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Article

Comparison of the influence of two types of plasma treatment of short carbon fibers on mechanical properties of epoxy composites filled with these treated fibers

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Abstract: The interfacial interface between the fibers and the matrix plays a key role for epoxy matrix composites and short recycled randomly arranged fibres. This study used short recycled carbon fiber (RCF) as a filler. Plasma treatment was used for carbon fiber surface treatment. This treatment was performed using radio (RF) and microwave (MW) frequencies at the same pressure and atmosphere. Appropriate chemical modification of the fiber surfaces helps to improve the wettability of the carbon fibers and, at the same time, allows the necessary covalent bonds to form between the fibers and the epoxy matrix. The effect of the plasma treatment was analyzed and confirmed by XPS analysis, Raman microscopy, SEM, TEM and wettability measurements. Composite samples filled with recycled carbon fibers with low concentrations (1 wt%, 2.5 wt% and 5 wt%) and high concentrations (20 wt% and 30 wt%) were made from selected treated fibers. The mechanical properties (impact toughness, 3PB) were analyzed on these samples. It was found that the modulus of elasticity and bending stress increase with the increasing content of recycled carbon fibers. A more significant change in impact strength occurred in samples with low concentration.

Keywords: recycled carbon fiber (RCF); fibers reinforced epoxy composites (FRE); plasma treatment; mechanical properties

1. Introduction

The plasma coating of carbon fibre (CF) is a dry reaction process and, depending on the process conditions, can have several effects that can coincide. The plasma cleans the surface of the fibers and creates a hydrophilic surface, removes the skinny surface layer by micro-etching, penetrates the top few molecular layers (about 10 nm) and creates a new surface chemistry, allowing for better interfacial adhesion in composites [1-3]. Due to the action of inert gases, crosslinking occurs, where two or more parallel polymer chains are linked together by O_2 or He or Ar, and the by-products allow the formation of bonds to adjacent chains [4].

For virgin CF, Morgan [5] gives model operating conditions for a plasma reactor: an RF generator with 100 W using at 2-50 Pa pressure and an operating time of 20 s to 20 min [2][6-7]. Donnet [8] points out that different results can be achieved depending on whether the plasma is conducted using radio (RF) or microwave (MW) frequency.

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In recycled RCFs, there are also surface changes due to plasma action, as reported by [9-10]. In these studies, it has been confirmed that the plasma action causes a slight deepening of the surface roughness of the recycled carbon fibers but no damage to the surface. However, it has been experimentally confirmed in a previous study [11] that changes in fiber topography can also occur, along with the incorporation of new surface functional groups, which SEM and XPS will further analyze.

This study analyzes the mechanical properties of carbon fiber reinforced epoxy composites (FRE) at low concentrations (1 wt%, 2.5 wt% and 5 wt%) and high (20 wt% and 30 wt%) concentrations: (a) RCF, (b) MW plasma-treated RCF, (c) RF plasma-treated RCF.

The mechanical properties were analyzed by impact toughness and flexural strength tests. The effect of plasma on RCF was analyzed by XPS analysis, wetting measurements and scanning electron microscopy (SEM), transmission electron microscopy (TEM) and Raman microscopy (RS).

2. Experiment

2.1. Materials

An epoxy matrix system made from a low viscosity bisphenol A-based epoxy resin L 285, together with a cyclic-aliphatic polyamine curing agent H 508, with a mixing ratio of 100:40 by weight was used. The density of the resin was 1,200 kg/m^3 and the density of the hardener was 1,030 kg/m^3. Commercially available PAN based Carbiso milled carbon fibre was used, with a bulk density of 400g/L. The fibres were 7.0 ± 0.3 μm in diameter and 100 ± 9 μm in length.

2.1.1. Plasma treatment of recycled carbon fibres

Based on the available data presented in [5-10], the following conditions were selected for the plasma application: source power: 30 W and 100 W, time: 0.5-50min. This work analyses two types of plasma treatments of recycled carbon fibers. The same pressure (100 Pa) is used for both treatments, but they differ in the treatment time and the frequency used.

Microwave plasma treatment

The first type of plasma treatment was carried out at microwave frequency (MW) under the influence of - oxygen 200 sccm and hydrogen 50 sccm at a power of 100 W. A particular fluidization device adapted for very light bulk materials was used to treat recycled carbon fibres. The device contained a unique hopper that ensures uniform dosing of the bulk filler to the plasma apparatus; the plasma action lasted 1-50 min. Small cracks were observed in the SEM images of the recycled plasma-treated carbon fibers (exposure time ≥10 min), as shown in Figure 1(c). Based on a pilot series of specimens containing these plasma-treated recycled carbon fibers (results published in [11]), where the majority of the plasma-treated fibre-filled composite specimens showed a deterioration in mechanical property values compared to the untreated fiber-filled specimens, a series of plasma-treated fibers were fabricated where the treatment time was reduced to 1 min. After this change, no more destruction was found in the SEM images of the fibers, see Figure 1 (b).

