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Effect of alloying elements on the mechanical behavior of microwave synthesized composites

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Abstract

In this research work three different composites are processed through powder metallurgy route using Aluminum Alloy (AA) 2900, 2024 and 2219 as matrix with SiC, Al₂O₃ and graphene as reinforcement. The objectives of this research work are to determine the influence of different weight percentage (i.e. 0 – 20 wt %) of ceramic reinforcements such as SiC, Al₂O₃ and graphene on the strength characteristics and tribological characteristics of the composites. The weight percentage of reinforcement materials and tribological studies at different loads are variable parameters in this experimental work. The average particle size used for both matrix and reinforcements are 10 microns and graphene with 10 nano microns. The wear surface morphology with strength correlation will be presented in the paper where the aluminium alloy possessing novel alloying element exhibits good strength properties, ductility properties and wear behavior. Al₂O₃ as a reinforcement exhibits good wettability and diffusion characteristics in the matrix compared to SiC and graphene which is reflecting on the mechanical properties and the wear behavior of the composites processed by microwave sintering.

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1. Introduction

Metal Matrix Composites with Ceramic particulate reinforcements are fetching good physical properties to increase the insufficiencies of lighter aluminium composites in terms of thermal resistance, stiffness characteristics, tensile strength and wear resistance. Important objective of developing metal matrix composites is to have a material with a better combination of toughness and stiffness and to increase the static and dynamic properties of the materials. Wear testing is widely used for research, development and control of surface quality for surface coatings. Among various reinforcements such as SiC, graphite and alumina, SiC upholds good thermal and chemical stability during synthesis and good strength at severe service conditions [1]. The poor mechanical properties of aluminium metal matrix composites produced by the other methods are attributed to the weak bonding between adjacent particles and to internal porosity. At higher volume portions, the strength of interfacial bonds, initiation and growth of voids and particle cracking all play an important role in controlling the mechanical properties. Sintering parameters, such as sintering atmosphere and moisture can also impact the sinterability. In a research work it is also confirmed that increased concentration of magnesium on the oxide surface film of the powder particles film supports sintering of aluminium by reduction of Al_2O_3 [2]. Likewise increasing the shear stresses during solid state consolidation processes can advance the particle–matrix interface strength due to added operative break-up of the oxide barrier [3]. The mechanical properties of the powder metallurgy processed (P/M) components are comparable to wrought and cast alloys. However, sintering of aluminium alloys are tough due to the presence of the balanced aluminium oxide layer covering the powder confining the atomic diffusion between each particle

There are several factors that affect the sinterability of aluminium, particularly the presence of magnesium as an alloying composition. In another research work it was justified that sintering parameters, such as sintering atmosphere and moisture can also influence the sinterability. Especially Microwave sintering technique is used to sinter the powder compacted samples. Most metals possess penetrating depths of micrometer order, so the direct heating remains superficial, using the metal powders of the same order might enhance to use Microwave in the heating process [7][5]. In Conventional sintering heat is generated from the heating source and the samples are heated up by conduction, radiation & convection. Moreover in microwave sintering the materials themselves absorb the microwave and convert them to heat inside their body [5]. Increase in volume fraction or reduction in size of reinforcement particle, the densification coefficient decreases indicating the inferior material deformation [9]. Relative improvements in hardness with respect to varying weight fractions of reinforcements are compared to identify the most applicable method. Similarly, the behavior of composite sintered with microwave sintering is studied to understand the functional behavior of the composite. Hence the AA-SiC, AA- Al_2O_3 and AA-Gr compacted samples are sintered in microwave furnace for which the testing's such as, Density, Rockwell hardness testing, Compression testing and Wear testing are compared and studied in correlation with the input parameters and sintering conditions. Field Emission Scanning Electron Microscope (FESEM) and Electro Diffraction Analysis (EDAX) micrographs are obtained to study the coefficient of friction behavior and the friction behavior of the microwave sintered samples.

