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Effect of $al_2o_3$and mwcnts nanofillers on the mechanical characteristics of epoxy-based polymeric matrix nanocomposites

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ABSTRACT

In aerospace applications research efforts are focusing on the design of advanced composite materials reinforced with ceramic nano-fillers. Such advanced materialscombine weight saving with multifunctional properties, including thermal, mechanical and electromagnetic ones. In the present investigation, the effect of dispersion of bothmulti-wall carbon nanotubes (MWCNTs) and Al_2O_3 nanoparticles on the mechanical characteristics of epoxy matrix was studied. The results revealed that, themicrohardness, impact energy and flexural strength were improved by dispersion of MWCNTs and Al_2O_3 nanoparticles. However, the tensile strength wassignificantly reduced by dispersion of the aforementioned nanofillers.

KEYWORDS

Polymer matrix nanocomposites;
MWCNTs;
Nano-sized Al_2O_3;
Mechanical characteristics.

INTRODUCTION

In the recent years, there is a great need for composites because the combination of two or more materials can lead to enhance performance and outstanding properties compared to their constituents. Especially, the polymer based composites reinforced with a small percentage of strong ûllers can signiûcantly improve the mechanical, thermal and barrier properties of the pure polymer. Moreover, these improvements are achieved through conventional processing techniques without any detrimental eûects on processability, appearance, density and aging performance of the matrix. The realization of their unique properties, has been considering for a wide range of applications including packaging, coating, sport, electronics, aerospace industries, aircraft and military, automotive, and marine engineering[1]. One part of composite material for technical applications may be represented by a thermosetting polymer matrix, e.g. an epoxy resin, which already covers alone some of the demanded properties[2]. Epoxy resins (EP) have been widely used in practical applications such as adhesives, construction materials, composites, laminates and coatings owing to their excellent mechanical properties, low cost, ease of processing, good adhesion to many substrates, and good chemical resistance[3]. However, because the polymer matrix must withstand high mechanical and tribological loads, it is usually reinforced with ûllers. These ûllers can be chosen as ûbres (glass, carbon and aramid) or particles such as ceramic powders[2].

Nanoparticles have been used as ûllers in polymeric composites for improving the mechanical per-

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formance of the materials. One of the distinct advantages of nanocomposites over microcomposites lies in that the performance improvement is often acquired at relatively low concentration of the nanoûllers. This is beneûcial to the mechanical properties, processability and aesthetic appearance of the end-products[4]. Carbon nanotubes (CNTs) are increasingly attracting scientiûc and industrial interest by virtue of their outstanding characteristics. The CNT walls resemble rolled-up graphite-like sheets. According to their graphitic structure, CNTs possess high thermal conductivity and an electrical conductivity that can be either semi-conducting or metal like[3]. Alumina (Al_2O_3)nanoparticles represents the ceramic nanocrystalline phase of the composites[2]. Ceramic–polymer composites are functional materials of a great potential for industrial applications. These composite materials combine hardness, stiffness and wear resistance of ceramics and elasticity of polymer[5,6].

The aim of the present work is to study the mechanical performance of epoxy based composites reinforcedwith MWCNTs and Al_2O_3 nanoparticles. Thevariation of tensile strength, hardness, impact energy and flexural strengthas a function of the content of the aforementioned nanofillers was evaluated.

EXPERIMENTAL PROCEDURES

Materials

Epoxy resin was used as a matrix material. Epoxy resin is a thermoset resin with good thermal and environmental stability. The type of epoxy resin used inthe present investigation is KEMAPOXY 150 manufactured by Chemicals forModern Buildings (CMB) Company, Egypt. Two nanoscale fillers, typically, the multi-walled carbon nanotubes (MWCNTs) and aluminium oxide (Al_2O_3) ceramic nanocrystalline phase in the form ofnanoparticles were used as reinforcing agents. The MWCNTs haveinner and outer diametersof20 and 30 nm, respectively. The Al_2O_3nanoparticulates have an average diameter of 10 nm. Figure Nanocomposites containing up to 1% weight percent of the nanofillers wereproduced. Figure 1 shows high resolution transmission electron microscope (TEM) micrograph of both the MWCNTs and Al_2O_3 nanoparticles.

Nanocomposites preparation

The nanocomposites were prepared using the following technique: (1) the epoxyresin and a certain weight percent of the nanofillerwere mixed together inplastic mould, and stirred mechanically SUPERMIX homogenizer (model DEPOSE) at 1000 r.p.m for twenty minutes in roomtemperature; (2) the hardener was added to the mixture by ratio 1:2 by volume andthen stirred mechanically again for three minutes; (3) The epoxy/nanofiller slurry was poured