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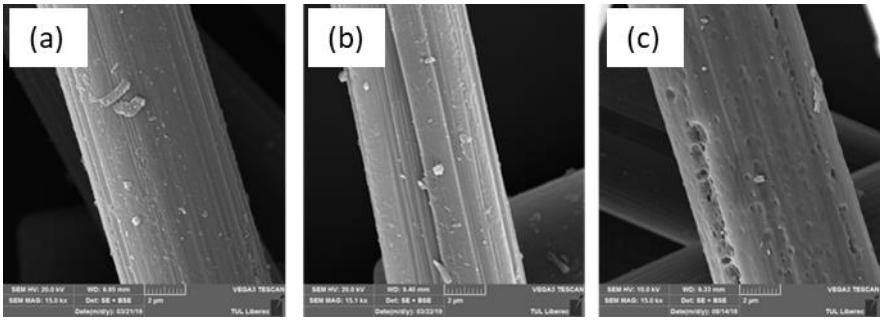

Figure 1. SEM photos of RCF: (a) without treatment, (b) MW plasma-treated for 1 min, (c) MW plasma-treated for 10 min.

Radiofrequency plasma treatment

Based on the recommendation already published by Donnet [8], an apparatus for the treatment of powder fillers by radiofrequency (RF) plasma treatment was developed. The duration of this treatment was 30 s. No defects were observed in the SEM images of the recycled fibres treated by radiofrequency plasma treatment, as shown in Figure 2. The recycled carbon fibers were plasma treated in a fluidized bed reactor. The working gas (air) flowed into the reactor at its bottom, fluidising the powder. Two outer ring electrodes were attached to the bottom of the reactor 1 cm apart to ignite the plasma discharge. The electrodes were connected to a Dressler Cesar 133 RF generator via a matching network. The airflow rate was 150 sccm.

The plasma treatment times (1xcycle = 30 s, 3xcycles = 90 s) and the applied power of 30 W and 100 W were compared. Based on the XPS analysis showing the elemental composition of the plasma-treated recycled carbon fibers shown in Figure 6, samples prepared using 100 W for 30 s were selected for application to epoxy composites.

2.1.2. Characterisation of recycled carbon fibres

SEM. Characterization of the effect of plasma treatments on recycled carbon fibers was performed by scanning electron microscopy (SEM; VEGA 3 TESCAN; ZEISS NEOPHOT 32).

TEM. High-resolution transmission electron microscopy (HR-TEM) images were obtained using an FEI Titan electron microscope operating at 80 kV.

Wetting measurements were performed by determining the water contact angle (WCA). It was measured using a DSA30 droplet shape analyzer (Krüss) with a water volume of 3 μl; RCF was spread on adhesive tape to measure the contact angle. Droplets were applied to each prepared sample and then the approximate WCA value was calculated.

XPS analysis. X-ray photoelectron spectra (XPS) were recorded using a Phoibos 100 hemispherical analyzer (from Specs) operated in FAT mode. An AlKα spectral line with an energy of 1486.6 eV was used and the spectra were referenced to aliphatic carbon bonds at 285 eV. Resolution spectra for a transition energy of 10 eV were used and CasaXPS software (including RSF factor) was used for calculations.

2.2. Preparation of FRE composite samples

For low fibre concentrations (1 wt%, 2.5 wt% and 5 wt%), the epoxy, hardener, and fibres were mixed for 10 minutes using a magnetic stirrer. At higher concentrations (20 wt% and 30 wt%), manual mixing was used. The plasma-treated RCF could be more easily mixed into the epoxy-hardener mixture, and magnetic stirring was also used for the 20

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wt% concentration. The mixture was then poured into moulds and left at room temperature for 24 hours. Subsequently, the mixture was left in the oven for 15 hours at 60°C. The overall sample preparation process is presented in [11].

2.3.Characterization of FRE composite samples

SEM analysis. Characterization of the fracture surfaces of the FRE composites was performed by scanning electron microscopy (SEM; VEGA 3 TESCAN); the images are shown in Figure 7.

