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EFFECT OF MHA ON TRIBO-BEHAVIOUR OF Al-MHA-Si_3N_4 HYBRID COMPOSITES

In the contemporary research community, hybrid composites with improved performance are emerging as a trend, overcoming the drawbacks of conventional composites and satisfying needs in tribological applications. In this work, Al-MHA-Si_3N_4 hybrid composites reinforced with various weight percentages of mustard husk ash (MHA), 0, 2.5, 5, 7.5% and 10%, produced by powder metallurgy techniques at 300, 400, 500, 600 and 700 MPa compaction pressure were analysed. The microstructural characterization of the metal matrix hybrid composites, followed by X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersive spectroscopy (EDS) investigations show the homogeneous distribution of the reinforcement in the metal matrix. A sliding wear study without lubrication was performed on a pin-on-disc wear testing machine under the following sliding conditions: sliding velocity (SV) of 1.5 m/s, sliding distance (SD) of 300 m and applied loads of 25 N and 35 N. The deformation of the worn surfaces was also investigated. It was found that the tribological characteristics of the composites were enhanced by increasing the weight percentage of MHA and the compaction pressure.

Keywords: **powder metallurgy, metal matrix hybrid composites, SEM, XRD, wear**

INTRODUCTION

The design, wear, friction, and lubrication of surfaces interacting in relative motion are all studied in the field of tribology [1]. Since metals and ceramics are common tribo-pair materials, metal-metal and metal-ceramic tribosystems had previously received a great deal of attention. Due to their quick manufacture, good chemical resistance, light weight, self-lubricating qualities, and lack of the need for maintenance, hybrid composites have quickly replaced conventional elements in mechanical components in recent decades. In recent years, aluminium metal matrix composites have become an attractive material owing to their worldwide applications in the aerospace, defence, agriculture, automotive, marine, mining and construction sectors [1, 2].

A composite material consists of two or more constituents that have different physical or mechanical properties. Human beings have been using composite materials since about 7000 BCE. The pots, bricks and mud plaster on the floors and walls of our predecessors houses are examples of products made of composites developed by mixing clay, sand and a binder (crop waste, wheat stalks, etc.). In 1200 AD, the Mongols used a composite bow that combined animal glue, wood and bone. Composites were introduced in automotive, medical and military applications to reduce costs and increase the strength of the employed metals. Composites of ashtadhatu and panchaloha, called octo-alloy, were used for casting metal idols for temples in the ancient world.

The demand for composites is increasing day by day because of their superior properties. The market for composites increases by 5% per year worldwide [3]. Aluminium metal matrix composites have excellent strength, stiffness and density properties. Their cost-effectiveness and light weight make them very attractive for industrialization [4-6]. The hardness, tensile strength, compressive strength, fatigue and wear behaviour properties of the developed composites are evaluated according to standards [7, 8]. The developed composites can be used in tribological applications like automobile brakes, piston rings, camshafts, rivets, and cylinder blocks to increase the strength of the these components [9-12]. The effect of hard ceramic particles like silicon carbide was studied and it was found that the wear resistance was enhanced due to the homogeneous distribution of the particles and good bonding with the matrix. Moreover, the wear rate can be decreased by increasing the sliding distance, velocity, and applied load [13].

Fly ash and red mud are industrial waste reinforcements containing SiO_2 and Al_2O_3, which enhance the properties of composites [14, 15]. The development of hybrid aluminium composites by using fly ash as reinforcement improves the tribological properties with a smaller impact on the cost. This impact on the tribological properties makes fly ash suitable for secondary reinforcement for hybrid composites [16-19]. The wear resistance can be enhanced by using fly ash as reinforcement in AA2024 metal matrix composites in [20].

Agricultural waste plays a very important role in developing aluminium hybrid composites. Generally, rice husk ash, MHA, ground net ash, bean shell bagasse ash, maize stalk ash, palm kernel shell ash, and corn cob ash are used today for the production of AMMCs [21-23]. In this era, agro wastes serve as encouraging reinforcement to improve the properties of composite materials. MHA was used as reinforcement in aluminium and it was found that the wear resistance was enhanced with the addition of MHA to pure aluminium [24]. The detailed specification of the employed powders and content specification of MHA are given in Tables 1 and 2, respectively.

