An Experimental Study on the Effect of Graphite Microparticles on the Mechanical and Tribological Properties of Epoxy Matrix Composites

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Abstract

In this article, mechanical and tribological properties of epoxy matrix composites filled with graphite particulates of 100μm at loadings between 0-1wt% were comprehensively discussed. The effects of the graphite filler on the wear surface of the specimens was investigated using an optical microscope. Results showed a large decrease in the tensile strength of the composite by more than 50% for 1wt% graphite loading. Flexural strength was seen to increase sharply with 0.1wt% graphite before reducing with higher graphite content. Impact and hardness values are both improved with higher graphite content. Friction coefficient was seen to reduce with the addition of graphite particles, this was attributed to the solid lubrication abilities of graphite. Wear rate was seen to sharply reduce with increase in graphite content as a result of the lubrication layer developed at the contact surface hence reducing the break-off of the specimen.

1.0 Introduction

Composite materials have opened doors to research for advanced materials and the opportunity to redesign conventional materials to make them cheaper or more aesthetically pleasing while also combining different properties to design materials with multiple functionalities. Polymeric materials filled with strong inorganic materials can possess high stiffness with low weight and excellent wear and corrosion resistance. This is one of the features that make polymeric composites a promising aspect of industrial application because of their ability to be tailored with functional fillers [1, 2]. It has been found that fibres in the polymer matrix can improve the strength and stiffness of the composite while particulates when homogenously mixed improve hardness and impact properties. Fillers generally improve the mechanical, thermal, and tribological properties of polymer composites [3, 4, 5]. Many materials like glass, carbon, and Kevlar have been made into fibres for reinforcing polymers, graphite, graphene and carbon black particles are also employed as fillers. Nanofillers are also widely used like silica, alumina, boron nitride, titanium oxide, and carbon [6].

Epoxy is a thermosetting polymer having unique mechanical properties and high chemical resistance. This makes it a versatile material for application in corrosive environments where metals can easily react and where weaker polymers cannot withstand them [7]. Fibre-reinforced epoxy matrix composites are one of the most used multi-phase materials mainly because of their outstanding strength-to-weight characteristics. Epoxy-matrix composite provides exceptional mechanical and tribological properties, outstanding dimensional stability, and adequate chemical and corrosion resistance [8]. A common method of improving matrix properties involves the incorporation of fillers into the epoxy resin. From literature, it has been shown that fillers like nanoclay [9], silica [10], TiO_2 [11], glass beads [12], fly ash [13], etc. have been used as fillers for epoxy matrix composites and an improvement in stiffness, toughness, hardness, mould shrinkage, heat distortion temperature, wear rate and fracture mechanism were all observed. In some instances, a significant reduction in processing cost was reported [14].

Integration of various functional fillers is an important route in the design of load-bearing and wear-resistant polymer composites. Baptista et al. [15] studied the mechanical properties of an epoxy-matrix composite containing graphite filler. Graphite fractions between 5-30wt% were added to the epoxy matrix and the tensile strength was observed to greatly decrease while the tensile modulus increased. This is associated with the high graphite particle content which leads to agglomeration and formation of both micro and macro pores. Kulkarni et al. [16] tried to enhance the mechanical properties of epoxy by adding graphite particles of about 75µm with weight fractions between 3 and 12%. They recorded an increase in the impact, flexural, and fracture toughness of the composite by 100%, 59%, and 19% respectively for a graphite content of 12wt%. El-Melegy et al. [17] investigated the synergistic effect of different nanoparticles hybridization on the mechanical properties of the epoxy composite where nanoparticles like graphite, silicon carbide, and alumina were dispersed in an epoxy matrix. Results showed a 37% and 195% increase in the tensile and elastic modulus of 2wt% SiC/epoxy composite, 132% and 200% for 3wt% Alumina and 85% and 32% for a 2wt% graphite/epoxy composite. Alajmi et al. [18] investigated the tribological characteristics of graphite epoxy composites under adhesive wear experiments. Composites with graphite content between 0-7wt% were prepared and tested, it was found that wear and friction were best at graphite contents between 3-4wt% making it the optimum amount, anything less than that results in high friction and wear and anything more than that resulted in higher friction and wear rates. Sakka et al. [19] studied the correlation between friction coefficient and temperature for epoxy matrix composites filled with either carbon nanotubes, graphite or a hybrid of both, they concluded that there is a strong correlation between friction and temperature because the flash temperature measured at the beginning of the experiment was higher than the degradation temperature of the composite hence affecting the friction coefficient of the whole system. Considerable attention has been given to the incorporation of particles and fibres into the polymer matrix to get better mechanical properties so as to be able to compete with metals and other high-strength structural members. This led to the more than 30 years of investigation by researchers. Such works are presented in articles by Shalwan [20], Kaushik [21], Yasmin [22], Novak [23], Suherman [24], and Yao [25] among others.

In this study, epoxy matrix composite filled with different fractions of graphite particulates having approximately 100µm were fabricated. Composites with layered structures were also developed after which the mechanical and tribological properties of both composites were investigated. The adhesion of the layered specimen is also considered. This is also an attempt to find cheaper alternatives to fillers in polymer matrices employed in a friction-intensive system.

2.0 Materials And Methods

2.1 Materials used

Epoxy resin (KEMAPOXY 150) sourced from Chemicals for Modern Building (CMB), Cairo, Egypt and a hardener ratio of 2:1 was used in this study. Graphite particles gotten from EGYCARBON, Cairo were sieved and graded to the particle size of 100µm.

2.2 Sample preparation

Two separate types of composite structures were developed in this study, the homogenously mixed (where the composite resin and the filler are mixed) and the layered structure (where a layer is first poured of neat epoxy before a layer of graphite/epoxy composite). For the homogenously mixed composite, epoxy and hardener are mixed in a container according to the ratio and direction of the manufacturer (2:1), it was mechanically stirred for two minutes with a drilling machine using a coupled metal stirrer head. Graphite particles are added slowly into the mixture according to the graphite content while stirring so as to get a homogenous mixture and avoid agglomeration. Liquid composite is poured into a mould after 5 minutes of stirring. Specimens cured for 24 hours at room temperature were removed from the mould and allowed to continue curing for 5 days to reach their maximum strength. For the layered composite, the first layer of neat epoxy is poured into the mould and allowed to cure for 1 hour before another layer of either 0.1wt% graphite/epoxy or 1wt% graphite/epoxy composite is poured and allowed to cure together. Samples are then cut according to the dimension of the test to be carried out. Figure 1 and 2 shows the homogenous and layered specimens. Tensile and flexural specimens were in a rectangular dimension of 25mm×8mm×270mm. Tribological specimens were cut into rectangular cubes of 10mm×10mm×30mm. Table 1 shows the designation of each omogenously mixed specimen and Table 2 shows the designation of the layered-specimen.

Table 1: Specimen designation (homogenously mixed)

Designation	**Epoxy (wt%)**	**Graphite (wt%)**
Neat	100	-
0.1% G	99.9	0.1
0.5% G	99.5	0.5
1% G	99	1

Table 2: Specimen designation (layered specimen)

Designation	Epoxy (wt%)	Graphite (wt%)
Neat + 0.1% G	**100 + 99.9**	**0.1**
Neat + 1% G	**100 + 99**	**1**
Neat + 0.1% + 1% G	**100 + 99.9 + 99**	**0.1 + 1**

2.3 Mechanical properties

The tensile and flexural measurements were carried out using a universal tensile testing machine (iBERTEST, Spain model TESTCOM-100) according to ASTM D638 and D790 respectively. The tensile and flexural tests were both performed with a crosshead speed of 1mm/min. Three specimens were tested for each specimen range (Neat, 0.1, 0.5, 1wt%) and an average was taken. Hardness value was determined using a Shore D hardness tester where five points were randomly tested on the specimen to get the average hardness value. Impact tests were carried out using a Charpy impact testing machine according to ASTM D256.

2.4 Friction and wear test

A Block-on-Ring (BOR) test setup as shown in Figure 3 was used for friction and wear testing in this study. The block specimen was 10mm×10mm×30mm in dimension and the ring was the stainless steel counterface (AISI 304, hardness 1250HB). Adhesive wear tests were carried out according to the G-77 ASTM standard at ambient conditions (temperature 25°C, humidity = 50±5%), a sliding speed of 200-400rpm and normal loads of 10,20 and 40N. friction coefficient was calculated by dividing the frictional force by the applied force.