The impact toughness was tested according to ASTM D 256. A 2.7 J pendulum was used at an impact velocity of 3.46 m²/s. The dimension of each specimen was 100 × 10 mm. The specimen thus prepared was placed on two supports with the larger specimen face downward and then broken with a pendulum hammer on the narrower side of the "edgewise" body. Samples with the same fibre concentration were measured ten times, and the arithmetic mean and standard deviation was calculated from the data obtained. The impact toughness measurements A [J/m²] are shown in Figure 8. The following formula was used for the calculation:

$$A = \frac{E_c}{h \cdot b}, \qquad (1)$$

where: E_c[J] is the impact energy required to break the sample, h [m] is thickness and b [m] is width of the composite samples.

The flexural strength was measured according to EN ISO 14125. The three-point bending test was carried out on a Tiratest 2300 testing machine. The dimension of each specimen was 100 × 10 mm. The movement of the standing transom, which applied the deformation to the test specimen, was set to a maximum displacement of 1.5 mm. Samples with the same fiber concentration were measured ten times, and the arithmetic mean and standard deviation were calculated from the data obtained. The resulting bending stress σ [MPa] and elastic modulus E [GPa] are shown in Figure 9, and are valid:

$$\sigma = \frac{3 \cdot F \cdot l}{2\, h \cdot b^2} \qquad (2)$$

Where F[N] is the loading force and l [mm] is the distance between the supports. To calculate the elastic modulus:

$$E = \frac{F \cdot l^3}{4 \cdot s \cdot h \cdot b^3} \qquad (3)$$

Where s is the deflection [mm].

3. Results and discussion

3.1 Analysis and morphology of fibres

SEM images. Together with XPS analysis, these images helped select a suitable filler type for the fabrication of the composite samples. Comparing the SEM images of RCF in Fig. 1, we can see the fiber surface destruction in the plasma-treated fibers that occurred during MW plasma treatment after 10 min exposure time, similar to [13-15]. After MW plasma treatment after an exposure time of 1 min, deepening and highlighting the striated fiber surface occurred. Similar highlighting of the striated surface was also reported in [16-18].

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Comparing the SEM images of RCF, Fig. 2(a) with the MW plasma-treated images in Fig. 2(b) and the RF plasma-treated images in Fig. 2(c), we can see that after MW plasma treatment, the striated surface of the fibers was highlighted. After both two types of plasma treatments, there was a loss of minor impurities formed on the recycled fibers during the recycling process.

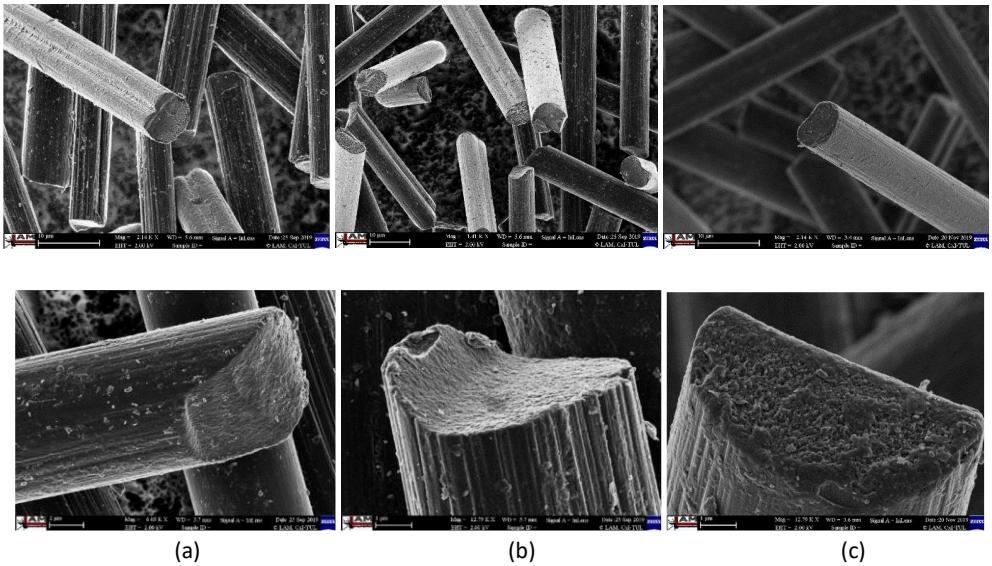

Figure 2. SEM photos of RCF (longitudinal views in the upper part of the figure, cross-sectional details in the lower part of the figure): (a) RCF, (b) MW plasma-treated for 1 min, (c) RF plasma-treated for 30s.

