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Impact damage in SiO_2 nanoparticle enhanced epoxy – Carbon fibre composites

M. Landowski [a,*], G. Strugała [a], M. Budzik [b], K. Imielińska [a]

[a] Gdańsk University of Technology, Faculty of Mechanical Engineering, Gabriela Narutowicza 11/12, 80-233 Gdańsk, Poland
[b] Aarhus University, Department of Engineering-Mechanical Engineering, Inge Lehmanns Gade 10, 8000 Aarhus C, Denmark

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ABSTRACT

Low velocity impact behaviour of nano-SiO_2 enhanced carbon fibre/epoxy composites for naval applications is reported. Epoxy resin matrix was enhanced by 1- 8 wt% SiO_2 nanoparticles, based on industrial surface-modified nanosilica. Impact parameters: force, deformation, energy, damage size were recorded. The most pronounced effect was damage size decrease; e.g. ~28% recorded by infrared thermography and X-ray computed radiography for 8% nano-SiO_2 content. Crack branching and crack deflection, due to the nanoparticles, were observed. Decreases in permanent deformation, by ~15%, and absorbed energy, by ~8%, were observed, as well as improved fibre/matrix interfacial strength. Flexural strength peaked at 2–5% at ~640 MPa and then decreased, probably due to nanoparticle agglomerations. There was very little effect of small nanoparticle contents on impact force, deformation and energy, with only a 6% increase of the peak impact force for 8% nano-SiO_2 content.

1. Introduction

Naval composites typically employ brittle thermoset matrix materials and may be highly susceptible to low velocity impact loading by hard objects, which causes damage that is difficult to detect. Hence it is important to characterize the impact response of candidate materials [1,2], e.g. nanoparticle enhanced epoxy matrix composites. Nanoparticles have been extensively used in polymer matrix composites as reinforcements on account of their favourable properties compared to corresponding microfillers, including ease of processing and higher property enhancement at 1–5% volume fractions [3–5]. Due to their small size and the absence of any larger aggregates, the nanoparticles can easily penetrate all fibre structures without compromising impregnation by excessive viscosity, thereby enabling all the state-of-the-art process technologies like resin infusion, RTM, or resin injection to be used.

One of the most attractive nanoparticles is SiO_2 which is relatively inexpensive, nontoxic, biocompatible, highly thermal resistant and especially effective as mechanically reinforcing. However, high hydrophilicity of nano-structured SiO_2 surface can induce the nanoparticles to be easily agglomerated and hardly dispersible in the polymer matrix. Thus one of the greatest challenges of producing polymer/SiO_2 nanocomposites is to find an effective method of controlling the dispersion of the nanoparticles in the polymeric hosts [6,7]. In 2002/2003 the first commercial grades of surface-modified silica nanoparticles, manufactured in situ directly in the epoxy resin by a modified sol-gel process, were introduced into the market. The spherical particles have an average diameter of 20 nm and a very narrow particle size distribution. Typical nanosilica levels in e.g. VARTM resin systems are 10% [8]. Nevertheless, it is difficult to determine the optimum addition level as a function of a resin and hardener system of choice.

Sprenger [9] presented a comprehensive review of the mechanical properties of composites with surface modified nanosilica, including the proposition that the modulus increases nearly linearly with increasing silica nanoparticle content. At 10 wt% nanosilica in a resin, an increase in modulus of 30–50%, improvements of 10–30% in compressive strength and in fatigue performance of 50–60% are expected. Experiments have shown [10] that Young's modulus and tensile strength were unchanged by incorporating 2% nano-SiO_2, and that for 8% nanosilica (Nanopox®), Young's modulus showed ~10% increase and there was a slight improvement in tensile strength [11]. At higher nanosilica contents the reported increases of Young's modulus were 23% [12] and 78% [13] for 20 and 25 wt% reinforcement, respectively. The latter (Nanopox® XP) reinforcement also showed 74% increase in Mode I fracture

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* Corresponding author.
E-mail address: mlandowski@gmail.com (M. Landowski).

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toughness K_{IC} [13]. However, at 2 wt% nano-SiO_2, only 16% improvement in K_{IC} was obtained [14].

When these resins are used as matrices for fibre-reinforced composites, however, much smaller improvements were generally found according to Sprenger [15]. He reported increases of 5–25% in fracture toughness for fibre-reinforced composites with an epoxy resin matrix modified with 10–20 wt% of silica nanoparticles. Zeng et al. [16] obtained 20–30% improvement in fracture toughness G_{IC} of carbon fibre/epoxy composite using 6 wt% SiO_2 nanoparticles (Nanopox®). Carolan et al. [17] concluded that toughness increases achieved in the bulk epoxy polymer by the addition of a combination of silica nanoparticles and/or CSR nanoparticles are readily transferable to composite toughness increases, due to its relationship with interlaminar fracture energy of the corresponding CFRP composite laminates. Uddin and Sun [18] showed that 15 wt% silica-nanoparticles enhanced the longitudinal compressive strength of E-glass fibre reinforced composite by 30–40%. Similarly 30% increase of flexural strength for 10–12 wt% SiO_2 was reported in Ref. [8] and improvements are noted for compressive properties in Ref. [15].