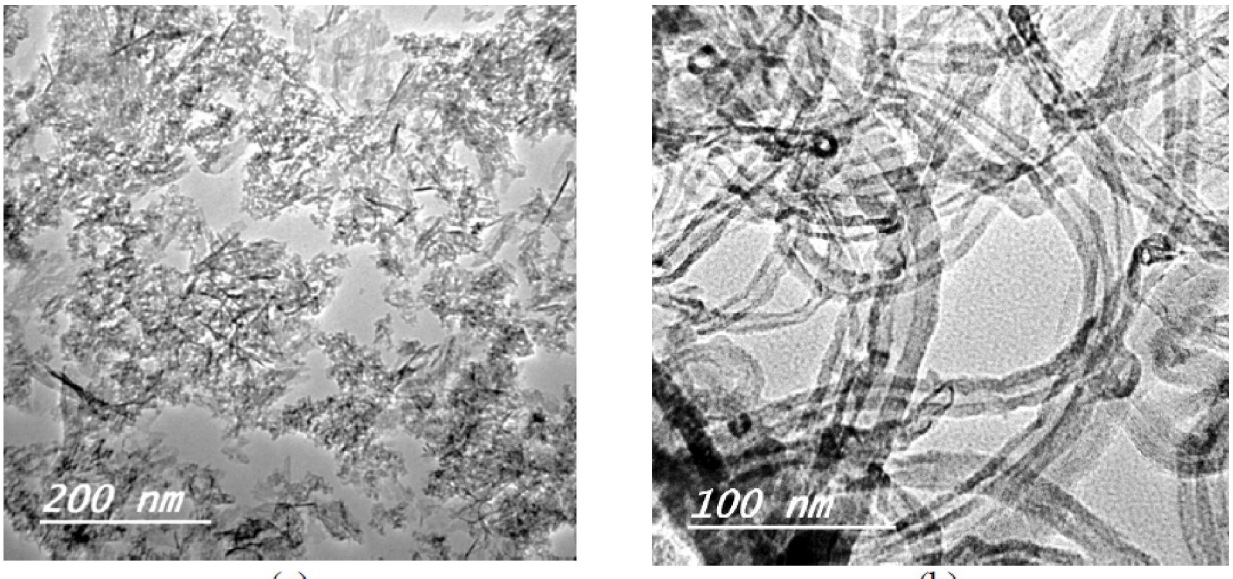

Figure 1 : TEM micrographs of the (a) Al_2O_3 nanoparticles and (b) MWCNTs

in silicon dies have different shapes according to the required tests; (4) finally, the mixture was allowed tofully hardened at room temperature. Nanocomposites were prepared by dispersing 0.25 wt.-%, 0.5 wt.-%, 0.75 wt.-% and 1 wt.-% of MWCNTs and Al_2O_3nanoparticles.

Mechanical tests

Tensile tests of the nanocomposites were performed according to ASTM D 638-03 standard (Standard Test Method for Tensile Properties of Plastics). The tensile tests were conducted using a universal testing machineat constant cross head speed of 10 mm/min and a dynamic extensometer to detect the strain of the specimens during tests. The shape and size of the tensile specimens shown in Figure 2a. The load-displacement results were analysed to calculate the tensile strength of nanocomposite samples. The microhardness of the nanocomposites was measured using Vickers indenterby applying a load of 200 g for 10 seconds. Minimum of five readings were taken for each sample and the averagevalue was determined.

CharpyImpact testing was performed using Zwick/Roellimpact tester according to DIN-ISO179 at room temperature. Figure 2b shows a schematic illustration of a typical notched impact specimen. In each condition, five specimens were tested and the average value of the data were reported. Three-point bending tests were performed using a universal testing machine. Bending specimens were fabricated according to ASTM D790-82, with dimensions shown in Figure 2c. The span distance was about 60 mm. The bending (flexure) strength, ($\sigma_{b)}$ was calculated from the load deflection curves using the following equation:

$$\sigma_b = 3P_{max}\ L/2bh^2 \quad (1)$$

Where: P is machine load, L isthe span length (mm),b is specimen width (mm) and h: specimen thickness

