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Synthesis and Characterization of Epoxy Matrix Composites Reinforced with Various Ratios of TiC

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Abstract

Epoxy matrix composites reinforced with various ratios of Titanium Carbide (TiC) have been synthesized and characterized successfully. Different ratio of (TiC) powder (0 wt%, 5 wt%, 10 wt%, 15wt%, 20wt%, and 25 wt%) has been used as reinforcements in epoxy matrix. The results obtained show improvement in both mechanical and tribological behavior of the composites. Hardness value, impact strength, tensile strength and wear rate was improved by the addition suitable titanium carbide powder ratio. Hardness and tensile strength values show increment with addition of 15 wt% of titanium carbide powder. Impact strength was found to be increased with increasing ratio to 20 wt% of titanium carbide. The wear behavior was investigated using a pin-on-disc wear testing machine with different sliding distance, wear rate improved greatly at 10 wt% of titanium carbide powder. Optical microscope images (OM) were taken for micro-pores that present on the specimens and for specimens after wear test with 2000m sliding distance. The mechanical properties such as hardness, tensile strength, impact, and wear resistance are observed to be increased considerably compared to the matrix composite.

Keywords: *Epoxy Matrix, Composites, Various Ratios of Particles, TiC.*

1. Introduction

The usage of epoxy resin has been a significant importance in the engineering field for a long period of time. Many components are made of epoxy based materials and are proven to be outstanding in many aspects such as mechanical properties, thermal conductivity, and electrical properties [1]. Mechanical properties have been a major scope since these aspects play an important role in industrial and material refurbishing in numerous field, such as automotive, aerospace, electronics, and biotechnology applications [2]. Epoxies, generally, have high chemical and corrosion resistance, good mechanical and thermal properties, outstanding adhesion to various substrates, low shrinkage upon cure, good electrical insulating properties, and the ability to be processed under a variety of conditions which make it suitable candidate as matrix materials for advanced composites applications [3] [4]. The curing reaction of the epoxy takes place after the addition of a hardener solution into the epoxy resin. During curing, the molecules form cross links with each another and grow in a three-dimensional network that finally forms a solid epoxy resin. Most reinforcing materials used nowadays, such as natural fiber, glass fiber, carbon fiber, etc., have a lower hardness and a higher wear rate compared to Titanium carbide. Composite reinforced with titanium carbide offer an outstanding properties such as high strength to weight ratio, high torsion stiffness, high hardness, good corrosion and wear resistance. Composite systems consisting of a polymer matrix and particles of titanium carbide have been considered as a novel class of smart materials. Researchers investigated abrasive wear behaviour of SiC and TiC fillers filled epoxy composites. They concluded that filled composite showed excellent abrasion resistance compared to unfilled Composites [5; 6; and 7]. Properties of the composites are determined by many factors such as the ratio, size, and shape of reinforced TiC particles. From the literature review it is observed that the researchers have studied the wear property of various inorganic fillers. Hence, the present study focuses on the effect of the contents ratio of TiC on mechanical and tribological behavior of the composites and to have better understanding of the synthesis and characterization the Epoxy- TiC composites.

2. Experimental

2.1. Raw Materials

The main material involved in the present study is the Epoxy which the matrix. The Epoxy is formed from two different components; the first is the resin and second is the hardener with the following specifications:

1. Density of Resin, and Hardener were 1.22, and 0.96 g/ml, respectively;
2. Viscosity of Resin, and Hardener (measured at 20-23◦C) were 800, and 400 mPa s, respectively;
3. Cure Time of mixture (23°C) 24 hours.

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In the present study, the reinforcement material was commercial TiC powder with average particle size 3µm supplied by Inframat® Advanced Materials LLC (USA). Titanium carbide is considered a promising reinforcing material towards the development of matrix composites due to its high hardness, melting point, chemical and thermal stabilities, wear resistance, solvency of other carbides, and good wettability and relative thermal stability with many binder materials [10].

2.2. Fabrication of Mold

Aluminum block (20cm ×10cm ×1.6cm) was used as a base for providing a perfect mold that can be reused several times in molding the specimen into required shapes.

The three main shapes that are required for testing were fabricated using the aluminum block. The dimensions, of specimens for Tensile and Impact test according to American Society for Testing and Materials (ASTM) D638, D6110 [8; 9], as shown in Fig. 1.

