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Effect of type, percentage and dispersion method of multi-walled carbon nanotubes on tribological properties of epoxy composites

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abstract

In this work the wear behaviour of epoxy matrix composites with different types and percentages of multiwalled carbon nanotubes (MWCNTs) has been studied. Three different types (NC3100, NC3150 and NC3152) and percentages (0.1 and 0.5 wt%) of MWCNTs were dispersed into an epoxy resin by a calendering process. The tribological properties of epoxy-MWCNTs nanocomposites were investigated using "pin-on disc" wear testing machine under different conditions (counterpart material, distances and sliding speeds test). Scanning electron microscopy and 3D optical profilometer were used to observe the worn surfaces of the samples. Compared with neat epoxy, the composites with MWCNTs showed a lower mass loss, friction coefficient and wear rate, and these parameters decreased with the increase of MWCNT percentage. Also, the results demonstrated that the epoxy composites with 0.5 wt% of amino-MWCNT (NC3152) have the best tribological properties.

1. Introduction

Polymers are being used in components subjected to wear due to their advantages such as easy processing, good corrosion resistance and low friction and vibration damping. However, the load carrying capacity and thermal resistance are lower than those of metals and ceramics. To improve these properties, nanoreinforcements such as carbon nanofibers or nanotubes could be added to the polymer. These nanoreinforcements exhibit excellent mechanical, electrical and thermal properties. For this reason, in recent years extensive research has been done on the improvement in the mechanical and electrical properties that is caused by the addition of these nanoreinforcements to the polymer matrices [1–3]. Besides, the carbon nanotubes (CNTs) have self-lubricating properties due to their structure which is similar to the structure of graphite and fullerenes. Therefore, the CNTs could be suitable for anti-friction materials and abrasion resistance.

Several studies on the influence of CNTs on the tribological behaviour of thermoplastic and thermosets polymers have been reported. All have reported a beneficial effect of carbon nanotubes reinforcement on the wear resistance of the polymers [4–13]. Dong et al. [9] studied the influence of different contents of MWCNTs (0–4 wt%) on tribological properties of the epoxy-MWCNTs nanocomposites in dry sliding against plain carbon steel. They found that the MWCNTs increased significantly wear resistance and reduced coefficient of friction of the nanocomposites, concluding that the nanocomposite with 1.5 wt% MWCNTs showed the best wear behaviour. Furthermore, Cui et al. [10] investigated the friction and wear behaviour of epoxy-MWCNTs nanocomposites at different sliding speeds under different applied loads (40–120 N). However, they investigated lower percentages (0–0.5 wt%) and the influence of the functionalisation of carbon nanotubes with amino and carboxyl group. They showed that compared with neat epoxy, the nanocomposites with MWCNTs had a lower friction coefficient and wear rate, and the wear rate decreased with the increase of MWCNTs loading. Recently, Yan et al. [11] evaluated the wear properties of aligned carbon nanotubes reinforced epoxy nanocomposites under water lubricated condition. They demonstrated that the wear rate and friction coefficients decrease in the nanocomposites with respect to the neat epoxy. And the wear mechanism of epoxy-CNTs nanocomposites was slightly abrasive, while that of neat resin was abrasive and fatigue wear. In all these studies, the wear behaviour against steel counterpart has been examined and low loads have not been used.

In order to make these improvements it is important to achieve a good dispersion of carbon nanotubes in the epoxy matrix. CNTs have a high tendency to form stable agglomerates. Several investigations have studied the influence of a dispersion method on tribological properties of carbon nanotube reinforced epoxy resin nanocomposites [10,12,13]. Results showed that the wear

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properties, in general, improve with increased dispersion and integrity of the carbon nanotubes. In these studies, different mechanical and chemical methods have been evaluated but none of them have used the calendaring approach of dispersion. The dispersion method by a calendering machine has proved to be effective to achieve homogeneous dispersion of MWCNTs in epoxy resins without causing major damage to the carbon nanotubes when compared with sonication and high shear mixing techniques. The latter two techniques have a limited effectiveness because of prolonged processing time required to achieve adequate dispersion and the resultant damage that occurs to carbon nanotubes [14,15]. The calendering method has been optimised in previous research and shown to be very effective in obtaining homogeneous dispersion of the carbon nanotubes in epoxy matrices [16–18].

In summary, there are several studies about the influence of the MWCNTs on the wear behaviour of the epoxy matrix nanocomposites. However, none used the calendering method for mechanical dispersion and the tribological properties in dry sliding against alumina counterpart at low loads have not been studied. The present work is focused mainly on the manufacturing of epoxy-MWCNT nanocomposites using a dispersion technique that allows suitable dispersion degree, while it is possible to scale it up to industrial size with high production rates. This is the main difference from previous researches focused on other dispersion methods such as sonication of nanoreinforcement in a solvent which is more complicated to implement at industrial scale. For these reasons, in this work, a study of the wear behaviour (mass loss, wear rate and friction coefficient) of the epoxy-MWCNTs nanocomposites was carried out, analysing the influence of dispersion by the calendering method and the percentage and type of MWCNTs. Furthermore, first wear parameters (speed and distance of sliding and counterpart material) have been optimised. And finally, an extensive study of the wear mechanisms that take place in the epoxy-MWCNTs nanocomposites was done by analysing the worn surfaces using scanning electron microscopy (SEM) and a 3-dimensional optical profilometer.

Table 1
Main parameters used in the calendering process.

Stage	GAP 1 (μm)	GAP 2 (μm)	No. cicles	Speed (rpm)
1	120	40	1	250
2	75	25	1	
3	45	15	1	
4–7	15	5	4	

2. Experimental procedure

2.1. Materials

The polymer matrix used was an epoxy resin, with the commercial name of Araldite LY556, based on bisphenol A mixed with a hardener based on an aromatic amine (Araldite XB3473) in a mass ratio 100:23. As nanoreinforcements, different types of MWCNTs supplied by Nanocyl with different lengths were used: long (NC3100), short (NC3150) and short functionalised with amino groups (NC3152). The percentage of carbon nanotubes has been modified between 0.1 and 0.5 wt%.