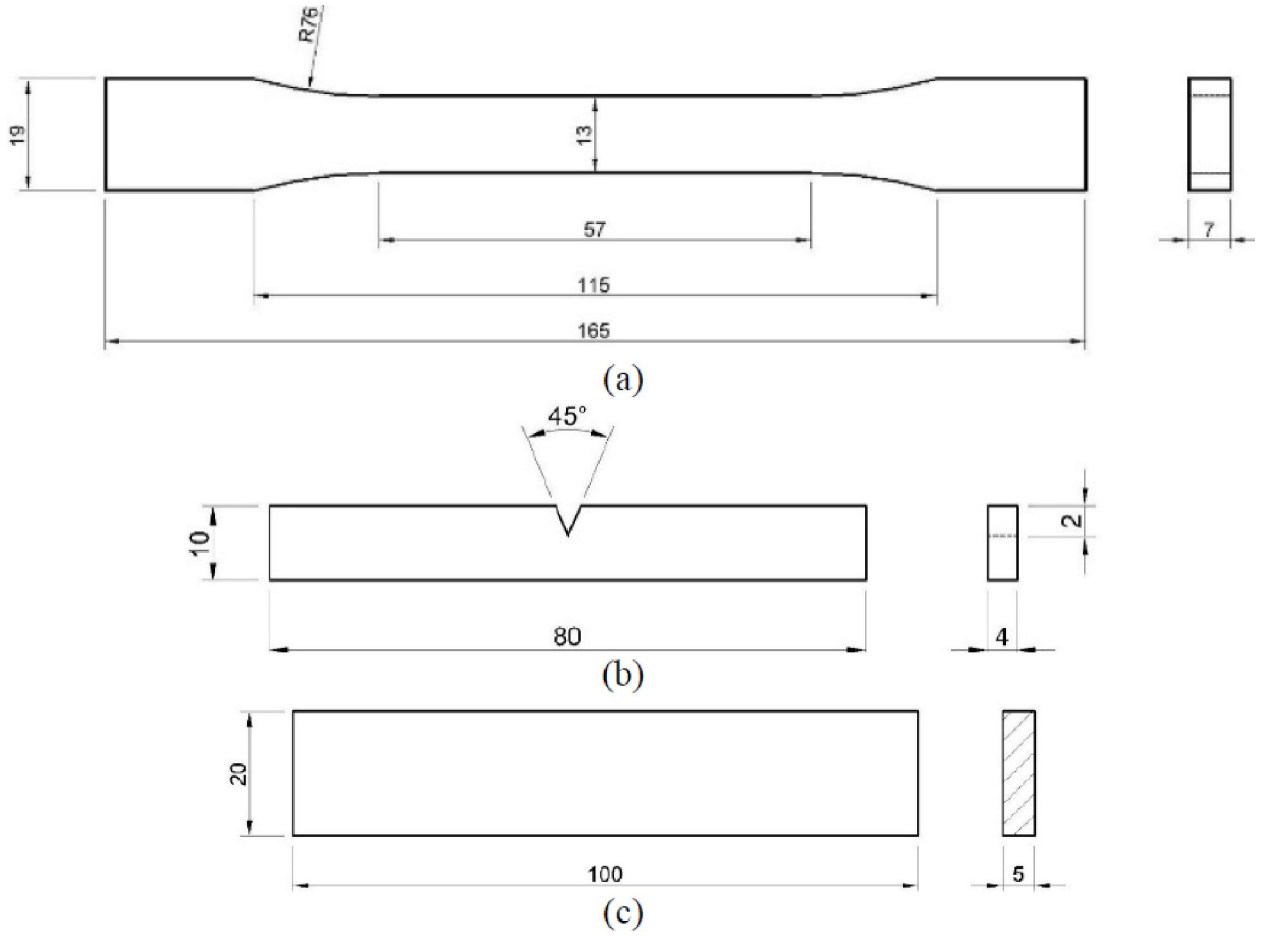

Figure 2 : Schemtic illustration of (a) tensile specimen, (b) impact specimen and (c) bending specimen (dimensions in mm)

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(mm). The morphology of fracture surfaces of tensile and bending failed specimens were examined using scanning electron microscope (SEM).

RESULTS AND DISCUSSION

Tensile strength of the nanocomposites

Figure 3 shows the variation of the ultimate tensile strength of the nanocomposites with the nanofillers weight percent. The results revealed that dispersion of both MWCNTs and Al_2O_3nanopaticles tend to reduces the ultimate tensile strength of the nanocomposites. The pure epoxy matrix exhibited higher ultimate tensile strength when compared with both epoxy/MWCNTs and epoxy/Al_2O_3 nanocomposites. For example, the ultimate tensile strength of nanocomposites decreasedby about 33% and 46% by dispersion of 0.25 wt.-% of MWCNTs and Al_2O_3nanopaticles, respectively. Increasing the weight percent of the MWCNTs and Al_2O_3 dispersed in the epoxy matrix reduces the ultimate tensile strength of the nanocomposites. The epoxy/MWCNTs nanocomposites exhibited slightly higher tensile strength when compared with the epoxy/Al_2O_3 nanocomposites. The reduction of the tensile strength with increasing the nanofiller weight percent may explained by the increase of the agglomeration sites of the nanofillersin the epoxy matrix,with the higher ûller contents. The agglomeration sites act as crack initiation sites which lead to the nanocomposite failure[7-9]. Moreover, increasing the nanofillercontent leads to the formation of agglomerations that are in the scale of microns within the nanocomposite. This inhibits the stress transfer from the resin matrix to the MWCNTs.

Figure 4 shows SEM micrographs of the fractured surfaces of the failed pure epoxy samples as well as the nanocomposites tensile specimens. The pure epoxy specimens showed a smooth fracture surfaces, as shown in Figure 4a, which indicates that brittle fracture was occurred. Figure 4b shows SEM micrograph of a typical fractured surface of epoxy/MWCNTs nanocomposite containing 1 wt.-% of MWCNTs. It is clear from the micrograph that the epoxy/MWCNTs nanocomposites failed specimens exhibitedrough fractured surfaces. Suchrough fractured surfacesmay attribute to the crack propagation that occurs when the crack meets the MWCNTs or an agglomerate of them. Figure 4c shows SEM micrograph of a typical fracture surface of epoxy/Al_2O_3 nanocomposite containing 1 wt.-% of Al_2O_3 nanoparticles. It is clearly seen that the fractured surface of the failed epoxy/Al_2O_3 nanocomposite tensile samples is smoother than the surface of epoxy/MWCNTs nanocomposites. However, clusters of the Al_2O_3nanoparticles are clearly seen on the fractured surface.