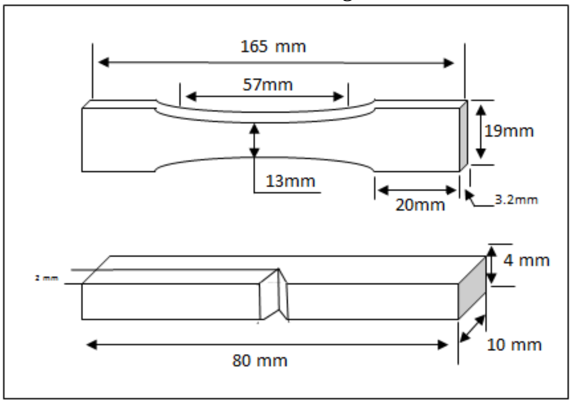


Fig. 1. The dimensions, of specimens for Tensile and Impact test

The dimensions of the specimens prepared for the Wear test were based on the available jig in the pin-on-disc machine. It was more convenient to fabricate rectangular shape specimens for this test. Mold was designed by using CATIA V.55 software. The design is later transferred to develop Computer Numerical Control (CNC) codes which will be used in the milling of aluminum block. The mold after milling and polishing processes is shown in Fig. 2.

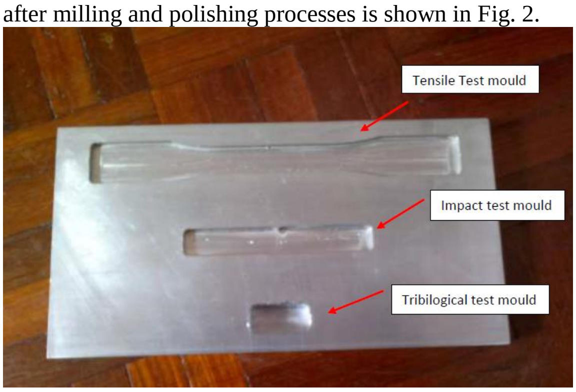


Fig. 2. Mold after milling and polishing processes

Mold was polished with abrasive paper several times until a smooth surface was obtained. The completed mold needs high attention to ensure a smooth release of the composite when it cures in the mold. For the first time five layers of wax were required to ensure that the surface is fully covered. After that only two layers of wax were sufficient to remove composite from the mold easily.

2.3. Preparation and Characterization of Composites

The composite was prepared according to wt% ratio. Pure Epoxy composite and the varying ratio of TiC are provided in Table 1.

Table 1. Epoxy-TiC composites specimens with different wt% ratio of TiC

Set of specimens	TiC content in composite,wt%
1	0wt% of TiC
2	5wt% of TiC
3	10wt% of TiC
4	15wt% of TiC
5	20wt% of TiC
6	25wt% of TiC

The fabrication of composites consists of three steps:

1. Mixing the Epoxy resin and TiC particle using a mechanical stirrer.
2. Mixing the hardener with the filled Epoxy resin.
3. Pouring the mixture into mold.

In the first and second steps, slow motion and steady stirring were applied to ensure that the mixing is done without formation of air bubbles (porosity). A fixed amount of Epoxy resin and hardener were used with a varying amount of TiC powder. In the last step, the mixtures were manually poured into previously prepared mold; the composites were cured at room temperature for 24h. Vickers hardness machine with load of 60kg for 5 seconds was used to investigate the influence of particulate weight fraction of TiC on the matrix hardness. Five different points were taken for each sample and the average value was taken to eliminate errors due to local non-homogeneity. The tensile test was carried out by Universal Testing Machine GUNT WP 300; the specimens were subjected to failure at a constant rate. Pin-on-disk machine shown in Fig. 3 was used to measure the wear resistance of samples. The test was carried out under dry sliding conditions, in ambient air at room temperature ≈ 25 ºC. The disk was rotated by DC motor with sliding rotation speed of 500 rpm and 40N load.

