Mechanical and morphology properties of titanium oxide-epoxy nanocomposites

Tayser Sumer Gaaz[1,*], Emad Kamil Hussein[1], Kussay Ahmed Subhi[1] and Ahmed Al-Amiery[2]

[1]Technical College Al-Musaib, Al-Furat Al-Awsat Technical University, Al-Musaib, Babil 51006, Iraq; [2]Energy and Renewable Energies Technology Center, University of Technology, Baghdad 10001, Iraq

Abstract

In the current work, the influences of titanium oxide (TO) on nanocomposites using epoxy (EP) resin have been investigated. EP resin was added to various contents in terms of volume of TO (6%). Based on the casting method, the TO-EP nanocomposites were prepared. Mechanical properties in terms of tensile, impact and hardness properties exhibited improvements in the mechanical results of the TO-EP nanocomposites. The experimental results exhibited that the enhancement in the tensile strength, impact, and hardness was found to be 14 MPa, 40.1 J/mm^2 and 228.9 HV, respectively. Moreover, the effect of the strength has decreased compared with pointing to brittleness that increased the TO-EP nanocomposites. Field emission scanning electron microscopy (FESEM) analysis was used to perform a microstructure analysis which in turn revealed how TO obstruction the cracks propagation in the nanocomposites. The interface between the TO and the EP nanocomposites was observed using the FESEM images. The results highlighted that the TO-EP nanocomposites can be used a wide range of industrial applications such as biomedical application.

Keywords: titanium oxide; nanocomposite; mechanical properties; impact strength; tensile strength;

*Corresponding author: taysersumer@atu.edu.iq

Received 2 June 2020; revised 10 July 2020; editorial decision 21 July 2020; accepted 21 July 2020

1. INTRODUCTION

Titanium oxide (TO) nanocomposites can be defined as carbon-based materials that can be used to deliver exceptional performance at low cost. Moreover, TO nanocomposites have high tensile strength and Young's modulus [1–4] where they were utilized in various aerospace and mechanical applications. Since the 1950s, carbon fiber composites have been used and found widespread where have considerable mechanical properties with good features such as easy to manufacture and lightweight [5–9]. In order to enhance the mechanical properties of the parent polymer material [10, 11], TO is utilized as fillers in polymer composite system [12]. TO-epoxy (EP) systems able to deliver strong and light materials with several advantageous characteristics. In an elastic EP matrix, the effect of TO was investigated in [13] by adding 6 vol.% of TO in order to improve the micro hardness, Young's modulus and impact strength. However, their proposed method to prepare the solution mixture was the inefficient to obtain efficient distribution of the TO within the EP substrate; hence, a microscopic specific test indicates that some conglomerate tens of a large micrometres afterward the mixing were found. The mechanical properties of EP-based nanocomposites have been investigated using sonication and surfactants after dispersing TO as reported in Kalali *et al.* [14, 15].

Li *et al.* [16] presented the modulus of EP-based composites. Ma *et al.* [17] achieved substantial progress in the dispersion and adhesion of EP matrix and carbon nanotubes based on the nanotubes functionalization. Where, TO can be made a portion of the EP network where the functionalization leads to increase the polarity. Previous studies showed that the homogeneous dispersion was achieved when TO was utilized to reinforce EP-based nanocomposites [14, 18–20]. While in the current study, the aim is to increase the content of the TO and the homogeneity of dispersion without using the solvent. Where, in the EP matrix, the part of the TO is increased leading to enhance the mechanical properties with improvement in the tensile strength and hardness. The TO-EP nanocomposites are brittle due to the reduction in the effect of strength.

International Journal of Low-Carbon Technologies 2021, 16, 240–245

doi:10.1093/ijlct/ctaa058 Advance Access publication 24 August 2020

Table 1. *Ratio of the nanocomposition in terms of EP resin, TO and hardener.*

TO-EP nanocomposites	Percentage cont. of TO (vol.%)	EP	
		Resin (vol.%)	**Hardness (vol.%)**
Intrinsic EP	0.0	75	25
EP + 1.5 vol.%	1.5	69	29.5
EP + 3.0 vol.%	3.0	68	29
EP + 4.5 vol.%	4.5	67	28.5
EP + 6.0 vol.%	6.0	66	28

2. MATERIALS AND METHODS

2.1. Material

2.1.1. *Titanium oxide*

In the current work, the TO was provided by the University Kebangsaan Malaysia, Malaysia. Where a modified Hummers technique was employed with potassium permanganate as oxidizing agents and sulfuric acid in order to get a natural dispersion of the TO. HCl (5%) and DI H_2O were used to purify the product and ultra-sonication was utilized to exfoliate it, where the TO dispersion has been obtained.