2.2. Preparation of MWCNT/epoxy composite samples

MWCNT/epoxy composite processing was performed by two steps: first, mechanical dispersion of carbon nanotubes in the epoxy matrix and, second, curing of the mixtures. Nanoreinforcements dispersion in the epoxy matrix was performed by a three-roll-mill machine or mini-calender according to a method developed in previous research activities [16–18]. Controlling the speeds and directions of the rollers, the forces exerted over the mixture were of pure shear, preventing compression forces that may damage the carbon nanotubes. In Table 1 the dispersion conditions used for the dispersion of MWCNTs in the epoxy matrix are summarised.

After the calendering process, the mixture was heated to 90 1C to reduce its viscosity and facilitate its subsequent processing. After reaching the set temperature, the liquid curing agent was added to the resin and the stirring continued for several minutes to obtain a homogeneous mixture. Finally, the resulting mixture was injected into an open mould placed in an oven for the isothermal curing cycle at 140 1C for 8 h.

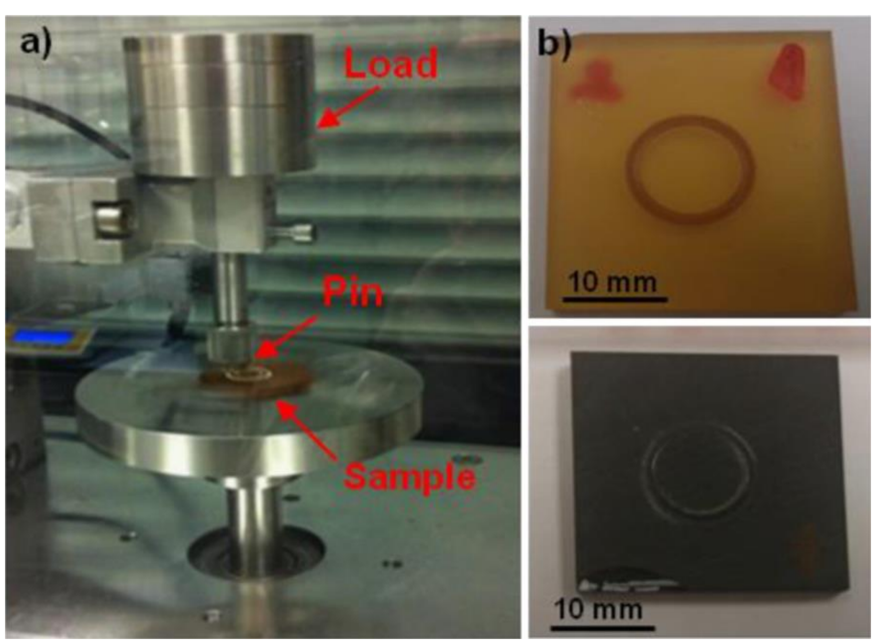

Fig. 1. (a) Pin-on-disc tribometre used for the wear tests and (b) wear track of the neat epoxy and epoxy-MWCNTs nanocomposite after the wear test.

2.3. Characterisation

To evaluate the dispersion of carbon nanotubes in the epoxy matrix, microstructural characterisation of epoxy-MWCNTs nanocomposites was done by optical microscopy in transmitted light mode (TOM). An optical microscope Leica DMR with a NIKON Coolpix 900 camera was used to obtain the images. The maximum size and percentage of agglomerates at different stages of the calendering process were evaluated by image analysis software (Image Pro Plus).

Before wear testing, a surface preparation of the samples was performed. The samples surfaces were ground with different emery papers up to 600 grit. The average roughness of the samples determined by a profilometer Mitutoyo SJ- 301 Surftest was 0.65 7 0.2. Later, the specimen surfaces were cleaned with acetone to avoid the presence of humidity and impurities on the surface. At least three samples were tested for each wear study.

Vickers microhardness measurements of the neat epoxy and epoxy-MWCNTs nanocomposites were taken using a microhardness indenter (Micro-Hardness Tester Shimadzu) applying a load of 300 mN (HV0.3) during 15 s. The microhardness values obtained are the average of at least five tests.

Wear tests were carried out on a pin-on-disc tribometre (Fig.1) under dry sliding condition and at room temperature. First, the influence of the counterpart material (alumina and steel ball), sliding distances (700 and 1000 m) and speeds (0.05, 0.09 and 0.13 m/s) were studied to optimise the wear test parameters. Second, the influence of percentage and type of MWCNTs in the tribological properties of epoxy resin nanocomposites were evaluated by dry sliding wear tests at load of 10 N, using an alumina ball as a counterbody, speed of 0.09 m/s and a sliding distance of 1000 m.

The wear testing machine continuously recorded the friction coefficient and the wear depth. The variation in height of the contact between ball and coating was registered using a LVDT with 7 1 μm of precision. The samples were weighted before and after the wear test in order to determine the mass loss during the test. Volume lost during the wear test was determined from the mass loss using the nanocomposite density to ascertain the wear rate. To evaluate the wear response of the material under different conditions, Archard's law was applied:

$$Q=\frac{V}{L}=K\frac{W}{H} \quad (1)$$

In this equation the coefficient Q is the wear rate and defines the wear volume (V) per the sliding distance (L). W is the applied load, H is the hardness of the sample and K is Archard's constant that compares the severity of wear between two different systems.

Finally, worn surfaces were analysed using a Scanning Electron Microscope (SEM) using a Hitachi S-3400N microscope and three-dimensional optical profilometer model Zeta 20 to define the main