3.0 Results And Discussion

3.1 Tensile behaviour

Tensile tests were performed on three specimens of each graphite/epoxy composite as well as the neat polymer. Typical engineering stress-strain curves are shown in Figure 4. These curves illustrate evidence of a brittle behaviour of all the specimens with and without graphite filler. It is clear that the mechanical properties of a composite are dependent on the characteristics of the filler used. In the present work, all composites exhibited lower tensile strength than the neat epoxy, the higher the graphite filler content, the lower the tensile strength measured. Tensile properties derived from the curves are given in Table 3. Results show that an addition of 0.1wt% graphite reduced the tensile properties of the neat epoxy by 32.7% (from 52.11MPa to 35.05MPa). This reduction is expected as graphite is a brittle material and its incorporation into the epoxy matrix makes the epoxy more brittle. A 43.5% reduction is also observed with the addition of 0.5wt% graphite particles. A final reduction of 56.03% was concluded with the specimen having 1wt% graphite particles. A similar trend was observed by Suresha et. al. [3] in their study on the role of micro/nanoparticles on the mechanical and tribological properties of polymer composites. The strain at break for each specimen as given in Table 3 shows a drastic reduction in elongation with the addition of graphite particles. This higher brittleness can be attributed to the increase in stress concentration areas as well as the reduced bonding between the matrix and the filler. It is expected that higher graphite content introduces agglomerates which in turn help in developing micro cracks as well as air bubbles from where there is larger interfacial distance between matrix and filler. This is shown in Figure 5. Additionally, the graphite microparticles may provide obstacles to cracks formed in the composite which results in a less convenient crack path which makes the fracture brittle.

Table 3: Tensile properties of graphite/epoxy composite

Specimen	Tensile strength (MPa)	Tensile Modulus (GPa)	Strain at break
Neat	52.11	2.96	0.0190
0.1% Graphite	35.05	3.0	0.0119
0.5% Graphite	29.41	3.08	0.0104
1% Graphite	22.91	3.26	0.0072

3.2 Flexural behaviour

3.2.1 Homogenously mixed composite

Flexural properties are of great importance for any structural element. Composite materials used in structures are prone to fail by bending and therefore the development of new composites with improved flexural characteristics is essential. Figure 6 shows the flexural strength of the homogenously mixed graphite/epoxy composites compared to neat epoxy. It is observed that all graphite epoxy composites exhibited higher flexural properties than the neat epoxy. This is as a result of the mechanism of load transfer from the matrix to the high-strength graphite particles in bending. This is not the case in tension because the stresses are in the opposite direction. The best improvement in the flexural properties of graphite/epoxy composite is with the addition of 0.1wt% graphite microparticles which increased the flexural strength of neat epoxy from 46.8MPa to 80.08MPa, this is a 71.1% increase. With a graphite content of 1wt%, an increase in flexural strength compared to neat epoxy was 59.79MPa which was a reduction from the 0.1wt% and 0.5wt% graphite content but still an increase of 27%.

A comparison between tensile strength and flexural strength is shown in Figure 7. It can be seen that the advantages of graphite as a filler are more prominent in bending than they are in tension. This was confirmed in literature where different inorganic fillers are added in a polymer matrix composite [26, 27].

3.2.2 Layered composite

The development of layered composites in this study is to determine the possibility of successfully bonding together different layers of epoxy and graphite/epoxy composites to have a new material that has different internal and external flexural properties. This investigation is so stiffer materials can be combined with weaker materials to reduce the general stiffness of the bulk material and to combine cheaper and expensive materials to reduce cost among other benefits. Figure 8 presents the flexural strength of the graphite/epoxy layered composites with regards to the neat epoxy. It is concluded that the best layered-specimen in terms of flexural strength is the composite with a layer of neat epoxy and 0.1wt% graphite/epoxy composite which had a 64.8% increase compared to the neat epoxy. This is close to the improvement noticed with the homogenously-mixed graphite/epoxy composite. The layered composite with neat+1wt% graphite had the least flexural strength compared to the other layered specimen which was 49.9MPa. This is still an improvement compared to the neat epoxy by 6.6%. The specimen with a triple-layer of Neat+0.1+1wt% graphite had a flexural strength of 61.28MPa. This is an encouraging output in the layered composites which means flexural properties can be improved with the layering of a composite with another composite that has better flexural properties. Failure mechanism of the layered specimens were noticed to be of a delamination type because of the clear interlayer debonding and the change of direction of the crack path when propagating between layers as seen in Figure 9.

3.3 Hardness and impact strength

The hardness and impact strengths of all graphite/epoxy composites were improved with the addition of higher graphite content. This is expected as the graphite particles have a higher density, surface hardness and mechanical strength compared to the neat epoxy. Table 4 shows the hardness and impact strengths of the composites. It can be seen that the Shore D hardness value of neat epoxy was 50SHD, with the final addition of 1wt% graphite, hardness values increased to 64SHD, this is a 28% increase in the hardness value. For the impact strength of the composite, a 1wt% graphite content increased the impact strength by 29%. These improvements can be attributed to the load transfer mechanism in a homogenously dispersed composite where the surface of the graphite particles become load-bearing during compression (Shore D hardness testing) and hence prove to be hard. On the impact strength, the change in direction created by graphite particles as a crack propagated during failure reduces the ease of fracture and hence increases the toughness of the material. It has been reported in [28, 29, 30] that the addition of high-density and high-strength inorganic particles enhances the surface hardness and the impact strength of a composite.

Table 4: Hardness and impact results

Specimen	Hardness value (SHD)	Impact value (J)
Neat	50	6.75
0.1% G	57	7.25
0.5% G	61	7.65
1% G	64	8.75

3.4 Friction and Sliding wear results

3.4.1 Friction

The friction coefficients of neat and graphite/epoxy composites were obtained under different conditions of operation. A sliding speed of 200 and 600rpm, applied loads of 10,20 and 40N, and a constant time of 300sec. In this study, it was observed that as applied load increases, the coefficient of friction increases for all composites and neat epoxy, this is to be expected as higher normal loads means the pressure exerted on the specimen and counterface increases and this increases the frictional force generated in the system. The lowest coefficient of friction was observed in the specimens with 1wt% graphite content for all applied loads and sliding speeds. For specimens tested at 10N applied load and 200rpm sliding speed, a reduction in the coefficient of friction of 14% was observed from the neat epoxy to the 1wt% graphite/epoxy composite as shown in Figure 10. For the specimens tested at 20N applied load and 600rpm sliding speed, it can be seen from Figure 11 that the composites with the higher graphite content started with lower friction coefficients, a decrease of 12% was observed between the neat epoxy and the specimen with 1wt% graphite content, this is as a result of the ease with which the graphite particles come off with higher graphite content and form the lubricating film as discussed earlier [23]. This is also comparable to results recorded in experiments by [31, 32]. Similar trends are noticed in all other operational conditions as shown in Figure 12. The reduction of the coefficient of friction is most noticeable when applied load is changed, despite the effect of sliding speed on the coefficient of friction of the composites, very little change is observed as seen in the figures presented. A more prominent effect is observed when higher loads are applied. An increase of 52% was noticed when the applied load is changed from 10N to 40N. A higher graphite content would certainly mean much lower friction coefficients but that will lead to instability of the composite as it becomes weak and crumbles easily due to the weak bonding between the matrix and the filler [33].

3.4.2 Wear

The variation in wear rates of the neat epoxy and the graphite/epoxy composites under different conditions were presented in Figure 13. The experimental results show that the wear rate reduces with an increase in graphite content as the specimen with 1wt% graphite exhibited a reduced wear rate compared to the neat epoxy of more than 75%. The difference was similar for the higher sliding speed of 600rpm which was 73% in reduction. This is a result of the solid lubrication abilities of graphite particles [34]. An interesting trend that was observed in this study is the reduction of wear rate with increasing sliding speed. This can be attributed to the faster formation of a sliding film on the specimen and counterface which then reduces further disintegration of the composite, the film generated stabilises the stick-slip process which becomes a low adhesion region where the wear rates are low. But this trend was not in agreement with the conclusions in the literature [35, 36]. However, a study by Tripathy et al. [37] stated that the wear rate increases with an increase in sliding velocity but with an exception. In their research, there was a sharp decrease in wear rate at velocities ranging between 3-4 m/s but then increases exponentially up to 7m/s. It could be argued that the reduction in wear rate with increase in sliding speed in this research is still between the 3-4m/s decrease observed in the research above and hence acceptable. Figure 14 shows the specific wear rate of graphite/epoxy composite for 10N applied load at different sliding distances.

The Worn surfaces of the specimens tested during friction were studied to determine the wear mechanism and surface morphology. From Figure 15, it can be seen that the difference between the worn surface of the neat epoxy composite was much more coarse and had deep scratches due to the high frictional force and the brittle nature of the epoxy while the composite with 1wt% graphite particle had a smoother surface even though some scratches were visible. These smooth scratches were as a result of the initial friction and break-off of the surface of the composite before a smooth lubrication film was generated which in turn becomes a layer with low friction and less debris formation. This is in agreement with results gotten in previous studies on the review of tribological properties of polymer composite [38]. The worn surface of the specimens with graphite filler in higher quantity can be described as quality surface [39] because there is no sign of extreme debonding or high porosity. The relative appearance of micro-cracks shows good resistance to shear stresses displayed by graphite particles. Similar things have been reported with carbon fibre and epoxy polymer where there was relatively less damage than the glass fibre epoxy polymer [40]. In other words, it can be concluded that graphite has excellent properties in reducing both the friction and wear rates in polymer composites.