2. Experimental Methods

3, 6 and 9% of SiC, Al_2O_3 and 0.25, 0.50, 0.75% of graphene with an average particulate size of 10 μm and 10 nano were used as reinforcements which were supplied by Carborundum Universal Limited, India. The average size of aluminium alloy 2900, 2024 and 2219 particles used was 10 μm with a purity of 99.97% which was sourced from AMPAL Inc. USA. Graphene (from Angstrom Materials) is used as reinforcement and ultrasonication is used to disperse it in metal matrix. Correct proportions of the powders were placed in a high-energy planetary ball mill for 30 mins at 100 rpm. Ball to powder ratio was maintained at 10:1. Uniaxial pressing, a conventional powder metallurgy process was used to compact the samples in universal testing machine. Pressure used for compaction was 550 Mpa for a cross sectional die diameter of 25 mm and holding time of 10 mins for each sample. The die used for the compaction was made of D_2 steel with tungsten carbide insert containing a lower and upper punch. The compaction rate was 2 ton per minute. To prevent cold welding of the aluminium sample to the die wall the walls and punch contact surfaces were coated with zinc stearate coating as a lubricating agent. Green compacts, thus

produced were sintered in sealed chamber at 500 °C for 30 mins. The sintering was carried out in a microwave-sintering furnace. The Microwave furnace used was with maximum working capacity of 1600 °C, up to 10 KW with 2.45 GHz of magnetron and 1°C accuracy at dwell temperature. Micro structural analysis was performed using scanning electron microscope to identify the effect of grain size and microwave sintering on the mechanical properties of the MMC's. Keller's agent was used as the etchant to obtain a clear microstructure. Density measurements were done according to ASTM standard B328 using Archimedes principle. Hardness measurements were performed in Rockwell hardness tester using B scale with a ball diameter of 1/16 mm and a load of 100 kg. Compressive tests were carried out according to ASTM standard E9 using Instron Testing machine and at 0.75mm per minute strain rate. The experiments were carried out in Instron tensile testing machine. The compressive strengths were measured on average of three samples and dimensions were maintained as 25 mm dia with 50 mm length. The reported experimental values are the average of five experimental sample values in order to reduce errors. The samples are separately prepared to carry out dry sliding wear studies where the test was carried out using PIN ON DISC wear testing machine for three different loads (i.e. 20, 40, 60 N) and the track dia of 70 mm, wear duration of 20 mins, disc rotation speed of 400 Rpm are maintained constant. The wear surface morphology with strength correlation is presented where the aluminium alloy possessing novel alloying elements exhibits good strength properties, ductility properties and wear behavior. The SEM morphology of the material is presented in fig 1.

Table 1: Chemical composition of aluminium alloy 2900, 2024 and 2219

Element	Cu	Mg	Si	Fe	Sn	Al
2900 Weight %	3.25	1.47	0.30	0.09	0.67	Remaining
2024 Weight %	4.12	1.62	0.50	0.50	-	Remaining
2219 Weight %	5.92	0.02	0.20	0.30	-	Remaining

