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Tribological Characterization of UHMWPE Composites Reinforced with MoS2 and Graphite: A Comparative Study

G. R. Jagannath[1], Dr. Basawaraj[2], A. J. K. Prasad[3]

[1.] Assistant Professor, RNS Institute of Technology, Bengaluru, Research Scholar Visvesvaraya Technological University, Belagavi.

[2]. Associate Professor, Aerospace Propulsion Technology,Visvesvaraya Technological University, Belagavi. , CGPS, VIAT, Muddenahalli,

[3]. Formerly Associate Professor, Sir MVIT, Bengaluru. Visvesvaraya Technological University, Belagavi.

Abstract

This study presents a comprehensive analysis of the microstructural, physical and tribological properties of Ultra-High Molecular Weight Polyethylene (UHMWPE) composites reinforced with micro MoS2 and nano Graphite. Microstructural evaluations through Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Analysis (EDAX) reveal intriguing insights into the morphology and elemental composition of the reinforcement materials. SEM images unveil the nanoscale hexagonal lattice structure of nano graphite, affirming its suitability for composite applications. EDAX spectra confirm the purity of both nano graphite and micro MoS2, reinforcing their efficacy as reinforcement agents.Density assessments illustrate an increase in composite density with rising reinforcement percentages for both materials, attributed to their inherently higher densities. This increase in density is complemented by reduced porosity, signifying improved packing efficiency within the composite.Wear evaluations under various load and speed conditions elucidate the tribological behaviour of the composites. UHMWPE composites with micro MoS2 exhibit reduced wear rates, frictional forces, and coefficient of friction (COF) compared to neat UHMWPE, emphasizing the effective lubricating properties of MoS2. Conversely, UHMWPE composites with nano graphite outperform micro MoS2 counterparts across all parameters, showcasing superior wear resistance and friction reduction. This is attributed to nano graphite's lamellar structure, which facilitates enhanced lubrication and reduced friction.

Keywords:-*UHMWPE, MoS2, Graphite, microstructural analysis, dry wear analysis, cera-wool blanket and electric band heaters*

1. Introduction

Ultra-High Molecular Weight Polyethylene (UHMWPE) has gained significant attention in materials science and engineering due to its exceptional mechanical properties, which include

high molecular weight, outstanding wear resistance, and remarkable chemical stability (Li et al., 2020; Wu et al., 2019). UHMWPE is renowned for its versatile applications in various industries, ranging from medical devices to automotive components, where its low friction, high abrasion resistance, and biocompatibility make it an attractive choice (Chen et al., 2018; Mokhtar et al., 2019). However, the quest for further enhancing its tribological characteristics has led researchers to explore the incorporation of solid lubricants, such as Molybdenum Disulfide (MoS2) and nano lubricants like Graphite (Borhan et al., 2021; Geng et al., 2017).

MoS2, known for its layered crystal structure and low friction properties, has been widely used as a solid lubricant in various applications (Berman et al., 2016). Additionally, nano-sized Graphite particles have demonstrated remarkable potential in improving the tribological properties of composites (Zhang et al., 2019). By introducing these solid and nano lubricants into the UHMWPE matrix, it becomes possible to create composites with enhanced wear resistance and reduced friction (Li et al., 2021; Yu et al., 2018).

Tribological characterization of such UHMWPE composites is essential to understand how the addition of MoS2 and Graphite influences their performance under different operating conditions. This characterization involves assessing critical parameters such as the specific wear rate, wear loss, and coefficient of friction (COF) (Borhan et al., 2021). These parameters are vital in determining the suitability of these composites for specific engineering applications.

Motivation behind this work arises from the need to optimize UHMWPE-based composites with solid and nano lubricants to meet the demanding requirements of industries where low friction and wear resistance are paramount, such as in automotive, aerospace, and medical devices (Li et al., 2021; Zhang et al., 2019). Understanding how these composites perform under different tribological conditions will facilitate their effective utilization, leading to improved product quality and longevity. This study aims to provide valuable insights into the tribological behaviour of UHMWPE composites reinforced with MoS2 and Graphite, ultimately contributing to advancements in materials engineering and industrial applications.

2. Materials and Methods

2.1 Materials: In this study, several materials were employed to investigate the tribological characteristics of composites. Ultra-High Molecular Weight Polyethylene (UHMWPE) served as the primary matrix material. UHMWPE, known for its exceptional properties, possesses a high molecular weight (typically 2-6 million g/mol) and a relatively low density (approximately 0.97 g/cm^3). Additionally, two distinct reinforcing agents were used to modify the UHMWPE matrix. Micro-sized Molybdenum Disulfide (MoS2) and nano-sized Graphite were directly sourced from Parshwamani METALS Bombay. MoS2, with a density of 2.8 g/cm^3, is renowned for its low friction properties. Nano Graphite, on the other hand, has a density of approximately 1.8 g/cm^3 and is known for its lubricating capabilities. These materials were chosen to create composites with improved wear resistance and reduced friction, aiming to enhance their suitability for various engineering applications.

Fig 1: Powders of a) UHMWPE b) MoS2 and c) Graphite

2.2 Fabrication of composites: The fabrication of Polymer Matrix Composites (PMCs) in this study relied on a well-established manufacturing process called hot compression moulding. This technique blends heat and pressure to shape a mixture of the polymer matrix material, Ultra High Molecular Weight Polyethylene (UHMWPE), and reinforcing particles into the desired form. During this process, the UHMWPE, typically in powdered form, is heated until it melts, allowing it to flow and thoroughly coat the reinforcing particles. The resulting blend is then compressed within a mould cavity to create the desired shape and density.

In the preparation phase, approximately 30 grams of the polymer-reinforcement blend were meticulously measured and placed in a mixing bowl. To ensure a consistent distribution of the matrix and reinforcing materials, dry mixing was initially performed. Following this, a precise amount of acetone (around 10-12 millilitres) was added to the mixture, and wet mixing ensued for 20 minutes to ensure thorough integration.To eliminate any remaining moisture and solvents and prepare the blend for further processing, it underwent drying at a controlled temperature of 180°C ± 2°C in a microwave oven for approximately 15 minutes. The resulting dry mixture was then carefully sealed in plastic covers, each labelled for easy identification during subsequent moulding.