TEM images. Due to the nature of TEM imaging, we focused on the ends of the fibers, where details of the longitudinal arrangement of the carbon fiber surface can be seen. No significant changes were observed between fibers without and with plasma treatment at the fibre ends. In Fig. 3, the turbostratic structure of the transverse fracture arrangement of the fibers can be seen, and it can be assumed that the original fibers had a moderately high modulus of elasticity.

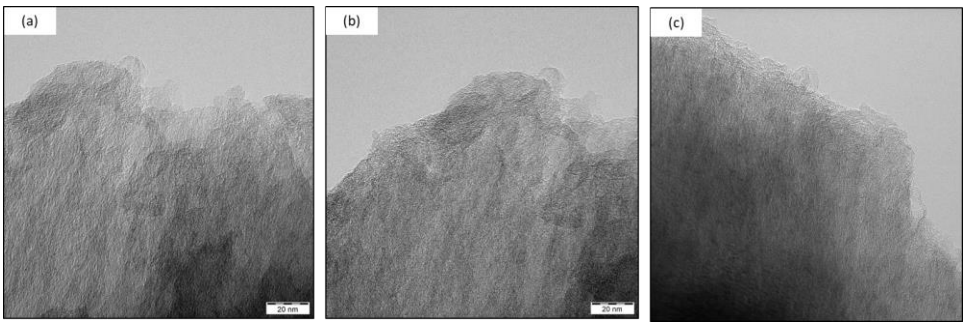

Figure 3. TEM images of the ends of the used RCF: (a) RCF, (b) MW plasma-treated for 1 min, (c) RF plasma-treated for 30s.

Measurement of wettability. The contact angle of WCA recycled carbon fibre is affected by plasma action. Fig. 4 shows that it can be observed that the plasma treatment has a significant effect on the changes in the hydrophobic character of the recycled carbon fibers. Both the type of plasma treatment and the duration of this treatment influence. In the case of MW plasma, the WCA value decreased with a longer plasma treatment time. Untreated fibers exhibit hydrophobic behaviour, while plasma treated fibers exhibit hydrophilic behaviour.

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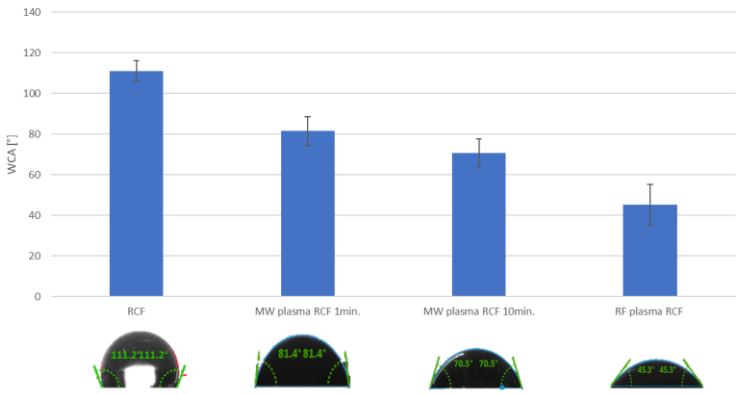

Figure 4. Measurement of wettability of RCF.

Comparing the WCA results shown in Fig. 4, we see that microwave plasma's longer plasma exposure time reduces the WCA angle value, i.e., the fibers are more wettable after more extended MW treatment. Nevertheless, considering the destructions demonstrated by SEM analysis, fibers with an MW treatment time of 1 min were chosen for application to composites.

Raman spectroscopy was used to assess the effect of plasma treatment on the structural changes of carbon fibres. The Raman spectra (RS) for different types of carbon structures show two peaks at approximately 1355 and 1580 cm^{-1}, corresponding to the defective carbon (D band) and graphite mode (G band). Fig. 5 shows the RS of RCF and the plasma-treated RCF. The intensities of the D and G oscillations are related to the graphite crystal size and the proportion of the amorphous carbon phase. These peaks correspond to the usual D and G peaks in PAN-based carbon fibers reported by Fitzer and Rozploch [19]. Recycling of carbon fibers does not change their RS spectra and thus probably does not significantly change their surface structure, as also pointed out by [20]. Comparing the RS results of RCF and those of plasma-treated RCF, it can be concluded that plasma treatment did not fundamentally change the crystalline structure of RCF because plasma only produces a surface effect in the nanometer range.