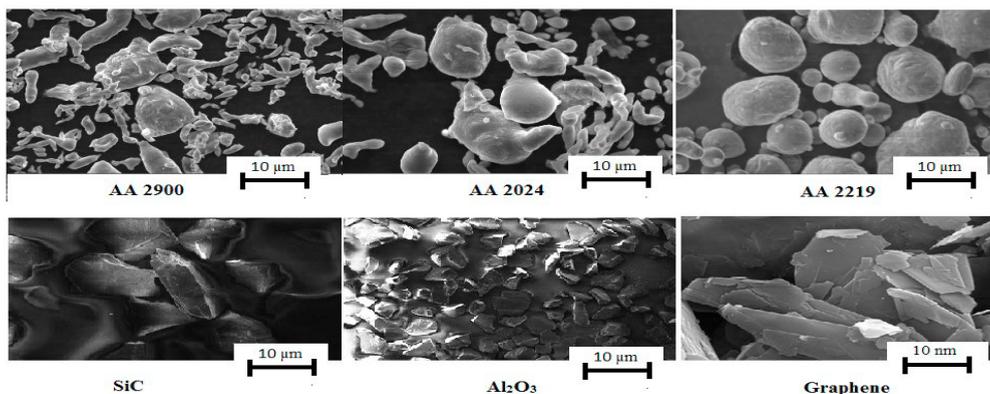


Fig 1: SEM micrographs of Aluminium alloys and reinforcements

3. Result and Discussions

3.1. Density

The influence of ceramic particulates percentage on the relative density of the composites samples fabricated using AA 2024, AA 2900 and 2219 are shown in Fig 2. It was observed that the sintered density is maximum for 3% of SiC and Al₂O₃ and reducing with the increase in percentage of ceramic particulates irrespective of the matrix material. It was observed that with the addition of lesser percentage of ceramic particulates mixed with the matrix material influence the change in densities; this is because of the variation in the densities of the matrix material and the reinforcement densities. Sintered densities were found to decrease as the graphene particulates percentage

increases as shown in fig 2. This reveals the reduction in compressibility in relationship with higher ceramic addition. It was observed that sintered density is reducing with an increase in reinforcement percentage, but the densification of the MMC's samples reinforced with Al_2O_3 has improved compared with samples that were reinforced with SiC, because the density of Al_2O_3 (3.97 g/cm^3) is higher than matrix alloy (2.7 g/cm^3) where the $10 \mu\text{m}$ Al_2O_3 particle occupies the voids and the matrix interface at higher compaction pressure.

But for the same processing condition graphene addition to the matrix exhibited a decreasing trend irrespective of the matrix material. Matrix material 2024, 2900 and 2219 when reinforced with Al_2O_3 shows a good improvement in sintered density compared to SiC particles reinforced samples. It is been evident from Fig 2 that the density of the MMC's reinforced with Al_2O_3 are increasing from 3 wt% ceramic content and reflects a decreasing trend in further addition of the ceramic reinforcements. But irrespective of the matrix material the 6 wt% Al_2O_3 MMC's samples exhibits good density compared closer to 3 wt%. Highest density was observed in the case of aluminium alloy 2900 because of 3.25 wt% of Cu present in the matrix alloy compared to the 2024 and 2219 monolithic alloy as shown in table 1, whereas 2219 has the least density compared to other matrixes. The increase in densities of the AA 2900 composites is related to Sn content as well. More over densities >98% can be achieved by the addition of Sn to the matrix alloy [2]. The difference in the melting point and compressive strength of Al_2O_3 and SiC act as a barrier to the rearrangement and diffusion of the particles leading to reduction in compressibility and reduction in density when there is increase in reinforcement percentage, irrespective of the sintering conditions [9][11].

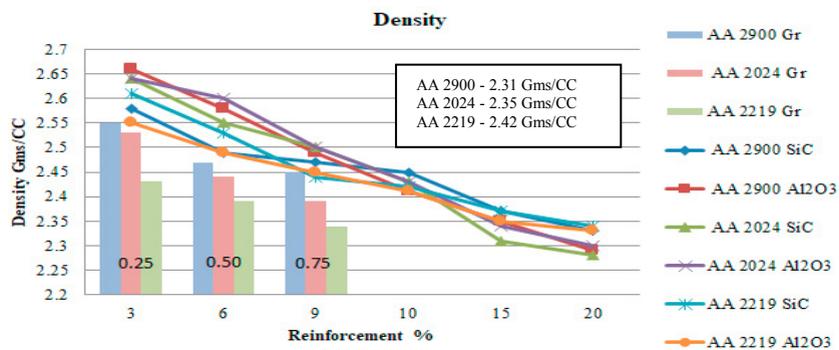


Fig 2: density of aluminium alloy 2900, 2024 and 2219 composites

3.2. Hardness Evaluation

Averages of 10 experimental data were taken for each sample to evade the inconsistency in the hardness data of the sample and to attain the accuracy of the experimentation results. The Rockwell hardness data are shown in fig 3 for AA 2900, AA 2024 and AA 2219 reinforced with SiC, Al_2O_3 and graphene particulates. Higher hardness values were observed in MMC's with increased weight percentage of SiC and Al_2O_3 particulates compared to graphene reinforced composites. The effect of microwave sintering also played a major role in improving hardness of the composites as the sintering mechanism deals with heating the composites from inside. Al_2O_3 have better bonding than SiC and graphene due to affinity and always aluminium alloy matrix particles have a layer of aluminium oxide to few nano meters.