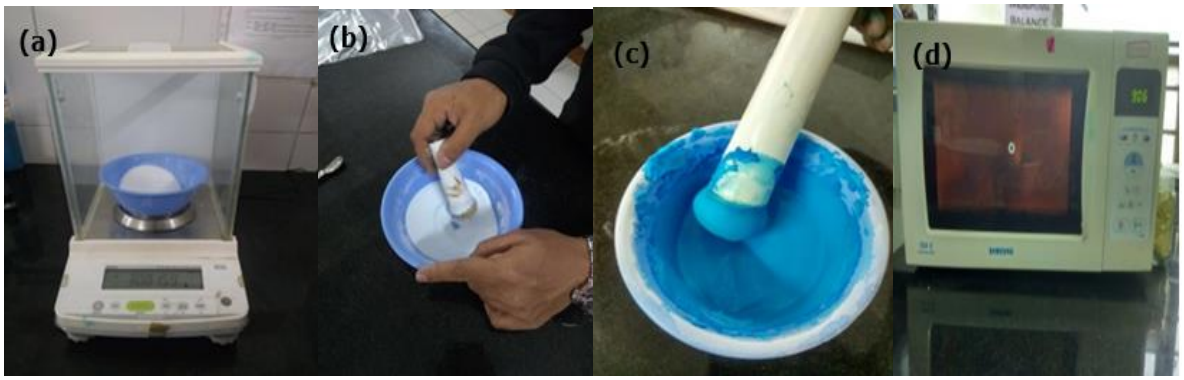

Fig 2: a) Weighing of matrix and reinforcements b) dry mixing c) wet mixing d) microwave oven e) labelling and packing

The actual hot compression moulding involved transferring the dried mixture, comprising UHMWPE and MoS2/graphite, into a multi-cavity die with precision. This loaded die was then securely placed on wooden slabs within a purpose-built mini hydraulic press. To facilitate the moulding process, a band heater, adequately insulated, was positioned around the die, with temperature control ensured by an iron-constantan thermocouple connected to a digital temperature indicator. Pressure application was achieved using a manual bottle hydraulic jack, with real-time pressure monitoring through a pressure gauge.

The fabrication process for UHMWPE composites adhered to rigorous standards, with hot compression moulding carried out at a precisely controlled temperature of 130°C ± 2°C for a specific duration of 90 ± 2 minutes. Electric band heaters were employed to ensure uniform heating across the multi-cavity setup, and to minimize heat loss, wooden slabs were positioned at the base of the die. The band heater was further insulated with a cera-wool blanket secured within a glass fibre cloth.

Photographs provided (see Figures 3 and 4) offer a visual representation of the multi-cavity die, band heater, cera wool cloth, neat UHMWPE samplemini hydraulic table-top press. These images provide clarity to the process. To maintain precision, polymer and lubricant quantities were measured as outlined in Table 1. Once the designated temperature and pressure conditions were met, compression continued for the prescribed duration. Subsequently, power to both the band heater and the digital temperature controller was cut. After cooling to room temperature, the die was carefully opened, and the samples were extracted, diligently stored in self-sealing plastic covers, each meticulously labelled for future testing and analysis.

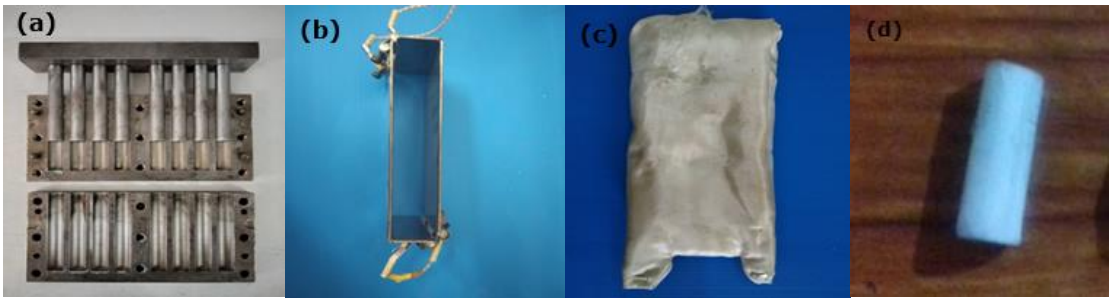

Fig 3: a) multi-cavity die b) band heater c) cera-wool cloth and d) neat UHMWPE sample

Fig 4: a) Hydraulic table-top press and Prepared composite samples

Table 1: Proportion of UHMWPE and reinforcements used

Sl. No.	Reinforcement Weight %	Weight of solid/nano lubricants (grams)	Weight of UHMWPE (grams)	Total Weight (grams)
1	1	0.3	29.7	30
2	2	0.6	29.4	30
3	3	0.9	29.1	30
4	4	1.2	28.8	30
5	5	1.4	28.5	30

2.3. Density measurement: Density, a pivotal property influencing various material characteristics, is defined as mass per unit volume. Understanding the density of the UHMWPE+MoS2 (micro)/Graphite (nano) composite is essential for several reasons. Firstly, it facilitates the calculation of the material's volume. Secondly, it aids in determining the required quantity of the composite for specific applications. Lastly, it enables a comparative analysis of the composite's density with other materials. To measure the density, a digital density scale with a maximum capacity of 5000 g, a resolution of 0.1 g, and an accuracy of ±0.01 g is used. The scale is placed on a level surface and zeroed. The empty sample container is weighed, filled with the composite, and reweighed. By subtracting the container's initial weight from the weight of the filled container, the composite's mass is determined.

Dividing this mass by its volume, which can be calculated by multiplying it by its density, provides the crucial density value. The scale operates within a temperature range of 0 - 40 °C, powered by AC 220-240 V, 50/60 Hz, and has dimensions of 350 x 240 x 120 mm, weighing 3.5 kg.