4.0 Conclusions

The mechanical and tribological properties of graphite filler epoxy matrix composite were investigated in this study and the following conclusions were drawn:

- Graphite can be successfully incorporated into the epoxy resin to fabricate a particulate filler epoxy composite with homogenous mixing before agglomeration at higher percentages.
- Mechanical properties are greatly influenced by the addition of graphite particles; tensile strength was reduced with an increase in graphite content with the addition of 1wt% graphite particle reducing the tensile strength by 56%, flexural strength was improved with the addition of graphite but not in a linear path as the composite with 0.1wt% graphite had the highest flexural strength, hardness and impact values are both improved with higher addition of graphite contents.
- Wear and friction properties were also influenced by the graphite particles; specific wear rate was seen to decrease with an increase in graphite content, this is as a result of the solid lubrication capabilities of graphite. Friction coefficient was affected by both graphite content and the applied load, as higher applied loads meant higher friction coefficients and higher graphite content means lower friction coefficient. Wear track studied under a microscope shows the worn surface of the composite with 1wt% graphite content to be smooth as the lubrication film developed during friction reduced the surface roughness of the system.

Declarations

Competing interest

The authors declare that they have no competing interests.

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Authors' contribution

MM carried out the material preparation (mixing and curing), carried out the mechanical and tribological tests as well as the microscopy. NS provided the epoxy resin and hardener and participated in the conceiving and design of the study. MS participated in the analysis of the results and the design of the experiment. All authors read and approved the final manuscript. **Availability of data and materials**

Datasets used in this study are available on a request basis. Further data can be made available on the journal submission page.

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COMPETING INTEREST

The authors declare no conflict of interest.