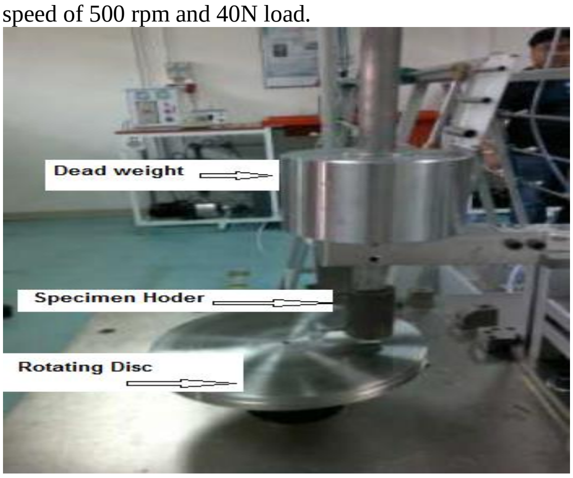


Fig. 3. Pin-On-Disk set-up

The impact test was performed by using Charpy Impact Test Instrument [Testing Machine INC, AMITYVILLE, New York Company] to investigate the influence of particulate weight fraction of TiC on the impact. Optical Microscope (OM) was used for analysis microstructure of different composites compositions.

3. Results and Discussions

3.1. Introduction

The dimensions, of specimens for Tensile and Impact test according to American Society for Testing and Materials (ASTM) D638, D6110 [8; 9]. The dimensions of specimens prepared for Tensile and Impact test were 165± 0.02, 19± 0.01, 3.2± 0.01, and 80± 0.02, 10± 0.01, 4± 0.01 (mm). The three specimens were used to determine the average value of each property.

3.2. Hardness Measurements

The hardness measurement is known to be one of the most informative and rapid methods to determine the mechanical behavior of composites. Yield and ultimate tensile strengths, fatigue strength, wear, etc., are often in good correlation with hardness [11]. A higher hardness was also associated with a lower porosity [12]. Improvement of hardness depends on the amount, particle size, and uniformity distribution of TiC particles (Fig. 6).

Average Hardness Vickers (H.V) for Epoxy with different wt% TiC ratios are shown in Fig. 4.

It is observed from the results of hardness that all reinforced specimens have a higher hardness than that of the matrix material. In general the hardness increased with increasing the hard particles content of TiC. The hardness for specimens with (20 wt% and 25wt% TiC) decreased due to the agglomeration of TiC particles (Fig. 6) and formation of porosity (Fig. 7) during mixing process which lead to big indenting and resulting in a low HV value even though the material has a high hardness value. Furthermore, conditions of a specimen surface also play an important role in determining the hardness value. The surface should be smooth and flat which allows a perfect indenting.

3.3. Tensile Test

Many researchers reported that adding ceramics nanoparticles in an epoxy had a significant effect on the tensile properties of the modified epoxies [13; 14; 15; and16].

In general, increasing the TiC content leads to an increase in the ultimate tensile strength comparing with matrix material. In Fig. 5, the matrix fails with strain value (0.0227); this result shows that the matrix was ductile with the absence of TiC particles, and indicates that the adding of Nano TiC particles support the matrix by absorbing stresses and bear some load before failing.

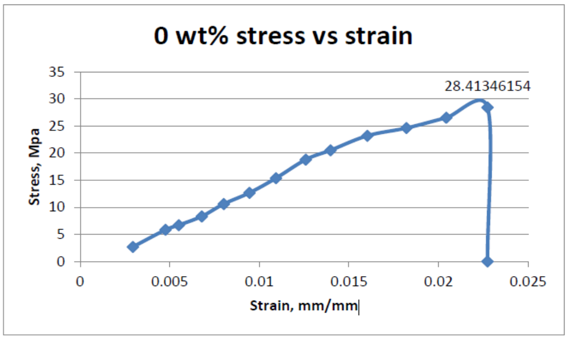


Fig. 5. Stress- strain diagram for 0 wt% of TiC

As shown in Table (2), due to the addition of different percentages of TiC, the tensile strength increased from (28.413 MPa) for the unreinforced matrix to (35.024MPa)) for 15 wt% TiC specimens and the strain failure reduces from (0.0227) for the unreinforced matrix to (0.0156) for 15 wt% TiC specimens. This indicates that the composite properties change from ductile to tough and brittle by good distribution of TiC particles in the resin, as shown in Fig. 6. Also the 25wt% TiC specimen exhibits lowest tensile strength (26.995MPa) comparing with other specimens due to the agglomerated TiC particles in the resin with higher TiC percentages. This indicates that the material became brittle and no plastic deformation was observed during fracture. The ultimate tensile strength, strain failure, and young modulus of elasticity for different specimens are given in Table 2.