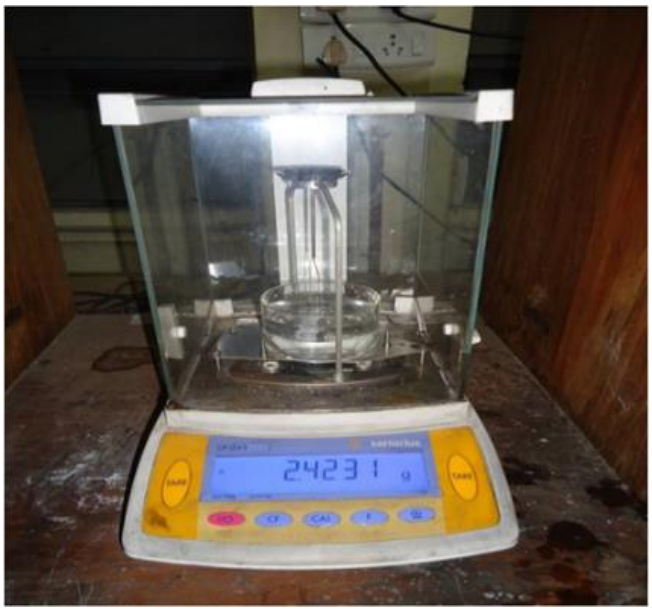

Fig 5: Digital density tester

2.4. Wear analysis: The wear tests, were meticulously conducted using the NOVUS PIN ON DISC WEAR TESTING MACHINE, adhering to the G99 standard. This state-of-the-art machine offers a wide range of technical specifications to ensure precise and comprehensive wear analysis. It encompasses parameters such as normal load, frictional force, wear measurement capabilities, disc speed variation, a robust 2KW AC motor with drive, and a preset timer for test duration. Additionally, the machine provides the flexibility of incorporating a pin heating module, recirculation lubricant system, and an environmental chamber for creating diverse testing environments. Specimen holders, accommodating a range from Ø4 to Ø12, allow for versatility in sample configurations. A data acquisition system, such as the National Instrument, can be optionally integrated to enhance data accuracy. With power requirements of 230V / 50Hz / 1Ø, 2KW, and machine dimensions measuring 3 feet by 2.5 feet by 3 feet, this advanced testing apparatus facilitates the precise determination of wear rate, wear loss, and coefficient of friction (COF) for UHMWPE composites reinforced with MoS2 (micro) and graphite (nano) reinforcements, contributing invaluable insights to the field of materials science and engineering. The table 2 gives the information about the conditions (parameters) selected for the test.

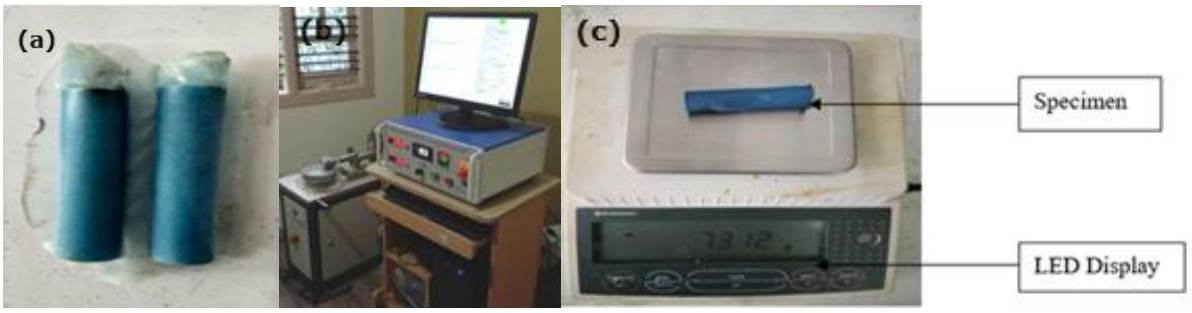

Fig 6: a) Samples for test b) Wear test rig and c) Weighing of samples

Table 2: Parameters considered for test

Sl No	Test Parameter	Value
1	Diameter of the pin specimen, mm	12
2	Length of the pin specimen, mm	36±0.5
3	Track radius, mm	50
4	Sliding speed m/s,& RPM (dry test)	18.84m/min , 22.61m/min,26.37m/min 500RPM, 600RPM and 700RPM,
5	Applied normal load (N)	40, 60 and 80
6	Duration of test (minutes)	10

3. Results and Discussions

3.1 Microstructural characterization

The microstructural characterization of individual elements in the work is done using SEM and EDX to confirm the constituents and the particle size analysis of the powders. The figures 7 gives the information about nano graphite powder considered for the study.

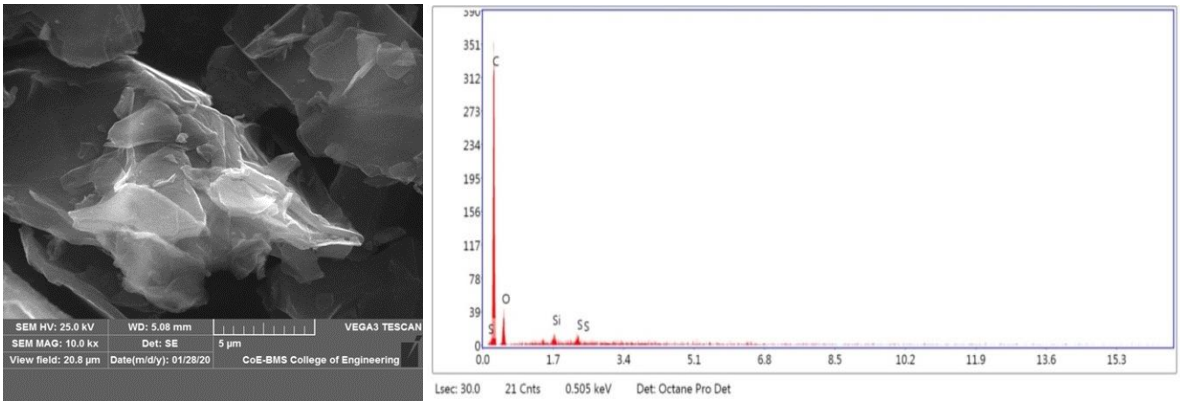

Fig 7: SEM image of graphite powder and EDAX graph of graphite

The SEM image of nano graphite powder reveals a fascinating microstructure characterized by a myriad of fine particles. Under high magnification, these particles exhibit a layered, hexagonal lattice-like structure, characteristic of graphene layers. The image showcases the

intricate arrangement of carbon atoms, highlighting the nanoscale dimensions of the graphite particles.