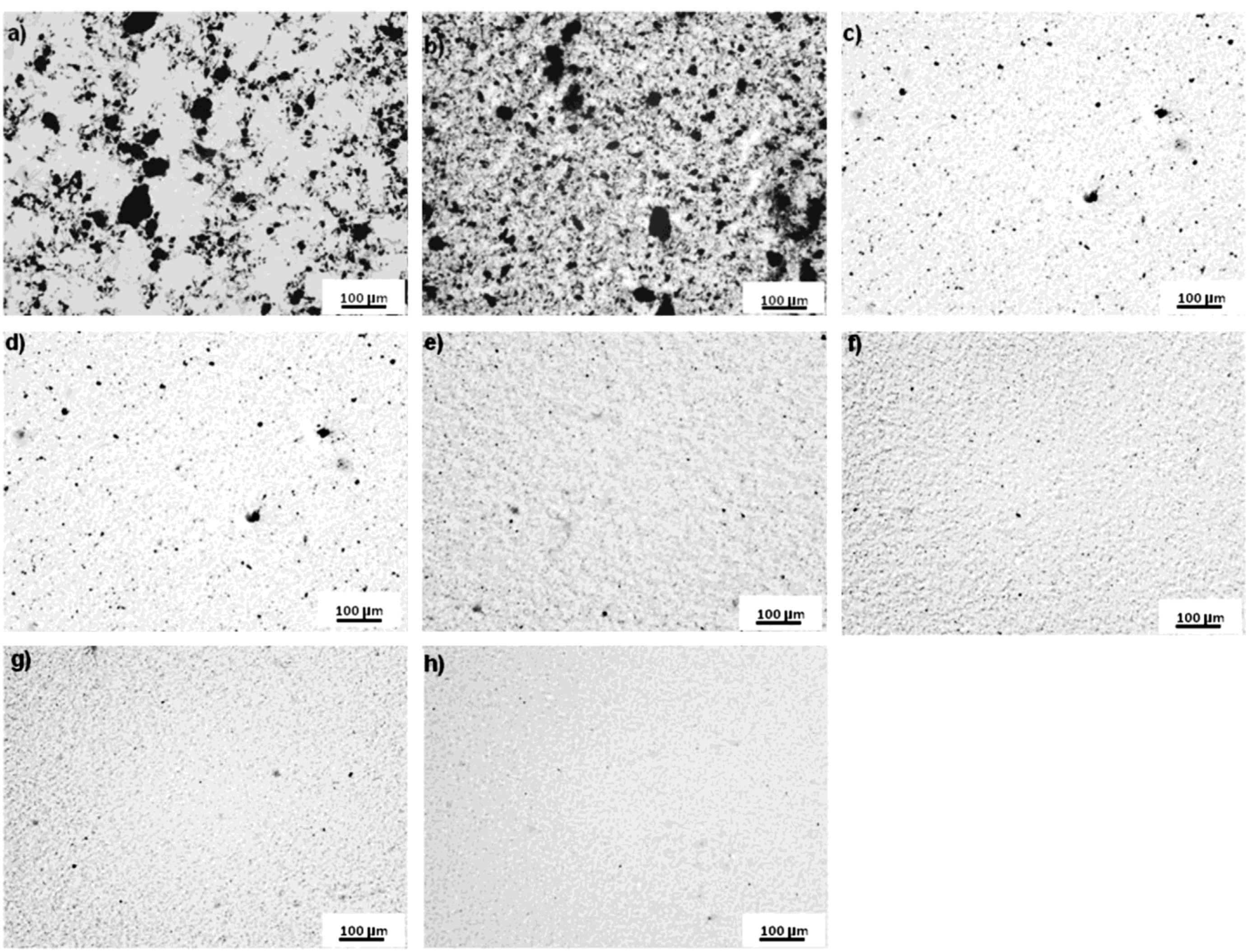

Fig. 2. Optical microscope images of the dispersion achieved in the different stages of the calendaring process.

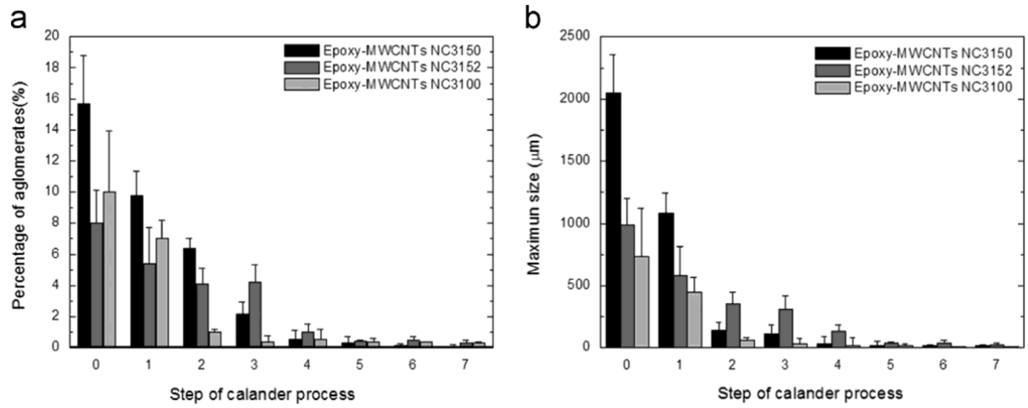

Fig. 3. Percentage (a) and maximum size of agglomerates (b) in the different stages of the calendaring process for the three epoxy-MWCNTs mixtures.