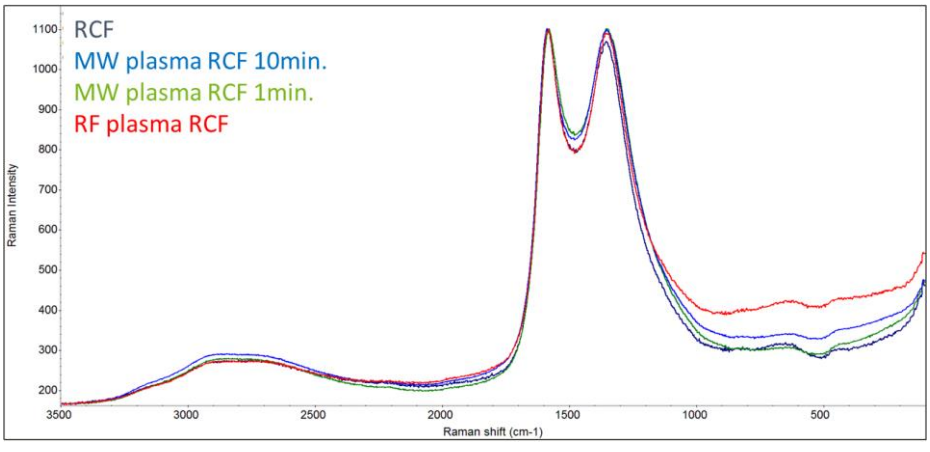

Figure 5. RS used RCF.

XPS analysis. The plasma treatments modify surface of carbon fibers significantly. The main observed effect is functionalization of fibers by introducing an oxygen functionalities on the surface. The plasma treatment increased oxygen content twice from 10 at.% on untreated fibers up to approx. 20 at.% on plasma treated in both types of discharges. The nitrogen content is not significantly changed by the plasma treatment.

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The detail study of chemical bonding on the surface was made on high resolution photoelectron spectra. Carbon related photoelectron peak C 1s consists of multiple component because bonds carbon to carbon, nitrogen and oxygen are expected. There is many possible types of bonds thus the components shall be interpreted as not comprehensive list. The main components were identified as follow: C-C at 284.5 eV, C-O-C ether-like at 286.2 eV, C=O carbonyl-like at 287.4 eV, C=O-O carboxyl/ester-like at 288.1 eV and p-p shake-up at 291.8 eV in agreement with literature [21-22]. The C to N bonds should be also present in C1s peak but due to the wide range of possible binding energies of this components and lower content of nitrogen compare to oxygen we have neglected C-N bonds in C1s spectra moreover the C-N components are overlapped by C-O components.

Literature reported Raman analyses showed both G and D bands typical for sp2 and defects/sp3 hybridization [23]. Therefore, we have introduced sp3 related component to C1s component model at binding energy about 0.8 eV shifted from the main C-C peak, then original C-C peak acts as sp2 related component. When model without sp3 component was used the C-C component was approximately in the amount of sp2 + sp3 components. The Raman analyses in literature [23] shows increase in ratio D/G for plasma treated samples and decrease for thermal treated samples. Although C1s component for sp3 and sp2 hybridization are not equal to D and G bands we observe similar behavior for MW plasma treatment ratio sp3/sp2 from XPS spectra is increasing from 0.23 up to 0.7 when RF plasma treatment leads to decrease in ratio sp3/sp2 to 0.17 for some conditions. The RF plasma treatment is similar to thermal treatment for some materials [24]. It is probably because higher energy particles are created in the discharge and they can modified the fibers more inside with more effective heating effect than low energy particles in MW plasma with dominant surface effect only.

The nitrogen related N 1s peek has two components [21] located at around 398.5 eV and 400.5 eV binding energy. Nitrogen present in the carbon fibers is from the polyacrylonitrile (PAN) used as precursor. Even after carbonization at temperatures above 1000 °C a small amount of nitrogen remains in the fibers, and it is incorporated in the graphite-like structure mostly in two ways: either at the center of three aromatic rings, bonded to three carbon atoms (graphitic nitrogen); or bonded to two carbon atoms at the edges of the graphite-like sheets or at defects (pyridine-like nitrogen) XPS study shows significant differences between plasma treated fibers [21]. The lower binding energy component represents the pyridine-like nitrogen and this type of nitrogen is reduced by both of studied plasma treatments. The pyridine-like nitrogen is reduced from initial 23% to 19% in 100 W and longer 30W RCF plasma-treated RF and to 12% in RCF plasma-treated MW for treatment times longer than 30 minutes. Therefore, we can control the N1s content on the surface in this range by plasma conditions.