Looking at the matrix material aspects, the aluminium alloy 2900 exhibited better hardness results compared to 2024 and 2219 because of novel alloying elements like magnesium, silica and tin at optimum levels. Since Sn is inert in nature with respect to monolithic alloy at ambient temperatures, it agglomerates in intergranular regions as isolated globules. Thus, a MMC's of sorts is created with the powder metallurgy processed aluminium alloy 2014 as the matrix and Sn as a softer secondary phase. While this would lead to the improved wear resistance, it would also lead to lesser bulk hardness of the MMC's [2]. 2900 alloyed with 0.67 wt% Sn have lead to a closer

trend in the hardness data compared to aluminium alloy 2024 and better than that of samples made of 2219 matrix material as shown in fig 3. The alloying elements like Mg have an effect on bonding which is one of the reasons where the 2219 composites failed to excel in strength properties.

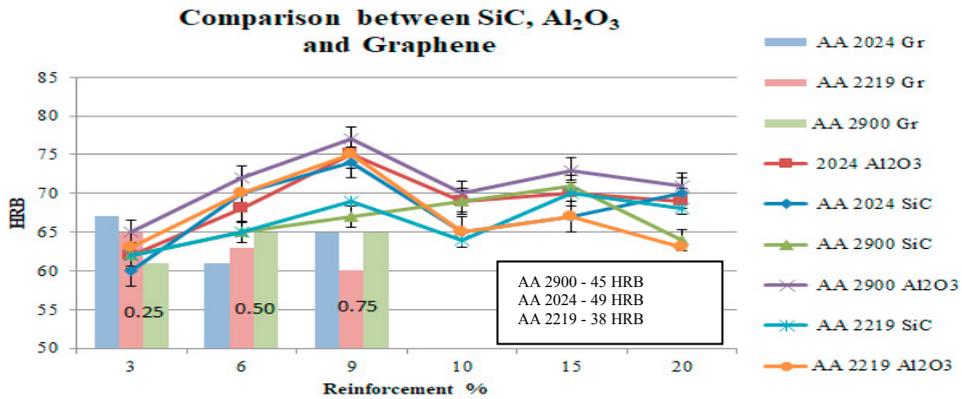


Fig 3: Effect of reinforcement wt% on the Rockwell hardness of the composites

3.3. Compressive Strength Evaluation

Figure 5 shows the compressive strength relation with reinforcement percentage for AA 2024, AA 2900 and AA 2219 reinforced with SiC, Al₂O₃ and graphene MMC's synthesized by microwave sintering method. The compressive strength of AA 2900-SiC, AA 2024-SiC and AA 2219-SiC increases with increase in weight % of SiC from 3 to 9 wt. %. On the other hand, for all composites AA 2024, AA2900 and AA 2219 reinforced with Al₂O₃ samples exhibit an increasing trend in the compressive strength from 3 to 6 weight % achieving a fall in its strength at 9% Al₂O₃. At higher amount of SiC particulates the compressive strength of the MMC's is increased while the strain to fracture initiation has decreased. SiC particles as the harder particulate in the composites can prevent the dislocation movements. Increasing wt% of SiC, Al₂O₃ particulates increased their strengthening effect, though SiC is the most effective strengthening particulates, for higher strength, hardness and reduction in grain size. 10 μm sized Al₂O₃ of 6 wt% when added to the matrix the dispersion of the particulates was enhanced because of microwave sintering and 0.30 wt% of Si alloyed in the AA 2900, 0.50 wt% of Si in A 2024 and 0.20 wt% of Si in case of AA 2219 as shown in table 1. AA 2024 with 0.50 % of Si is expected to show better material behavior and good diffusion of the ceramic reinforcement but failed to produce good compressive strength because of over diffusion of the reinforcement in the alloy which lead to agglomeration leading early failure.