TABLE 1. **Detailed specification of employed powders**

Element powder	Mean diameter	Density [g/cm^2]
Al	44	2.67
MHA	75	2.46
Si_3N_4	44	3.44

The density and porosity of composites depend on the morphology, shape, size and compaction pressure applied during the fabrication of composites [25]. A review of the literature found that very few papers have been published on Al-MHA hybrid composites developed by the powder metallurgy route using different compaction pressures.

TABLE 2. **Content specification of MHA**

Element	Weight [%]
SiO_2	46
MgO	4.5
Al_2O_3	7.9
FeS_2	2
KCl	2.9
MAD-10 Feldspar	5
Wollastonite	12.87

EXPERIMENTAL PROCEDURES

Aluminium was employed as the matrix to fabricate the hybrid composites. It was purchased from Otto Chemika Reagents Ltd, Mumbai. The density of the aluminium is 2.67 g/cm^3. The fine aluminium powder has 99.7% purity with a melting point of 660.37°C. The size of the powder grains is ≤44 μm.

α-Si_3N_4 was used as the reinforcement (Aldrich Chemicals Pvt. Ltd., USA). The size of the α-phase of the reinforcement is ≤ 44 μm and it has a density of 3.44 g/cm^3. The mustard husk was collected from farms in the villages of Nasirbas, Haryana and was dried in a dry atmosphere for 48 h to reduce the moisture content. After that, the mustard husk was converted into ash to remove the carbonaceous and volatile components by means of a muffle furnace at 600°C for 60 min in the materials science laboratory at Mewat Engineering College, Nuh, Haryana. After cooling at room temperature, the ash was sieved to a particle size of ≤ 75 μm. In this study, firstly, the powders of the aluminium metal matrix Al, Si_3N_4 and MHA were analysed. Secondly, analytical scales KERRO P7 BL-2204 having a least count of 0.0001 g, were used for weighing the quantities of the essential powders. In order to prepare the Al-Si_3N_4-MHA/x% powder mixtures (x = 0, 2.5, 5, 7.5 and 10 wt.% and fixed 5 wt.% Si_3N_4), ball milling (Ball mill 1 kg, Bexco export, India) was performed with a stainless steel ball to powder ratio of 10:1 at 100 rpm for 180 min. To avoid cold welding and for homogenous mixing, 3 wt.% ethanol was added as a process control agent during milling. According to standard ASTM G99-95, cylindrical specimens of diameter 10 mm and length 25 mm were fabricated by means of a 100 kN hydraulic press applying uniaxial loads of 300, 400, 500, 600, 700 MPa, and a dwell time of 30 seconds [28]. Acetone was used for cleaning and zinc stearate was the lubricant for the punch and die and their use was repeated after producing every green sample. A long steel needle was also used for removing cold weld metal from inside the die hole. The obtained green samples were sintered at 500 °C for 45 minutes in an inert atmosphere of nitrogen supplied at 50 ml/min to avoid oxidation of the aluminium matrix. The heating and cooling rate was maintained at 50°C/minute.

During sintering, nitrogen gas was supplied throughout the process, followed by cooling to room temperature. The details of the specimens are given in Table 3.

TABLE 3. **Specimen details**

Specimen	Weight		
	Al	Si_3N_4	MHA
A	100	0	0
B	92.5	5	2.5
C	90	5	5
D	87.5	5	7.5
E	85	5	10

Wear test

The wear test was performed on a pin-on-disc machine (DUCOM, Model No.TR-20L-PHM800-SHM850) according to standard ASTM G99-95. The experimental laboratory setup of the wear test machine is shown in Figure 1 and the parameters of the wear test are given in Table 4.