Complementing this visual examination, the accompanying Energy Dispersive X-ray Analysis (EDAX) graph offers quantitative insights into the elemental composition of the nano graphite powder. Carbon (C), as expected, dominates the composition, reflecting the material's primarily carbonaceous nature. The absence of other significant peaks underscores the purity of the nano graphite sample, further affirming its suitability as a reinforcement material in composite applications. Together, the SEM image and EDAX analysis provide a comprehensive understanding of the nano graphite's morphology and composition, crucial for its effective utilization in composite materials.

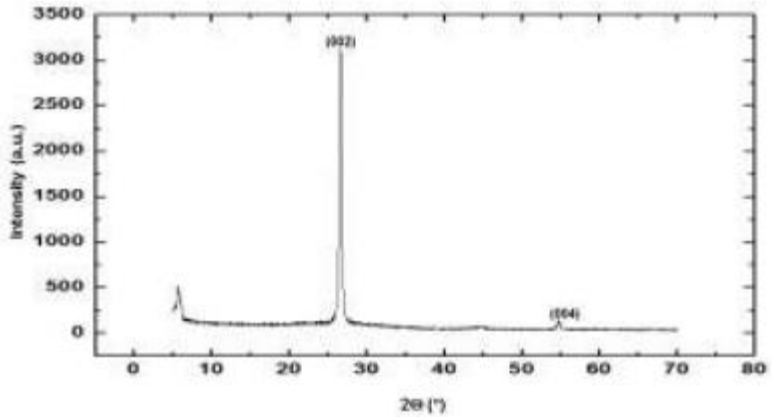

Fig 8: XRD graph of nano graphite powder (as received)

For graphite nano powder, the XRD graph (fig 8) typically displays a series of sharp and intense diffraction peaks, indicating a highly crystalline structure. The most prominent peak corresponds to the (002) plane of graphite, which is a typical feature of well-ordered, layered graphite structures (JCPDF card number 75-1621).To determine the particle size of graphite nano powder from the XRD pattern, the Scherrer equation is employed and the particle size is found to be 70nm.

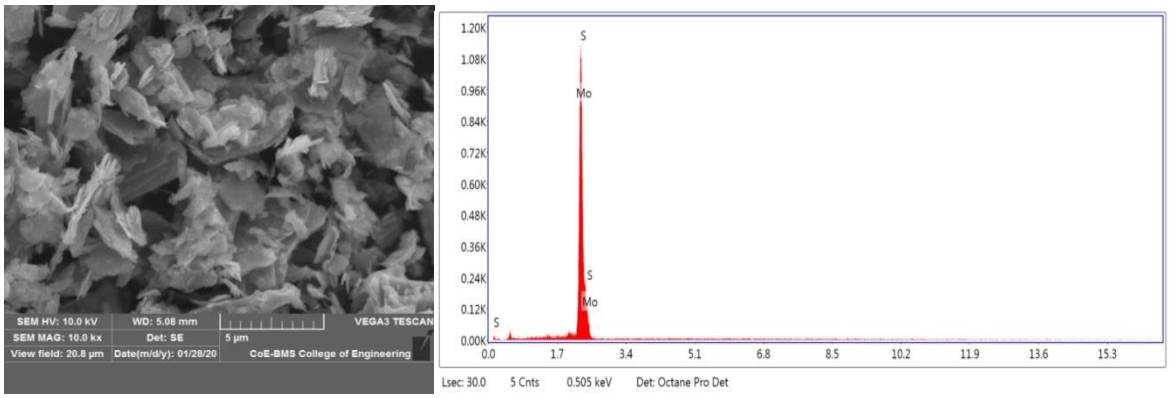

Fig9: SEM image of MoS2 powder and EDAX graph of MoS2

The SEM image of micro MoS2 powder (fig 9) presents a captivating glimpse into the particle-level structure of this solid lubricant. Under high magnification, the image showcases irregularly shaped particles with varying sizes and surface features. The micro MoS2 particles appear as agglomerates, exhibiting a complex surface texture. These structures are indicative of the material's layered crystalline nature, a characteristic feature of molybdenum disulfide.

Complementing the SEM image, the Energy Dispersive X-ray Analysis (EDAX) graph provides valuable quantitative insights into the elemental composition of the micro MoS2 powder. Molybdenum (Mo) and sulphur (S) peaks dominate the spectrum, as expected, confirming the presence of these elements in the material. The absence of other significant peaks underscores the material's purity and highlights its suitability as a solid lubricant. This SEM-EDAX combination not only offers visual information on the microstructure of MoS2 but also verifies its elemental composition, critical for its effective use in enhancing the tribological properties of composite materials.

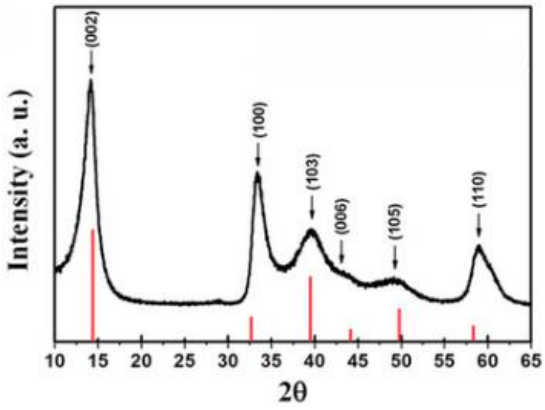

Fig 10: XRD graph for micro MoS2 (as received)

Typically, the XRD pattern for MoS2 micro powder displays multiple peaks. The most prominent peaks are associated with the (002) and (100) planes of MoS2, indicating the crystalline nature of the material. The (002) peak is usually the most intense and sharp, suggesting a well-ordered layered structure in the MoS2 crystal lattice (JCPDF card number 37-1492). Using Scherrer equation is employed and the particle size is found to be 6.8µm.