Micro-hardness of nanocomposites

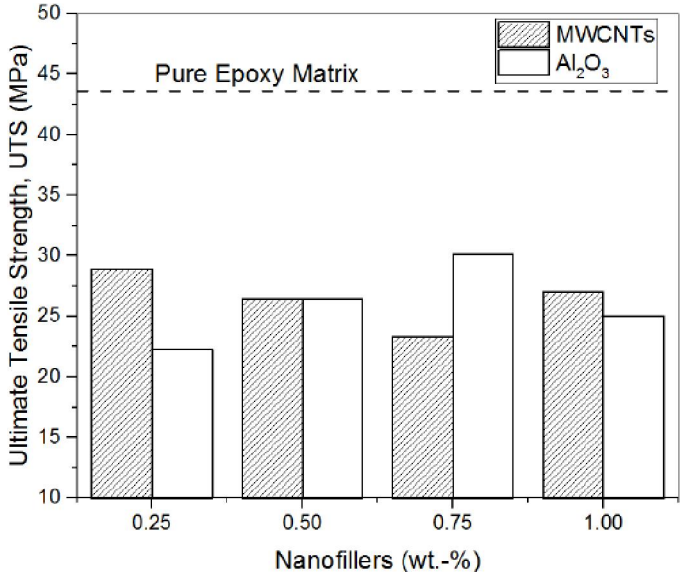
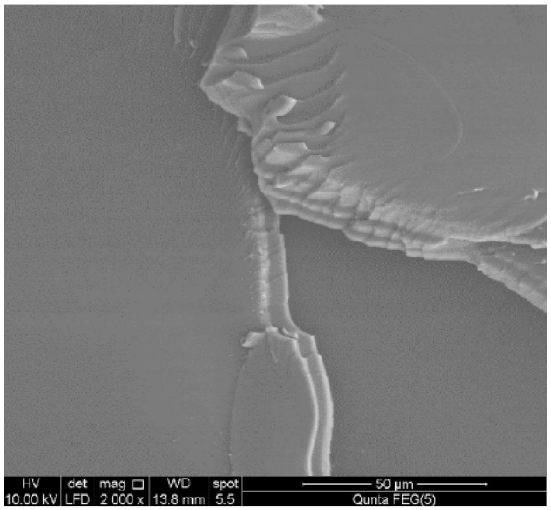
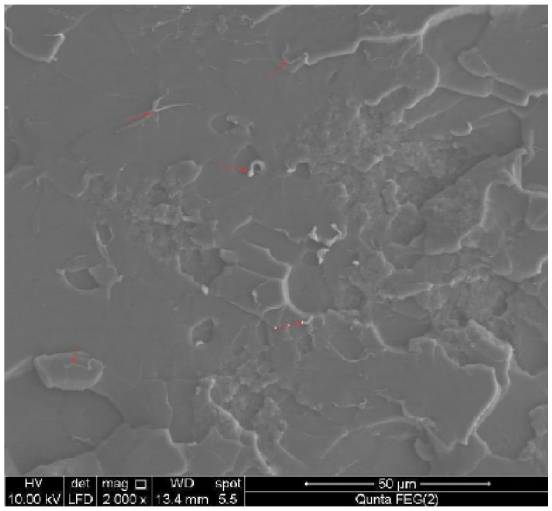


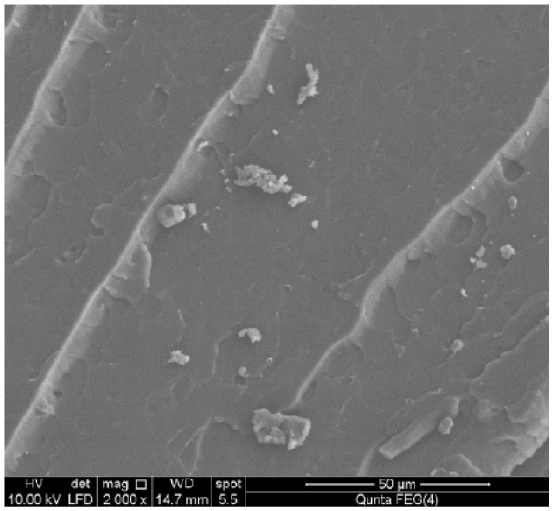
Figure 3 : The variation of the ultimate tensile strength of the nanocomposites with the nanofillers weight percent



(a)



(b)



(c)

Figure 4 : SEM micrographs showing the fracture surfaces of failed tensile specimens of (a) pure epoxy matrix, (b) epoxy/1 wt.-% MWCNTs nanocomposites and (c) epoxy/1 wt.-% Al_2O_3 nanocomposites

The variation of the average micro-hardness of the nanocomposites with the weight percent of the nanofillers is illustrated in Figure 5. Both of the epoxy/MWCNTs and epoxy/Al_2O_3 nanocomposites showed slightly higher average microhardness than the pure epoxy matrix. In most cases the epoxy/Al_2O_3 nanocomposites exhibited higher microhardness than the epoxy/MWCNTs nanocomposites. The pure epoxy matrix exhibited an average microhardness of about 16.3 VHN. A maximum microhardness value of about 18 VHN was observed for epoxy/MWCNTs nanocomposites containing 0.5 wt.-% of MWCNTs. For epoxy/MWCNTs nanocomposites, increasing the weight percent of MWCNTs up to 0.5 wt.-% increases the microhardness of the nanocomposites. Further increase in the MWCNTs weight percent, up to 1 wt.-%, reduces the microhardness of the nanocomposites even lower than the pure epoxy matrix. For example, epoxy/MWCNTs nanocomposites containing 1 wt.-% of MWCNTs exhibited microhardness of about 15.6 VHN. The reduction of micro-hardness of epoxy/MWCNTs containing 0.75 and 1 wt.-% of MWCNTs may attribute to the weak bonding strength between MWCNTs and epoxy resin at high concentration, and also more possibility of void formation. For epoxy/Al_2O_3 nanocomposites, increasing the Al_2O_3 nanoparticles weight percent up to 1 wt.-% did not significantly increase or reduce the microhardness of the nanocomposites. The epoxy/Al_2O_3 nanocomposites exhibited practically the same microhardness.