Table 2. The relation between TiC powder and tensile strength of the composites

Specimens	Tensile strength MPa	Strain failure	Young modules, GPa
0wt% of TiC	28.413	0.0227	1.25
5wt% of TiC	29.688	0.0108	2.761
10wt% of TiC	31.178	0.0125	2.492
15wt% of TiC	35.024	0.0156	2.241
20wt% of TiC	30.385	0.0125	2.436
25wt% of TiC	26.995	0.0114	2.375

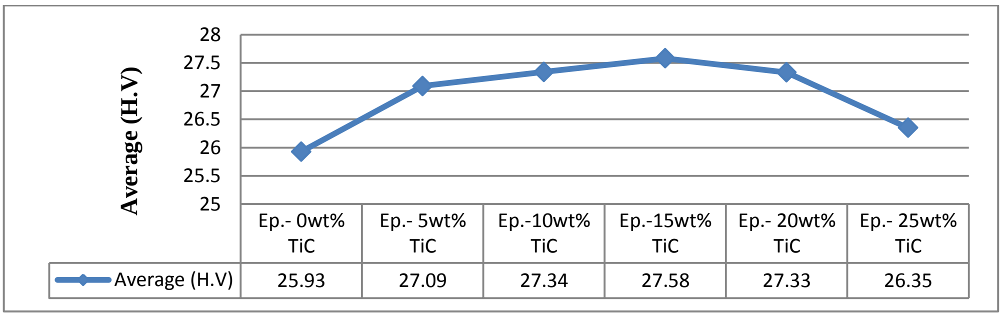


Fig. 4. Average hardness value for epoxy matrix composites reinforced with various ratios of TiC

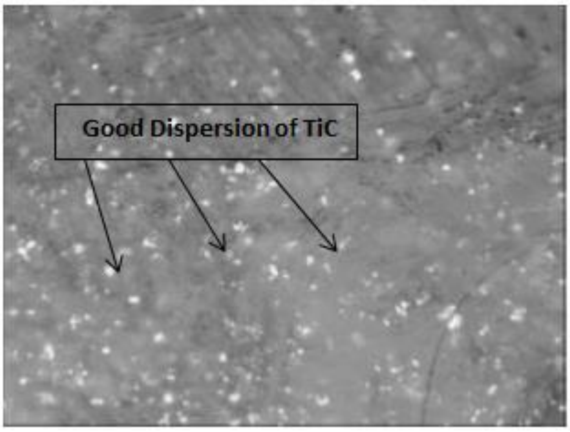


Fig. 6. OM image at 50x showing even dispersion of TiC powder for 15wt%

The shiny particles in Fig. 6 are the TiC powder that is dispersed in the composite uniformly. This proves that the better particles dispersion lead to enhance composite properties. For the 20wt% TiC specimen, a decrease in tensile strength is observed and there is further decreasing in tensile strength for 25wt% TiC specimen. This drop in tensile strength can be explained by the agglomerations and porosity of TiC powder, as shown in Figs. 7 and 8.

The agglomeration of TiC occurs due to high specific surface area which causes an increase in the surface energy of powder and lead to rapid recombination of micro-particulate into agglomerates to compensate unsaturated surface forces via surface reconstruction [17]. The agglomeration leads to creating large pores among agglomerates which lead to decrease in the tensile strength. In general the failure of specimens is caused by cracks around pores that are formed during fabrication and agglomerations. Pores that are trapped in the composite forms empty places in the structure of the composite and help to propagate cracks and allowing the composite to fail at a low stress load. A slight deformation would propagate to crack when even a small load is applied to the composite. The addition of TiC particle increases the brittleness of the epoxy. This is proven by the smooth and flat fracture of the entire composite when it fails. Almost all the fracture was observed to be perpendicular with the direction of the load applied to each composite.

3.4. Effects TiC Ratio on Impact, and Wear

Inorganic particles are frequently employed to improve the mechanical performances of epoxy for engineering applications. Toughness can be improved by the addition of inorganic particles. The resistance to impact (amount of energy absorbed by each specimen before fracture) is one of the key properties of materials. In fact, there is almost a constant rate of increase in material toughness and fracture toughness with the increase of reinforcement material content, as shown in Fig. 9.