In terms of impact behaviour of nanoparticle enhanced matrix composites, Hosur et al. [19] showed that an addition of nanoclay to carbon/epoxy composites decreases impact damage, as a consequence of the higher stiffness and resistance to damage progression of the nanophased laminates. According to the studies of Reis et al. [20–22], in glass and Kevlar/epoxy composites, nanoclay promoted higher impact loads, lower displacements, the best performance in terms of elastic recuperation and maximum residual tensile strength. Assessment of nanofillers (carbon nanotubes, nanoclay, aluminum oxide, silicon carbide) in Kevlar/vinylester composites showed that nanoclay fillers were the best in enhancing impact and mechanical properties of composite materials with 4.3 wt% of nanoclay being the optimum percentage [23]. Uddin and Sun reported considerable increase in low velocity impact strength for

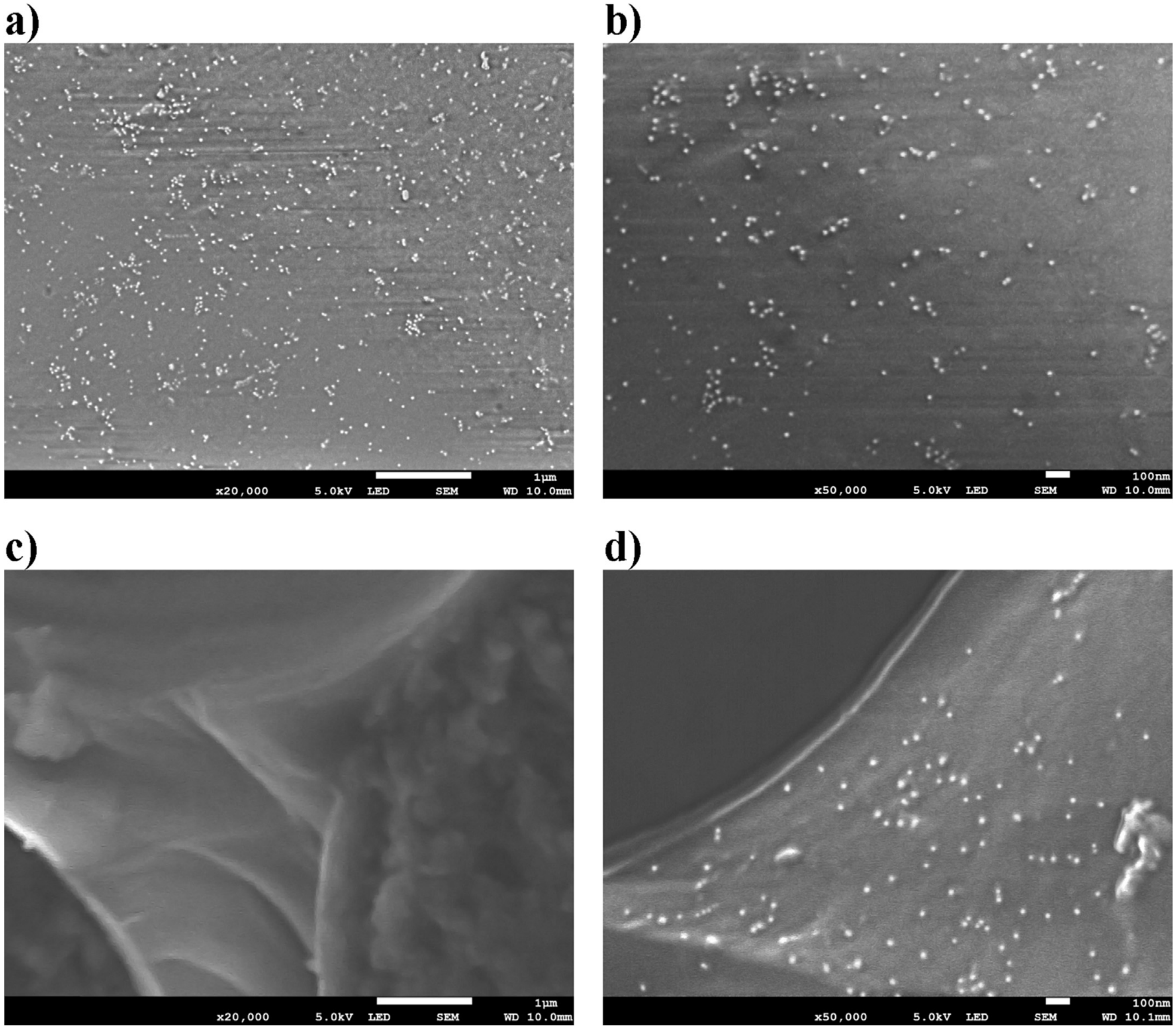

Fig. 1. SEM micrographs of the matrix in carbon fibers/epoxy composite with 8 wt% nanosilica: at 20000× (a) and 50000× (b). Unmodified (c), (20000×) and modified (8 wt% nano-SiO_2) (d), (50000×) matrix/fibre interface.

15 wt% SiO_2 enhanced epoxy and glass fibre reinforced composites [24], but a study of SiO_2 enhanced carbon fibre/epoxy composites showed only a small increase of impact energy absorption [25]. Modification of carbon epoxy composites with silica particles (of the order of 20 nm diameter) and rubber resulted in 50% smaller delamination area at 7 J impact energy and about 30% improvement in residual compressive strength after impact [26].