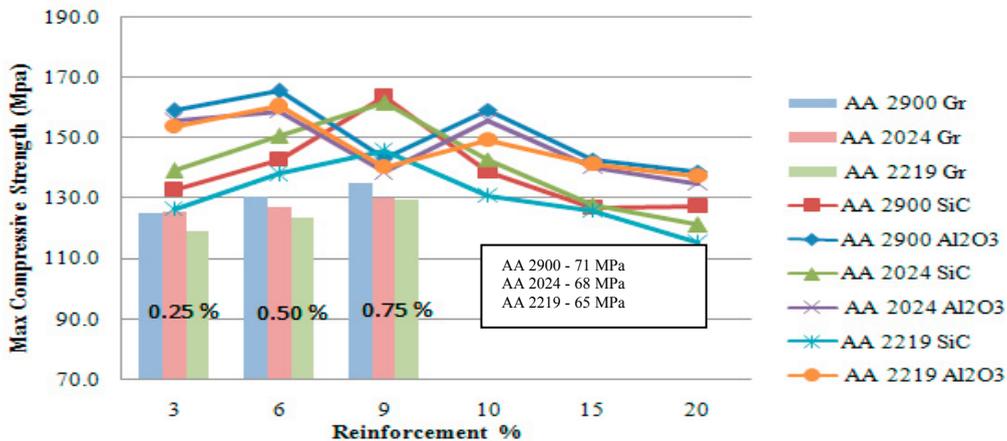


Fig 4: Effect of reinforcement wt% on the Compressive strength of the composites

3.4. Tribological Evaluation

From fig 5, 6 and 7 it is found that, as the reinforcement percentage increased the coefficient of friction values are increased irrespective of the loading condition. It is observed that increased content of SiC in alloy matrix results in increased frictional coefficients. It is also observed that AA–Al₂O₃ composite (i.e. 6 wt. % of Al₂O₃) exhibit lesser frictional coefficients when compared with that of composites fabricated of AA 2024 and 2219 matrix. It confirms that SiC particulates pulled out slide at the contact surface creating a third body abrasion and thereby increases the frictional coefficients. This can be attributed to the fact that during dry sliding, silicon carbide particles (rigid particulates) may get removed from the matrix and get trapped between the surfaces leading to elevated frictional coefficients.

The lower value of coefficient of friction of AA–Al₂O₃ composite can be mainly recognized because of the good bonding nature of Al₂O₃ with the aluminium matrix and good load bearing capacity compared to SiC and reduced sharp surfaces. Similar results are addressed by [16], [17] and [18]. Comparing AA 2024, AA 2900 and 2219, 2900 with 6 wt. % Al₂O₃ performed in good resistance to wear loss because of the Sn content in the matrix as shown in the fig 5, 6 and 7 irrespective of loading conditions. The coefficient of composites had a constant increasing trend for the compositions AA2900-SiC, 2219 with SiC and Al₂O₃, and AA 2024-SiC as observed from the fig 5, 6 and 7 irrespective of the loads used for wear testing with increase in the ceramic content from 0–20 wt %.

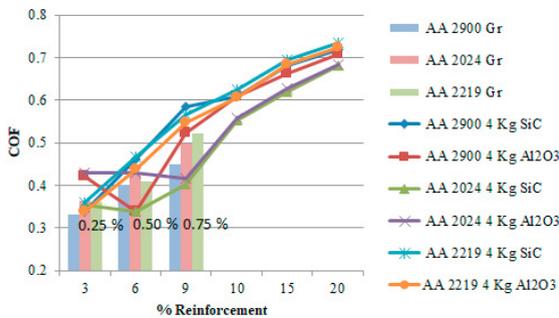


Fig 5: Effect of reinforcement wt% on the coefficient of friction of the composites loaded at 20 N

