Tribological Behaviour of Carbon Fibre Polymer Composites Reinforced with Nano-fillers

Shylesh K. Siddalingappa, Bhaskar Pal, M. R. Haseebuddin and K. Gopalakrishna

Abstract Nano-fillers reinforced with polymer-based composites have found to enhance the tribological properties in polymer composites. Alumina $(A₁, O₃)$ and molybdenum-di-sulphide $(MoS₂)$ were selected as nano-fillers in the present research work. Al_2O_3 and MoS_2 fillers were mixed with the epoxy resin in the concentration of 0.1, 0.2, 0.4 and 0.7% by volume of the epoxy resin. The reinforced epoxy resin was used as a matrix material along with the aircraft grade carbon fabric as the primary reinforcement. Test panels were fabricated using vacuum-assisted resin transfer moulding technique (VARTM), and samples were extracted using water jet cut as per the dimensions given in the ASTM standards of three-body wear (ASTM G-65) and two-body wear (ASTM G-99). Scanning electron microscopic (SEM) studies revealed the even distribution of the $MoS₂$ when disbursed with ultrasonicator than by manual mixing. The three-body and two-body wear tests were carried out at a load of 24 and 48 N. The composite reinforced with 0.4% of MoS₂ particulate has shown the decrease in wear rate by 20.45% as compared to that of the Al_2O_3 particulate-based composites in three-body wear test when the applied load was 48 N, whereas in two-body wear test, the composite reinforced with 0.4% of MoS₂ particulate has shown the decrease in wear rate by 16.13% as compared to that of the Al2O3 particulate-based composites. SEM studies of worn-out surfaces revealed that the mode of failure in worn-out samples was due to delaminate at the fibre matrix interface. SEM images also revealed that better bonding between fibre and matrix can be well achieved for the 0.4% concentration of nano-reinforcements as compared to that of the composites with 0.2% concentration of nano-reinforcements.

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1 Introduction

Carbon fibre-reinforced polymer composites command the major share in the structural polymer composite industry. It is widely used for various applications such as aerospace, aeronautical, automobile, locomotive, and recreational applications. Amongst the various advantages, the polymer composites offer one noticeable advantage which is its flexibility in allowing the user to tailor the properties as per the end application needs. The few amongst them are higher strength-to-weight ratio, higher stiffness and higher fatigue resistance. Profound tests were carried out to determine the array of properties related to structural as well as tribological properties of the carbon fibre-reinforced polymer composites [\[1\]](#page-9-0). However, the research work to evaluate the significance of nano-sized reinforcements on the tribological properties of the carbon fibre-reinforced polymer composites has not been dealt in an intense manner [\[2\]](#page-9-1). Hence, this research work aimed at studying the significance of reinforcing the carbon fibre-reinforced composites with alumina $(A₁O₃)$ and molybdenum-disulphide $(MoS₂)$ separately and compared their effect on the tribological properties based on test results that obtained by three-body wear tests and two-body wear tests.

2 Literature Review

GuangShi et al. had assessed the impact of nano-sized alumina fillers on the sliding wear behaviour on the epoxy-based polymer composites. The researchers were successful in reducing the specific wear rate by 97% when the volume of the nanoalumina used was 0.24 vol.% than compared to the bare epoxy resin [\[3\]](#page-9-2). B Suresha et al. evaluated the tribological properties of carbon–epoxy and glass–epoxyreinforced polymer composites using pin-on-disc apparatus. It was found that carbon–epoxy samples showed lower friction and lower slide wear loss [\[3,](#page-9-2) [4\]](#page-9-3). A Shafiei-Zarghani et al. had developed the Al_2O_3 nano-composite surface layer on an Al alloy using friction stir welding (FSP). FSP made significant enhancement in the mean microhardness of the nano-composite layered composite by three times as compared to the bare aluminium substrate [\[5\]](#page-9-4).

V.P. Gordienko et al. studied the significance of adding additives less than one micrometer $(1 \mu m)$ with polyolefin such as polyethylene and polypropylene by hot pressing under a pressure of 35 MPa for 20 min by adding inorganic additive molybdenum-di-sulphide in the range of 0.1 to 10% by volume. It was found that there was increase in the hardness due to the addition of molybdenum in a tracer quantity of 0.3 to 0.5% which enhanced the wear resistance of the composite by 8 to 10% [\[6\]](#page-9-5). Zhu Peng et al. conducted the study of the tribology performance

of molybdenum-di-sulphide reinforced thermoplastic polyamide under dry and wet lubrication conditions. It was found that mechanical performances were superior the thermoplastic filled with molybdenum-di-sulphide [\[7\]](#page-9-6). P. H. Sivaraman et al. studied the influence of molybdenum-di-sulphide composition on the wear characteristics of the composites fabricated. SEM images were used to study the worn surfaces. It was found that the there was a significant improvement in the composite properties which were doped with molybdenum-di-sulphide than compared with that of the bare composite [\[8\]](#page-9-7).