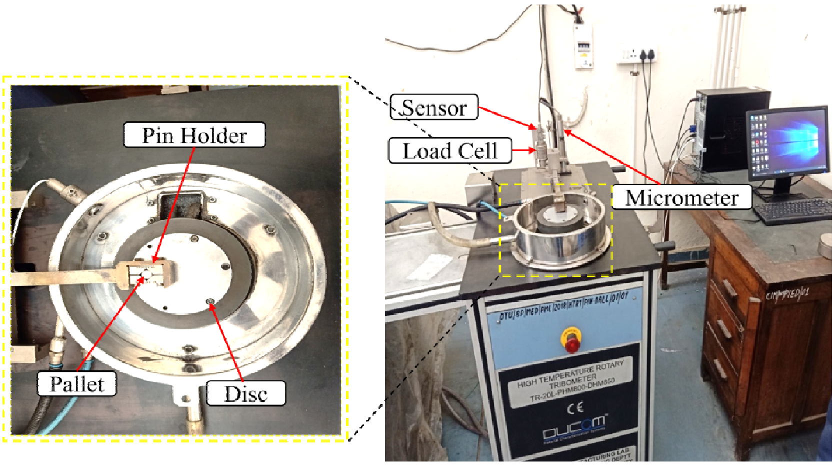


Fig. 1. Laboratory experimental setup of pin-on-disc tribometer

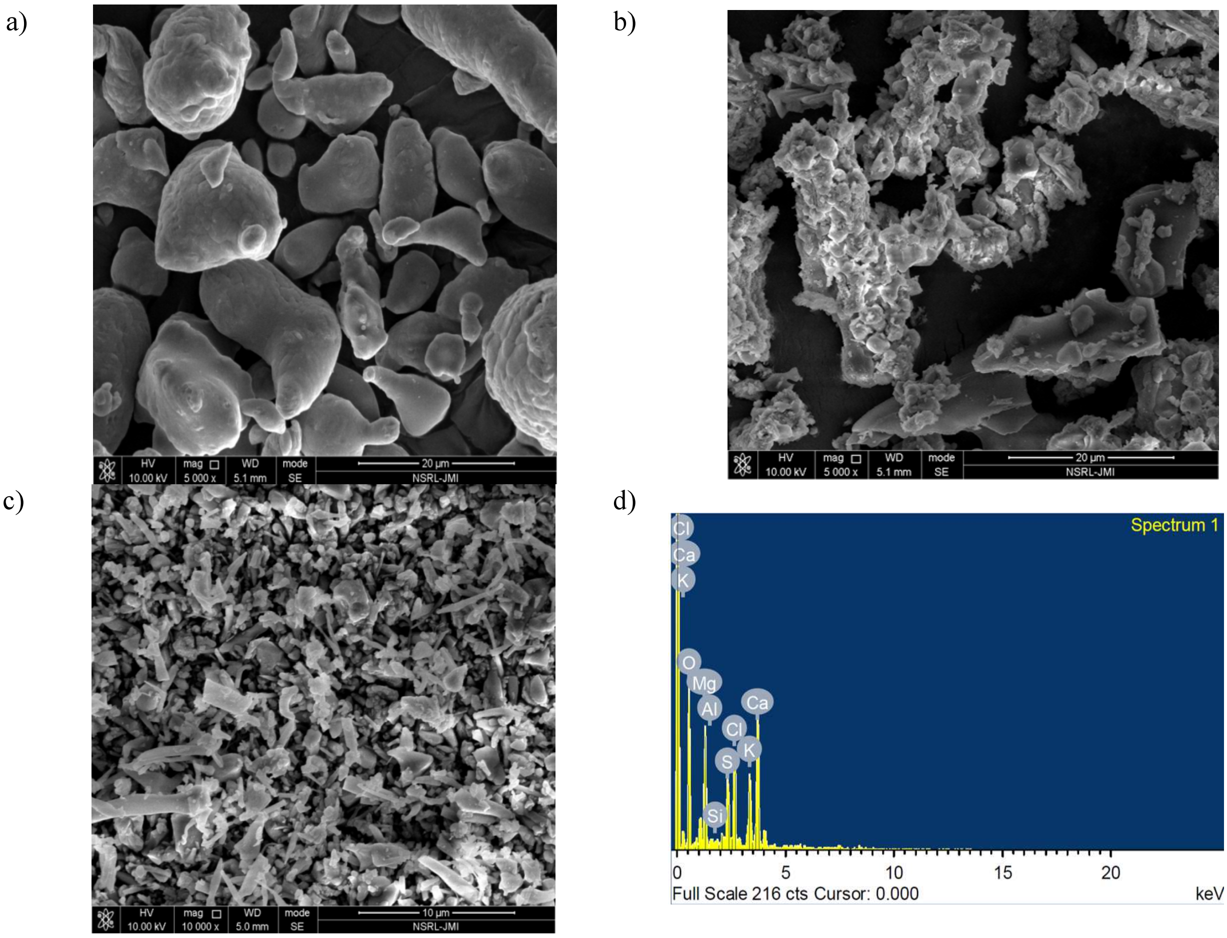


Fig. 2. SEM micrographs: a) aluminium, b) MHA, c) Si_3N_4, d) EDS of MHA

TABLE 4. **Wear test parameters**

Test variables	
Dimensions of test specimen	10 mm x 20 mm
Sliding distance	300 m
Track (diameter)	70 mm
Applied load	25 N, 35 N
Disc RPM	410 rpm
Disc material	EN31 steel, 65 HRC
Temperature, relative humidity	30°C, 60-65%

The test specimens were grinded with grade 1000 emery paper. The EN31 wear test disc was cleaned with acetone to remove any abrasive debris before and after performing the wear test. Every sample was also weighed before starting the wear test. The wear rate was measured by the weight loss of the samples measured by a weighing machine and calculated with the formula discussed in [26]. The wear rate is directly proportional to the average weight and inversely proportional to the density and sliding distance.

CHARACTERIZATIONS

The density of the composite is measured by the Archimedes principle to determine the actual density of the fabricated samples. The mass of the developed sample was calculated in air and, after that, suspended in distilled water. Experimentally, the density is calculated with equation (1) [27].

$$\rho_a = \left\{ \frac{M_a}{M_a - M_d} \right\} \times \rho \tag{1}$$

where:

ρ_a – actual density, ρ_d – density of distilled water, M_a – mass of the sample in air,