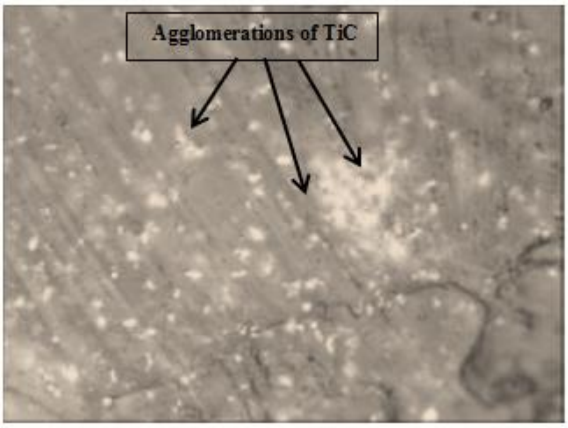


Fig. 7. OM image at 50x of agglomeration of TiC particle in 25wt%

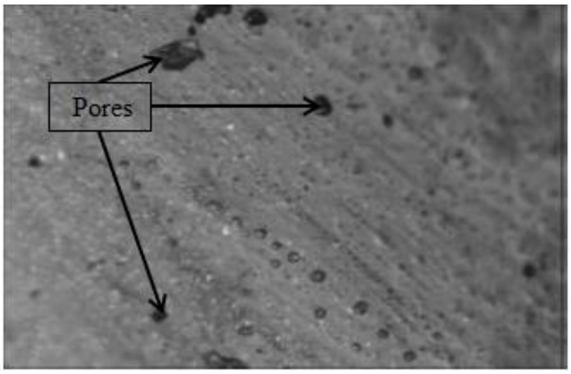


Fig. 8. OM image at 50x of air bubbles in composite

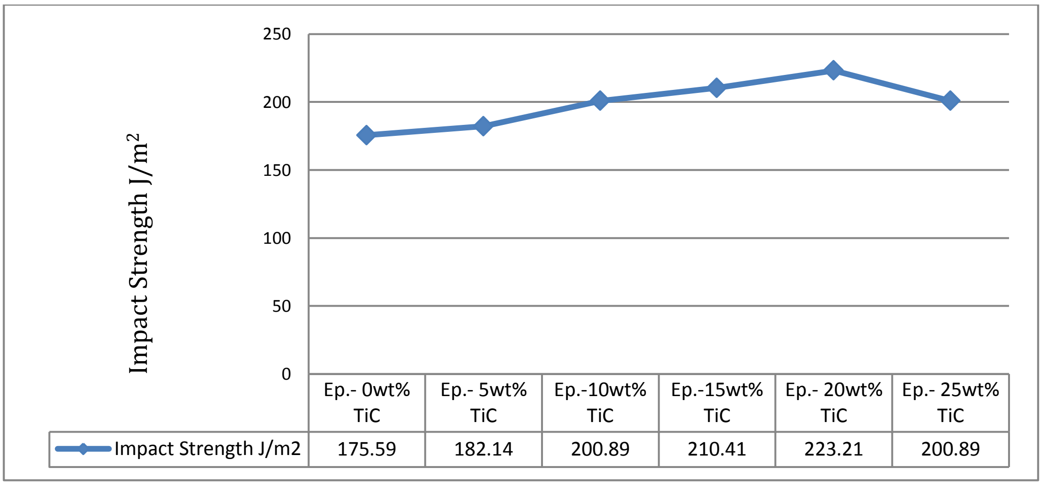


Fig. 9. Average Impact strength value for epoxy matrix composites reinforced with various ratios of TiC