2.1.2. *Matrix*

The EP system consists of 25% hardener and 75% clear EP resin where the amounts of TO is varied to get the nanocomposites. The viscosity of the clear EP resin was ranged between 11 000 and 14 000 cps. In addition, a non-methylene dianiline aromatic amine that is prepared by the Daemyung Chemical Technology to be used as a curing catalyser (i.e. Amicure 101) has been employed in order to obtain an accurate wetting boundary condition to the TO strengthening filler.

2.1.3. *Preparation of nanocomposites (TO-EP)*

The sample preparation and the experimental procedure were conducted according to the flow chart shown in Figure 1. In terms of volume, various percentages of TO namely: (0, 1.5, 3, 4.5, 6) have been utilized to get blends of EP with equivalent ratio of EP of 75:25 where the mixture of the nanocomposites was accomplished after 10 min at a temperature of 25°C where the prepared sample was placed in the oven at 3.5 h. The oven has been set at 40°C in order to dispose of moisture that can be adsorbed on the surface. The room temperature at 25°C for 48 h, the reaction between TO and EP was achieved. Table 1 shows the contents of TO and EP.

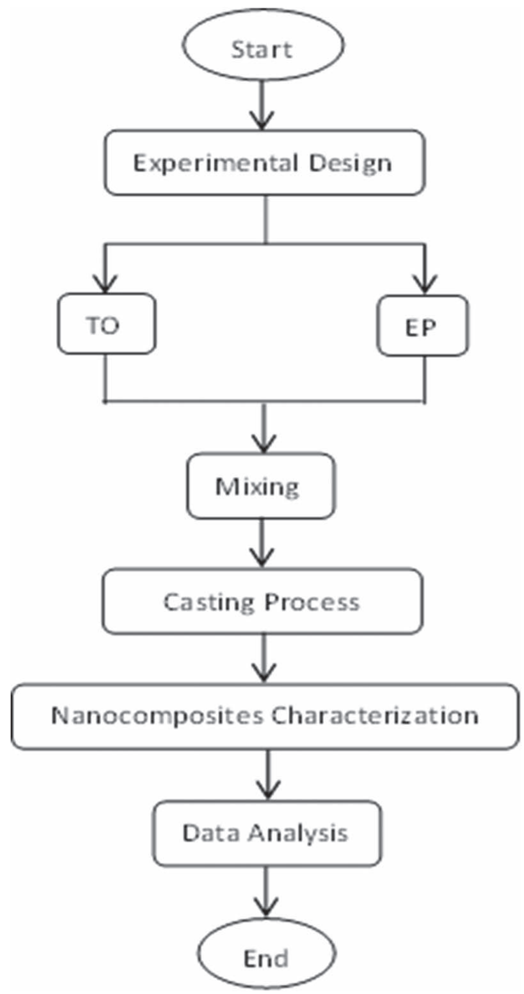

Figure 1. *Sample preparation and experimental set-up.*

2.2. Mechanical properties

2.2.1. *Tensile properties*

For the tensile test specimen purpose, dog bone-shaped as shown in Figure 2. A machine from Instron Company was utilized to carry out the mechanical ductile test at a constant temperature of 25°C and fixed extension rate of 1 mm/min as shown in Figure 3. In order to enable a reasonable analysis with materials for flexible and ductile, the loading percentage ratio at a slight value was kept. Moreover, the tensile strength, strain, modulus and % elongation were measured based on Bluehill software. The American Society for Testing and Materials (ASTM) standard D638 was applied after the tensile test where five samples of the contents of TO from 0 to 6 vol.% were involved.