The oxygen photoelectron peek O1s can be successfully fitted by 3 components. Where the component at lowest energy 531 eV is O=C, component at 532.9 eV is O-C similarly as in polymers [21], the last weak component about 536.4 eV is adsorbed water. The plasma treatments are significantly reducing amount of lowest energy component O=C from initial 40% to approx. 17% for MW or 20% for RF plasma. The simultaneously O-C component is increased in relative and also in absolute values because of the increase in total amount of oxygen.

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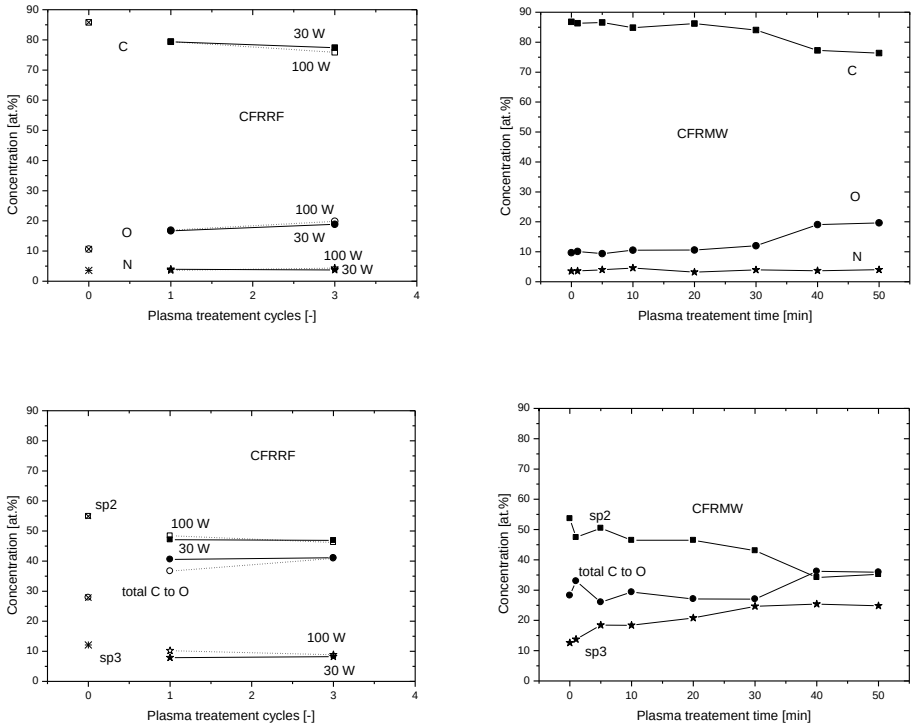

Figure 6. XPS analysis of plasma-treated RCF: (a) RF plasma-treated, (dotted line = fibres treated at 30W, solid line = fibres treated at 100W), (b) MW plasma-treated.

3.2 Characteristics of FRE composites

Characteristics of fracture surfaces. SEM analysed the surface of the fracture surfaces. Figure 7(a) is a snapshot of the pure resin without filler. Figure 7(b)-(d) are microphotographs of the composites filled with recycled carbon fibers at 1%wt.

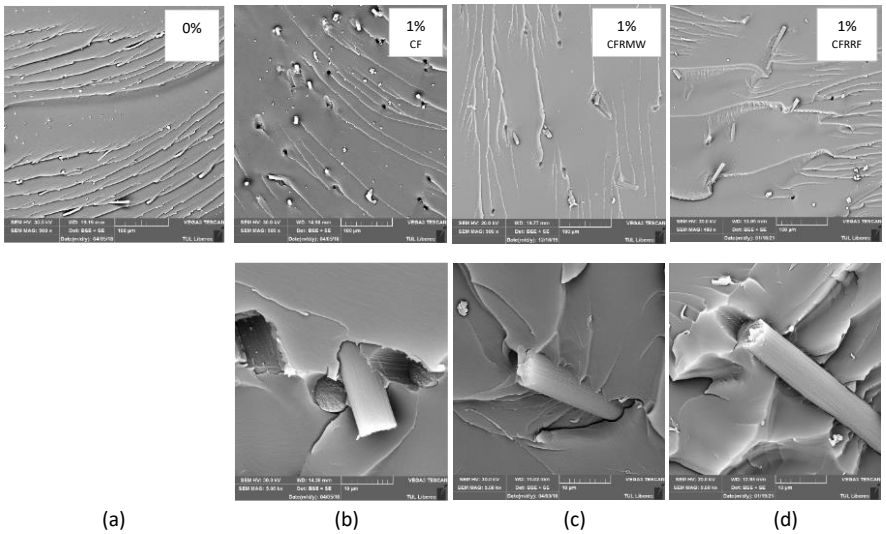

Figure 7. SEM photos of FRE (view of the total fracture surface in the upper part of the figure, details of the fiber-resin interfacial interface in the lower part of the figure): (a) Neat resin, (b) RCF – 1 %wt, (c) MW plasma-treated RCF - 1 wt%, (d) RF plasma-treated RCF - 1 %wt.