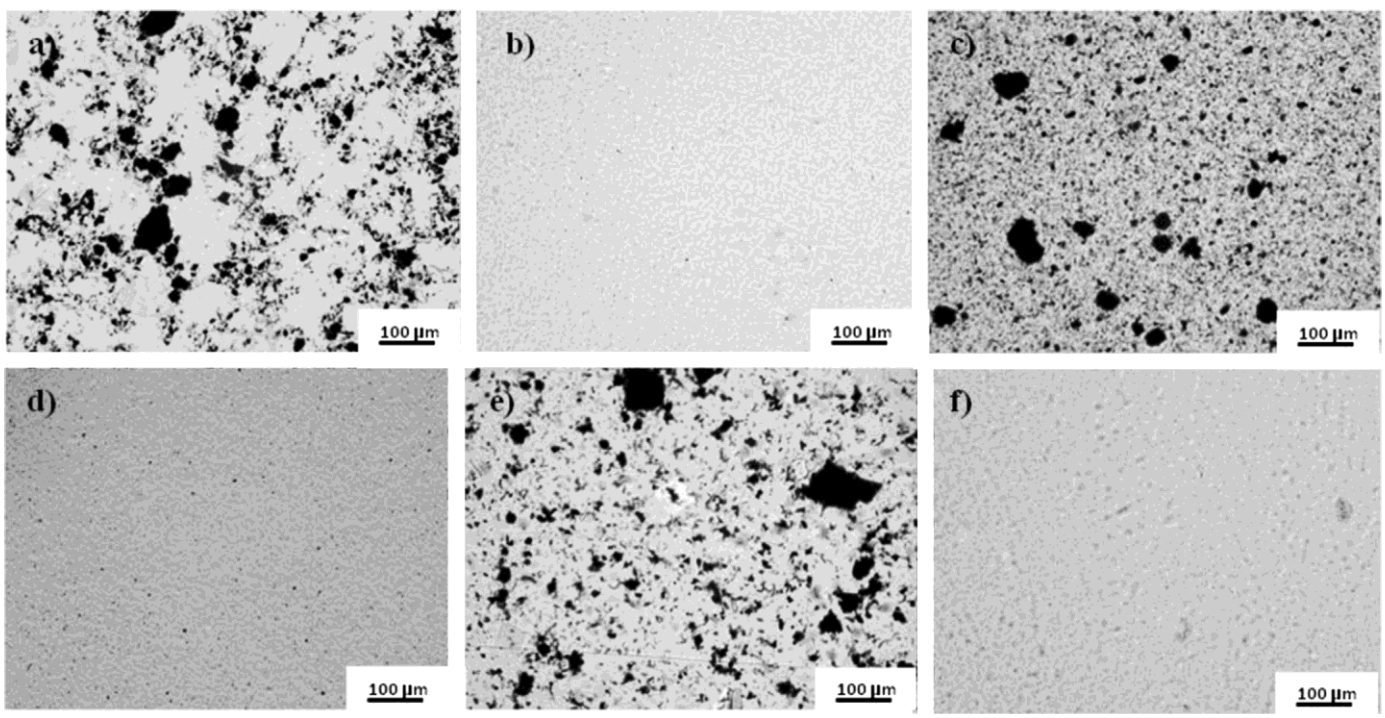

Fig. 4. Optical microscope images of the dispersion achieved in the nanocomposites with 0.5 wt% of different types of MWCNTs: NC3150 (a, b), NC3152 (c, d) and NC3100 (e, f).

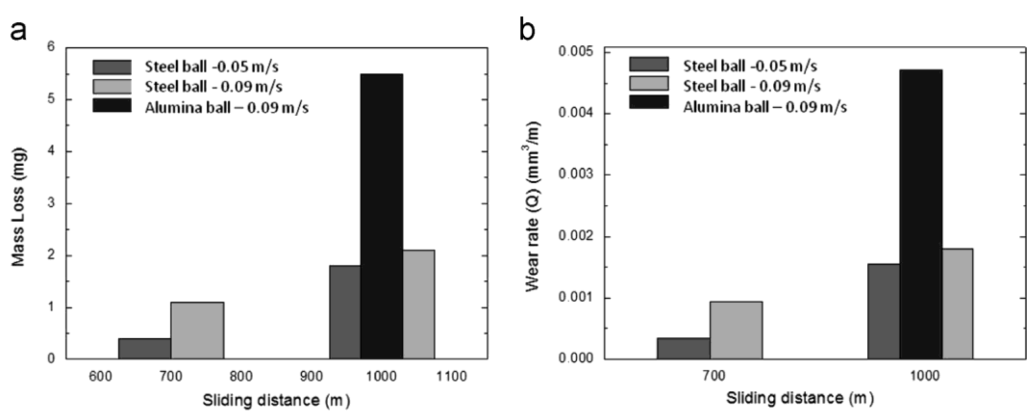

Fig. 5. Effect of the material counterpart, distance and speed sliding in the mass loss (a) and wear rate (b) of the neat epoxy under dry conditions at 10 N.

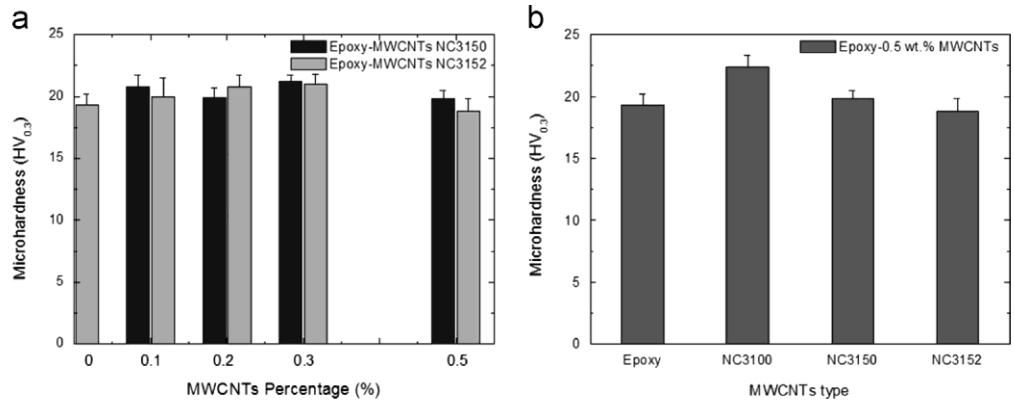

Fig. 6. Variation of the microhardness of epoxy-MWCNTs nanocomposites as function of MWCNTs percentage (a) and MWCNTs type (b).

wear mechanisms. The width of the wear track was measured by optical microscopy using image analysis software.

3. Results and discussion

3.1. Microstructural characterisation

Fig. 2 shows the dispersions achieved in epoxy-MWCNTs (NC3150) mixtures in the different stages of the calendaring process. We can observe the breakdown of the agglomerates and homogeneous dispersion of carbon nanotubes into the resin at the end of the calendering process. During this dispersion method, disaggregation of the agglomerates and homogeneous dispersion of carbon nanotubes in resin occur in two phases. The first occurs in the early stages (1–3) of the process (Fig. 2b–d) and it is observed as larger agglomerates present in the initial mixture (Fig. 2a) are disintegrated obtaining a homogeneous dispersion but with the presence of small agglomerates. In subsequent steps (4–7), with distance constant between the rollers (Fig. 2e–h), the main effect is the homogeneous dispersion of MWCNTs in resin, although agglomerate size is also reduced.

Fig. 3 shows the results of maximum size and percentage of agglomerates for the three types of carbon nanotubes studied. As discussed above, the disaggregation of the larger agglomerates is produced in the early stages (1–3) because the maximum agglomerate size decreases considerably. In the four last stages (4–7), when the gap between the rollers is kept constant, a homogenisation and disintegration of smaller agglomerates occur. The maximum agglomerate size after the last step is 11.4 μm, 15 μm and 21.1 μm for NC3100, NC3150 and NC3152 MWCNT respectively. These results allow concluding that the best dispersion is obtained for the nanocomposite with longer MWCNTs (NC3100), while the worst dispersion is for functionalised MWCNTs (NC3152).