2.2.2. *Impact properties*

In order to carry out the Charpy impact tests, CEAST 6545 pendulum has been utilized based on the common standard

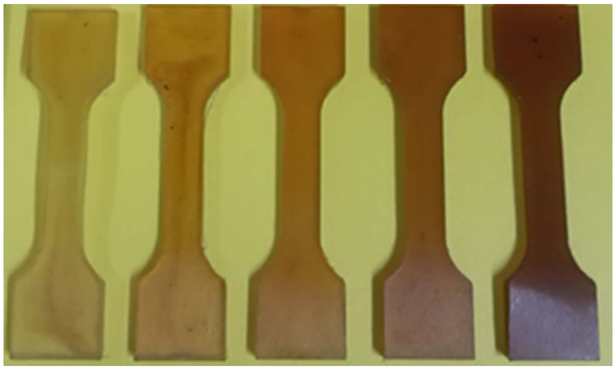

Figure 2. *Tensile sample for various contents of TO vol.%.*

Figure 3. *Universal testing machine.*

(ASTM D 638). The specimens' sizes of 11.4 cm × 1.2 cm × 0.2 cm were installed in a straight manner with the long axis is complying with the horizon as a simple beam from both ends as displayed in Figure 4. The nick was shaped at the center of each sample. The total fracture energy based on the Charpy impact strength in order to break the sample where it is calculated for a sample with a single nick based on the following equation:

$$(a_c u) = \frac{w}{h.b} \times 10^{-3}, \tag{1}$$

2.2.3. *Hardness properties*

A micro-hardness tester was used to obtain average readings of hardness by measuring two points. The unit was set based on to

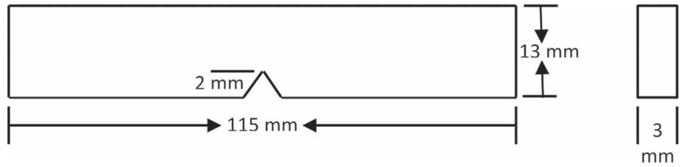

Figure 4. *Charpy model impact specimen.*

Figure 5. *Shore hardness specimen.*

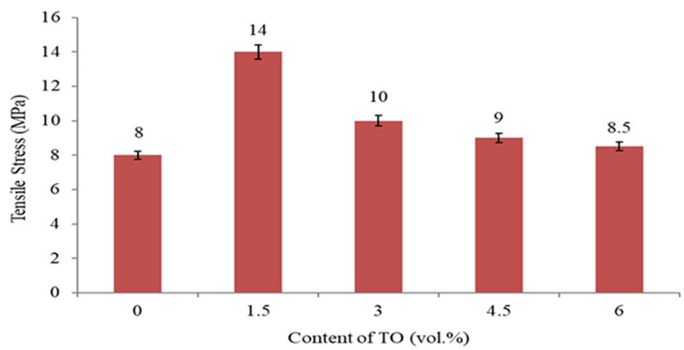

Figure 6. *Tensile strength of TO-EP nanocomposites at various contents of TO.*

Vickers hardness and the indentation average diagonal length was measured as indicated in Figure 5. Vickers hardness was obtained based on Equation (2) as follows:

$$\begin{aligned} HV &= 2F\sin 136^\circ/2/d^2 or \\ \mathrm{HV} &= 1.854F/d^2\ (\text{approximately}) \end{aligned} \tag{2}$$

3. RESULTS AND DISCUSSION

3.1. Tensile properties

According to the tensile tests results, the tensile strength, Young's modulus, load and elongation increased with adding TO, while the fracture strain was decreased [21, 22]. As can be seen in Figure 6, tensile strength increased from 8 to 14 MPa when the TO was added into the EP nanocomposites. Where the maximum strength of 14 MPa was obtained at the content of TO of 1.5 vol. while the increasing of the content of TO beyond this value leads

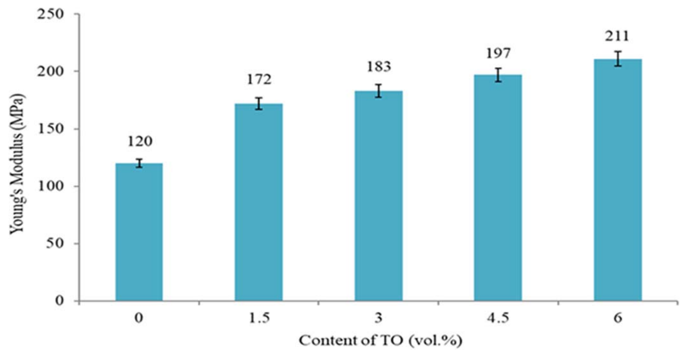

Figure 7. *The effect of TO-EP of nanocomposites on Young's modulus at various TO contents.*