$$\rho_{AHC} = \rho_{Al} \times V_{Al} + \rho_{MHA} \times V_{MHA} \tag{2}$$

ρ_{AHC} – density of the composite, ρ_{Al} – density of aluminum, V_{Al} – volume friction of aluminum, ρ_{MHA} – density of mustard husk ash, V_{MHA} – volume friction of mustard husk ash.

The theoretical density of the sintered composites A to E was calculated by the rule of mixtures as given in Equation (2). The microstructure of the powders was analysed by scanning electron microscopy in a nanoscience laboratory (Fig. 2). The sintered composites and XRD pattern are shown in Figure 3. The peaks of Al seen at 38.230, 44.500, 64.850, and 78.220 have the Miller indices of crystallographic planes (111), (200), (220), and (311). The small peaks show phases like alumina silica wollastonite in the sample. With anincrease in the wt.% of MHA in the aluminium, the diffraction peaks of MHA become wider and the peaks also shift slightly from their original position. This process happened by distortion of the reinforcement particles during incorporation in the AMCs. Therefore, it can be concluded that the synthesized hybrid composites are reinforced with MHA and Si_3N_4. The EDS study of the developed composite was also analysed and the peaks of Al, Si_3N_4, as well as SiO_2, MgO, KCl, feldspar and wollastonite were observed. Every sample was weighed before and after the test was performed on the scales to determine the weight loss due to wear. The generated EDS report details of MHA are given in Figure 4.