Charpy impact energy of nanocomposites

The variation of the impact energy of the nanocomposites with the weight percent of the nanofillers is illustrated in Figure 6. The results revealed that the both of epoxy/MWCNTs and epoxy/Al_2O_3 nanocompositesoûer higher impact energy (i.e. better impact resistance) than the pure epoxy. The MWCNTs and nano-Al_2O_3 particles are able to provide epoxy with higher impact toughness. The pure epoxy samples exhibited average impact energy of 0.052 J. It has been found that increasing the weight percent of MWCNTs increases the impact energy of the epoxy/MWCNTs nanocomposites. For example,

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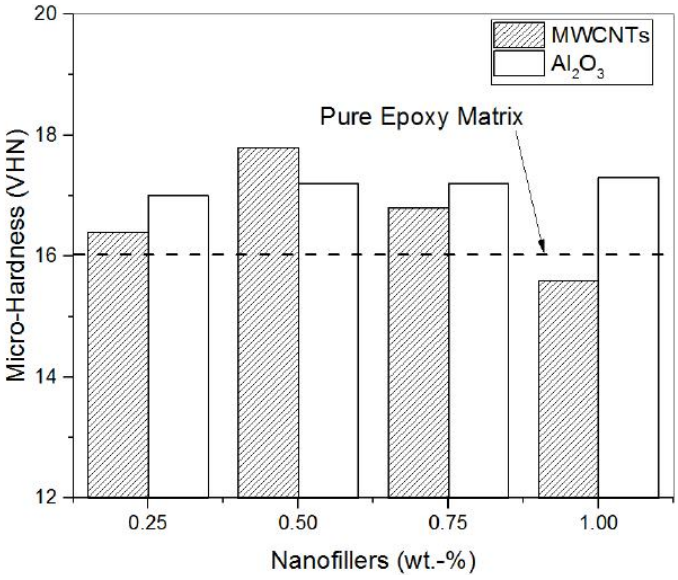

Figure 5 : The variation of the microhardness of the nanocomposites with the nanofillers weight percent

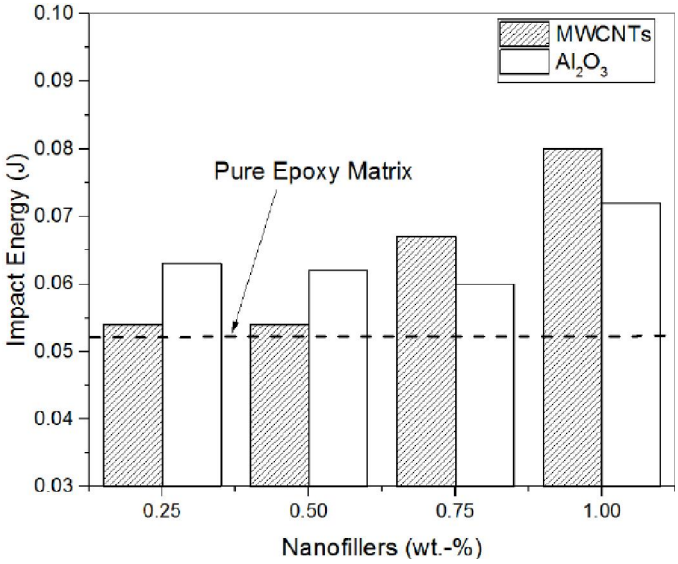

Figure 6 : Variation of the impact energy of the nanocomposites with the nanofillers weight percent

increasing the weight percent of the MWCNTs from 0.25 to 1 wt.-% increasing the impact energy from 0.054 to 0.08 J. Moreover, it has been observed that, for epoxy/Al_2O_3 nanocomposites, increasing the weight percent of nano-Al_2O_3 particles dispersed in the epoxy matrix up to 0.5 wt.-%, increasing the impact energy even higher than the MWCNTs at the same values of filler content. This may attribute to the good bonding between the nano-Al_2O_3particles and the epoxy matrix. Good particle/matrix interface prevents the cracks initiation effectively. For nanocomposites containing 0.75 and 1 wt.-% of nanofillers, the epoxy/MWCNTs nanocomposites exhibited higher impact energy than epoxy/Al_2O_3 nanocomposites.