Finally, Fig. 4 shows the epoxy resin mixtures with 0.5 wt% of the three types of carbon nanotubes in reception state (Fig. 4a, c and e) and after mechanical dispersion by the calendaring process (Fig. 4b–f). It can be observed that after the calendering process, the MWCNTs are homogeneously dispersed throughout the matrix to the different percentages and types of carbon nanotubes studied.

3.2. Tribological properties

3.2.1. The effect of the wear test parameters

Wear test parameters used in the present research were selected based on wear tests on the neat epoxy resin. Fig. 5 presents the mass loss and the wear rate obtained by modifying

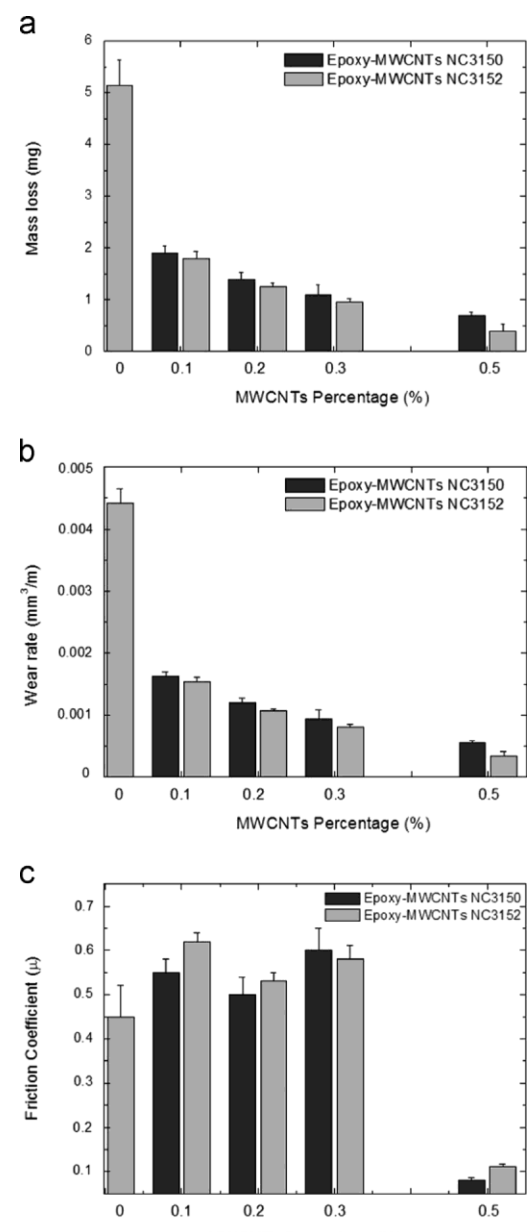

Fig. 7. Mass loss (a), wear rate (b) and friction coefficient (c) of epoxy-MWCNTs nanocomposites with different contents of MWCNTs.

the sliding distance (700 and 1000 m), speed (0.05, 0.09 and 0.13 m/s) and counterpart material (steel or alumina). Results obtained for a speed of 0.05 and 0.13 m/s were very similar to those of 0.09 m/s. In addition, the errors in the measurements were greater at high speed (0.13 m/s), while at low speed (0.05 m/s) the test was too slow. It can be observed, as expected, that the mass loss and the wear rate increase with increasing the speed and sliding distance. Furthermore, the values are much higher when an alumina ball is used instead of steel.

The following wear parameters were selected: a distance of 1000 m, speed of 0.09 m/s and alumina ball as counterpart, with the aim of studying improvements produced by the carbon nanotubes on the more aggressive conditions evaluated.

The wear rate is dependent on the hardness of the material (Eq. (1)). Therefore, first microhardness of the studied materials was evaluated to assess its influence on the wear behaviour (Fig. 6). When the content of MWCNTs is increased (Fig. 6a), the microhardness values slightly decrease. However, these differences, considering the error, are very small and could be due to defects during fabrication of the samples such as porosity, distribution of nanoreinforcement or the presence of matrix areas without MWCNTs. Regarding the influence of the type of MWCNTs (Fig. 6b), we can see that the microhardness of the material with NC3100 carbon nanotubes was slightly higher than that of the epoxy resin, but for carbon nanotubes NC3152 and NC3150, it was similar to that of the resin without nanoreinforcement. The highest microhardness value for the nanocomposite with NC3100 carbon nanotubes is attributed to its better dispersion in the epoxy resin. However, we can conclude that the microhardness values are not a determining factor in the wear behaviour as these values are very similar for all studied materials.

3.2.2. The effect of the MWCNT percentage

The influence of MWCNTs content on the mass loss, wear rate and friction coefficient is shown in Fig. 7. It is clear, for both types of MWCNTs that the mass loss and wear rate decrease with respect to the neat epoxy with the increase of MWCNTs percentage. This reduction is more pronounced with only 0.1 wt% of MWCNTs and gradually decreases for percentage between 0.1 and 0.5 wt%. The properties of carbon nanotubes give to the resin epoxy an improvement in the wear behaviour due to being extremely hard in the direction of the axis, but at the same time flexible, and also to its lubricating effect.

Comparing two types of carbon nanotubes, we can observe that for the same percentage of MWCNTs, the values are slightly lower in the case of functionalised MWCNT (NC3152). This difference is greater for higher percentages (0.5 wt%). The result indicates that stronger interaction between matrix and MWCNTs has a great influence on the wear behaviour, which is particularly noticeable at higher MWCNTs contents.

Fig. 7c shows the effect of MWCNTs content on the friction coefficient. It can be observed that at low MWCNTs percentage, it remained almost constant, being slightly higher for materials containing NC3152 MWCNTs. However, when the percentage is higher (0.5 wt%) a sharp drop in the value occurs in both cases. This behaviour could be due to the tribological layer (debris) formed by the epoxy resin detached and the carbon nanotubes torn during the test. These carbon nanotubes are present in the debris having a lubricating effect which produces a reduction in the friction coefficient. The low coefficient of friction observed in the materials with a 0.5 wt% could be due to the higher amount of carbon nanotubes in the sample and in the debris, which leads to greater lubricating effect. On the other hand, the increase of the friction coefficient that occurs in the materials with 0.1, 0.2 and 0.3 wt% in relation to the epoxy matrix may be because debris with MWCNTs can act as a third body in the wear mechanism, thus further increasing real contact surface, and producing a slight increase in the friction coefficient [19,20]. This effect is diminished as the amount of MWCNTs increases, where the property as a lubricant effect of MWCNTs is higher.