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Figures

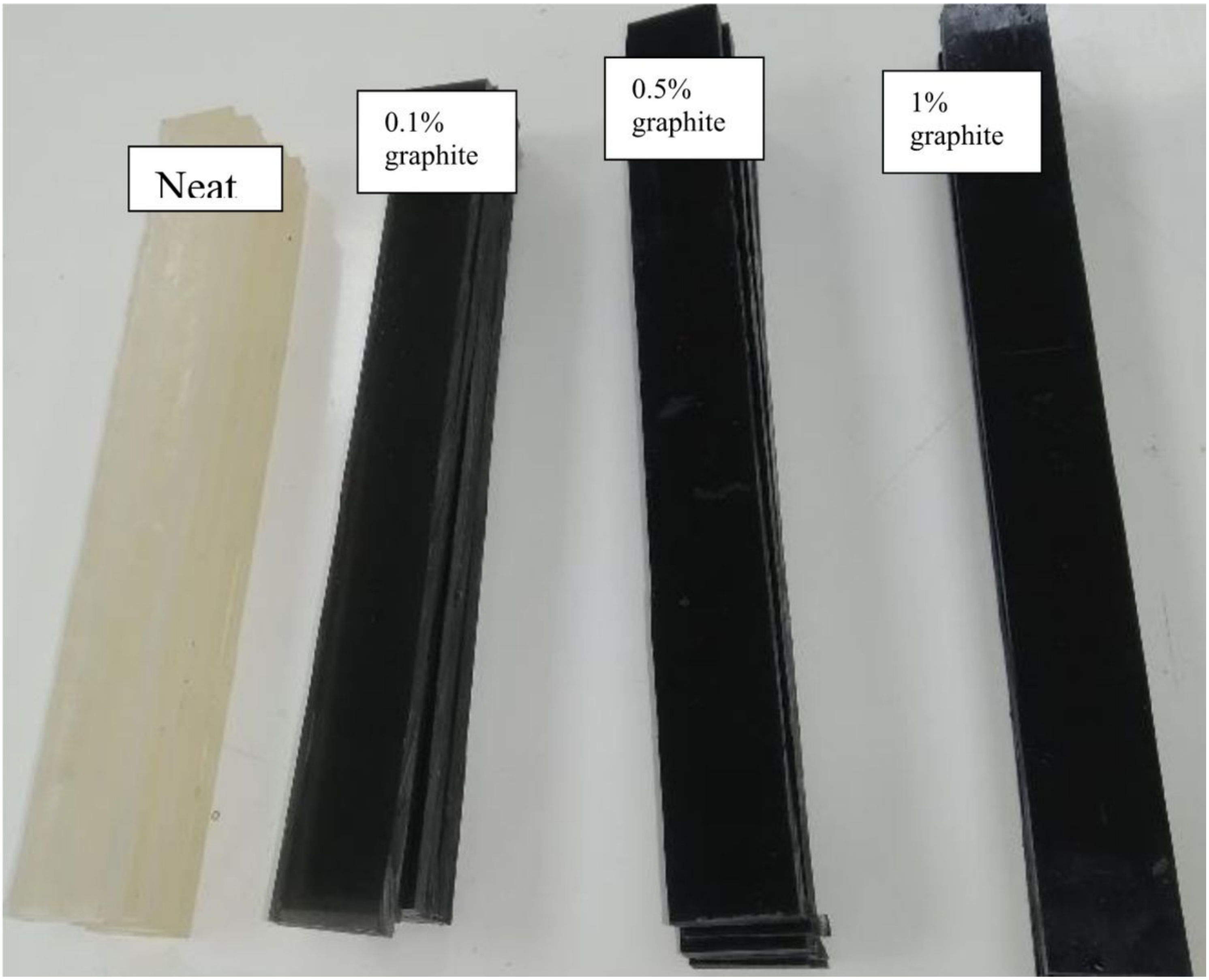

Figure 1

Homogenously mixed specimen

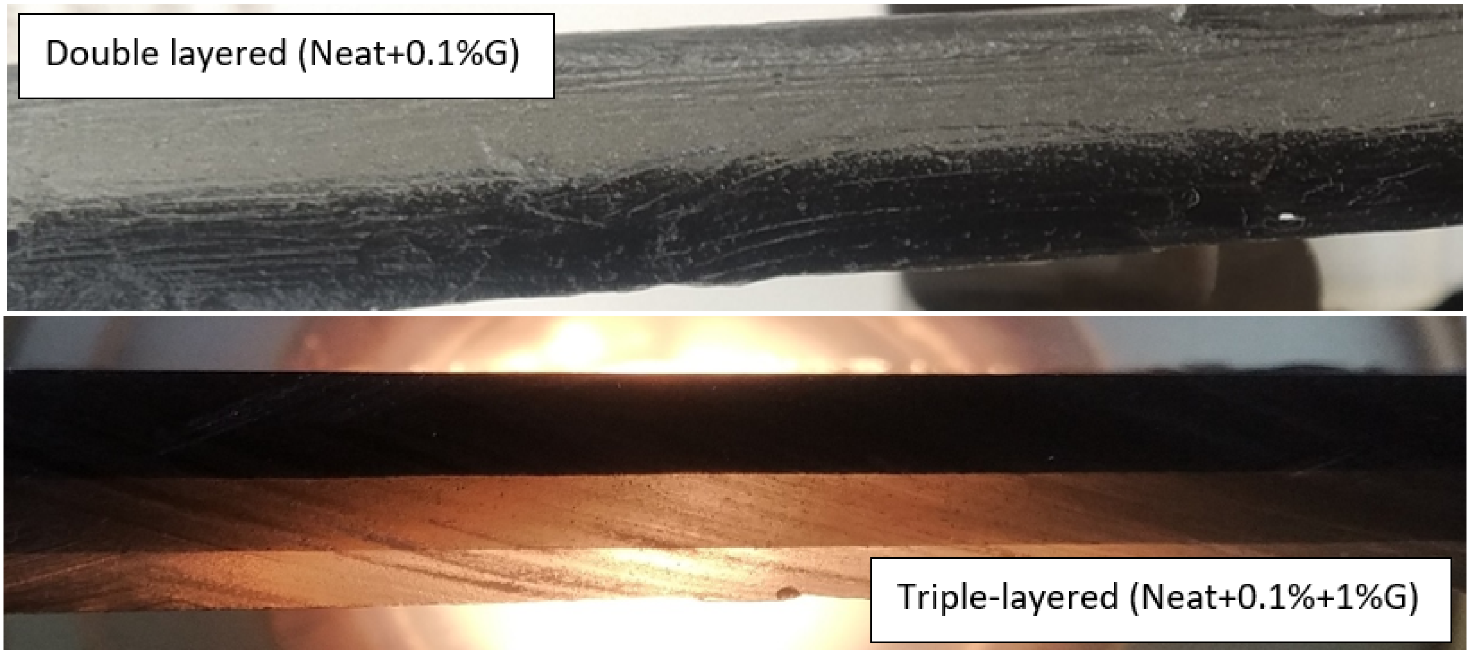

Figure 2

Double and triple-layered specimen

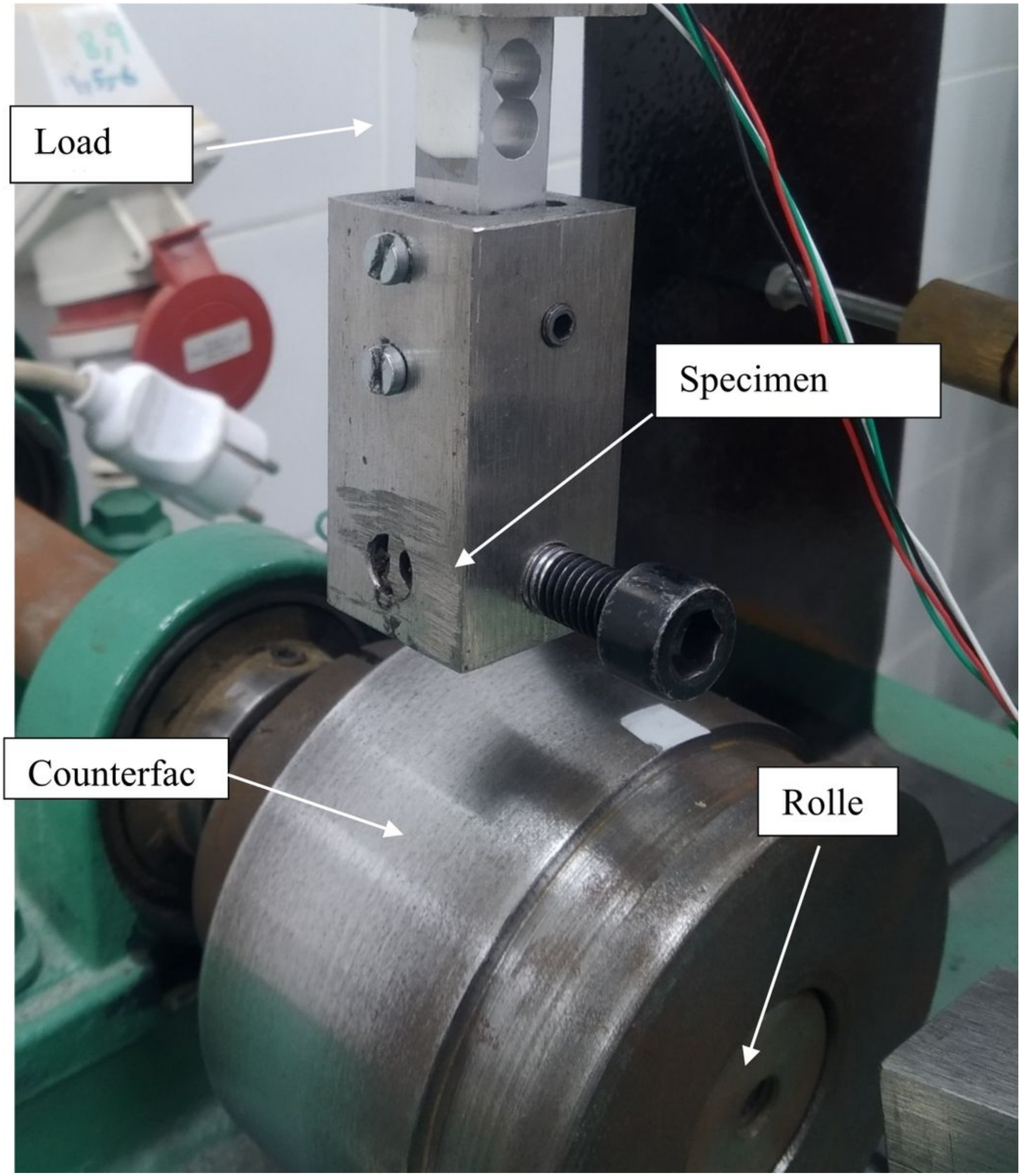

Figure 3

Outline of the block-on-ring tribometer.