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After mechanical tests on FRE composite specimens, the fracture surface images obtained by SEM were analyzed. The dispersion state of the recycled carbon fibers in the epoxy resin was evaluated. Fig.7 (a) Pure resin shows a brittle homogeneous fracture surface. The images of composites containing filler in the form of RCF in Fig. 7 (b)-(d) show fibers that are dispersed continuously in the matrix. According to the morphology of the carbon fibers, these short fibers can transmit stress, which leads to the absorption of external energy during mechanical testing. When cracks form in the composite, the dispersed fibers of the modified RCFs can prevent the propagation of these cracks, but only to a certain extent and at lower fiber concentrations, as also reported by Altay [22]. The improved mechanical properties of epoxy composites with plasma modified RCFs are due to the inhibition of crack growth, which was also confirmed in [25-26].

Impact toughness of FRE composites. One of the possible disadvantages of using micro fillers in epoxy matrices is the potential formation of microscopic cracks. Microcracks in the materials can propagate under repeated loading or a combination of different loads and lead to the destruction of the composite, as pointed out by Teh [27]. If the measured values of impact toughness of composites filled with recycled carbon fibre are compared with the impact toughness of pure resin (in Figure 8). The RCF plasma treatment improves the impact toughness of the samples with 1 wt% RCF. In contrast, plasma treatment of fibers has no significant effect on samples with high filler concentrations. Low fiber concentrations (1 wt%a 2.5 wt%) increased the tested composites' impact toughness values compared to the unfilled resin's impact toughness values. Compared to other studies [25-27], there was no stable increase in impact toughness values for the composites filled with recycled carbon fibers; the composites with the lowest filler concentration achieved the best results. While higher concentrations resulted in a deterioration of impact toughness compared to the impact toughness value of unfilled resin. This is probably due to the shape of the filler in the form of cylinders, and it may also be due to the formation of clusters after reaching a suitable fibre concentration. In contrast, at higher fibre concentrations, on the contrary, the influence of micro-layers reduces the impact toughness value, as also pointed out by [26].

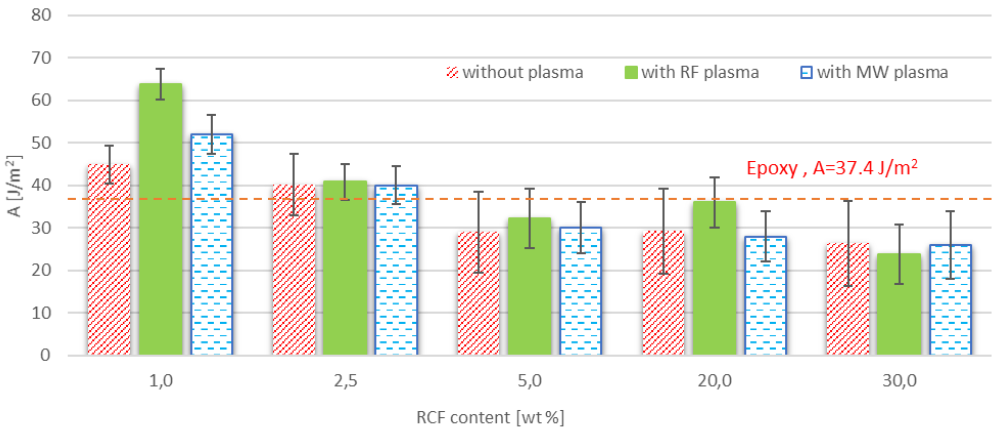

Figure 8. The impact toughness of FRE composites.

Three-point bending. Flexural strength is the ability of a material to resist a bending force applied perpendicular to the longitudinal axis of the composite specimen. The stress-induced by the bending load is then a combination of compressive and tensile stresses [27]. Figure 9 compares the tested composite specimens elastic modulus and bending stress values. It can be seen that the addition of fibers to the resin improves the elastic modulus and bending stress for all fiber concentrations and types. The effect of plasma treatments is negligible except at the lowest concentrations. At low concentrations of 1 wt%, an increase in elastic modulus is observed, namely by 10% for samples with RCF

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fibers without plasma treatment and by, 29% for samples with RCF with RF plasma treatment, and 21% for samples with RCF with MW plasma treatment. The improvement in bending stress values at the same concentration was 15% for samples with RCF without plasma treatment and 31% for samples with RCF with RF plasma treatment, and 26% for samples with RCF with MW plasma treatment. The scatter of the elastic modulus and bending stress values is lower for samples with fibers treated with both types of plasma treatments than for samples with untreated fibers.