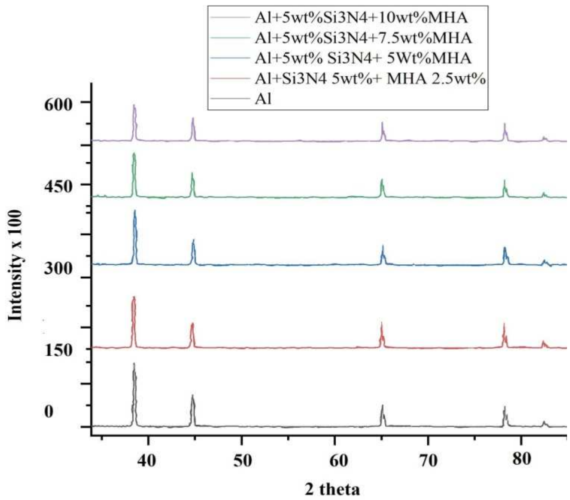
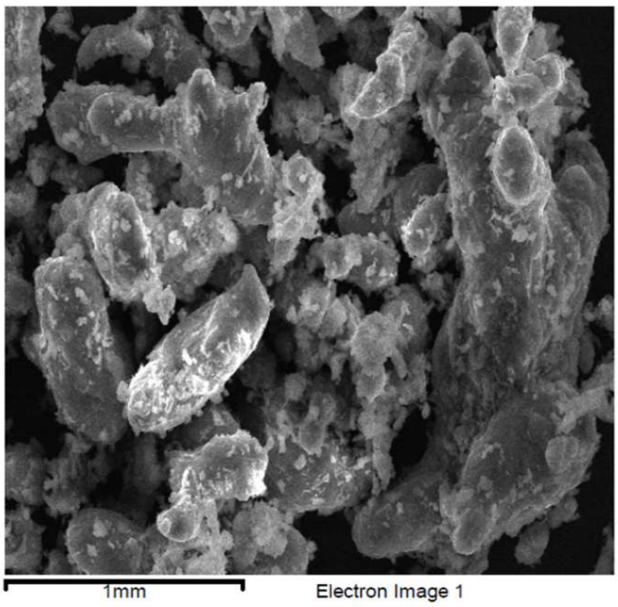
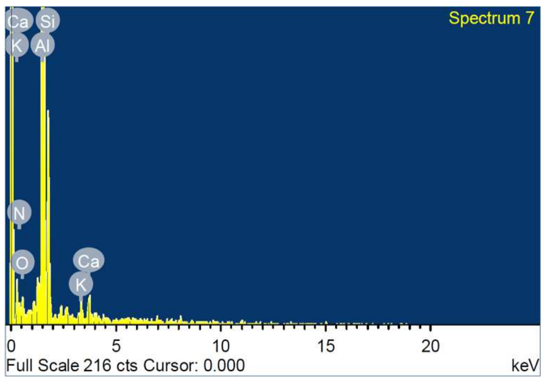


Fig. 3. XRD pattern of produced specimens





Element	Weight [%]	Atomic [%]
NK	12.71	21.29
OK	6.90	10.11
AlK	62.56	54.38
SiK	15.07	12.59
KK	1.17	0.70
CaK	1.59	0.93

Fig. 4. Generated EDS report details of MHA

TRIBOLOGICAL BEHAVIOR

The wear resistance of the proposed composites is enhanced with the increase in the wt.% of reinforcement and pressure. Significant improvement in the wear resistance of composites is a consequence of the high hardness of the ceramic particles present in the reinforcement and the higher density which is achieved by high compaction [27-29]. The effects of changing the wt.% of MHA on the wear loss at different pressure is shown in Figure 5. Similarly, the effect of the reinforcement on the coefficient of friction is given in Figure 6.

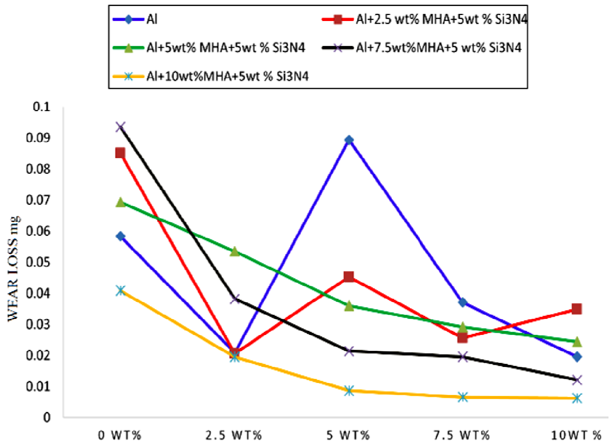


Fig. 5. Effect of varying wt.% of MHA on wear loss at different compaction pressures