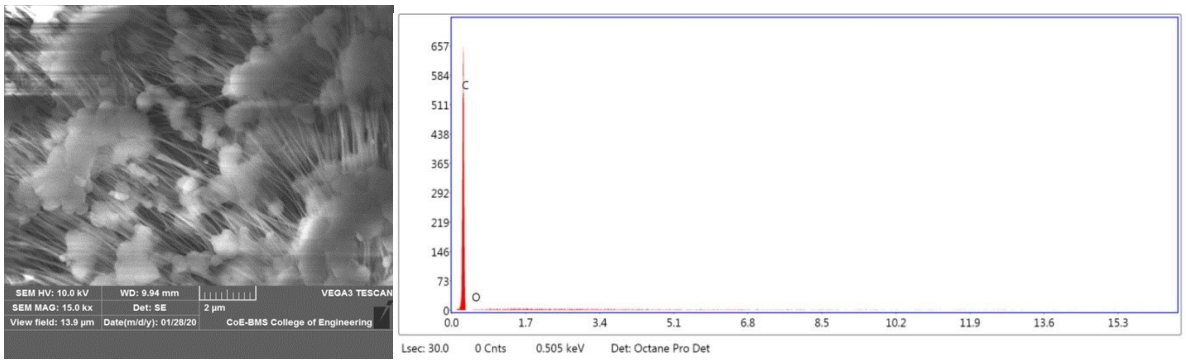

Fig11: SEM image of UHMWPE powder and EDAX graph of UHMWPE

The SEM image of UHMWPEpowder (fig 11) provides a detailed view of the material's microstructure. Under magnification, the image reveals a myriad of irregularly shaped particles, varying in size and exhibiting a rough, textured surface. These particles appear to agglomerate, forming clusters and interlocking structures. The SEM image underscores the granular and porous nature of UHMWPE powder, characteristics that can significantly influence its material properties.

Complementing the SEM image, the Energy Dispersive X-ray Analysis (EDAX) graph offers quantitative data regarding the elemental composition of UHMWPE powder. Carbon (C) peaks dominate the spectrum, as expected for a polyethylene-based material. Additionally, the presence of hydrogen (H) and oxygen (O) peaks is evident, reflecting the chemical composition of the polymer. The absence of other significant peaks indicates the relative purity of the UHMWPE powder. This SEM-EDAX combination provides essential insights into the microstructure and chemical composition of UHMWPE, which are fundamental for understanding and optimizing its performance in composite materials and tribological applications.

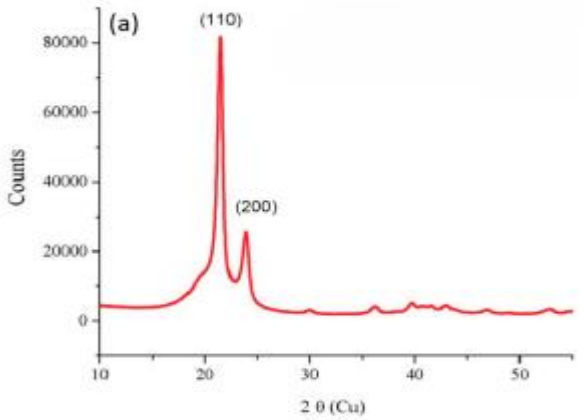

Fig 12: XRD graph of UHMWPE (as received)

Typically, the XRD pattern of UHMWPE powder exhibits a series of sharp peaks, indicating its semi-crystalline nature. The most prominent peak, often seen around 21.4 degrees 2θ (angle of diffraction), corresponds to the (110) crystallographic plane of UHMWPE. This peak is a characteristic feature of UHMWPE and is used for identification purposes.

3.2 Density Measurements

Table 3 Density and porosity values for both the composites

Reinforcement %	Density-g/cm3 (micro MoS2)	Porosity-% (micro MoS2)	Density-g/cm3 (nano graphite)	Porosity-% (nano graphite)
0	0.95	1.68	0.95	1.57
1	2.41	1.16	1.45	1.02
2	2.85	1.11	1.53	0.94
3	2.92	1.12	1.67	0.9

4	3.26	0.96	1.79	0.86
5	3.48	0.84	1.92	0.79

The provided data in the table 3 and figures 13 and 14 illustrates the density and porosity variations in UHMWPE composites with micro MoS2 and nano graphite reinforcements at different reinforcement percentages. Upon analyzing the values, some notable trends and differences between the two composites emerge.

In the case of UHMWPE composites with micro MoS2, there is a steady increase in density as the reinforcement percentage rises from 1% to 5%. This trend can be attributed to the relatively high density of micro MoS2, which is 5.06 g/cm³. As the proportion of this denser material increases in the composite, it naturally results in a higher overall composite density. Conversely, the porosity of the composites with micro MoS2 exhibits a slight decrease as the reinforcement percentage increases. This can be attributed to improved packing efficiency as more micro MoS2 is added, reducing the void spaces within the composite structure.

On the other hand, in the UHMWPE composites with nano graphite, a similar pattern is observed but with distinct values. The density increases as the reinforcement percentage grows, primarily due to the relatively high density of nano graphite, which is 2.26 g/cm³. As with micro MoS2, the increase in density is a consequence of incorporating a denser material into the composite. Additionally, the porosity of the composites with nano graphite decreases as the reinforcement percentage rises, indicating improved packing efficiency and reduced void spaces within the composite structure.

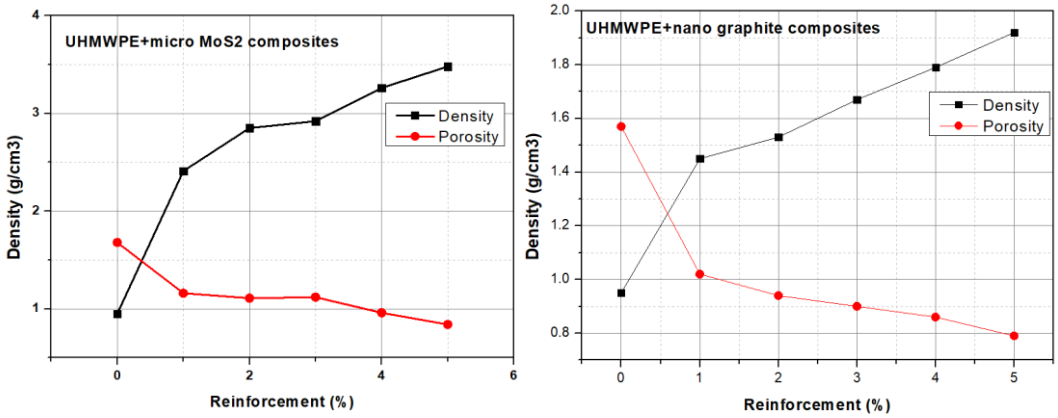

Fig 13 and 14: Density and porosity variations in UHMWPE+micro MoS2 and UHMWPE+Nano graphite composites