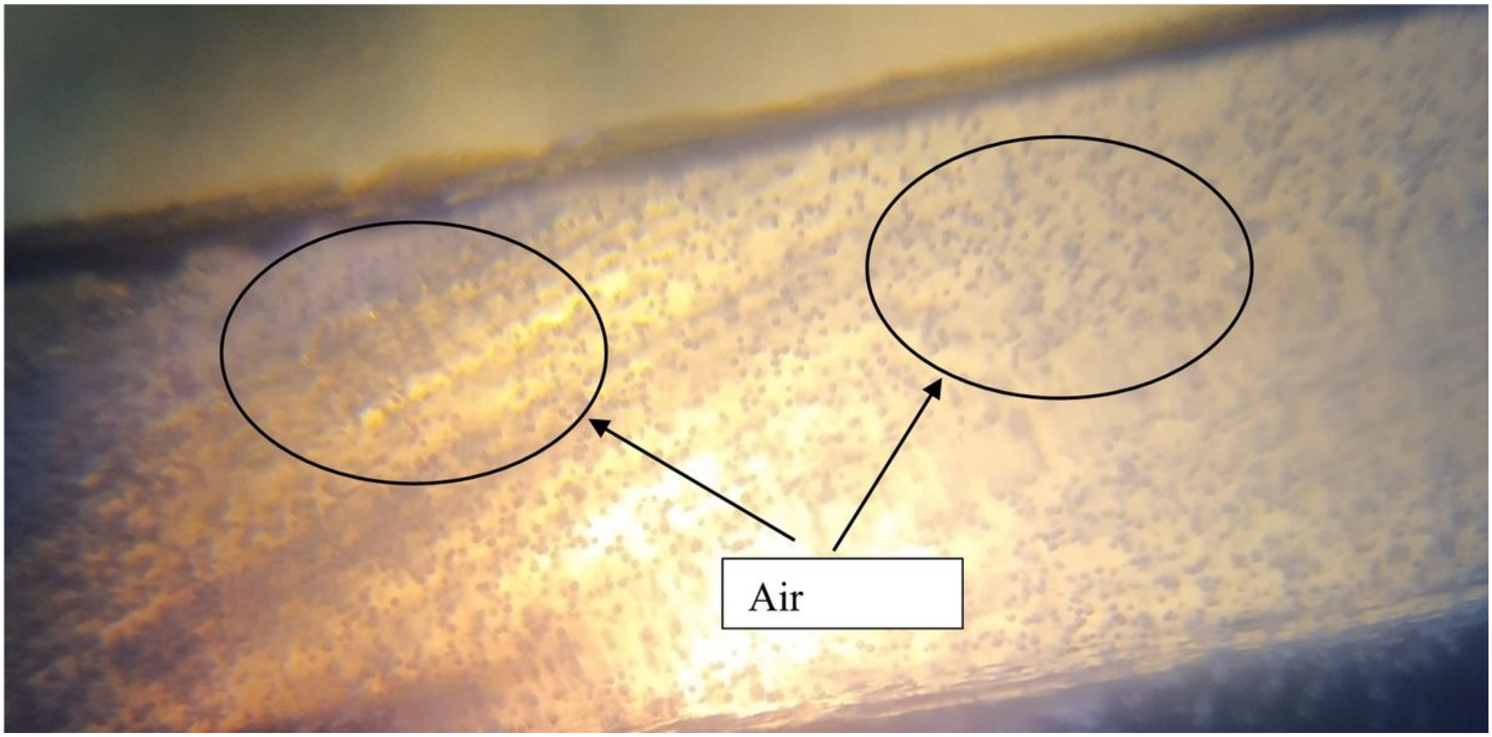

Figure 5

Visible air bubbles in the neat epoxy at 4× magnification

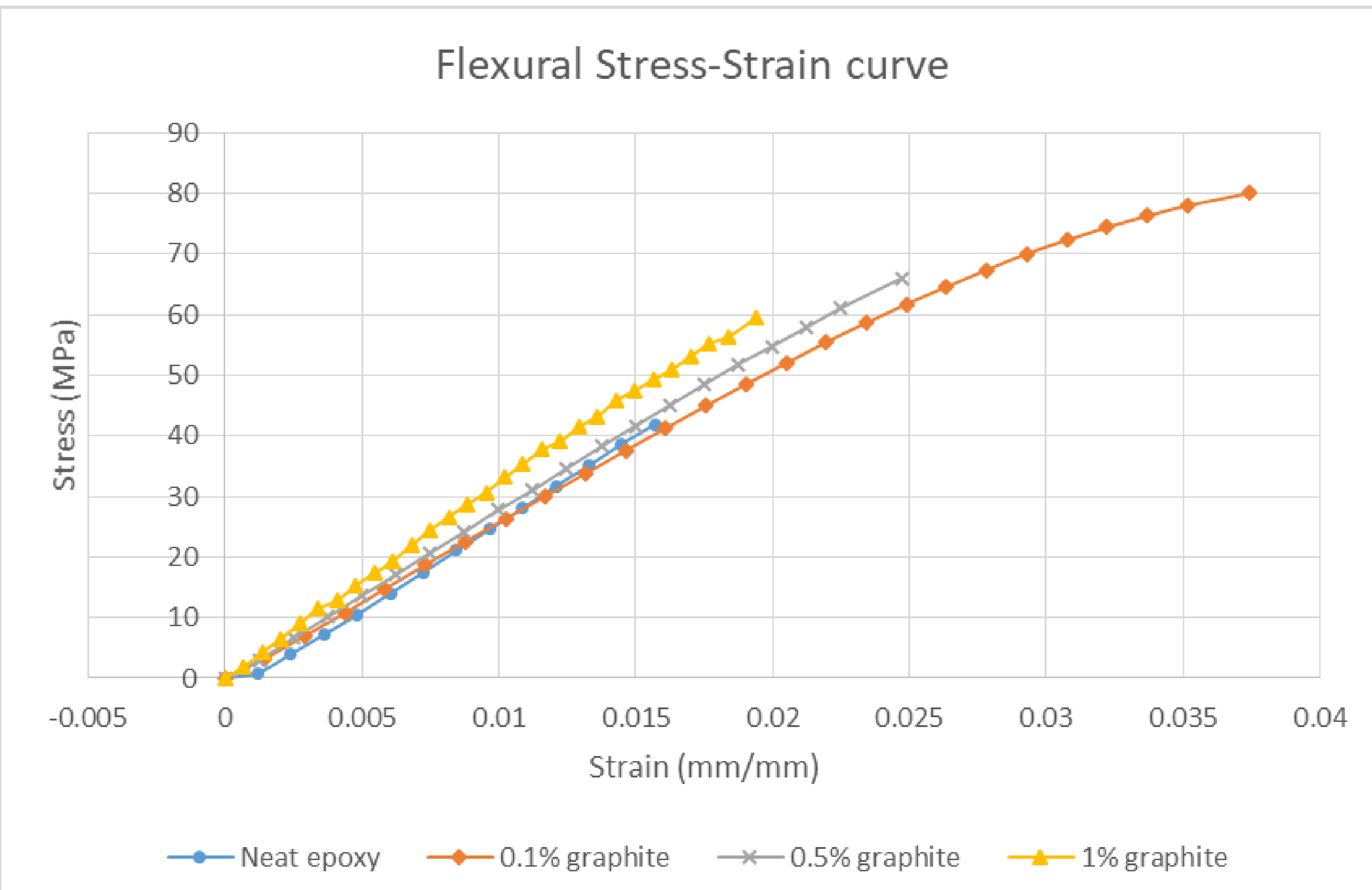

Figure 6

Flexural stress-strain curve

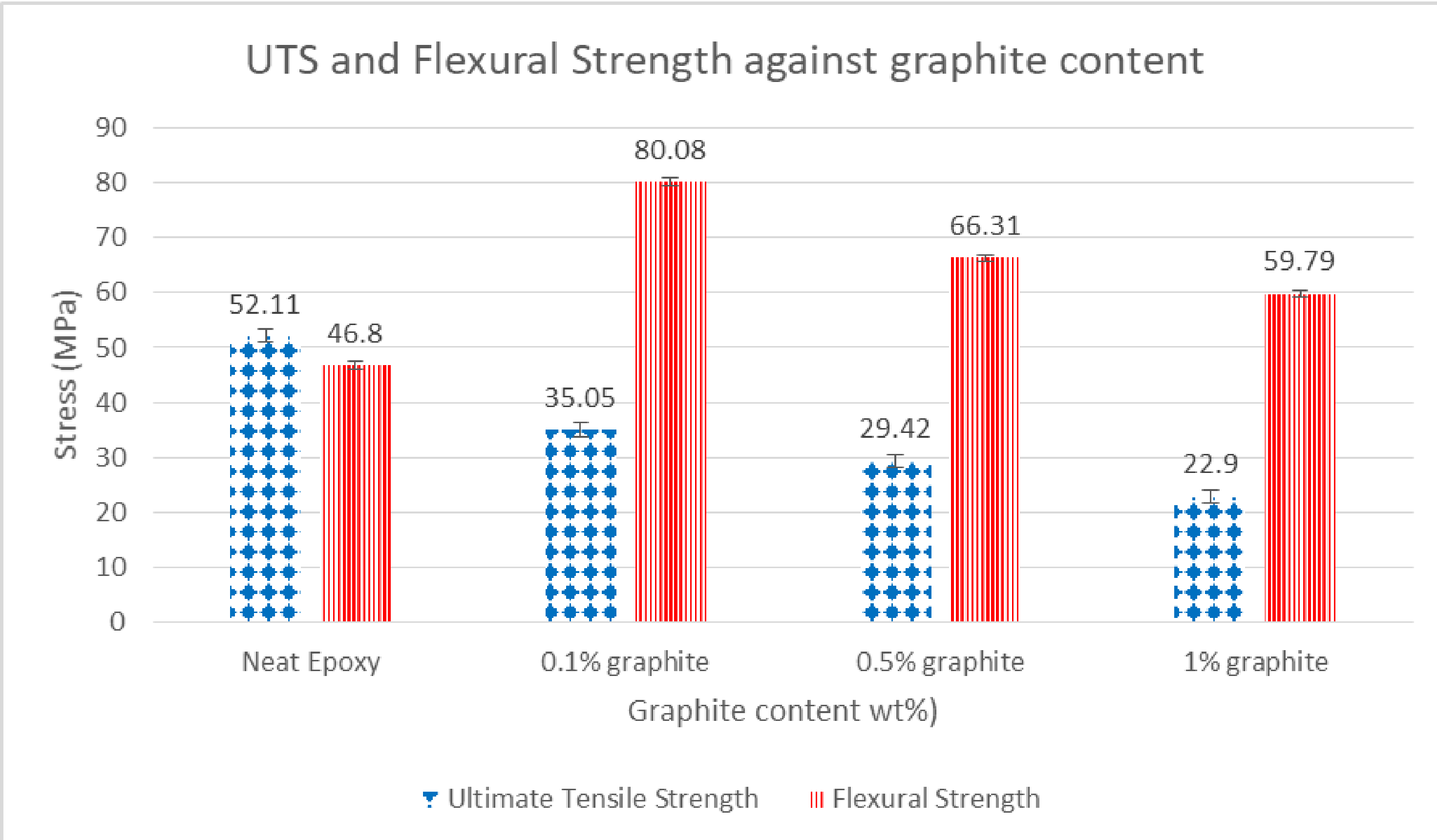

Figure 7

Effect of graphite content on the tensile and flexural strength of the graphite/epoxy composite.

