminum hybrid composite/nanocomposite are shown in below table 12. It is noteworthy that the developed aluminum hybrid nanocomposite reinforced with both Al_2O_3 and GO displayed significant tribological performance compared to the other literature shown in Table 12.

Composite	**Method**	**Relative density (%)**	**Hardness**	**COF**	**Wear**	**Particle size of Reinforced material**	**Reduction of Wear Rate %**	**Ref**
Al-6082	Stir casting	98.31	74 BHN	--	62 µg		0	[8]
Al-6082+10% Al_2O_3		97.55	78 BHN	--	45 µg	NA	27.41%	
Al-6082+15% Al_2O_3		97.33	81 BHN	--	35 µg	NA	43.35%	
Al-6082+20% Al_2O_3		96.91	87 BHN	--	30 µg	NA	51.61%	
6061Al	Stir casting	--	95 HV	--	0.035 gm		0	[10]
6061Al+6% Al_2O_3		--	105 HV	--	0.028 gm	125 µm	20%	
6061Al+9% Al_2O_3		--	150 HV	--	0.023 gm	125 µm	34.28%	
6061Al+12% Al_2O_3		--	188 HV	--	0.020 gm	125 µm	42.85%	
Al6061	Stir casting	--	28 BHN	0.5	$1.4x10^{-3}$ (mm^3/m)		0	[43]
Al6061+5% Al_2O_3		--	33 BHN	0.44	$0.9x10^{-3}$ (mm^3/m)	40 nm	35.71%	
Al6061+10% Al_2O_3		--	38 BHN	0.35	$0.64x10^{-3}$ (mm^3/m)	40 nm	54.42%	
Al6061+15% Al_2O_3		--	39 BHN	0.35	$0.66x10^{-3}$ (mm^3/m)	40 nm	52.85%	
7075 Al	SPS	99.05	96.8 HV	0.5	0.0034 (mm^3/m)		0	[48]
7075 Al/GNPs		99.04	124.9 HV	0.15	0.00275 (mm^3/m)	5-10 µm (2-4 nm) sheet	19.11%	
A356	Stir and squeeze casting	--	--	0.78	44 Vol loss (mm^3)		0	[44]
A356 +0.5% Al_2O_3		--	--	0.45	27 Vol loss (mm^3)	30 nm	38.63%	
A356 +1% Al_2O_3		--	--	0.32	25 Vol loss (mm^3)	30 nm	43.18%	
A356 +1.5% Al_2O_3		--	--	0.47	32 Vol loss (mm^3)	30 nm	27.27%	
Al	Sintering process	--	53 HRB	0.57	0.039 (g)		0	[34]
Al+ 0.01wt%Greaphene		--	54 HRB	0.49	0.037 (g)	Nano sheet	5.12%	
Al+ 0.5wt%Greaphene		--	62 HRB	0.43	0.021 (g)	Nano sheet	46.15	
Al+ 1wt%Greaphene		--	58 HRB	0.42	0.055 (g)	Nano sheet	-41.02%	

Al+ 2wt%Greaphene		--	49 HRB	0.36	0.105 (g)	Nano sheet	-169.23%	
Al+ 5wt%Greaphene		--	45 HRB	0.33	0.170 (g)	Nano sheet	-335.89%	
AA 6061	Microwave sintering	--	65 HV	0.30	0.075(gms)		0	[45]
AA 6061+0.3%Graphene		--	77 HV	0.25	0.062(gms)	10 μm (Flake)	17.33%	
AA 6061+0.6%Graphene		--	75 HV	0.24	0.062(gms)	10 μm (Flake)	17.33%	
AA 6061+0.9%Graphene		--	73 HV	0.23	0.066(gms)	10 μm (Flake)	12%	
AA 6061+1.2%Graphene		--	71 HV	0.23	0.070(gms)	10 μm (Flake)	6.66%	
Al 2024	Stir casting	--	--	--	0.109 (g)		0	[47]
Al 2024+0.25wt% Graphene		--	--	--	0.104 (g)	NA	4.58%	
Al 2024+0.50wt% Graphene		--	--	--	0.0998 (g)	NA	8.44%	
Al 2024+0.75wt% Graphene		--	--	--	0.0942 (g)	NA	13.57%	
Al 2024+1wt% Graphene		--	--	--	0.0892 (g)	NA	18.16%	

Table 12: Summary of Comparison studies.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

Aluminum (Al), Al+10Vol% Al_2O_3, Al+10Vol% Al_2O_3+(0.25, 0.5 and 1wt%), and Al+0.25wt% GO have been successfully produced, tested, and characterized as follows.

- Received material was sonicated and mixed using ball milling for 24 hours for Al + 10 Vol% Al_2O_3 and Al+ 0.25wt%GO and for 48 hours for Al + 10 Vol% Al_2O_3 +(0.25, 0.5 and 1 wt% GO)
- SEM and XRD were conducted and observed a great distribution of graphene oxide along the grain boundaries and alumina particles were distributed in the grains.
- Spark plasma sintering method was used to produce all samples. Samples with diameters of 20 mm and 7 mm thickness were produced successfully.
- Samples were surface finished by grinding with different grids up to 800 grid size and then polished with alumina paste.

All samples were measured for densification; adding 10 Vol% of alumina or 0.25 wt% GO into the Al matrix showed about the same results, whereas adding different fractions of GO (0.25, 0.5, and 1 wt%) to Al+10 Vol% Al_2O_3 shows more reduction due to the higher porosity of the hybrid composite.

Hardness tests were conducted on all samples and revealed that introducing 10 Vol% alumina to the aluminum matrix increased the hardness by 70%. The highest hardness found in all samples was in Al + 10% Al_2O_3 and Al + 0.25 wt% GO, which increased twofold over pure aluminum.

Tribological tests were conducted to evaluate the specific wear rate and friction of all samples. Tests were conducted under a load of 3N and a speed of 0.1 m/s for 100 meters. Wear tests were running for 938 seconds.

The coefficient of friction of all samples measured was determined and observed that as GO fraction increases, the COF decreases, indicating that GO acts as a solid lubricant between mating surfaces. The least COF was measured in the hybrid nanocomposite containing Al + 10 vol% Al_2O_3 + 1 vol% GO.

The optimum hardness and specific wear rate value were found on an aluminum hybrid nanocomposite made from Al + 10 Vol% Al_2O_3 + 0.25 wt% GO, where the hardness was two times greater than pure aluminum and the specific wear rate was 55% lower than pure aluminum. It was confirmed that the wear mechanism mainly was abrasive, with clear ploughing marks in the sliding direction. It is also apparent that the scar mark on the counter face ball is considerably smaller than other samples.

As the GO content increased over 0.25 wt% in Al + 10 vol% Al_2O_3, the hardness and specific wear rate decreased. In hybrid samples, GO agglomerates formed two phases of morphology, resulting in a loss of hardness, thereby lowering wear resistance. According to SEM images, abrasive wear was the principal mechanism for the wear. Furthermore, the scar marks on the balls sliding against the counter face were more pronounced than those on the other samples.

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