3.3 Wear Analysis

The wear analysisconducted on the UHMWPE composites reinforced with MoS2 (micro) and graphite (nano) involved a systematic exploration of different load and speed conditions to assess the tribological behavior of these materials. The study focused on three distinct load conditions: 40N, 60N, and 80N, and three different speed conditions: 500rpm, 600rpm, and 700rpm to study the wear rate, frictional force and COF. To facilitate a comprehensive

analysis, the experimental design kept the speed constant while varying the loads for each speed value.

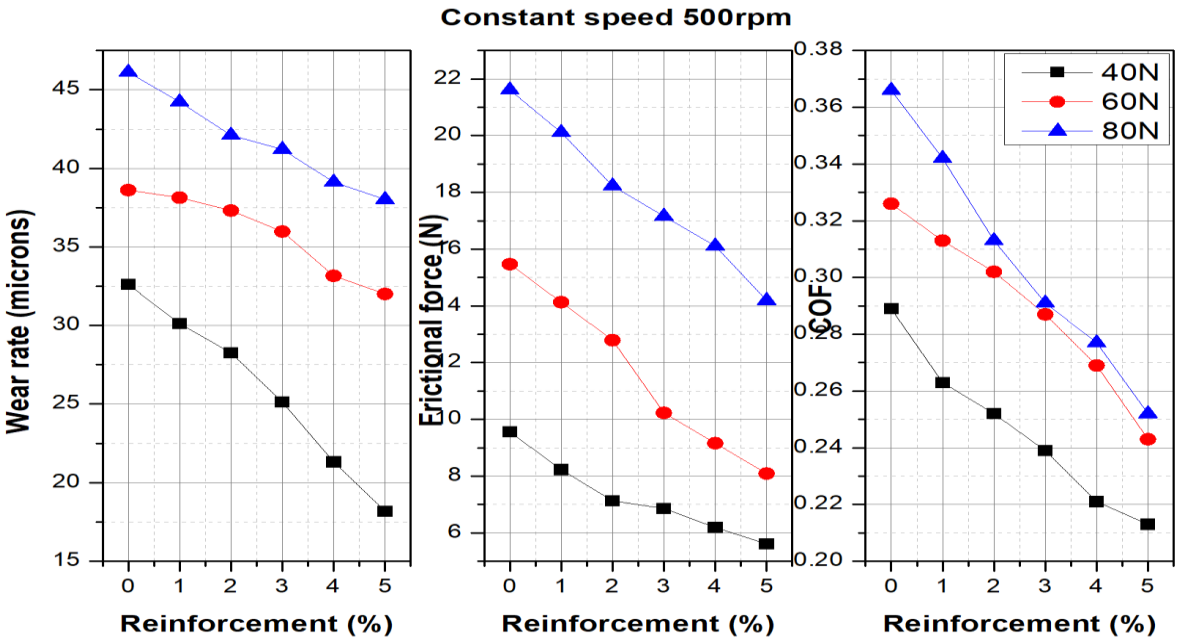

Fig 15: Wear analysis of UHMWPE+MoS2 composites for 500rpm speed

The presented results (fig 15) for wear rate, frictional force, and coefficient of friction (COF) under varying load conditions (40N, 60N, and 80N) and constant speed (500rpm) for UHMWPE composites reinforced with micro MoS2 provide valuable insights into the tribological behaviour of these materials.Firstly, when examining the wear rate, it's evident that as the load increases, the wear rate also tends to increase. This is a typical behaviour observed in tribological studies, where higher loads lead to increased material deformation and contact stresses, resulting in more significant wear. However, a noteworthy observation is the influence of micro MoS2 reinforcement on wear rate. As the percentage of micro MoS2 in the composite increases, there is a consistent reduction in wear rate. This reduction can be attributed to the excellent lubricating and anti-wear properties of MoS2. Its presence in the composite forms a solid lubricating film between the sliding surfaces, reducing direct contact and thus lowering wear.Secondly, the frictional force data follows a similar trend as the wear rate. Higher loads result in increased frictional forces, which is expected due to the greater contact pressures. However, the inclusion of micro MoS2 also leads to a reduction in frictional forces, particularly notable at higher load levels. This reduction can again be attributed to the lubricating effect of MoS2, which reduces the resistance to sliding and hence frictional forces.Thirdly, the COF values demonstrate that, as expected, higher loads correspond to higher COF values. This indicates increased resistance to sliding. However, the incorporation of micro MoS2 consistently leads to lower COF values, especially at higher loads. This is indicative of reduced friction and smoother sliding behaviour due to the presence of MoS2, which acts as an effective solid lubricant.