One of the most important factors affecting mechanical behaviour of fibre-reinforced composites is fibre/matrix interfacial strength. It was observed [27–29] that fracture mode changes from interfacial failure between glass or carbon fibre and epoxy matrix to a combination of interface and matrix failures for laminates with nanosilica, which should provide mechanical properties enhancement. Also changes were investigated in laminate properties with silica nanoparticles only present in the interphase between fibre and resin [30]. Debonding strength and total energy absorption were improved also by fibre sizing containing 1% of 22 nm silica nanoparticles [15]; cracks were deflected, which apparently increased energy absorption.

The preceding short survey shows that it is clear that SiO_2 nanoparticles have positive effect on mechanical properties of fibre-reinforced polymer composites. There are, however, only few reports in the open literature on the impact behaviour of nano-SiO_2 enhanced carbon/epoxy composites. In the investigations now reported this subject was accordingly investigated for industrial surface-modified nanosilica - carbon fibre/epoxy system.

2. Experimental procedures

Carbon fibre-reinforced polymer (CFRP) laminates with Araldite LY1564 DGEBA epoxy resin matrix (curing agent XB3486), supplied by Milar Ltd, were the base materials. The reinforcement was plain weave (1/1) carbon fiber fabric 200 g/m^2 (HexForce® PrimeTex™ 43199 UB 1250 ST). 1, 2, 3, 5, 6, 7, 8 wt% SiO_2 nanoparticles (Nanopox® F400 supplied by Evonik Hanse GmbH) were added to the matrix. Nanopox® F400 is a suspension of 20 nm diameter (40 wt%) SiO_2 in epoxy resin DGEBA to be mixed with epoxy resins [8].

The base resin was mixed with nano-SiO_2 suspension for 30 min at 2800 rpm, using a mechanical mixer. It was then degassed: first