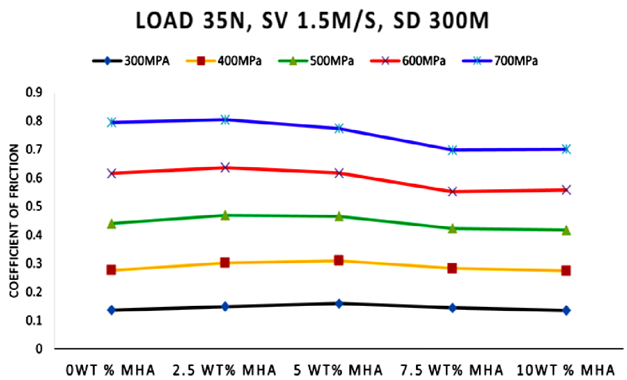


Fig. 6. Impact of reinforcement on coefficient of friction

Influence of reinforcement on coefficient of friction

Increasing the wt.% of mustard husk ash as reinforcement enhances the coefficient of friction. The coefficient of friction improved for two reasons; firstly, the thermal softening effect and secondly, the hard ceramic particles present in the reinforcement. As the compaction pressure is augmented and the wt.% of the MHA reinforcement is increased up to 7.5 wt.%, the coefficient of friction rises. After 7.5 wt.% MHA, there is a slight descent in the coefficient of friction and then it becomes stable after 10 wt.% reinforcement. The test was performed at applied loads of 25 and 35 N, sliding distance 300 m, and sliding velocity 1.5 m/s.

Analysis of worn surfaces

The worn surfaces of the developed composites with 0, 2.5, 5, 7.5 and 10 wt.% MHA were investigated by SEM. Every composition exhibits its own wear mechanism. The wear surface of the developed composites shows pits and grooves. All the SEM micrographs of the surfaces worn under the 35 N load were examined. These SEM micrographs of the developed composites produced at different compaction pressures are given in Figures 7-11. In Figure 7, the wear debris of pure aluminium can be observed on the worn surface. Cracks and pits appear because of plastic deformation during wear. Previous researchers also found similar observations [30, 31]. In the SEM micrographs of the composite with 2.5 wt.% MHA in Figure 8, it can be seen that delamination occurs due to the hard ceramic particles present in MHA. The generation of heat as a consequence of friction leads to plastic deformation and wear particles adhering to the surface are visible [31]. The SEM micrographs of the composite with 5 wt.% MHA revealing a flow of smooth grooves are shown in Figure 9. The conducted observations of the SEM micrographs of the compacts show that the wear resistance rises with increasing compaction pressure. Furthermore, with the increase in compaction pressure and wt.% of reinforcement the hardness improves. The hard ceramic particles present in the reinforcement improve the wear resistance, and formation of an oxide layer takes place. The oxide layer contributes to the high wear resistance, which correlates with the high hardness. The SEM micrographs of the composites with 7.5 wt.% MHA in Figure 10 show fine abraded particles, thin and elongated wear debris, which indicate the low rate of wear. The micrographs of the composite with 10 wt.% MHA presented in Figure 11 clearly show the high wear resistance of wt.% MHA in the developed composites. MHA contains a variety of ceramic particles, including alumina and silica. The mentioned ceramic particles embedded in aluminium metal matrix composites lead to improved wear resistance. MHA exhibits excellent bonding with the aluminium matrix at high compaction pressure.