From the above discussion, it was concluded that there is enough documented research work carried out in fabrication polymer-based nano-composites using nanosized alumina and nano-sized molybdenum-di-sulphide. Most of the work involved fabricating the samples from hand layup and compression moulding. But no evident was found about fabrication of composite using VARTM method and testing the composites with three-/two-body abrasive wear test and traditional pin-on-disc apparatus. Hence, the present research work concentrated on developing the VARTM fabricated nano-composites reinforced with nano-sized filler in different proportions and conducting three-body abrasive wear and two-body abrasive wear for performance study.

3 Materials and Method of Fabrication

Structural grade carbon fabric used as a primary fibre and epoxy resin HTR 212 were mixed with hardener HTR-386-99 for preparing the composites. These materials were procured from Aircraft Spruce, USA. It is noted from the supplier that the size of alumina $(A₁, O₃)$ particles is about 18 nm and the size molybdenum-di-sulphide $(MoS₂)$ particle is in range of 80 nm–100 nm with 99.9% purity. These particles were used as nano-fillers in the volume of 0.1, 0.2, 0.4 and 0.7% to develop nano-fillerreinforced polymer composites. Vacuum-assisted resin transfer moulding (VARTM) method was used for fabricating the composite panels. The ultrasound powered ultrasonicator is used for uniform distribution of the nano-fillers in the pure epoxy resin. The whole set-up is shown in Fig. [1.](#page-2-0) Panels were fabricated resembling the size of the VARTM mould, and samples were extracted by water jet cutting as per the

Fig. 1 Vacuum-assisted resin transfer moulding

ASTM standards. The photographs of the extracted samples are as shown in Fig. [2.](#page-3-0) The scanning electron microscope (SEM) studies were conducted to understand the significance of using the ultrasonicator to mix the nano-reinforcements into the epoxy resin as compared to that of the manual mixing. The SEM micrographs reveal that $MoS₂$ nano-filler in the epoxy resin is evenly distributed when it is mixed with ultrasonicator. The SEM micrographs also disclose that the porosity level is almost nil due to ultrasonication.

4 Experimental Testing

4.1 Three-Body Abrasive Wear Test

Three-body abrasive wear tests were conducted in accordance with ASTM G-65. The equipment used for conducting the three-body wear test is shown in Fig. [3.](#page-4-0) The initial weight of the specimen was recorded. The velocity and test time were set. Abrasive particles from the hopper were introduced between the test specimen and rotating cholorobutyl rubber tyre abrasive wheel (hardness; Durometer-A 58-62). At the end of the set duration, specimen was removed and cleaned thoroughly. The final weight of the tested specimen was recorded for measuring the wear loss. The above procedure was repeated for varying abrading radius for different concentrations of the nano-fillers. These experiments were carried out at two different loads, i.e., 24 N and 48 N [\[8\]](#page-9-7).

Fig. 3 Three-body abrasive wear test set-up

4.2 Two-Body Wear Test

Two-body abrasive wear tests were conducted in accordance with ASTM G-99. The rotating disc of the apparatus was cleaned using the acetone to remove any dirt, grease or any material debris attached from the previous test. Initial weight of the sample was measured and recorded, and the specimen was fixed to the sleeve based upon the preselected abrading radius. The specimen was loaded by the predefined magnitude of weight and brought in contact with the rotating disc. As the machine was switched on, the specimen in contact with the rotating disc abraded gradually. The test was performed for a fixed duration of time as per standard, and finally, the weight of the specimen measured after the test was conducted, thus measuring the wear loss. The above procedure was repeated for varying abrading radius for different concentrations of the nano-fillers used at two different weights of 24 N and 48 N [\[9\]](#page-9-8) (Fig. [4\)](#page-4-1).

Fig. 4 Two-body abrasive wear test set-up: 1. Pin holder with screw, 2. specimen with holder, and 3. rotating disc

Fig. 5 Scanning electron microscope images showing the distribution of the reinforcement in the composite structure when the reinforcements were mixed manually (**a**) and by ultrasonicator (**b**)

5 Results and Discussions

5.1 Scanning Electron Microscope (SEM) Studies

Figure [5](#page-5-0) shows the SEM images of M_0S^2 dispersement into the epoxy resin matrix done manually as well as by using the ultrasonicator. The figure reveals composites developed by the dispersing the reinforcements by manually mixing (Fig. [5a](#page-5-0)) is more porous that the dispersement done using the ultrasonicator (Fig. [5b](#page-5-0)).