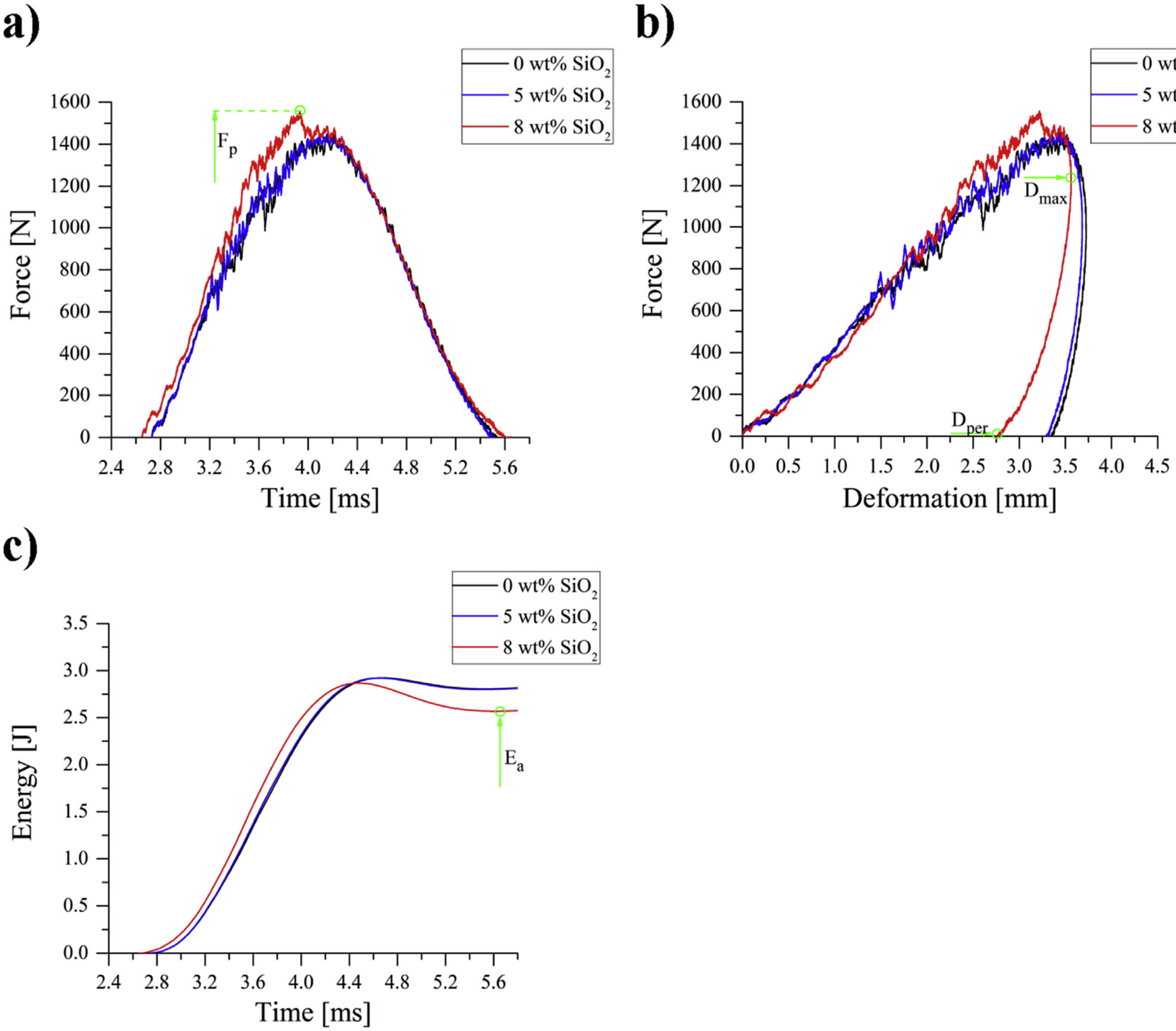

Fig. 2. a) Force-time, b) Force-deformation, c) Energy-time plots for 0, 5, 8 wt% SiO_2.

by allowing it to stand for 24 h and next in a vacuum chamber for another 24 h (−95 kPa, 80 °C), then cooled. Subsequently a curing agent (34 wt%) was added and finally the material was mixed for 1 min at 1000 rpm and degassed in a vacuum chamber for 30 min 4-ply carbon fibre laminate plates of 0.9 mm thickness were formed using the vacuum infusion method.

In order to investigate the morphology of the composite material at 8 wt% SiO_2 a 10 mm × 50 mm specimen was placed in liquid nitrogen for 2 min and fractured. The fracture surface was examined at 20000× and 50000× magnifications on a JEOL JSM-7800 F scanning electron microscope with a LED detector, at 5 kV acceleration voltage.

For each configuration four specimens with dimensions of about 100 mm × 100 mm were cut from the plates using a mechanical saw with a diamond disc. All specimens were postcured at 80 °C for 26 h to obtain maximum strength of the matrix. Impact tester was a Drop Weight Tower Instron CEAST 9310 with a 20 mm spherical tup. Dynamic impact characteristics were recorded using DAS 64 K-SC recorder. The impact energy was 3 J. Flexural tests were performed using an Instron 1112 machine. Five specimens for each configuration were tested according to PN-EN ISO 14125 standard to obtain flexural strength and modulus.

The extent of impact damage was studied using X-ray computed radiography and infrared thermography, which was performed with a GE phoenix v/tome/xs using direct tube (40 kV, 330 μA). The resolution was 0.0104mm/px. For each radiograph presented 64 radiographic integrations were performed with the exposure time of 1000 ms. The length of the longest crack on the horizontal and vertical axes was measured for each sample using Auto-Cad.

In the active thermography test, samples were located within the 2.2 mm distance from the heat source, four Peltier cells, which were heated to 100 °C. Heating was fast (several seconds), and with a high heat intensity. The samples were heated so as to show the area of changed thermal transmission rate due to impact damage. The entire sequence was recorded by an infrared camera Flir A325 with 320x240px detector with a temperature resolution of 0.05 °C.