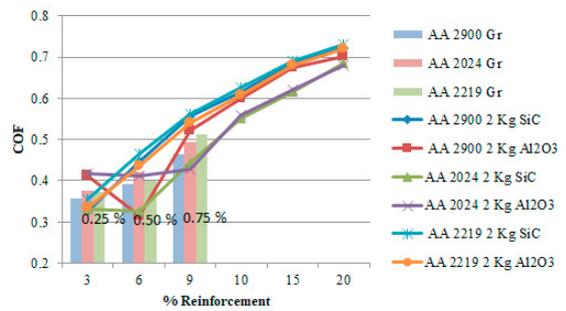


Fig 6: Effect of reinforcement wt% on the coefficient of friction of the composites loaded at 40 N

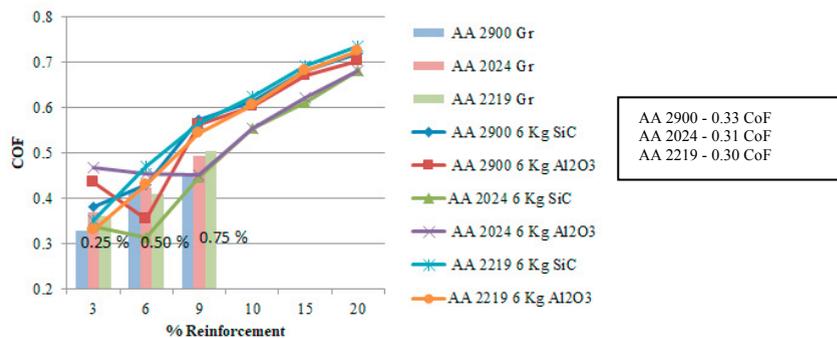


Fig 7: Effect of reinforcement wt% on the coefficient of friction of the composites loaded at 60 N

3.5. SEM and Microstructure Evaluation

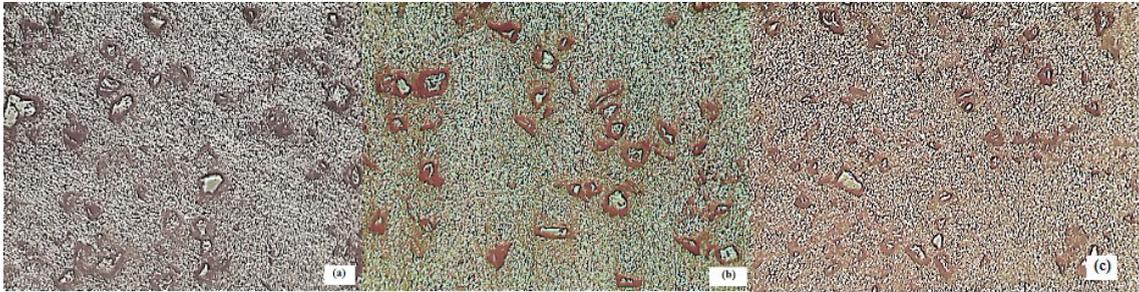


Fig 8: Microstructure of the samples microwave sintered for 6 wt% Al₂O₃ with (a) AA 2900 and (b) AA 2024 and (c) AA 2219

Fig 8 shows the microstructure of the microwave sintered sample for 6 wt% Al₂O₃ with AA 2900 and AA 2024. Fig 8 (a) and (b) clearly depicts that ball milling has improved the homogeneous dispersion of the reinforcement particulates in the matrix. Very less porosity is being observed in the microstructure which is evident from density values as shown in fig 2 irrespective of the metal matrix used. From fig 8 (a) and (b), it is evident that reinforcement particles have bonded firmly with the matrix metal during microwave sintering process. From fig 8 (c), it is observed that very less reinforcement particles are observed on the surface of the 2219 alloy composites. This is attributed that lesser diffusion of the reinforcement particulates into the matrix is obtained because of lack in Si content which leads to reduction in hardness and compressive strength as shown in fig 3 and fig 4.