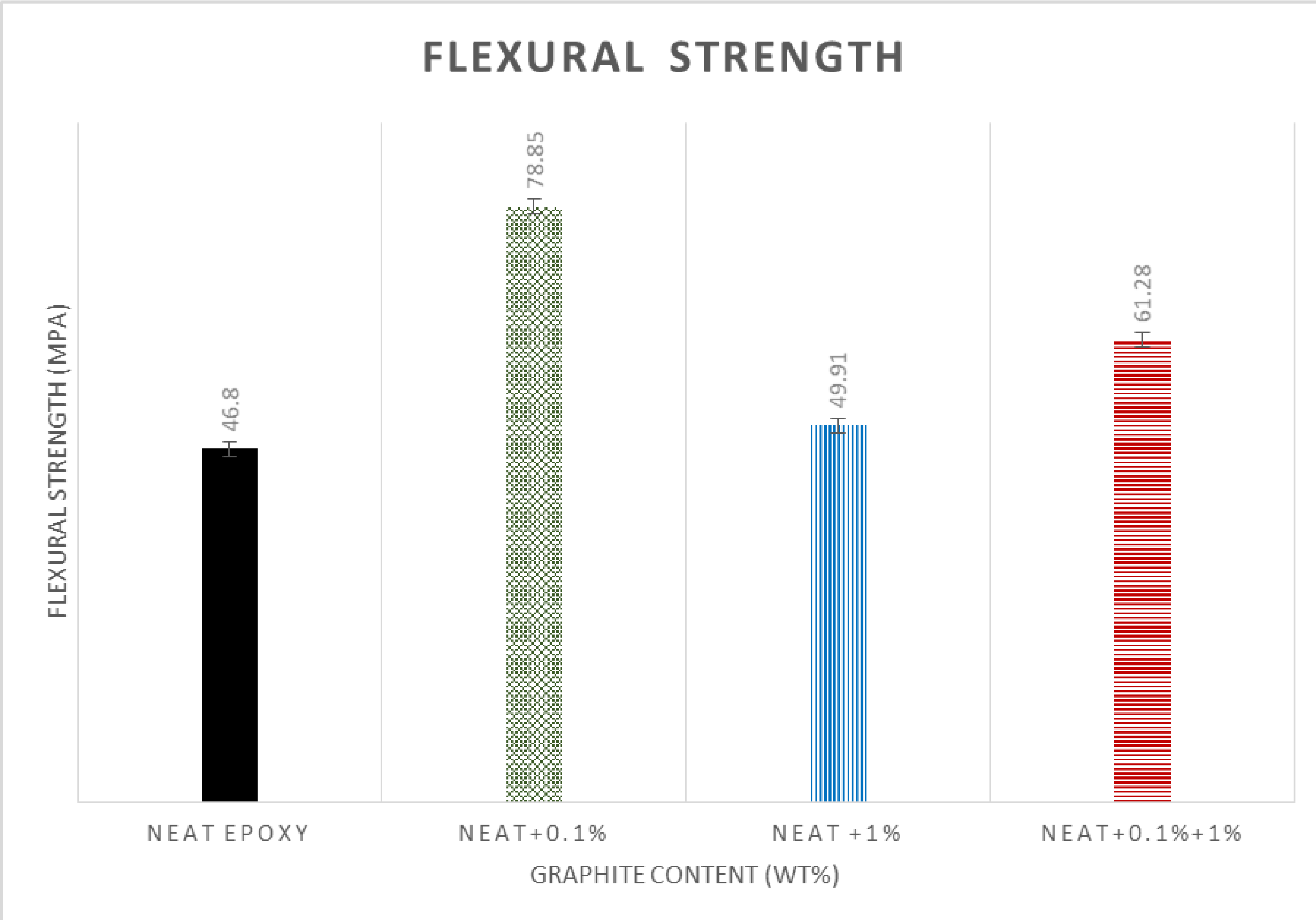

Figure 8

Flexural strength of graphite/epoxy layered composites

Figure 9

Failure mechanism of Neat+0.1%G layered specimen in bending.

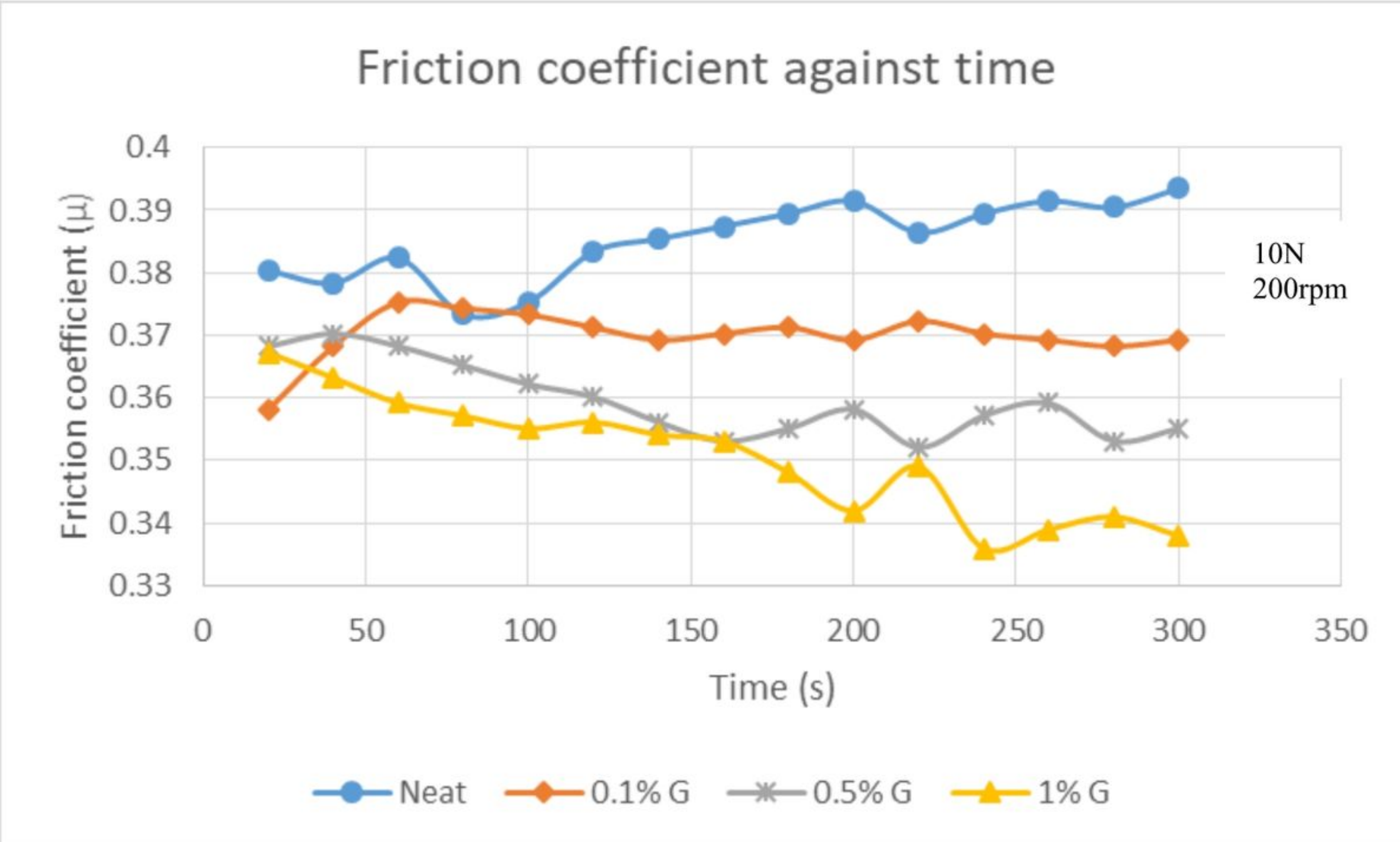

Figure 10

Friction coefficient for neat epoxy and graphite/epoxy composites at 10N applied load and 200rpm sliding speed.