3. Results

3.1. Microscopy

Fig. 1a shows SiO_2 nanoparticles (8 wt%) generally evenly

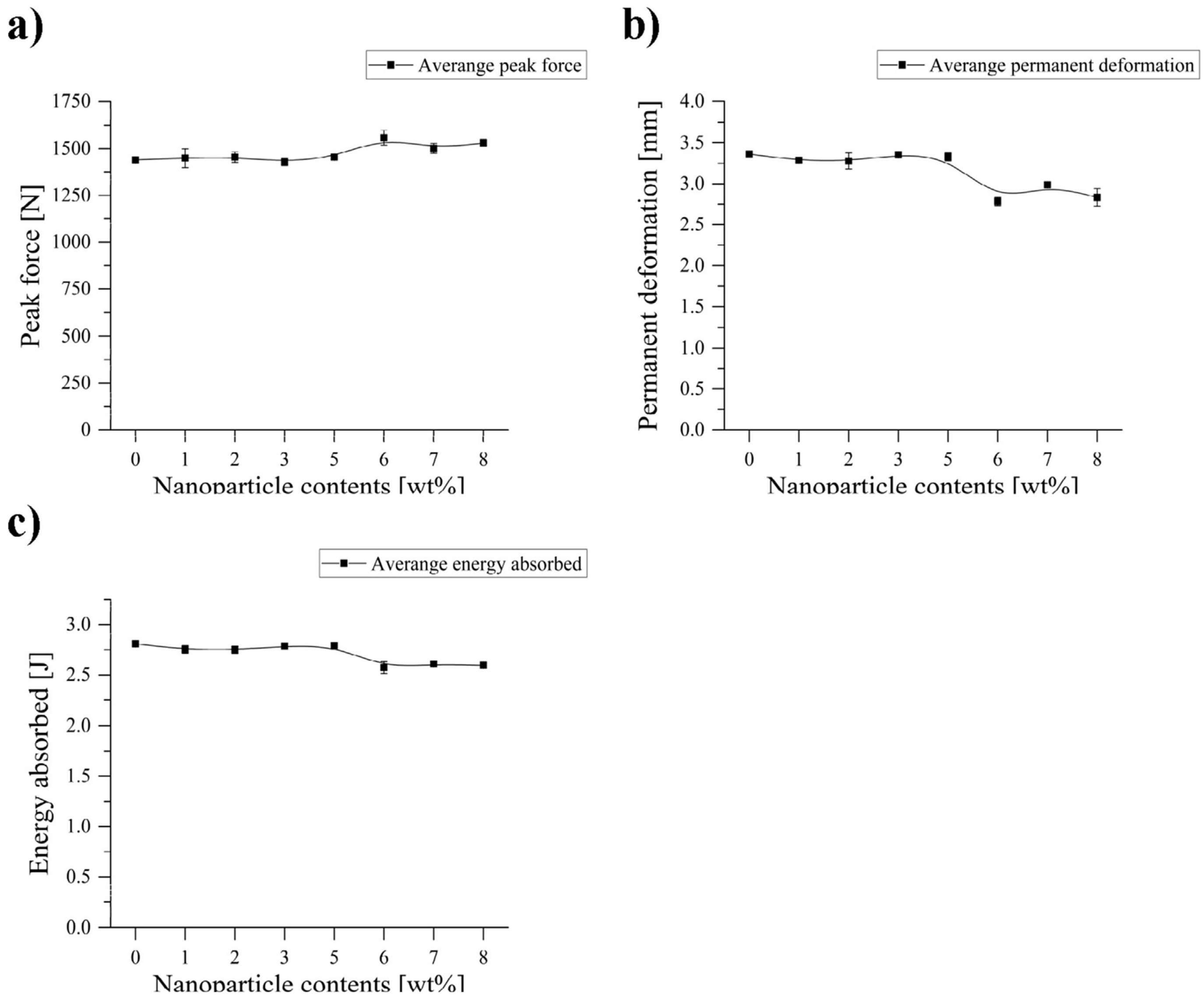

Fig. 3. a) Peak force, b) permanent deformation, c) absorbed energy vs nanosilica content.

distributed in the epoxy matrix. It should be noted, however, that there are some nanoparticle clusters or agglomerates (Fig. 1b). These can lead to initiation of damage under stress and a reduction in strength of the material, to be considered later. The area at the fibre/matrix interface is illustrated in Fig. 1c and d for the unmodified matrix (c) and the matrix with 8% nanoparticles (d), showing lack of visible defects at the particle/matrix interface. SiO_2 nanoparticles are located very close in the interfacial zone.

3.2. Static flexure tests

The flexural tests did not detect a significant change in E, at ~13 GPa, due to incorporation of SiO_2. There was a variation in strength, ±20 MPa: 555, 642, 618, 586, 549 MPa for 0, 2, 5, 6, 8% SiO_2 wt% nanoparticles content, respectively. Flexural strength thus appeared to peak at above 630 MPa for 2–5% nanoparticles and then decrease. This decrease is tentatively associated with nanoparticle clusters, which can play a significant role in initiating and propagating fracture. This role is to be associated with crack deflection and arrest in impact tests, to be now considered.

3.3. Impact tests

The impact behaviour of SiO_2 nanocomposites is characterized by the following parameters: a peak impact force (F_p), permanent deformation (D_{per}) and absorbed energy (E_a) [31,32] obtained from the impact curves (Fig. 2). These curves are characterized by an increase in the load, up to a peak value, followed by a drop. The impactor deformed the specimens and rebounded.

Fig. 2a–c illustrates these data showing force-time, energy-time

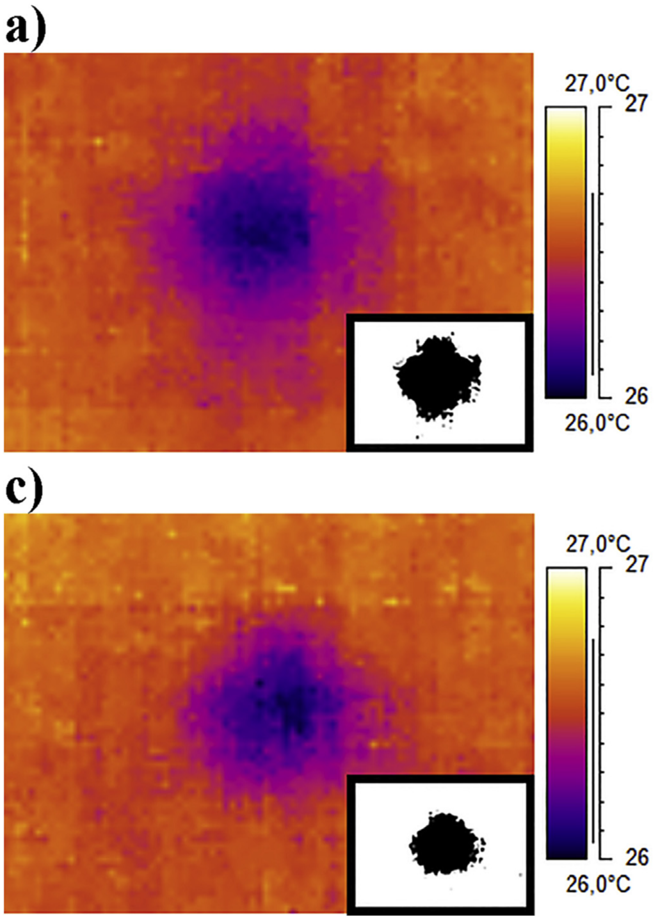

Fig. 4. Impact damage area obtained using active thermography method with threshold set at 210 of the grey scale, a) 0% SiO_2, b) 5% SiO_2, c) 8% SiO_2.