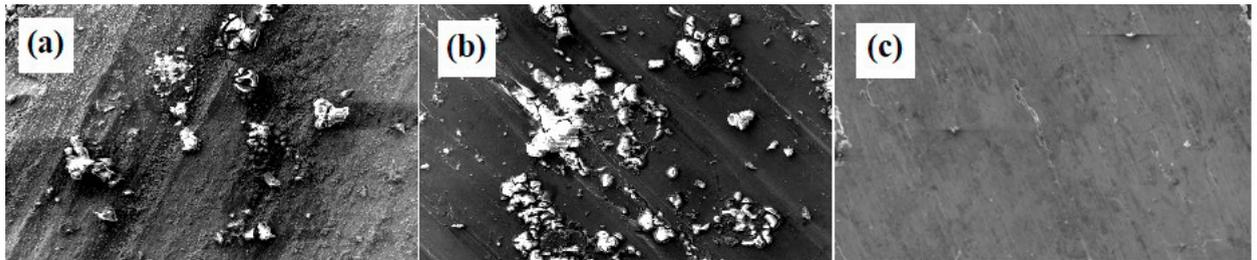


Fig 9: Wear track SEM micrographs (Scale 20 μm) of AA 2900 with 6 wt % Al₂O₃ at (a) 20 N and (b) 40 N forces and (c) 0.5 wt% Graphene at 40 N

Fig 9 depicts the SEM micrographs of AA2900- 6 wt% Al₂O₃ MMC's showing the effect of microwave sintering on the wear behavior. Figs 9 (a) showing the movement restriction of the particles and are observed to be firmly bonded with the matrix material and uniformly dispersed in the matrix. Fig 9 (b) showing the particulate trapped in matrix material even worn at higher load of 40 N confirming the bonding strength of Al₂O₃ with the aluminium matrix. A frictional mechanism can be understood to explain their relationships between the wear resistance and the material removal of the composites. Enhanced shear strengths of the composites processed by microwave sintering with reinforcements is one of the reasons for the resistance to wear sliding them against the steel disc of 45 HRC. It also evident that a higher particle weight (wt %) fraction leads to a higher hardness and thus results in a larger frictional coefficient as shown in fig 5, 6 and 7 irrespective of the loading conditions. Similar results have been addressed by zhang et. al [8].

Particle size of 10 μm is also a major reason for good wear performance of the composites. The AA 2900 shows grain boundary precipitates as well, and reinforcement particulates surrounded by precipitate, where the composite does not give the particulates pull out when the load is increased to 40 N. Under these conditions the presence of alumina particulates (Al₂O₃) seems to be beneficial to the tribological performance of the composites

rather using SiC as reinforcement. Similar results have been reported by Narayan and Bai [19]. From fig 9 the graphene addition facilitated the frictional behavior of the composites by leaving negligible grooves and surface failures after the wear process. But for the same composite the strength behavior was noticed to be less compared to SiC and Al₂O₃.

4. Conclusions

SiC, Al₂O₃ and graphene reinforcements found to increase the hardness of the matrix, though the difference is not much and Al₂O₃ has better influence than SiC and graphene due to better bonding. Hardness is improved from the 3 – 9 wt % of ceramic content beyond which the decreasing trend is observed. Compressive strength increase upto 6% of the ceramic content and then decreases with further addition. Hence there exists a threshold reinforcement percentage for achieving maximum compressive strength. Hence addition of higher reinforcement percentage can be employed wherever the objective is to increase hardness and wear resistance. Just by adding graphene there was not a significant contribution to the improvement in strength properties were noticed.

Si alloyed with matrix material improved wettability and enabled homogeneous distribution of the reinforcement particles especially in case of composites that are reinforced with 6 wt% Al₂O₃ when microwave sintered. 2900 with the optimal Si content compared to AA 2024 and AA 2219 exhibited good strength and tribological behavior because of the optimal alloying content in the matrix alloy. Increasing wt% of SiC increases matrix structure defects such as pores and reinforcement particle coarsening which lead to lesser relative density, strength and hardness of the composites. Al₂O₃ addition in the metal matrix gave better hardness results and compressive strength results compared to SiC addition. And 6% Al₂O₃ reinforced MMC's samples was exhibiting improved hardness results, strength behavior and stress-strain behavior when the samples are microwave sintered. Microwave sintering process also played a significant role in enhancing the mechanical property of the MMC's and good Microstructural refinements are observed in the MMC's when the samples were microwave sintered.

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