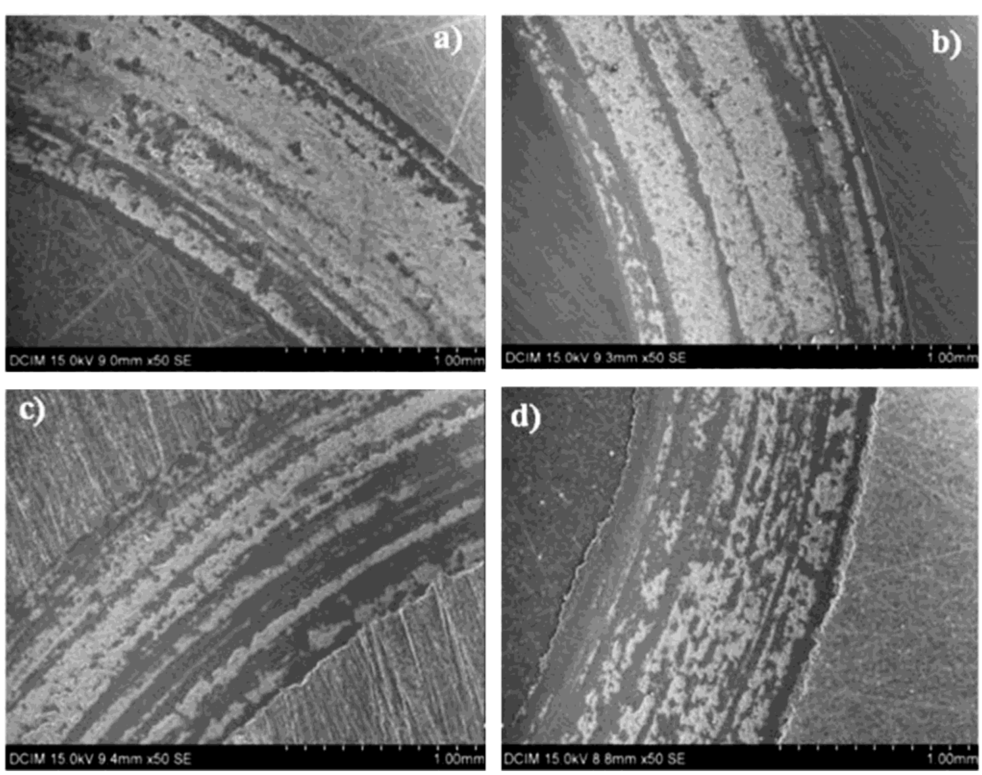

Fig. 8. SEM images of worn surfaces of neat epoxy (a) and 0.2 wt% (b), 0.3 wt% (c) and 0.5 wt% (d) epoxy-MWCNTs nanocomposites.

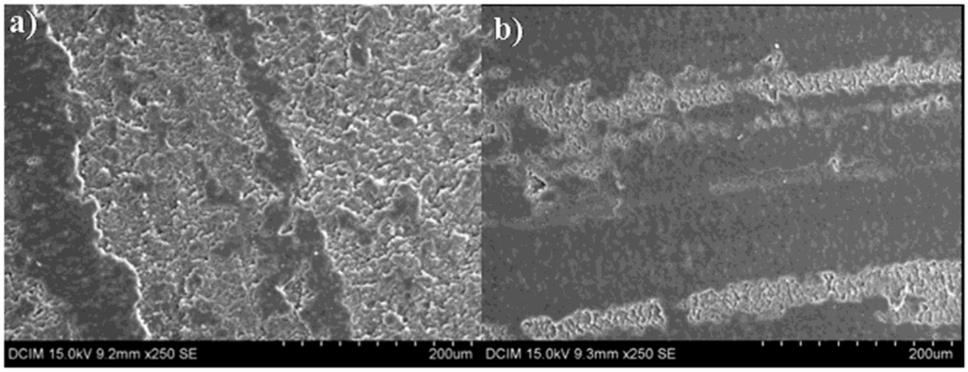

Fig. 9. SEM images of worn surfaces at higher magnification of neat epoxy (a) and 0.5 wt% (b) epoxy-MWCNTs nanocomposites.

The SEM morphologies of the worn surfaces of the neat epoxy and nanocomposites with 0.2, 0.3 and 0.5 wt% MWCNTs were selected to evaluate the effect of the MWCNTs content on the worn surface (Figs. 8 and 9). In general, in all surfaces two zones were shown on the wear track: a darker one which corresponds to accumulation of a tribological layer (debris) formed by the epoxy matrix and carbon nanotubes torn during test; and a clearer one with greater depth and which corresponds to the surface from where the material is torn (abrasive zone). This surface morphology is characteristic of abrasive and adhesive wear mechanism.

The surface of the epoxy resin wear track (Figs. 8a and 9a) is very rough and exhibits a larger amount of ploughed furrows justifying their lower wear behaviour. By contrast, when MWCNTs are added to the resin, the abrasive wear and adhesion on the worn surface is significantly reduced (Fig. 8b–d). This indicates that addiction of MWCNTs is an effective method to decrease adhesion and abrasion wear and to improve the wear resistance of epoxy resin. The wear mechanism is less abrasive due to the lubricating effect of the carbon nanotubes. However, at low percentages this lubricating effect is not very important because the surfaces are rough and have a greater number of blocky fragments on the surface (Fig. 8b and c), while at higher percentages (0.5 wt%) the worn surface is smoother and has a lower exfoliation and plastic deformation justifying the low friction coefficient measured for the nanocomposite with 0.5 wt% MWCNTs (Fig. 9b).

These results are in agreement with other reports [4,21,22], which attributed the lower friction coefficient and improved wear behaviour of the nanocomposites to two different effects; first, the improvement in mechanical properties of nanocomposite by the addition of MWCNTs; and, second, pull out of MWCNTs uniformly dispersed from the resin during wear test that act as a layer of separation between the ball and the material. This effect together with the lubricating property of the carbon nanotubes causes a decrease of the friction coefficient, mass loss and the wear rate.