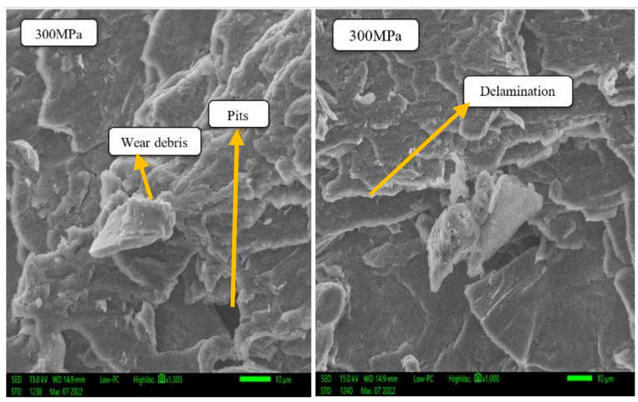


Fig. 7. SEM analysis of 0 wt.% MHA at load 35 N, SD 300 m, SV 1.5 m/s

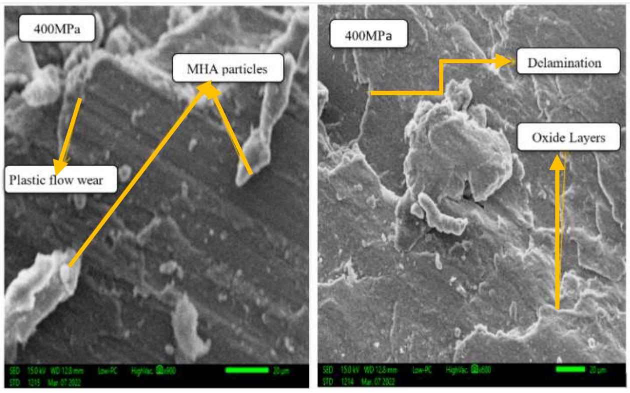


Fig. 8. SEM analysis of 2.5 wt.% MHA at load 35 N, SD 400 m, SV 1.5 m/s

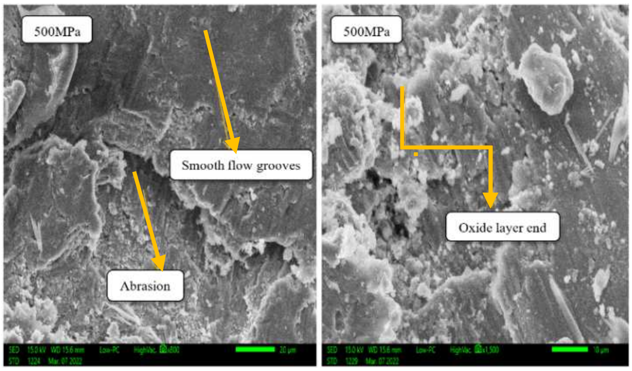


Fig. 9. SEM analysis of 5 wt.% MHA at load 35 N, SD 500 m, SV 1.5 m/s

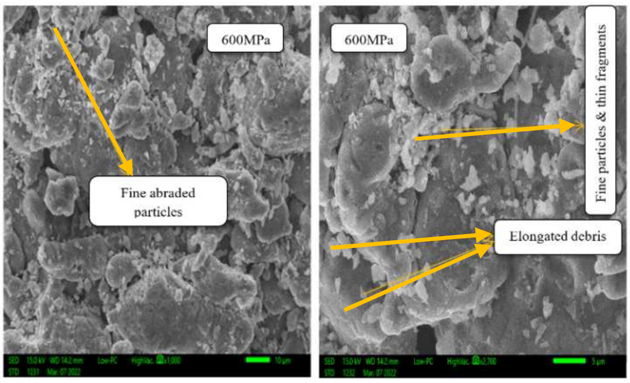


Fig. 10. SEM analysis of 7.5 wt.% MHA at load 35 N, SD 600 m, SV 1.5 m/s

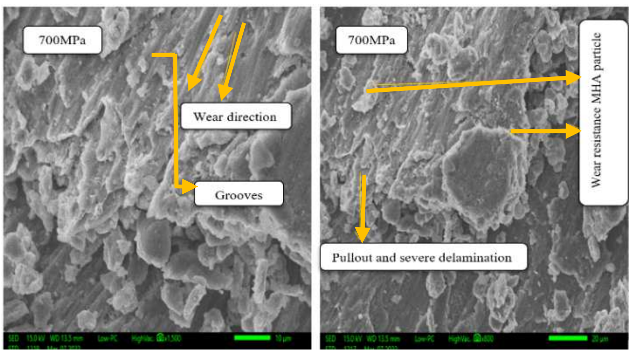


Fig. 11. Analysis of 10 wt.% MHA at load 35 N, SD 700 m, SV 1.5 m/s

CONCLUSIONS

In this work, aluminium metal matrix hybrid composites were developed by the powder metallurgy route at 300-700 MPa compaction pressure with different wt.% of MHA described in Table 3. SEM, XRD and EDS investigations were performed on the powders as well as the produced specimens and the tribological properties were also analysed. The SEM results of the sintered composites validate that the microstructure of the composites is fine, homogeneous, and that the porosity is low, which results in better properties of the composites. The produced Al-5wt.%Si3N4-10 wt.% MHA composite exhibits higher wear resistance as compared to the pure aluminium metal matrix. The presence of hard ceramic particles in the form of SiC and alumina greatly enhances the tribological properties of the composite. This is preferred for higher wear resistance requirements in the automobile sector. Applying mustard husk as reinforcement allows the production of light, less expensive, high strength hybrid composites, instead of conventional reinforcement materials like SiC, alumina, etc. The wear behaviour of the produced composite is directly related to the compaction pressure and sliding distance. The wear loss is reduced by increasing the compaction pressure and wt.% of MHA in the production of hybrid aluminium metal matrix composites.

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