Flexural strength of nanocomposite

Figure 7 shows the variation of the bending (flexural) strength of the nanocomposites with the weight percent of MWCNTs and nano-Al_2O_3nanofillers. Both epoxy/MWCNTs and epoxy/Al_2O_3 nanocomposites exhibited better flexural strength than the pure epoxy matrix. The pure epoxy exhibited an average flexural strength of 36 MPa. It has been found that the flexural strength of the nanocomposites increases with increasing the MWCNTs and nano-Al_2O_3nanofillers weight percent-

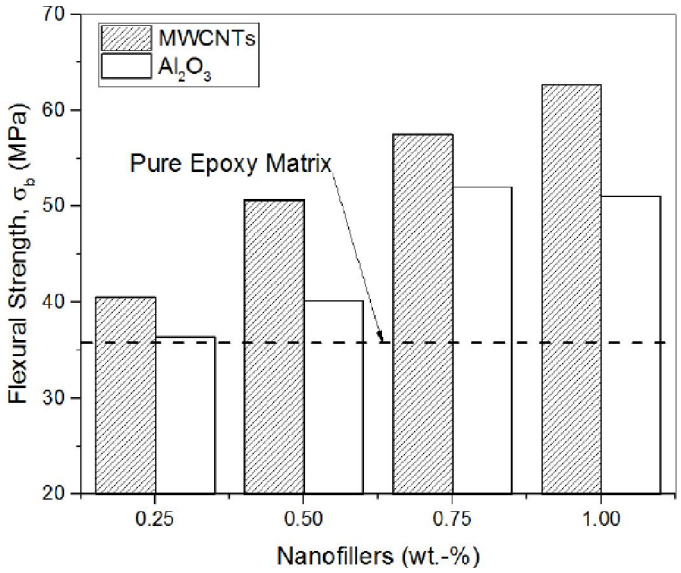

Figure 7 : Variation of the flexural strength of the nanocomposites with the nanofillers weight percent

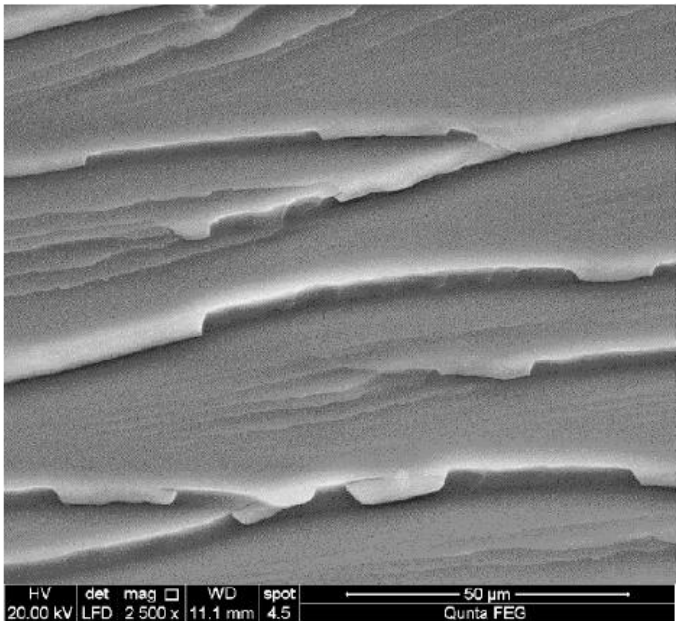

Figure 8 : SEM micrograph of the fractured surface of pure epoxy matrix after ûexural testing

age. For example, increasing the MWCNTs from 0.25 to 1 wt.-% increases the flexural strength of the epoxy/MWCNTs nanocomposites from about 40.5 to 62.7 MPa. The epoxy/MWCNTs nanocomposites exhibited better flexural strength than the and epoxy/Al_2O_3 nanocomposites. For example, the epoxy/MWCNTs and epoxy/Al_2O_3 nanocomposites containing 1 wt.-% of the nanofillers showed flexural strength of 62.7 and 51.03 MPa, respectively.

For Al_2O_3 compositesthe inûuences of rigid particulate ûllers on the stress–strain behaviour of polymers are well known, at least for ûllers in the size of micrometers and larger. The ûexural strength of microparticle flled composites is known to be reduced with rising fller content[10]. The results measured for the nanocomposites in this study oûer an apparent conûict with the known behaviour, where the ûexural strength behaves an increasing with the filler concentration increased. Some important characteristics of composites have to be considered in order to explain this phenomenon. The quality of the interface in composites, i.e. the static adhesion strength as well as the interfacial stiûness, usually plays a very important role in the materials' capa-