Table 1
Average impact damage area vs SiO_2 nanoparticle contents measured using thermography method.

SiO_2 nanoparticles contents [wt%]	Damage area with standard deviation [mm^2]	% decrease vs 0 wt% SiO_2 [%]
0%	38.4 ± 3.3	–
1%	37.0 ± 2.7	−3
2%	38.7 ± 0.5	1
3%	37.7 ± 3.9	−2
5%	32.1 ± 2.5	−17
6%	34.4 ± 0.2	−10
7%	31.5 ± 4.2	−18
8%	27.4 ± 1.8	−29

and force-deflection graphs respectively, for 0%, 5%, 8% SiO_2. It is to be noted that there is only a slight (ca. 6%) increase in the maximum force for 8% SiO_2 compared to 0%, while permanent deformation decreased by ca. 15%, and also that the absorbed energy decreased by 8–9% for 6–8% SiO_2, compared to the reference specimens.

Fig. 3 compares the peak impact force (a), permanent deformation (b), absorbed energy (c) vs nanosilica content for all specimens tested.

3.4. Impact damage

3.4.1. Thermography

Fig. 4 illustrates the impact damage obtained by infrared thermography [33–35] for specimens with 0%, 5% and 8% SiO_2 nanoparticles. It can be seen that the damage area decreased with increasing nanoparticle content. In order to analyse the full impact damage zone accurately image thresholding was used. The threshold was set at 210 on the grey scale and subsequently a

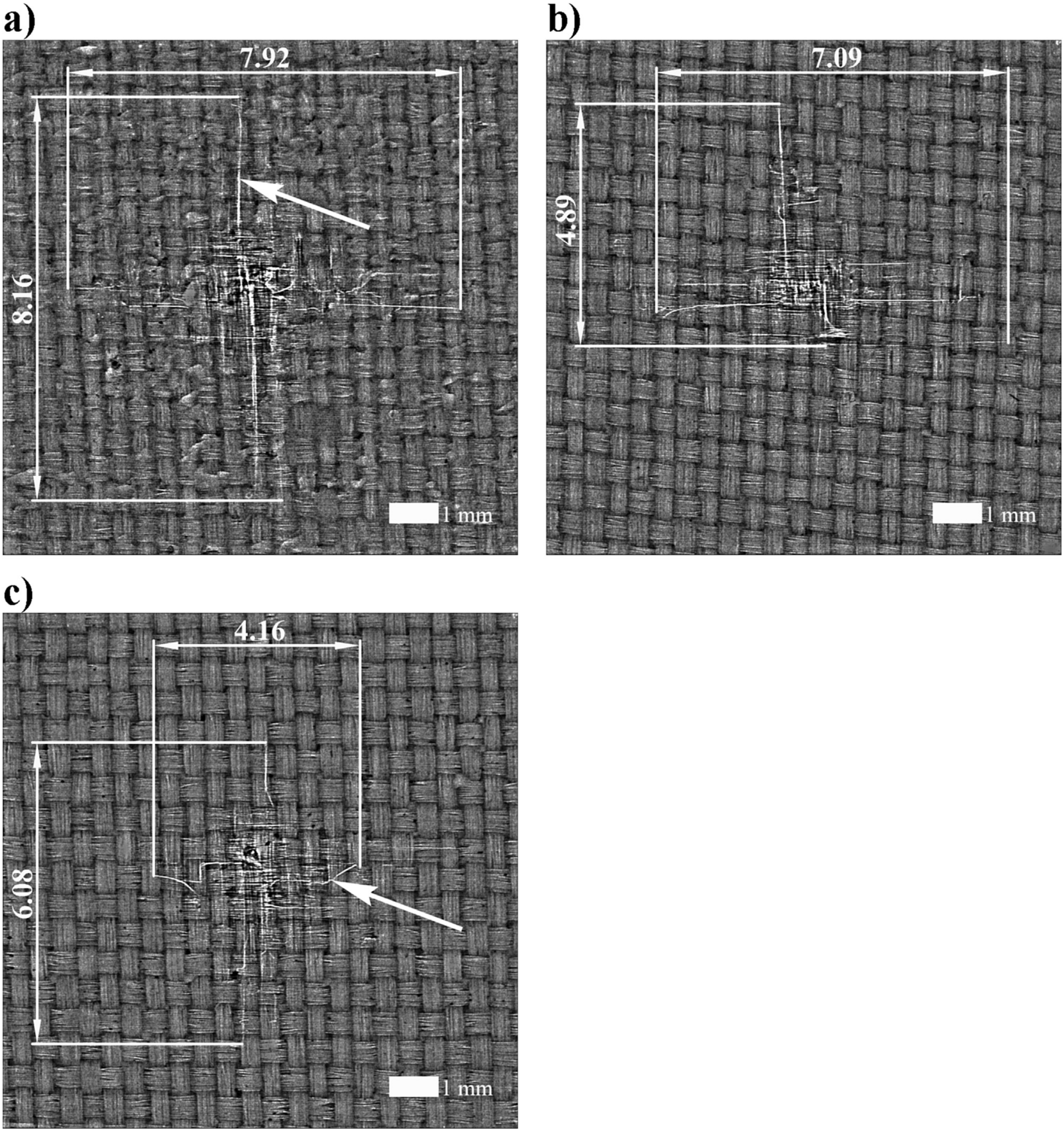

Fig. 5. Impact damage size examined using X-ray computed radiography for a) 0% SiO_2, b) 5% SiO_2, c) 8% SiO_2. Arrows indicate straight (a) and branched (c) crack paths.