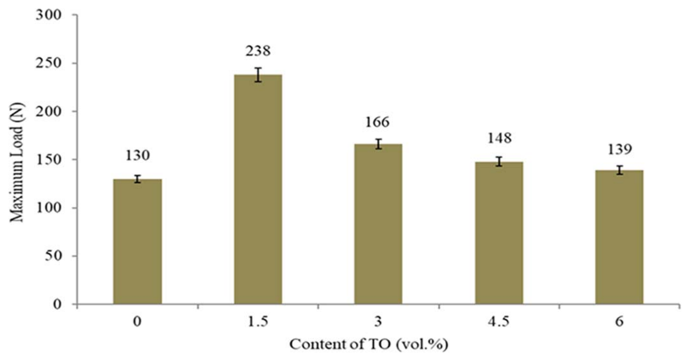

Figure 8. *The effect of TO-EP nanocomposites on max. Load at various TO contents.*

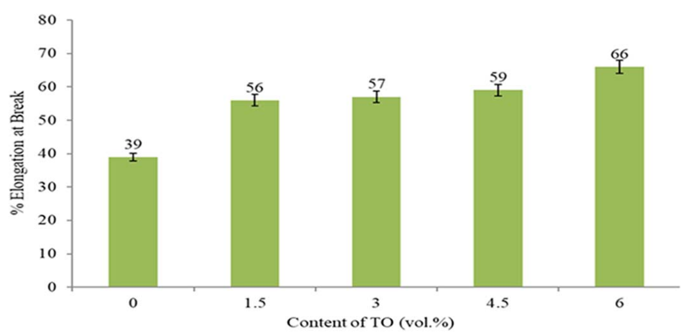

Figure 9. *The effect of TO-EP nanocomposites on elongation at break at TO various contents.*

to decrease the tensile strength to 8.5 MPa. In order to carry out parametric studies, the effects of addition of TO on the nanocomposites mechanical properties were investigated in terms of oxidative treatment, structure and concentration level. Where, the TO content influence on the EP nanocomposites Young's modulus is shown in Figure 7. For the concentration range of the TO content between 0 vol.% and 6 vol.%, Young's modulus obviously increased from 120–211 MPa. While the dispersion and surface functionality of the material is firstly insufficient, the properties of the nanocomposites were improved by adding the TO. Moreover, the compatibility between the EP matrix and TO led to improve the interfacial adhesion. The maximum tensile strength of 238 N