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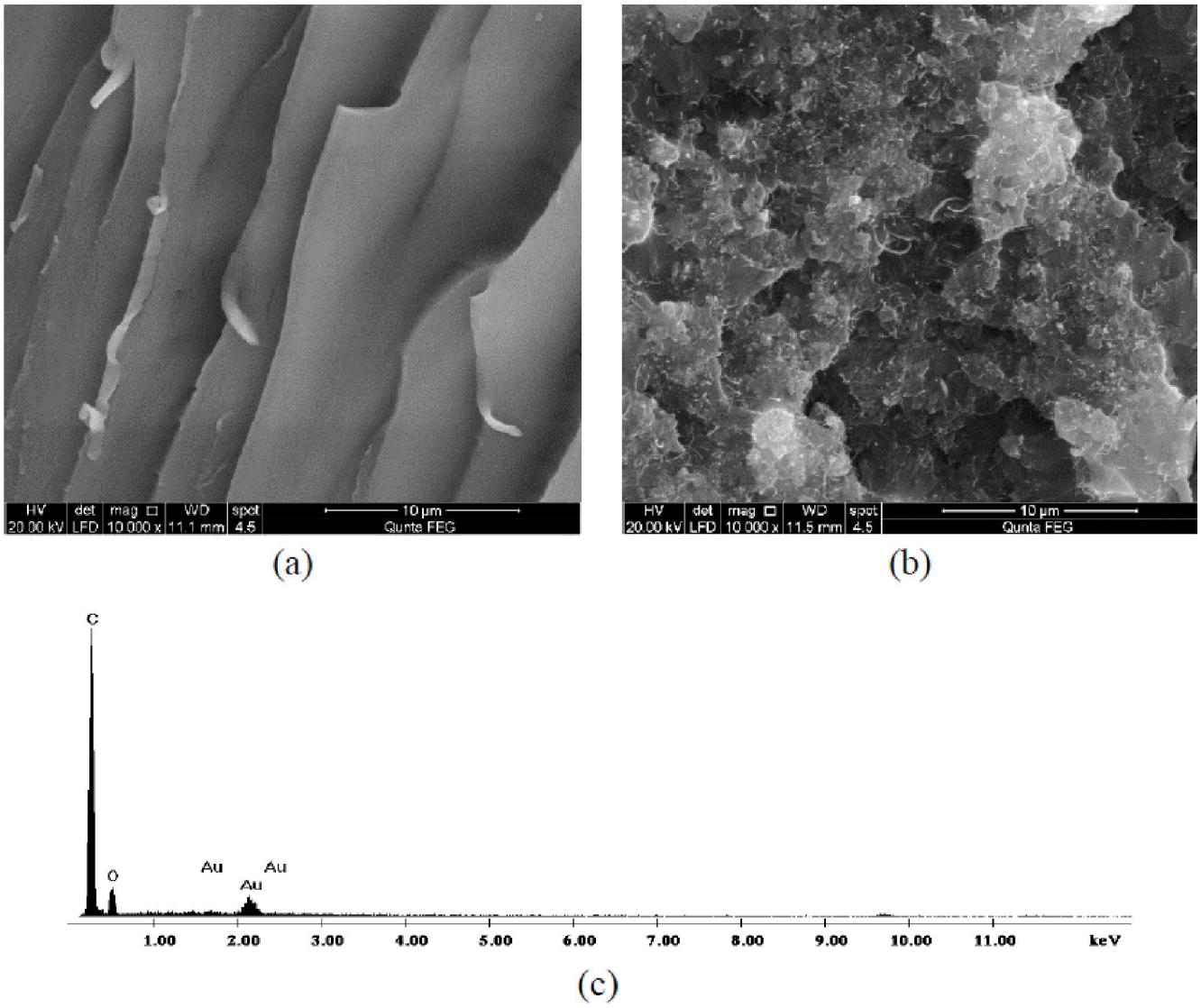

Figure 9 : SEM micrograph of the fractured surface of epoxy/MWCNTs nanocomposites containing 0.25 wt.-% (a) and 1 wt.-% (b) MWCNTs. (c) EDS spectrum of MWCNTs observed in (b)

bility to transfer stresses and elastic deformation from the matrix to the ûllers[11]. If ûller matrix interaction is poor, the particles are unable to carry any part of the external load. In that case, the strength of the composite cannot be higher than that of the neat polymer matrix. Hence, the gradual increase in stiûness and ûexural strength, as observed for the nanocomposites, reveals that stresses are eûciently transferred via the interface.

Figure 8 shows a SEM micrograph of the fracture surface for the neat epoxy matrix which reveals a brittle behaviour characterised by large smooth areas, ribbons and fracture steps in the direction of crack propagation. Figure 9 and 10 show SEM micrographs of the fracture surfaces of failed bending samples for epoxy/MWCNTs and epoxy/Al_2O_3 nanocomposites, respectively. In contrary to the neat polymer, it is clear that nanocomposite fractured surfaces showed rougher structured. Figures9a and 9brepresent SEM micrographsof the fractured surface of epoxy/MWCNTs nanocomposites containing0.25 and 1 wt.-% of MWCNTs, respectively. The fracture surface shown in Figure 9a indicates that nanocomposites containing low content of MWCNTs exhibited similar fracture topography of the pure epoxy matrix. However, at the highest weight percentage of MWCNTs (i.e. 1 wt.-%), see Figure 9b, the large size of epoxy layers was not observed, because MWCNTs become more effective in preventing theepoxy from growing into large size. Figure