Table 2
Average crack lengths in the horizontal and vertical axes measured using X-ray computed radiography.

SiO_2 nanoparticles contents [wt%]	Average crack length: horizontal axis [mm]	Average crack lengths: vertical axis [mm]	Average crack lengths with standard deviation: both axes [mm]	% crack length decrease vs 0% SiO_2 [%]
0%	8.19	7.62	7.9 ± 0.6	–
1%	7.51	7.79	7.6 ± 0.4	−3
2%	8.43	7.76	8.1 ± 0.5	2
3%	7.96	7.53	7.7 ± 1.0	2
5%	7.31	6.37	6.8 ± 1.3	−13
6%	6.74	7.42	7.1 ± 0.5	−10
7%	6.77	6.72	6.7 ± 0.4	−15
8%	5.57	6.04	5.8 ± 0.8	−27

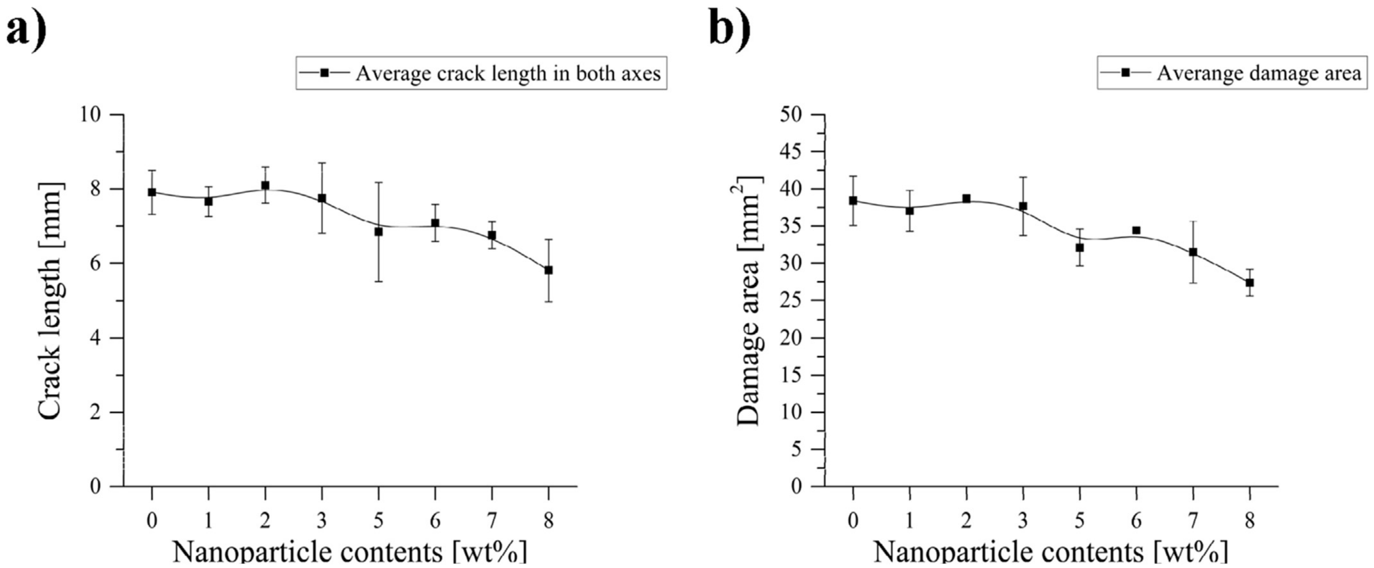

Fig. 6. Results of impact damage assessment using: a) X-ray computed radiography and b) active thermography.

histogram was used to count the number of pixels in the entire damage area. Afterwards, the damage areas counted in pixels were converted to square millimetres. The average results for all samples are shown in Table 1. It is worth noting that only at 5 wt% of SiO2 a significant reduction of the damage area occurs (- 16%) while it rises up for systems containing 8 wt% of the same filler.

3.4.2. *X-ray computed radiography*

Fig. 5 illustrates the impact damage zones obtained on the radiographs using X-ray computed radiography [35] for 0, 5, 8 wt% SiO_2 (a,b,c). To be noted are characteristic main, straight cracks [36] propagating along the orthogonal fibre weave (Fig. 5a, the arrow). The average crack lengths are given in Table 2, showing their significant decrease with increasing nanoparticle content. The crack lengths decreased by 10%–15% for 5%–7% SiO_2 and by 26% for 8% SiO_2, again the effect of SiO_2 starting at 5%. In the 8% SiO_2 specimen main crack branching is illustrated (Fig. 5c, the arrow). This contrasts with the reference specimens, where main cracks were long and straight. This is to be associated with crack arrests and deflections due to nanoparticle clusters at high nanoparticle content. There is good correlation between crack size [X-ray computed radiography] and damage area [active thermography] observations, as illustrated in Fig. 6.