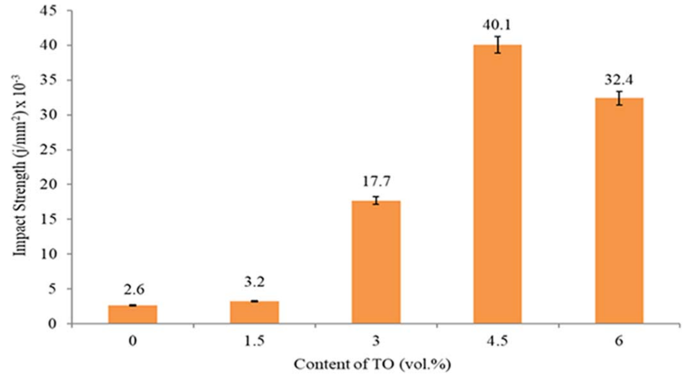

Figure 10. *The variation of impact strength with TO content.*

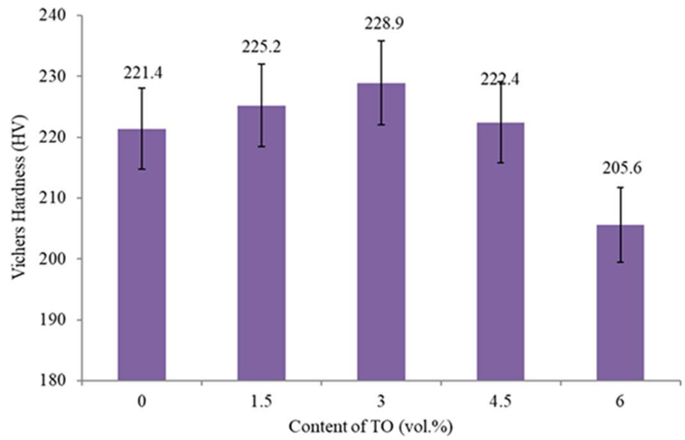

Figure 11. *Effects of TO content on nanocomposites hardness.*

in terms of applied load on the TO-EP nanocomposites was at 1.5 vol.% content of the TO. At the TO content of about 1.5 vol.%, where the applied load is increased as of 130 up to 238 Newton. As can be seen in Figure 8, the applied load has been decreased intensely from 238 reaching to 139 Newton as the content of the TO was varied from 1.5 vol.% to 6 vol.%. The effect of the content of TO on the elongation was shown in Figure 9 where the elongation increased with increasing the break. At TO content of 6 vol.%, the elongation value was 66% compared with 56% at TO content of 1.5 vol.%.

3.2. Impact properties

Figure 10 presents the results of the test of the Charpy impact where five samples of single-notched were utilized. When TO was added with the content of 4.5 vol.% that led to increase the total gained impact energy from 2.6 as an initial value up to 40.1 J/mm^2. While the impact strength reduced to a confirmed value of 32.4 J/mm^2 when TO composition was 6% (volumetric percentage ratio%). The EP nanocomposites matrix combined with TO led to enable the formation of the chemical bond with EP the filler and resin [23].

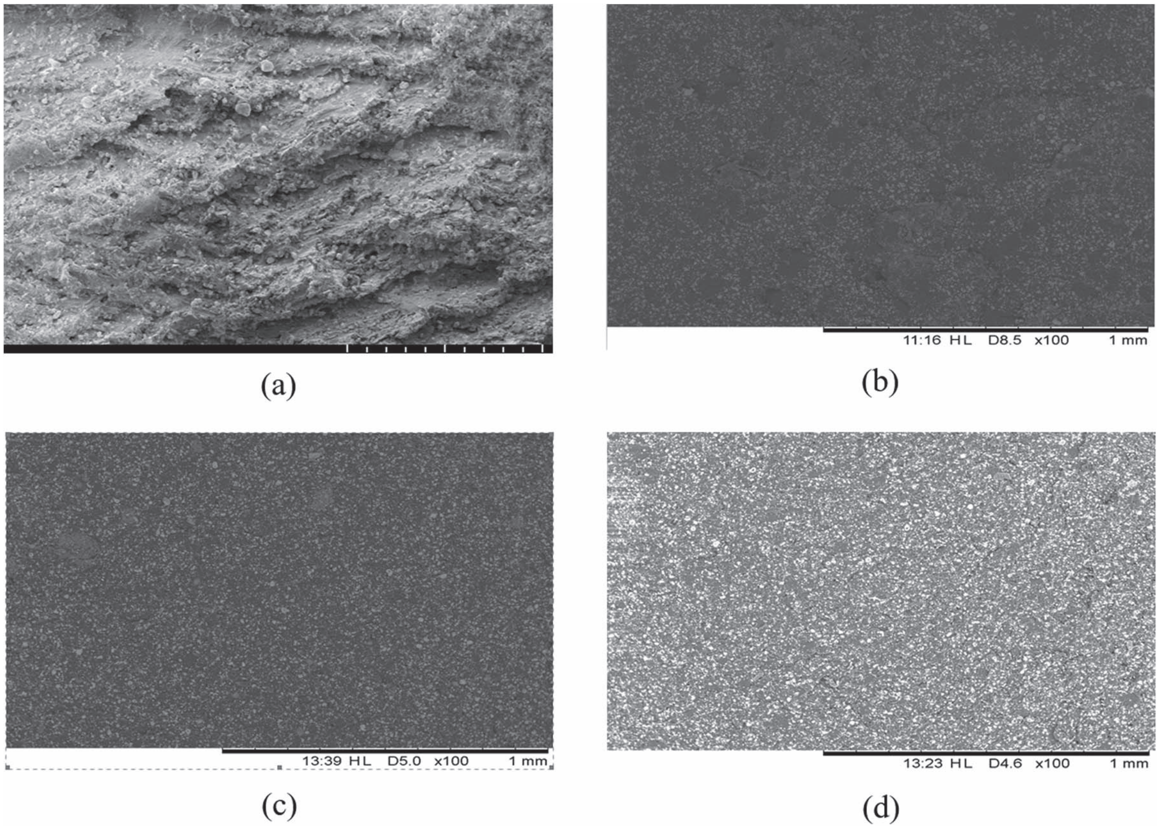

Figure 12. *Tensile samples images of FESEM fracture surface (**a**) at pure EP (**b**) at TO content of 3 vol.% (**c**) at TO content of 6 vol.% (**d**) influence fracture surface at TO content of 6 vol.%.*