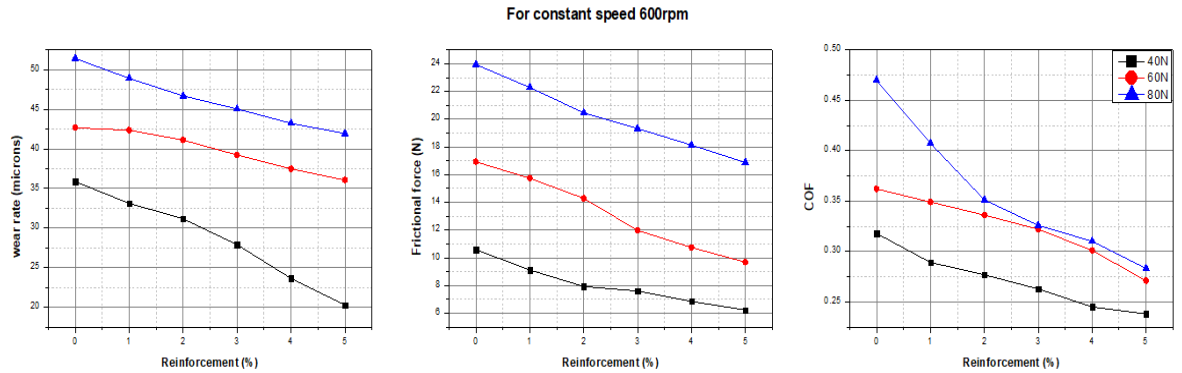

Fig 16: Wear analysis of UHMWPE+MoS2 composites for 600rpm speed

The results of wear rate and frictional force at 600rpm for UHMWPE composites reinforced with micro MoS2 (% ranging from 0 to 5%) under different loads (40N, 60N, and 80N) provide valuable insights. As the load increases, we observe a consistent trend of higher wear rates, which is a typical behaviour in tribological studies. This phenomenon can be attributed to the increased contact pressure between the sliding surfaces, leading to more material removal and thus higher wear.Conversely, the addition of micro MoS2 to the UHMWPE matrix shows a clear trend of reducing wear rates across all load conditions. This reduction can be attributed to the solid lubricating properties of MoS2, which act as a protective barrier between the sliding surfaces. As the percentage of micro MoS2 increases, this lubricating effect becomes more pronounced, resulting in lower wear rates.In terms of specific values, when we compare the wear rate at 0% micro MoS2 reinforcement and 5% micro MoS2 reinforcement under the 80N load condition, we see a substantial reduction from 51.42 microns to 41.95 microns. This demonstrates the effectiveness of micro MoS2 in enhancing wear resistance.

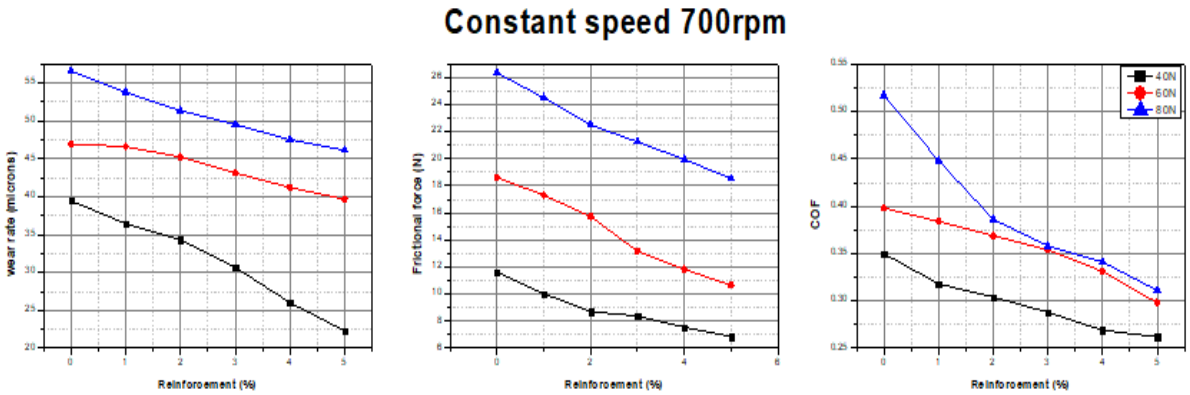

Fig 17: Wear analysis of UHMWPE+MoS2 composites for 700rpm speed

Similar trend of values can be seen for 700rpm also but the values are quite high about 10-12% which is obvious due to the increase in sliding speed and also the values of the considered parameters are seen to be decreased due to the addition of micro MoS2. In summary, the incorporation of micro MoS2 in UHMWPE composites proves to be effective in reducing wear rates and improving tribological performance under various operating

conditions, regardless of rotational speed or load. These findings highlight the potential of micro MoS2 as a valuable reinforcement for enhancing wear resistance in composite materials.