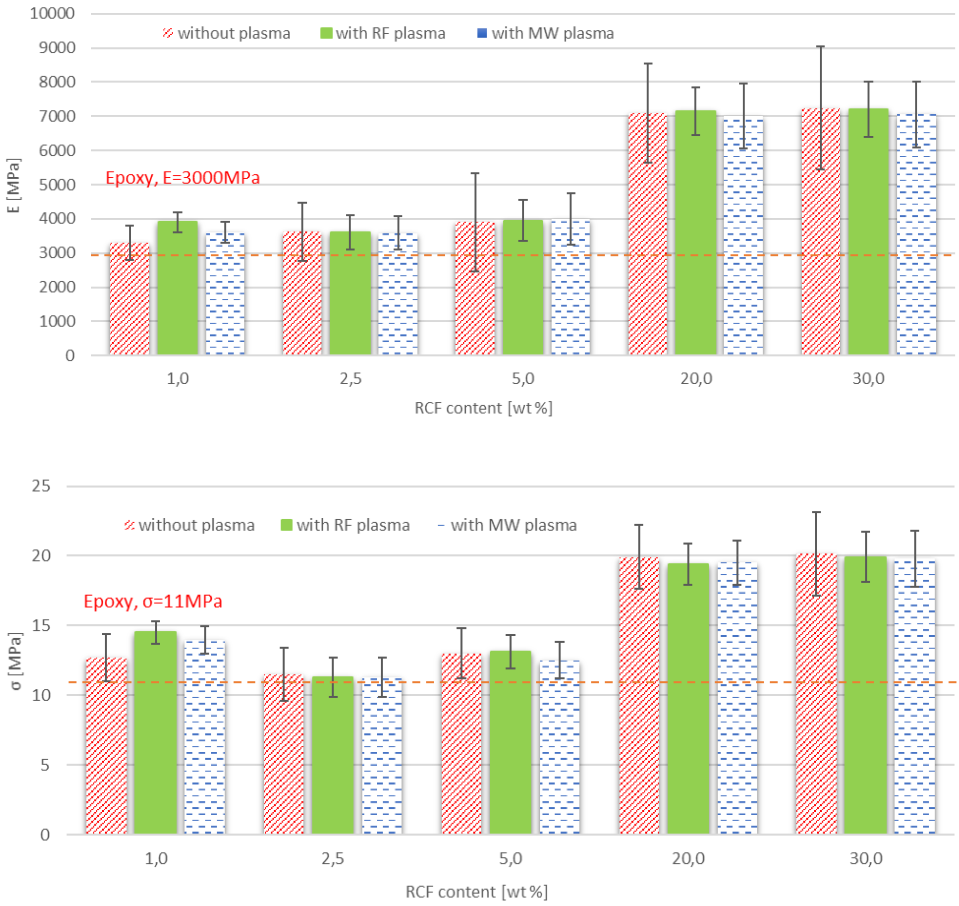

Figure 9. The flexural strength of FRE composites.

4. Conclusion

This study analysed the mechanical properties of epoxy composites filled with short RCF. Two types of plasma treatments were used to treat RCF surface. These treatments were performed using radio frequency (RF) and microwave (MW) at the same pressure and atmosphere. WCA measurements and XPS analysis confirmed that plasma treatments change the surface layers of carbon fibers, as reported in many studies [10-11]. As expected, based on a pilot study [11], using both types of plasma treatments increases the wettability of recycled carbon fibers. At the same time, both types of plasma treatments contribute to the easier implementation of the fibers into the epoxy matrix.

The suitability of plasma treatment was verified during mechanical tests performed on composite samples prepared from these fibres. It was found that the elastic modulus and bending stress increased with the increasing content of recycled carbon fibers. For impact toughness, it was seen that there is an optimum limit. A suitable balance between elasticity and toughness was achieved at composite samples with 1 wt% RCF, both types

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of plasma treatment further improved the tested mechanical properties by this concentration of RCF. However, the RF plasma treatment of the fibres had a more significant effect on the improvement of impact toughness and modulus.

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