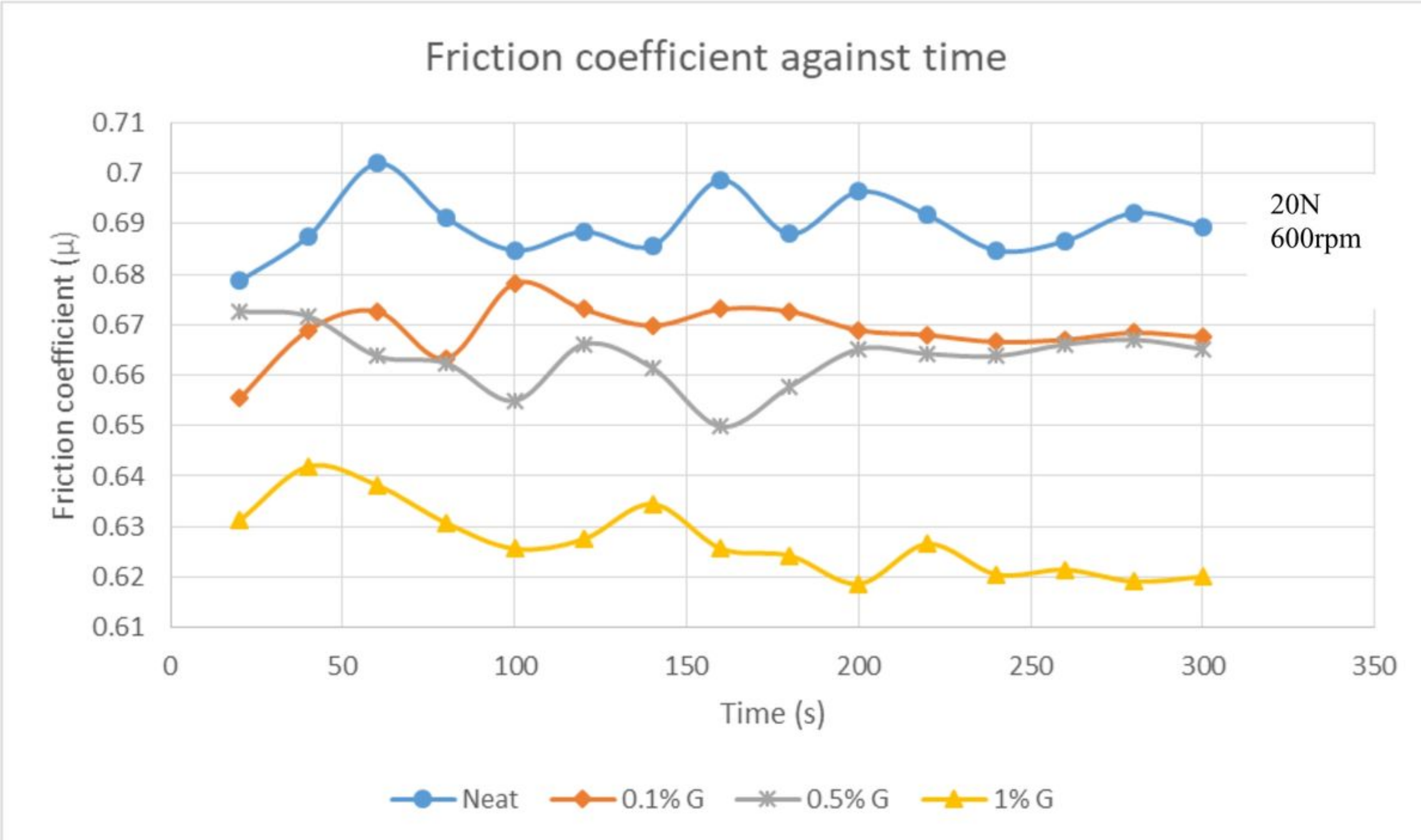

Figure 11

Coefficient of friction of graphite/epoxy composites at 20N applied load and 600rpm sliding speed.

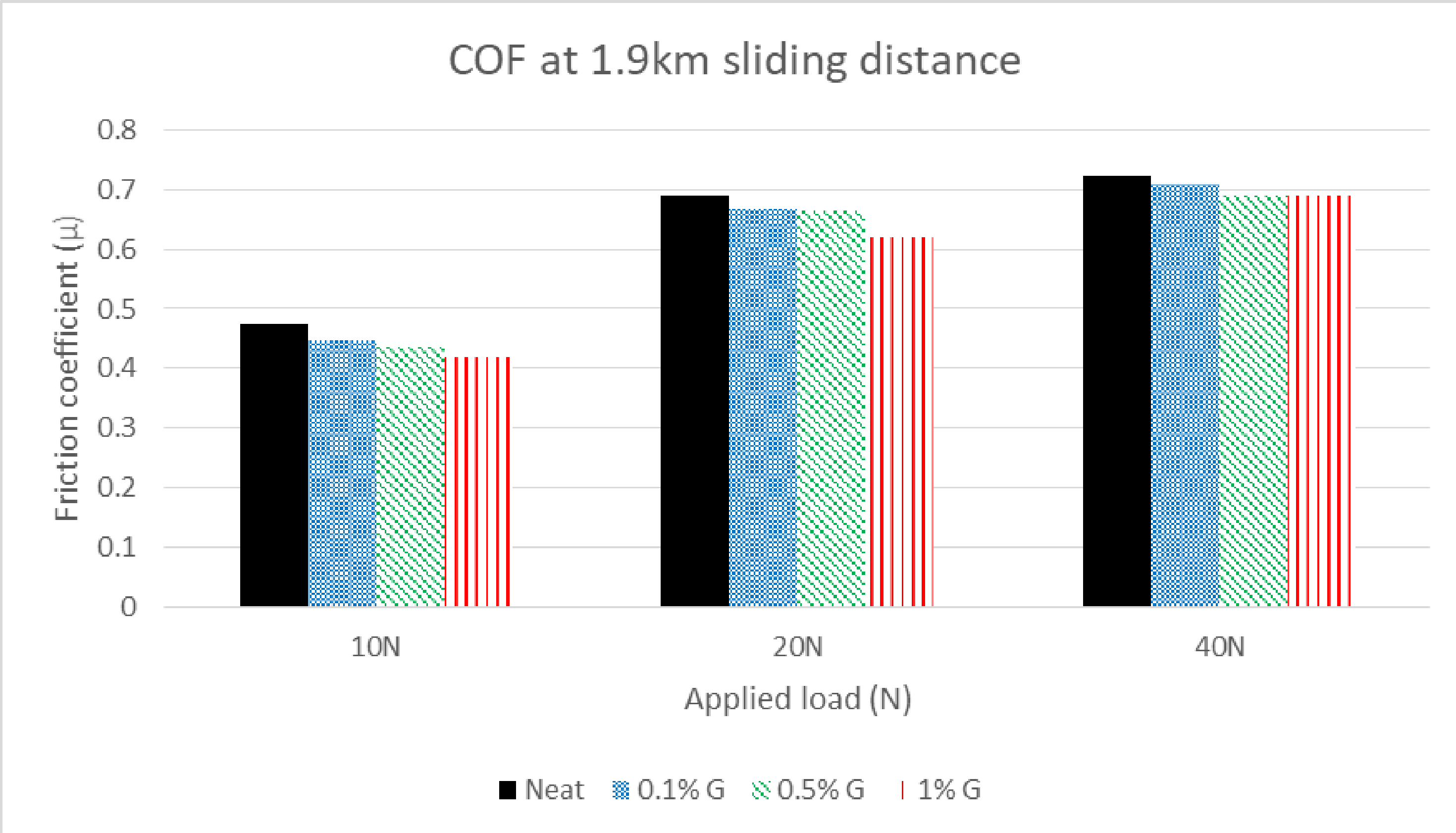

Figure 12

Friction coefficient at 1.9km sliding distance for 10N, 20N, and 40N applied load.

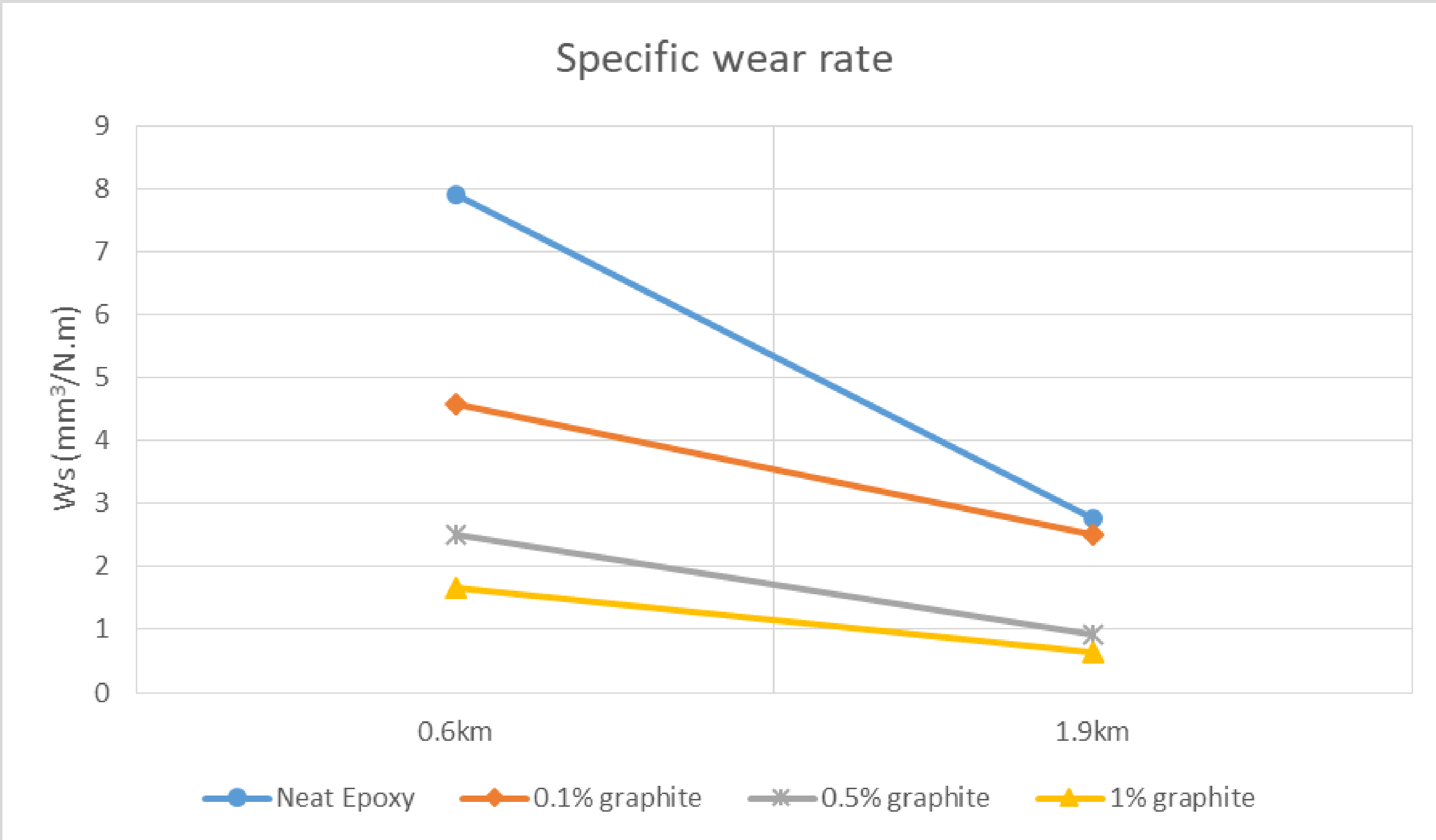

Figure 13

Specific wear rate against sliding distance with respect to graphite content at 40N applied load and 600rpm sliding speed.