3.2.3. The effect of the MWCNT type

Fig. 10 shows the mass loss, wear rate and friction coefficient corresponding to epoxy-MWCNTs nanocomposites at a percentage of 0.5 wt% with different types of MWCNTs. The mass loss and wear rate decrease sharply in all samples compared with the neat epoxy resin. This decrease is slightly lower for nancomposites with long carbon nanotubes (NC3100) and short functionalised carbon nanotubes (NC3152). In the case of nanocomposite with long MWCNTs (NC3100), this decrease is due to their higher microhardness value possibly caused by the better dispersion of the MWCNTs in the matrix. In the case of nanocomposite with functionalised MWCNTs (NC3152), this effect is due to the better interaction between matrix and carbon nanotubes. Regarding the values of the friction coefficients are very similar for the three types of carbon nanotubes (Fig. 10c). It can be concluded that the type of nanotube has not had a great influence on the wear behaviour.

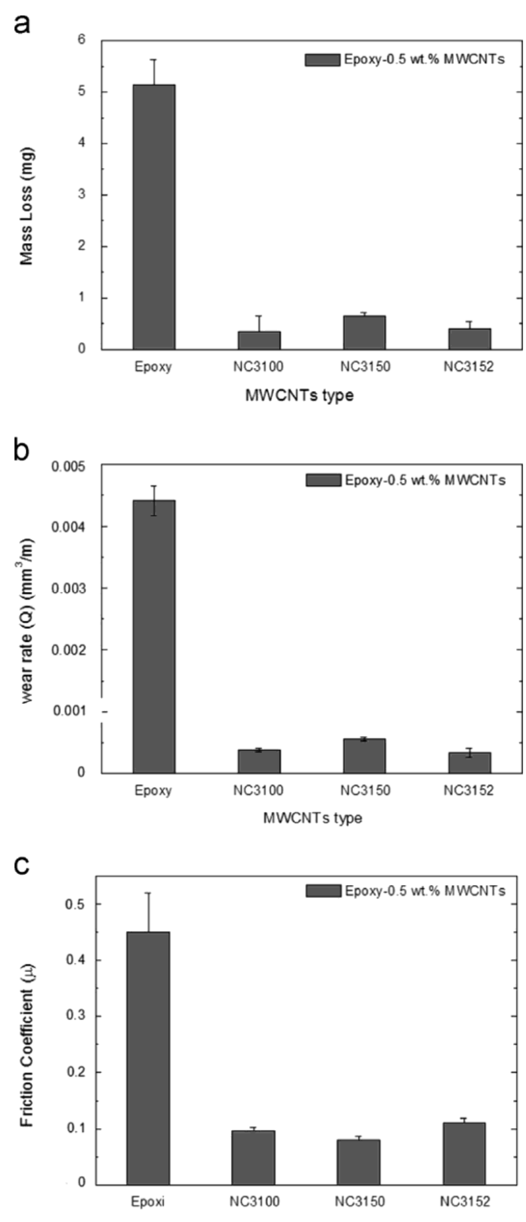

Fig. 10. Effect of the type of MWCNT on the mass loss (a) wear rate (b) and coefficient friction of the composites with 0.5 wt% MWCNTs.

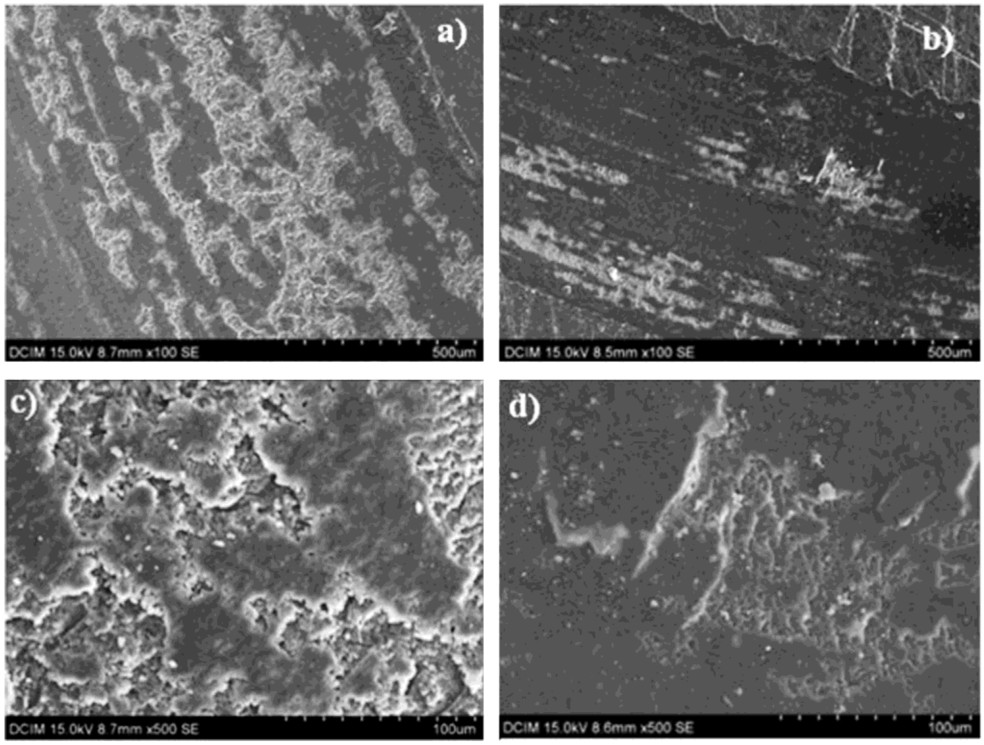

Fig. 11. SEM images of worn surfaces of composites with 0.5 wt% of MWCNTs (a, b) and amino-MWCNTs (c, d).

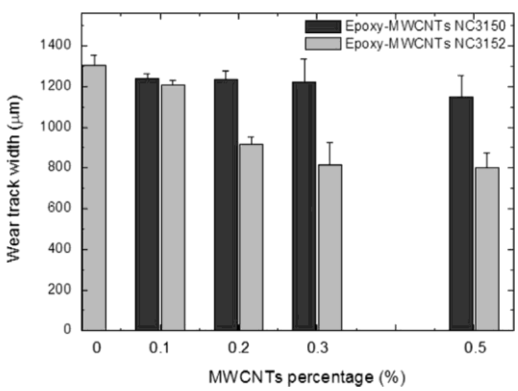

Fig. 12. Wear track width of epoxy-MWCNTs nancocomposites as function of MWCNTs content.