5.2 Three-Body Wear Test

The plots shown in Figs. [6](#page-5-1) and [7](#page-6-0) show the variation of wear rate against the abrading distance for the various reinforcements $(Al_2O_3$ and $MoS_2)$ used at an operating load of 24 N and 48 N, respectively. The three-body wear test was conducted at abovementioned loads at various $(0.0, 0.1, 0.2, 0.4 \text{ and } 0.7\%)$ concentrations of Al_2O_3 and $MoS₂$. In the plots shown in Figs. [6](#page-5-1) and [7,](#page-6-0) it can be observed that the wear rate was

Fig. 6 Three-body abrasive wear test results showing the variation of wear rate against the abrading distance for different concentrations of Al_2O_3 and MoS_2 at 24 N

Fig. 7 Three-body abrasive wear test results showing the variation of wear rate against the abrading distance for different concentrations of $A₁$ O₃ and MoS₂ at 48 N

directly proportional to the abrading distance; hence, the gradual increase in the wear rate can be observed for all the concentrations of reinforcement studied irrespective of whether it was Al_2O_3 reinforced composites or MoS_2 reinforced composite. But however the detailed observation of the plots demonstrated that the wear rate in $MoS₂$ reinforced composites is substantially lower as compared to that with composited reinforced with Al_2O_3 . For instance, at 0.4% concentration Al_2O_3 sample wear rate was 0.135 units whereas that of the $MoS₂$ was 0.1 units; i.e., the wear resistance of the $MoS₂$ was approximately 25% more than that of the $Al₂O₃$ reinforced composites. This behaviour of the material could be attributed for the fact that Al_2O_3 being a hard reinforcement gets wore out and material gets removed from the specimens in the form of flakes; hence, the volume of material removed is higher, whereas in the case of MoS₂, the very nature of the MoS₂ is to act as a lubricating agent which in turn reduces the friction during the abrasion and also $MoS₂$ is a soft reinforcement; hence, the material gets removed in the form of small powders, thereby reducing the rate of the material removal and hence the lesser wear rate. The behaviour of the samples tested at a load of 48 N has shown the similar trend.

5.3 Two-Body Wear Test

With reference to the above discussion in Sect. [5.2,](#page-5-2) the results obtained in the twobody wear test are shown in the plots given in Figs. [8](#page-6-1) and [9.](#page-7-0) The results showed

Fig. 8 Two-body abrasive wear test results showing the variation of wear rate against the sliding distance for different concentrations of Al_2O_3 and MoS_2 at 24 N

Fig. 9 Two-body abrasive wear test results showing the variation of wear rate against the sliding distance for different concentrations of Al_2O_3 and MoS_2 at 48 N

the similar trends as that in case of three-body wear test. However, the wear rate obtained was far more less as compared to that of the three-body wear results. The abnormalities in the last plot shown in Figs. [8](#page-6-1) and [9](#page-7-0) can be attributed for the reason of loose gripping of sample in the pin-on-disc machine.

5.4 Surface Morphology

Figures [10](#page-7-1) and [11](#page-8-0) show the SEM images of the worn-out surfaces of the composites with Al_2O_3 and MoS_2 as nano-reinforcements with 0.2% and 0.4% of the concentrations. The severity of the wear in composites with 0.2% alumina is more than compared to that of composites reinforced with 0.2% MoS₂. In the SEM micrographs shown in Fig. [10a](#page-7-1), b it is evident that the failure is due to delamination at the fibre matrix interface. However, in the composites reinforced with 0.4% of Al_2O_3 and

Fig. 10 Scanning electron microscope images showing the abraded surfaces of composites reinforced with 0.2% Al₂O₃ concentration (**a**) and composites reinforced with 0.2% MoS₂ concentration (**b**) during three-body wear test with an applied load of 48 N

Fig. 11 Scanning electron microscope images showing the abraded surfaces of composites reinforced with 0.4% Al₂O₃ concentration (a) and composites reinforced with 0.4% MoS₂ concentration during three-body wear test with an applied load of 48 N

 $MoS₂$ as nano-reinforcements, the bonding is better as compared to the composites with 0.2% of the nano-reinforcements of Al_2O_3 and MoS_2 as shown in Fig. [11a](#page-8-0), b.

6 Conclusions

- i. Composite panels with A_2O_3 and MoS_2 nano-fillers were fabricated using vacuum-assisted resin transfer moulding (VARTM) in the varying concentrations discussed above, and performance study has been carried out in three-body and two-body abrasive wear test.
- ii. The nano-reinforcement of $MoS₂$ was well distributed by the use of ultrasonicator as shown in the figure.
- iii. The results obtained clearly depicted that composites reinforced with $MoS₂$ showed better wear resistance at all the concentrations studied as compared to that of the composites reinforced with Al_2O_3 nano-filler.
- iv. The Al_2O_3 being a hard reinforcement gets wore out in the form of flakes; hence, the wear rate is more as compared to $MoS₂$, which by very nature acts as a lubricating agent which in turn reduces the friction during the abrasion as shown in Figs. 6.4 and 6.5.
- v. $MoS₂$ is a soft reinforcement; hence, the material gets removed in the form of small powders, thereby reducing the rate of the material removal and hence the lesser wear rate.
- vi. The SEM images shown in Fig. 6.4 depict the severity of the worn-out surfaces of the composites 0.2% of nano-reinforcement of Al_2O_3 and MoS_2 signifies that the mode of failure is due to fibre and matrix debonding.

vii. The SEM micrographs shown in Fig. 6.5 reveal that the composites fabricated with 0.4% of nano-reinforcement of Al_2O_3 and MoS_2 showed better bonding than that of the composites with 0.2% of nano-reinforcements.

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