3.3. Properties of microhardness

The TO effect on the EP matrix hardens is clearly illustrated in Figure 11 below. It can be seen that the hardness was 225.2 HV at 1.5 vol.% TO. While the hardness increased to reach the maximum value of 228.9 HV at TO content of 3.0 vol.%. Moreover, it can be found that the lowest value of Vickers hardness was obtained at TO of 6 vol.%, so this increment may be accredited to the proposed value of the aspect ratio of TO and high modulus. The voids generated through the fabrication increased at higher TO content. Moreover, the results of the hardness test indicated that the cooled samples were better than the sample at normal environment conditions (i.e. at room temperature).

3.4. Field emission scanning electron microscopy

Figure 12 shows the obtained images from the field emission scanning electron microscopy (FESEM) for tensile fracture surfaces with pure EP resin. From Figure 12a, it can be noted that a smooth fracture surface was obtained at ×100. The sample with 1.5 vol.% TO is shown in Figure 12b where the fracture surface comes to be cloud-like and rougher due to adding more TO. The growth of a micro cracks in a random manner are indicating that the TO grid considered as an undesired case as a results of wide specific surface area. Moreover, the rising adhesion component with the matrix was identical solid. Consequently, the addition of TO led to enhance the nanocomposites strength, while the TO addition more than 1.5 vol.% led to the development of clusters through the TO network in the micron scale. The strength of the nanocomposites was deteriorated due to the stress transfer from the resin matrix to the TO network which was discovered in more details in [24]. The cross sectional generated area of the fracture surface of the nanocomposites beyond a tensile test at 6 vol.% TO is shown in Figure 12c. From FESEM image, it can be seen from FESEM image that TO was normally discrete suitably in the matrix [25]. For the impact test at 4.5 vol.% TO, the morphology of the fractured surfaces is shown in Figure 12d and indicated that the fracture surface was non-uniform. Due to the TO agglomerates, the polymer flow was hindered. The indicator of brittleness with a coarser topography surface can be seen in the surface of the nanocomposite than neat EP. Also, in the EP matrix, the pull-out of the TO decreased.

4. CONCLUSIONS

Based on its outstanding mechanical features and excessive aspect ratio, TO has great potential. Therefore, in the current work, the effects of TO on EP matrix nanocomposites was investigated. The results showed that adding TO with volume of 1.5% to the EP matrix led to improve the nanocomposites in terms of Young's modulus, hardness and the tensile strength compared with the pure EP matrix. The nanocomposites homogeneity showed a considerable impact on the mechanical properties of nanocomposites. Where it showed that Young's modulus increased with increasing the TO volume from 1.5% to 6%. Moreover, adding TO, the impact and tensile strength increased. The results showed the potential advantages of using TO-EP nanocomposites in various applications such as biomedical applications.

AUTHOR CONTRIBUTIONS

T.S.G. did all of the experiments as part of his project and wrote the manuscript. E.K.H. and K.A.S. were the principal investigators and supervised the whole work. A.A.-A. helped with characterization of the manuscript. All authors are aware of this manuscript and have agreed to its publication.

CONFLICTS OF INTEREST

The authors declare no conflict of interest.