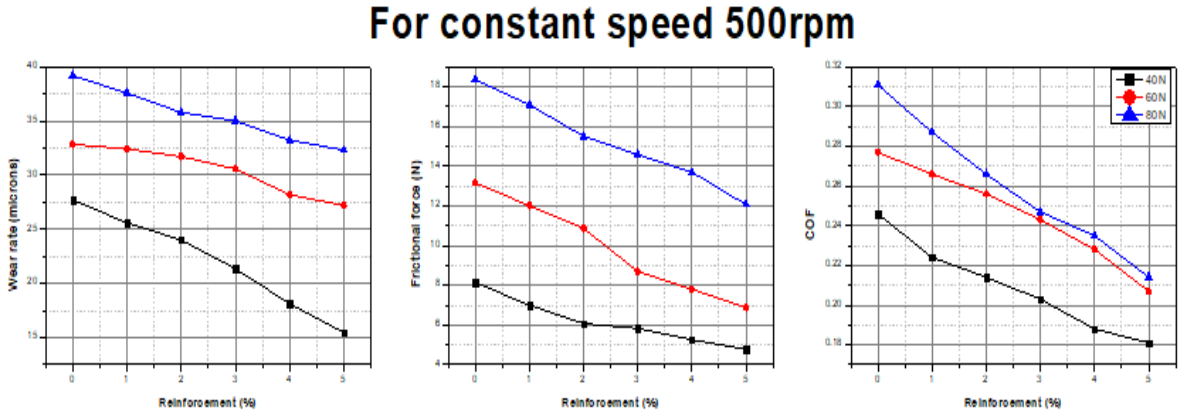

Fig 18:Wear analysis of UHMWPE+Graphite composites for 500rpm speed

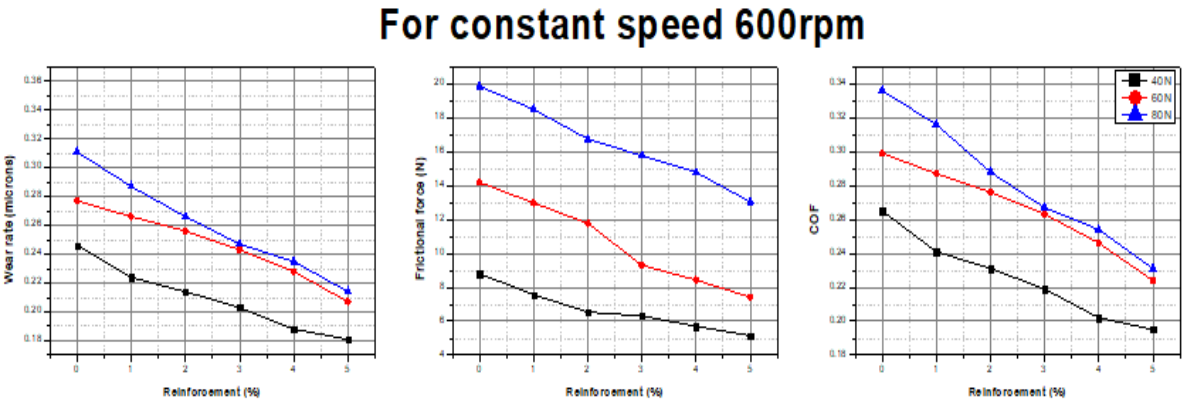

Fig 19:Wear analysis of UHMWPE+Graphite composites for 600rpm speed

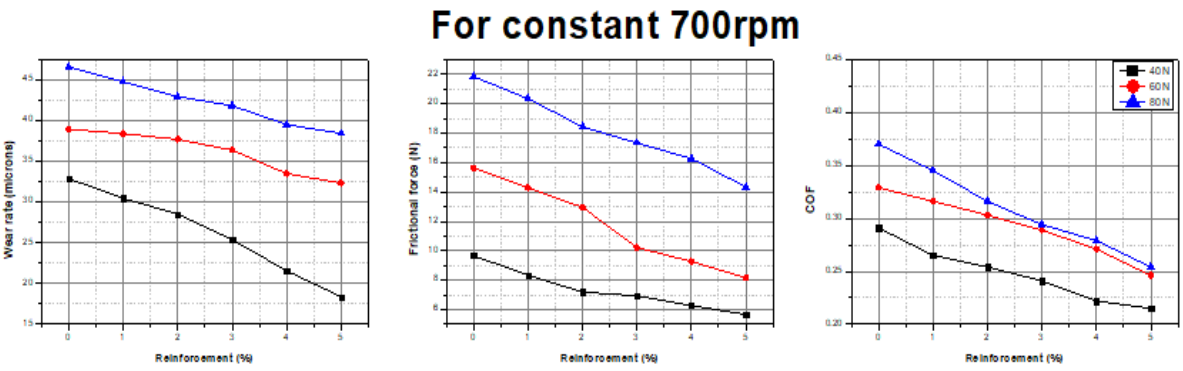

Fig 20:Wear analysis of UHMWPE+Graphite composites for 700rpm speed

The presented data (fig 18, 19 and 20) reveals valuable insights into the tribological properties of UHMWPE composites reinforced with nano graphite under varying conditions. It is evident that as the percentage of nano graphite reinforcement increases, the wear rate,

frictional force, and COF all tend to decrease. This is attributed to the improved lubrication and reduced frictional contact between the sliding surfaces due to the introduction of graphite particles. Graphite, with its unique lamellar structure, acts as a solid lubricant, reducing the direct contact between UHMWPE and the counter surface, thereby lowering wear and friction. The decrease in these parameters with increasing graphite content suggests that the distribution of the reinforcement within the UHMWPE matrix is relatively homogeneous. This even dispersion ensures that the benefits of graphite's lubricating properties are consistently realized across the composite material.Moreover, the increase in wear rate, frictional force, and COF with higher loads and speeds is expected behaviour in tribological studies. Increased loads result in greater contact stresses and higher frictional forces, leading to elevated wear rates and COF. Similarly, higher speeds intensify the relative motion between the sliding surfaces, also contributing to increased wear and friction.

Comparing the results of wear rate, frictional force, and COF for UHMWPE composites reinforced with micro MoS2 and nano graphite at different speeds (500, 600, and 700rpm), it becomes evident that nano graphite is the superior reinforcement material in terms of wear resistance in UHMWPE. When looking at the wear rate, UHMWPE composites with nano graphite consistently exhibit lower wear rates compared to those with micro MoS2 across all load and speed conditions. This indicates that nano graphite provides a more effective reduction in wear, which is a crucial factor in tribological applications. The fine particle size and lamellar structure of nano graphite allow for better lubrication and reduced friction, resulting in improved wear resistance. In terms of frictional force, UHMWPE composites with nano graphite also consistently demonstrate lower frictional forces, which aligns with the reduced wear rates. The reduced friction is attributed to the solid lubricating properties of nano graphite, which facilitates smoother sliding and less energy dissipation as heat.Additionally, the coefficient of friction (COF) values for UHMWPE composites with nano graphite are generally lower than those with micro MoS2. This is another indicator of the superior lubricating ability of nano graphite, resulting in a more efficient reduction in friction.The reasons behind the superior performance of nano graphite can be attributed to its unique structural properties. Nano graphite consists of thin, flat particles that easily slide over one another, providing excellent lubrication and reduced friction. In contrast, micro MoS2, while effective to some extent, may not achieve the same level of reduction in wear and friction due to its larger particle size and different structural characteristics.

4. Conclusion

- Microstructural Analysis: SEM images and EDAX analysis of nano graphite and micro MoS2 reveal their respective structures and purity, essential for their effectiveness as reinforcement materials.
- Density and Porosity: Both nano graphite and micro MoS2 composites show increased density and reduced porosity with higher reinforcement percentages, attributed to the dense nature of these materials.

- Wear Analysis (Micro MoS2): Increasing micro MoS2 content reduces wear rates, frictional forces, and COF, thanks to MoS2's solid lubricating properties, improving tribological performance.
- Wear Analysis (Nano Graphite): Nano graphite composites consistently exhibit lower wear rates, frictional forces, and COF across various conditions, highlighting their superior lubrication and wear-resistant characteristics.
- Nano Graphite vs. Micro MoS2: Nano graphite outperforms micro MoS2 in wear resistance due to its fine particle size and lamellar structure, providing efficient lubrication and reduced friction.
- Overall Conclusion: Nano graphite is the superior reinforcement material for enhancing wear resistance in UHMWPE composites, offering better tribological performance compared to micro MoS2 across varying load and speed conditions.

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