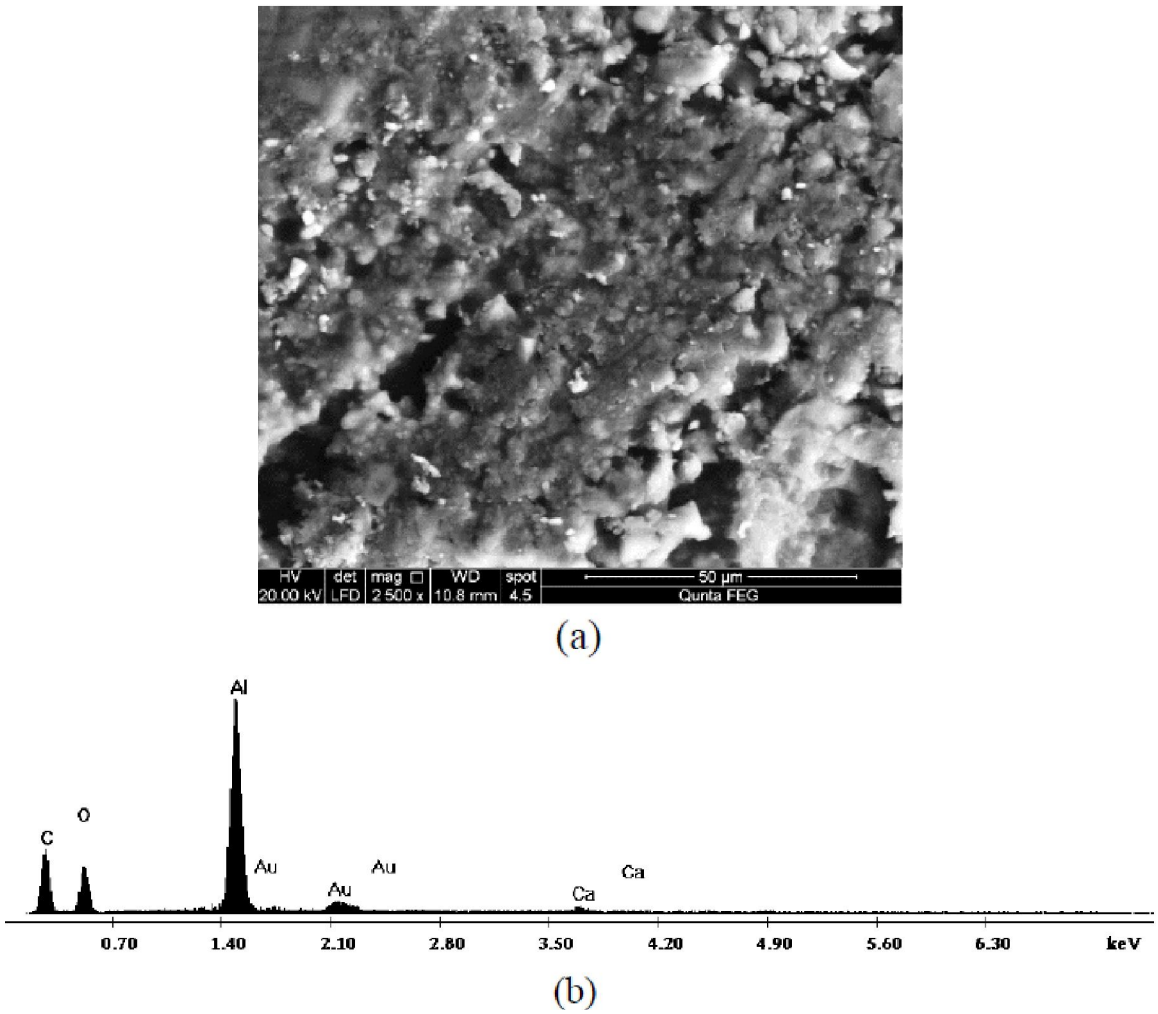

Figure 10 : SEM micrograph of the fractured surface of epoxy/Al_2O_3 nanocomposites containing 1 wt.-% Al_2O_3nanoparticles (a). (b) EDS spectrum of Al_2O_3nanoparticlesobserved in (a)

9c shows EDS spectrum of MWCNTs presented on the fractured surface. Figure 10a shows SEM micrograph of the fractured surface of 1 wt.-% epoxy/Al_2O_3 nanocomposite sample. It is clear that fractured surface is very rough and the agglomerations of Al_2O_3 nanoparticles are observed on the fractured surface.

In the present investigation, the improvement of the flexural strength of the epoxy matrix due to the addition of MWCNTs and Al_2O_3 nanoparticles is observed. The nanofillers plays an important role in preventing the cracks from propagating. TheMWCNTs and Al_2O_3 nanoparticles are diûcult to be broken by propagating cracks due to their high hardness. In addition, it is expected that other energy consuming mechanism act, such as crack front pinning and crack deviation. The mechanisms mentioned above indicate a higher energy consumption of the nanocomposites in comparison to the neat epoxy matrix during fracture and explains the superior mechanical properties of the nanocomposites.

CONCLUSIONS

Based on the results obtained from the present investigation, the following conclusions can be drawn:

The epoxy/MWCNTs and epoxy/Al_2O_3 nanocomposites exhibited lower ultimate tensile strength when compared with pure epoxy matrix. However, the epoxy/MWCNTs nanocomposites exhibited slightly higher tensile strength when compared with the epoxy/Al_2O_3 nanocomposites.

The epoxy/MWCNTs and epoxy/Al_2O_3

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nanocomposites showed slightly higher average microhardness than the pure epoxy matrix. Generally, the epoxy/Al_2O_3 nanocomposites exhibited higher microhardness than the epoxy/MWCNTs nanocomposites.

The epoxy/MWCNTs and epoxy/Al_2O_3 nanocompositesoûer higher impact energy than the pure epoxy. Increasing the weight percent of MWCNTs up to 1 wt.-% increases the impact energy of the epoxy/MWCNTs nanocomposites. While, increasing the weight percent of nano-Al_2O_3 particles dispersed in the epoxy matrix up to 0.5 wt.-%, increasing the impact energy even higher than the MWCNTs at the same values of filler content. Further increase in the weight percent of the nano-Al_2O_3 particles reduces the impact energy of epoxy/Al_2O_3 nanocomposites.

The epoxy/MWCNTs and epoxy/Al_2O_3 nanocomposites exhibited better flexural strength than the pure epoxy matrix. The flexural strength of the nanocomposites increases with increasing the MWCNTs and nano-Al_2O_3nanofillers weight percentage.

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