The SEM micrographs of the worn surfaces of the nanocomposites with 0.5 wt% of MWCNTs NC3150 and amino-MWCNTs (NC3152) are shown in Fig. 11. It is clearly seen that the worn surface of the composites with amino-MWCNTs (Fig. 11b and d) is much smoother than that of the composites with non-functionalised MWCNTs (Fig. 11a and c). In addition, the amount of the material peeled off from the worn surface is smaller. The signs of adhesion, plastic deformation and exfoliation of epoxy resin are reduced in nanocomposites with amino-MWCNTs.

Fig. 12 shows the results of the width of the wear track as a function of MWCNTs percentage for nancomposites with non-functionalised (NC3150) and amino functionalised (NC3152) MWCNTs. We can observe for non-functionalised MWCNTs that the width decreases slightly with the addition of MWCNTs to resin, but remains practically constant with increasing percentage of MWCNTs. However, for nanocomposite with functionalised MWCNTs from 0.2 wt% the track width decreases considerably. This result could be due to their greater interaction with the matrix that makes more difficult to pull out the carbon nanotubes and hence the worn area is smaller. That is to say, the nanocomposites with 0.5 wt% of NC3152 MWCNTs, besides decreasing the amount of removal material, they also decrease the worn area.

Finally, the profilometry 3D image (Fig. 13) confirms the findings obtained. The neat epoxy resin has a wear track wider and deeper showing a higher amount of material removal and more abrasive wear (Fig. 13a). However, in the case of epoxy-MWCNTs composites the wear track is less wide and deep (Fig. 13b–d). The results prove that carbon nanotubes decrease the abrasive wear and the loss of resin for adhesion.

4. Conclusions

This study investigated the effect of type and percentage of MWCNTs on the wear behaviour of the epoxy-MWCNTs nanocomposites in dry sliding against alumina ball. The main conclusions of the research are the following:

1. The mechanical dispersion of carbon nanotubes by the calendering process allows obtaining homogeneous and uniform mixtures for the three types and different percentages of carbon nanotubes studied.
2. The addition of MWCNTs to the epoxy matrix significantly improves its wear behaviour. Compared with neat epoxy, the composites with MWCNTs showed lower mass loss, wear rate and friction coefficient. These improvements are greater with increasing percentage of MWCNTs and for longer carbon nanotubes (NC3100) and functionalised with amino group (NC3152).
3. The wear mechanisms of neat epoxy and nanocomposites with MWCNTs are mainly abrasion and adhesion wear. The adhesive and abrasive mechanisms wear decreases with the increase of MWCNTs percentage and the amino-MWCNTs.
4. The nanocomposites with 0.5wt% amino MWCNTs (NC3152) had the better wear behaviour. For this material, the amount

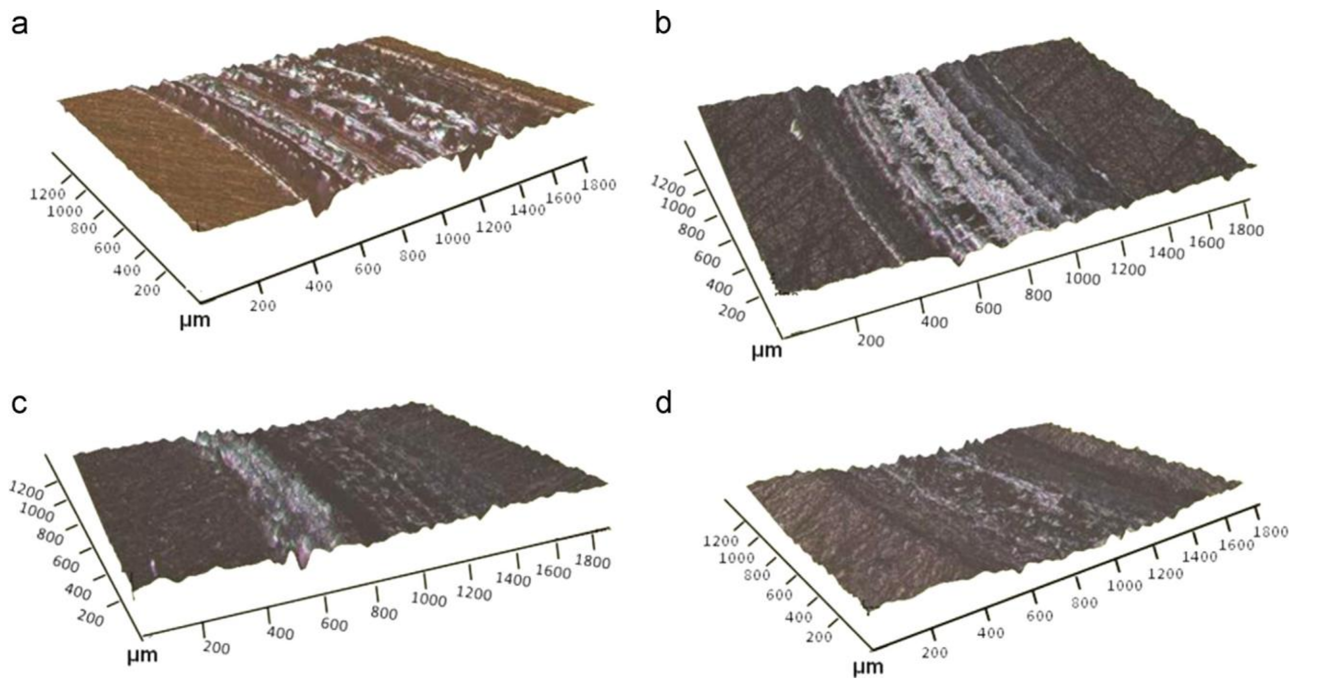

Fig. 13. 3D profilometer images of worn surfaces of neat epoxy (a) and epoxy-MWCNTs with 0.1 wt% MWCNTs NC3150 (b), 0.1 wt% MWCNTs NC3152 (c) and 0.5 wt% MWCNTs NC3152 (d).

removed and the worn surface was smaller due to the greater interaction of these carbon nanotubes with the epoxy matrix.

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