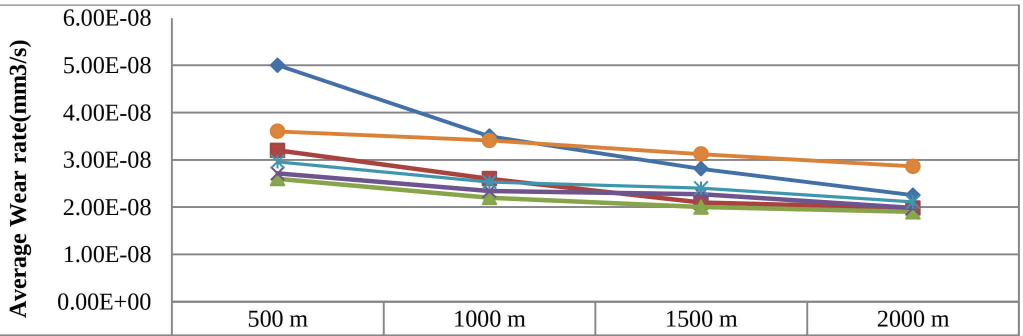
From Fig. 8, it is observed that the impact strength of the pure epoxy composite shows the lowest impact strength at 0.176 kJ/m^2, whereas the 20 wt% has impact strength of 0.223 kJ/m^2. There is a slight increment in the impact strength when the epoxy is reinforced with different ratios of TiC powder. Homogeneously distributed TiC particles are able to improve simultaneously the toughness of the epoxy. Also high specific surface area for TiC particles promotes stress transfer from matrix to TiC particles. With small particle size of TiC, the interparticle distance reduces under a certain filler content. This may activate particle–particle interactions and result in a three-dimensional physical network structure of interphase in a polymer matrix. Good particle–matrix bonding is normally a prerequisite for enhanced fracture toughness [18]. The fracture area of each specimen is flat and smooth regarding wt% of TiC powder. The material behavior that changes after 20 wt% of TiC can be explained by increase of pores which creates a path for crack to propagation. This can be proven by the formation of micro pores that arise during the fabrication of the specimen. Since the composite was mixed manually, formation of micro-pores could not be avoided, even after the use of ultra-sonic vibrator. Furthermore, the decrease in availability of epoxy material to bond all the Titanium Carbide powder is also another reason for the decrease of impact strength after 20 wt%. Addition of hard ceramic TiC particles increases the hardness of the composites and enhances the resistance of the epoxy to the indenter penetration and reduces subsequent removal of material. By increasing TiC to 10wt.% in epoxy matrix the wear rate was lowest, as shown in Fig. 11, and small plough groove, as shown in Fig. 11 (b), compared with pure epoxy and 25wt.% TiC. There are several factors that affect the wear resistance of the composites such as the type of matrix material, the type of reinforcement, additives (graphite), surface roughness, processing technique (powder metallurgy or casting), sliding speed, load, type of friction (dry or lubricated)[19]. As in Table 3, most of these factors are constant to compare between different specimens compositions.

Table 3. Factors that are affect the wear resistance of the composites

Load, P (N)	**40**
Speed, N (rev/m)	500
Test Distance, d (m)	500, 1000, 1500, 2000
Time for each distance, t (seconds)	173, 347, 520, 694
Room temperature, °C	25±2
Contact condition	dry

From Fig. 10, the wear rate for all specimens with different TiC ratios were decreased with increasing sliding distance due to high interfacial adhesion of remaining TiC to synthetic matrix and could be due to filling of the abrasive paper while the rubbing process. Generally, the wear rates begin a gradual decline with the increasing sliding distance until a steady state condition is reached.

During the friction process bigger TiC particles on the friction surface are impressed deeper to the tested surface and are not easily releasable from the surface. On the other hand, smaller TiC particles on the friction surface are easily released from the surface of composite. The larger size of reinforcing particles can offer protection to the matrix during sliding. Once the reinforcing particles fracture or loosen from the matrix, they can be removed easily from the matrix, resulting in a certain amount of material loss. The two surfaces are brought into sliding contact at the beginning; the soft ductile epoxy matrix between TiC particles undergo deformation. The uniform distribution decreases the matrix between TiC particles which lead to decrease the wear and hardness. Generally, some of the wear debris is lost from the system and some entrapped between the contact surfaces. The entrapped debris particles produce further damage on both surfaces; however increasing the amount of hard TiC particles in the debris leads to more damage on both surfaces, as shown in Fig. 11(c). In order to investigate the wear mechanism, the surfaces of the worn composites were examined under OM.