4. Discussion

To complement the earlier studies of the effect of nanoclay particles (1.5–6 wt%) [19–22] and nanosilica [24–26] on the impact behaviour of fibre reinforced composites, the effect of small quantities (1–8 wt%) of nano-SiO_2 was examined. Due to the presence of nanoparticles, a peak force increase and absorbed energy decrease were thus anticipated. However for 8% nano-SiO_2 only a 6% increase in the peak impact force was noted and 15% and 8% decreases in permanent deformation and absorbed energy, respectively. In contrast, in a similar study for glass fibre/nanoclay-enhanced epoxy, Reis [19] obtained ca. 16% increase in the peak load for 3% nanoclays in GFRP composites. In Kevlar fibre reinforced epoxy modified with 1.5, 3 and 6% of nanoclays and subjected to 21 J impact, the peak load increased by ~5; ~19 and ~23%, respectively [21]. Similarly 15–17% increase in the peak load for 20 J was observed for 0.2% CNF in glass fibre/polyster composites [4].

The comparatively lower improvements in the impact force obtained in this study may be attributed to interfacial adhesion properties of fibre/matrix. Accordingly, to examine the nature of these interfaces, the reference material and that containing 8% silica were examined by SEM following failure in static flexure test (Fig. 7a and b). In unmodified material the surfaces of the fibres are relatively smooth, but with adhering resin, which indicates a moderate fibre/matrix interfacial bond strength. For 8% nanoparticles, greater roughness of the surface is noticeable and the fracture is adhesive-cohesive, suggesting better adhesion of fibres and the nanoparticle-modified matrix. Jianing Zhang at al. however, reported that inclusion of nanoparticles did not significantly affect fibre-matrix interfacial bonding strength in carbon fibre-nanosilica enhanced epoxy composites [37].

Our important observation is a significant, by ~28% for 8% SiO_2,

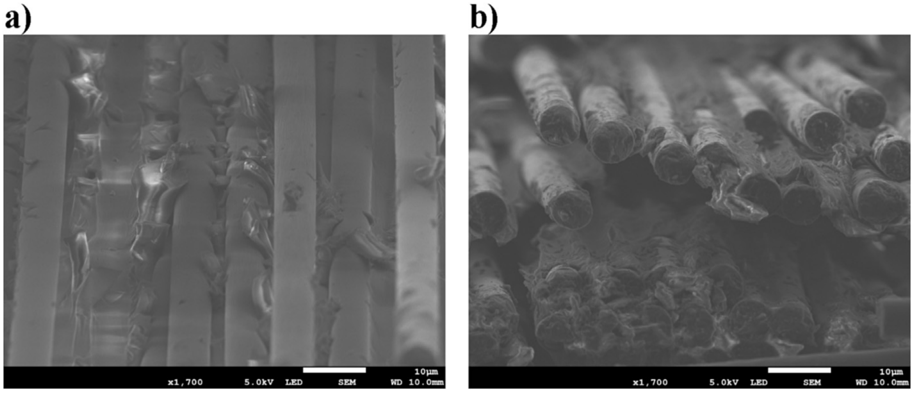

Fig. 7. Fractures of specimens in static flexural test for (a) 0% and (b) 8% nanoparticles.

damage size decrease. This is associated with crack branching and arrests (observed by X–rays, Fig. 5c), in contrast to the straight crack paths in the reference samples (Fig. 5a). The reason for such behaviour are probably nanoparticle clusters, noted in Fig. 1b, formed during mixing of the ingredients. These agglomerates and associated cracking may also be responsible for decreasing fracture strength above ~5% silica loading.

It is therefore suggested that, under impact loading, nanoparticle agglomerates, which provide sites of easy matrix/nanoparticle debonding, deflect the crack tip, which leads to branching and some crack arrests. These mechanisms absorb energy, therefore cause the impact damage area to decrease. This is in agreement with observations of Quaresimin et al. [5] that "some toughening mechanisms like crack deflection and pinning are likely to occur only in the presence of filler aggregates of sufficiently large size; a single nanosized reinforcement being unable to mechanically act to deflect the crack front".

The reduction of impact damage area was not reported previously, e.g. by Sprenger [38] in GFRP laminates subjected to 30 J impact and Kuhn et al. [39] in 25% nano-SiO_2 modified CFRP laminates at 20 J, 30 J, 40 J. The reason for the difference between their results and ours is possibly related to the presence of nanoparticle agglomerates in our study.

5. Conclusions

1. There was very little effect of small nanoparticle contents (1–8% nano-SiO_2) on impact force, deformation and energy for the epoxy-carbon laminates.
2. A transition in properties appeared at ~5% nano-SiO_2 loading: peak strength and commencement of decrease in impact damage size.

3. The damage size decrease at 8% nano-SiO_2 reached ~28%. There is no report of any previously recorded decrease for GFRP and CFRP laminates.

4. This damage size decrease is associated with energy absorbing mechanisms of crack deflection and crack branching due to nanoparticle agglomerates.

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