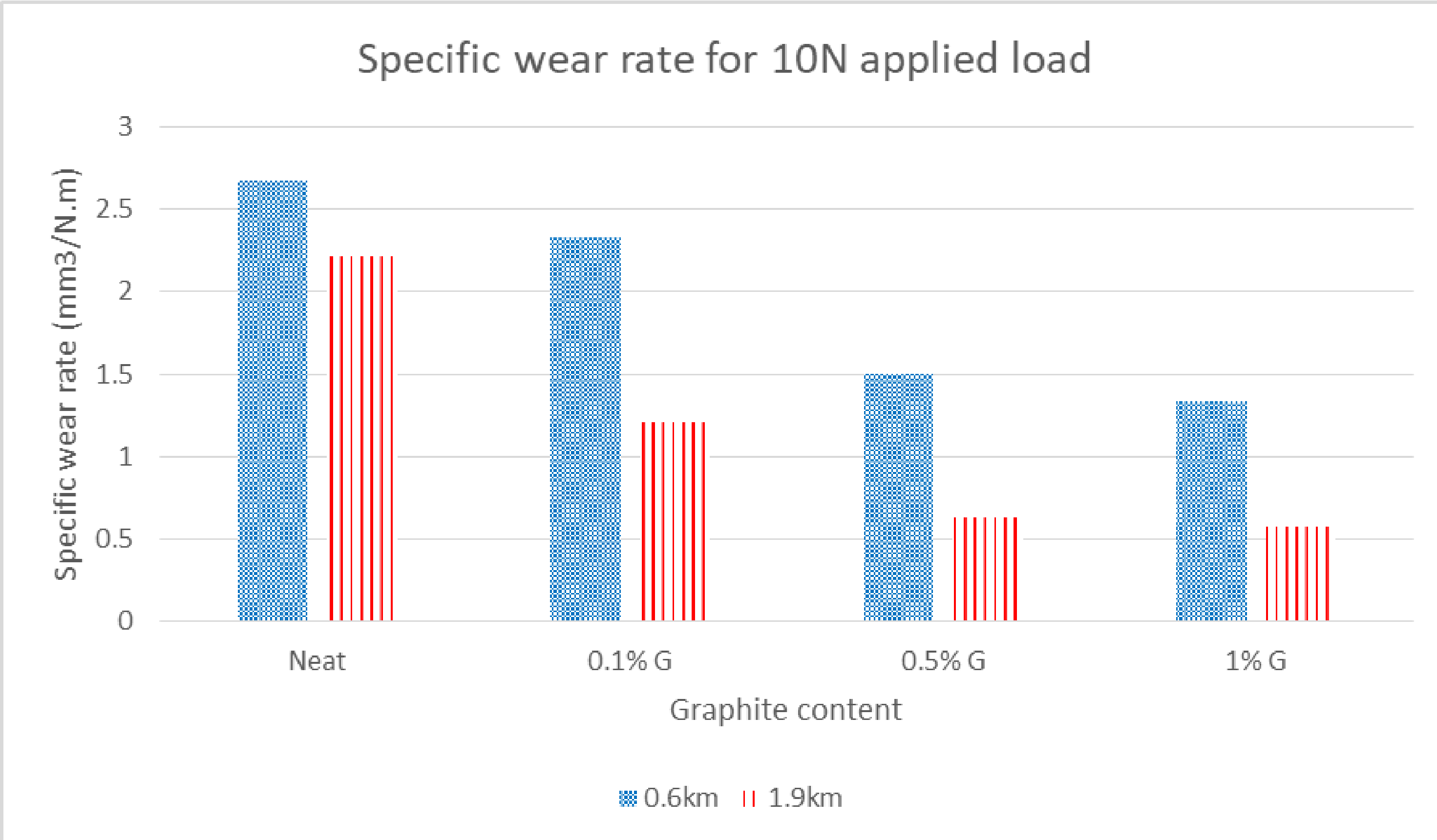

Figure 14

Specific wear rate of graphite/epoxy composite for 10N applied load at different sliding distances

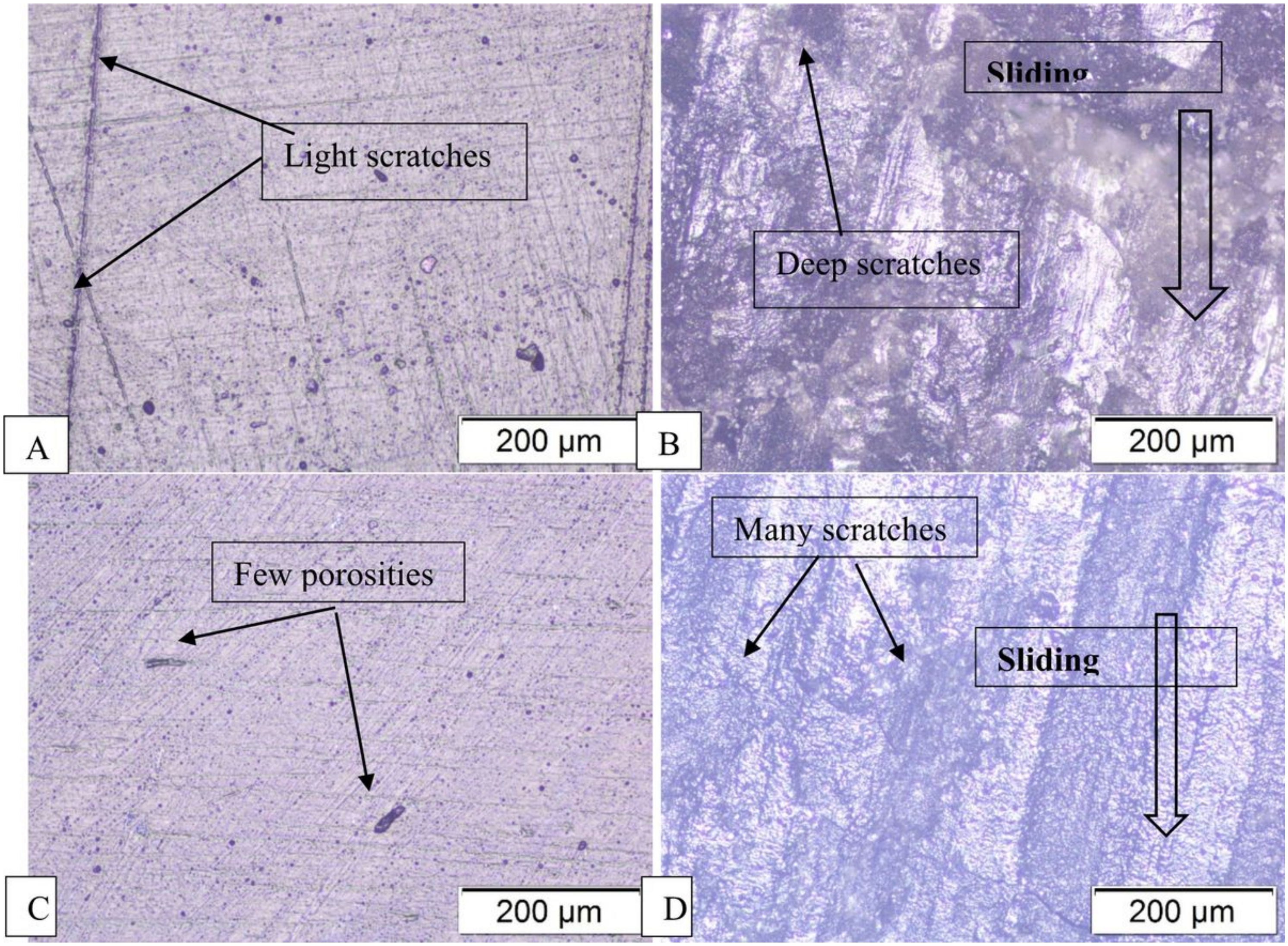

Figure 15

Wear surface of (A) neat epoxy before the test (B) after the test (C) 1% graphite/epoxy composite before the test (D) after the test. All at 10X magnification.