REFERENCES

[1] Brennan LJ, Byrne MT, Bari M *et al.* Carbon nanomaterials for dye-sensitized solar cell applications: a bright future. *Adv Energy Mater* 2011;**1**:472–85.
[2] Hauert R, Patscheider J. From alloying to nanocomposites—improved performance of hard coatings. *Adv Eng Mater* 2000;**2**:247–59.
[3] Nakane K, Kurita T, Ogihara T *et al.* Properties of poly (vinyl butyral)/TiO2 nanocomposites formed by sol–gel process. *Compos Part B Eng* 2004;**35**:219–22.
[4] Shirkavand S, Moslehifard E. Effect of TiO2 nanoparticles on tensile strength of dental acrylic resins. *J Dent Res Dental Clin Dent Prospects* 2014;**8**:197.
[5] Fratzl P, Gupta H, Paschalis E *et al.* Structure and mechanical quality of the collagen–mineral nano-composite in bone. *J Mater Chem* 2004;**14**:2115–23.
[6] Soutis C. Carbon fiber reinforced plastics in aircraft construction. *Mater Sci Eng A* 2005;**412**:171–6.
[7] Clyne T, Hull D. 2019. *An Introduction to Composite Materials*. Cambridge University Press.
[8] Mallick PK. 2007. *Fiber-Reinforced Composites: Materials, Manufacturing, and Design*. CRC Press.
[9] Kumar A, Sharma K, Dixit AR. A review of the mechanical and thermal properties of graphene and its hybrid polymer nanocomposites for structural applications. *J Mater Sci* 2019;**54**:5992–6026.
[10] Gaaz T, Sulong A, Ansari M *et al.* Effect of halloysite nanotubes loading on thermo-mechanical and morphological properties of polyurethane nanocomposites. *Mater Technol* 2017;**32**:430–42.
[11] Wu Y, Song L, Hu Y. Fabrication and characterization of TiO2 nanotube–epoxy nanocomposites. *Ind Eng Chem Res* 2011;**50**:11988–95.
[12] Masood S, Song W. Thermal characteristics of a new metal/polymer material for FDM rapid prototyping process. *Assembly Autom* 2005;**25**:309–15.
[13] Li L, Zou H, Shao L *et al.* Study on mechanical property of epoxy composite filled with nano-sized calcium carbonate particles. *J Mater Sci* 2005;**40**:1297–9.
[14] Kalali EN, Wang X, Wang D-Y. Functionalized layered double hydroxide-based epoxy nanocomposites with improved flame retardancy and mechanical properties. *J Mater Chem A* 2015;**3**:6819–26.
[15] Liao S-H, Liu P-L, Hsiao M-C *et al.* One-step reduction and functionalization of graphene oxide with phosphorus-based compound to produce flame-retardant epoxy nanocomposite. *Ind Eng Chem Res* 2012;**51**:4573–81.
[16] Li W, Shang T, Yang W *et al.* Effectively exerting the reinforcement of dopamine reduced graphene oxide on epoxy-based composites via strengthened interfacial bonding. *ACS Appl Mater Interfaces* 2016;**8**:13037–50.
[17] Ma P-C, Siddiqui NA, Marom G *et al.* Dispersion and functionalization of carbon nanotubes for polymer-based nanocomposites: A review. *Compos A: Appl Sci Manuf* 2010;**41**:1345–67.
[18] Pinto D, Bernardo L, Amaro A *et al.* Mechanical properties of epoxy nanocomposites using titanium dioxide as reinforcement—a review. *Construct Build Mater* 2015;**95**:506–24.
[19] Song P A, Yu Y, Zhang T *et al.* Permeability, viscoelasticity, and flammability performances and their relationship to polymer nanocomposites. *Ind Eng Chem Res* 2012;**51**:7255–63.
[20] Yuan B, Bao C, Qian X *et al.* Synergetic dispersion effect of graphene nanohybrid on the thermal stability and mechanical properties of ethylene vinyl acetate copolymer nanocomposite. *Ind Eng Chem Res* 2014;**53**:1143–9.
[21] Long M, Rack H. Titanium alloys in total joint replacement—a materials science perspective. *Biomaterials* 1998;**19**:1621–39.
[22] Kim Y-W. Intermetallic alloys based on gamma titanium aluminide. *Jom* 1989;**41**:24–30.
[23] Fellahi S, Chikhi N, Bakar M. Modification of epoxy resin with kaolin as a toughening agent. *J Appl Polym Sci* 2001;**82**:861–78.
[24] McAllister MJ, Li J-L, Adamson DH *et al.* Single sheet functionalized graphene by oxidation and thermal expansion of graphite. *Chem Mater* 2007;**19**:4396–404.
[25] Avramovic-Cingara G, Ososkov Y, Jain M *et al.* Effect of martensite distribution on damage behaviour in DP600 dual phase steels. *Mater Sci Eng A* 2009;**516**:7–16.