	500 m	1000 m	1500 m	2000 m
0wt% of TiC	5.00E-08	3.50E-08	2.81E-08	2.25E-08
5wt% of TiC	3.20E-08	2.60E-08	2.10E-08	1.98E-08
10wt% of TiC	2.60E-08	2.20E-08	2.00E-08	1.90E-08
15wt% of TiC	2.71E-08	2.34E-08	2.27E-08	1.98E-08
20wt% of TiC	2.96E-08	2.53E-08	2.40E-08	2.11E-08
25wt% of TiC	3.60E-08	3.41E-08	3.12E-08	2.86E-08

Fig. 10. Average wear rate for epoxy matrix composites reinforced with various ratios of TiC particles

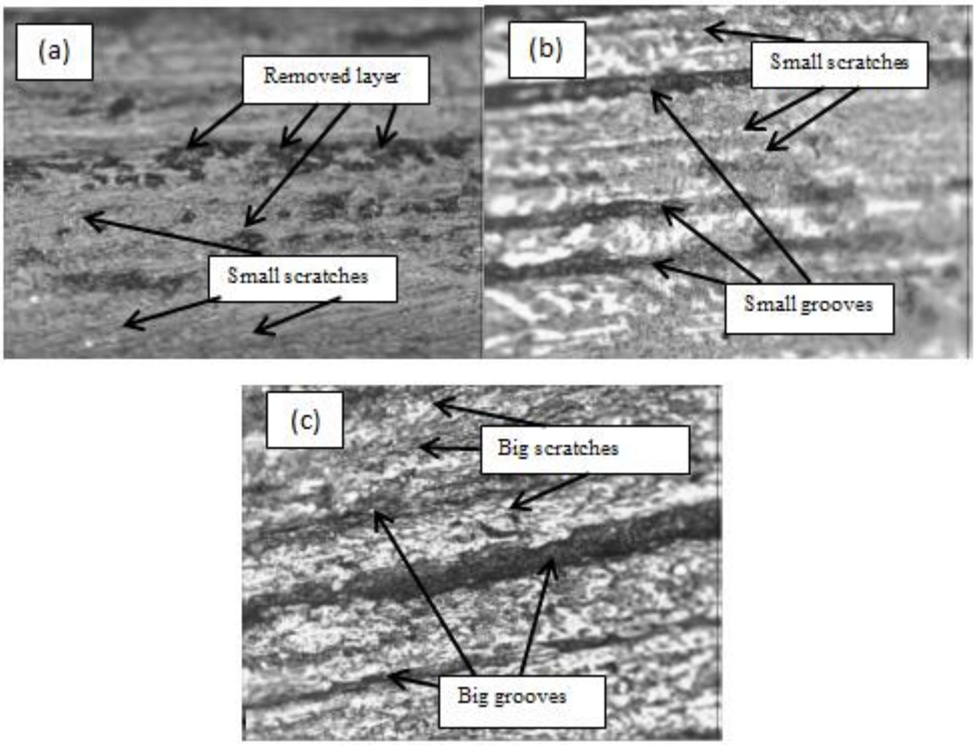


Fig. 11. OM of 0 wt%, 10 wt%, 25 wt% composite at 2000m sliding distance

Fig. 11(a-c) shows typical worn surfaces of the 0 wt.%, 10 wt.% , 25 wt.% TiC composites, respectively. OM for 0 wt. % TiC composite (Fig. 11 (a)) shows a very thin layer removed from some areas with grooves and scratches on the worn surfaces.

OM for 10 wt.% TiC composite (Fig. 11 (b)) shows very small grooves and scratches on the surface, whereas OM for 25 wt.% TiC composite (Fig. 11 (c)) shows many scratches and big grooves on the worn surfaces due to, but with little evidence, particulate fracture, even in composites with the highest weight fraction.

4. Conclusions

Composites reinforced with various ratios of TiC have been synthesized and characterized successfully. The mechanical properties such as hardness, tensile strength, impact, and wear resistance are observed to be increased considerably compared to the matrix composite. Hardness and tensile strength values show increment with addition of 15 wt% of TiC powder whereas hardness and tensile strength values decreased for specimens with (20 wt% and 25wt%) TiC due to agglomeration of TiC particles (Fig. 6) and porosity (Fig. 7) during mixing process. Impact strength had increment with ratio of 20 wt% of TiC. Wear rate improved greatly at 10 wt% TiC. OM for 0 wt.% TiC composite (Fig. 10 (a)) shows a very thin layer removed from some areas with grooves and scratches on the worn surfaces, whereas OM for 25 wt.% TiC composite (Fig. 10 (c)) shows many scratches and big grooves on the worn surfaces.

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