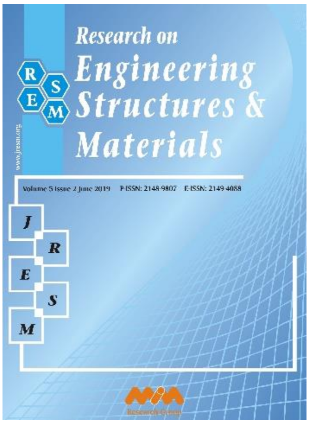
Research on Engineering Structures & Materials
journal homepage: http://jresm.org
RESEARCH GROUP



Evaluation of low-velocity impact behavior of epoxy nanocomposite laminates modified with SiO2 nanoparticles at cryogenic temperatures

Ahmet C. Tatar, Halil B. Kaybal, Hasan Ulus, Okan Demir, Ahmet Avcı

Online Publication Date: 25 Feb 2019
URL: http://dx.doi.org/10.17515/resm2018.55is0704
DOI: http://dx.doi.org/10.17515/resm2018.55is0704

Journal Abbreviation: *Res. Eng. Struct. Mat.*

To cite this article
Tatar A C, Kaybal H B, Ulus H, Demir O, Avcı A. Evaluation of low-velocity impact behavior of epoxy nanocomposite laminates modified with SiO2 nanoparticles at cryogenic temperatures. *Res. Eng. Struct. Mat.*, 2019; 5(2): 115-125.

Research on Engineering Structures & Materials

journal homepage: http://jresm.org

MIM Research Group

Research Article

Evaluation of low-velocity impact behavior of epoxy nanocomposite laminates modified with SiO_2 nanoparticles at cryogenic temperatures

Ahmet C. Tatar[a,1], Halil B. Kaybal[b,2], Hasan Ulus[*,c,1], Okan Demir[d,1] and Ahmet Avcı[e,3]

[1]*Department of Mechanical Engineering, Konya Technical University, Konya, Turkey*
[2]*Department of Mechanical Engineering, Amasya University, Amasya, Turkey*
[3]*Department of Biomedical Engineering, Necmettin Erbakan University, Konya, Turkey*

Article Info

Article history:
Received 4 Jul 2018
Revised 11 Dec 2018
Accepted 25 Jan 2019

Keywords:

Glass fiber;
Epoxy composite;
Cryogenic temperature;
Low-velocity impact

Abstract

Epoxy based fiber reinforced composites are widely utilized in aerospace applications due to mechanical properties, thermal stability and, chemical resistance. However, it is known that materials become brittle and due to the poor crack resist restricts their applications in cryogenic engineering applications. The purpose of this paper is to experimentally investigate the cryogenic temperatures' effect on the low-velocity impact (LVI) test of composite laminates. In addition, the effect of matrix modification in the studied composites was investigated. The LVI tests were conducted at RT (room temperature), 0 °C, -50 °C, -150 °C and -196 °C (liquid nitrogen temperature) on the composite laminates to measure influence on their energy absorption capacity. LVI tests performed according to ASTM-D-7136 standard under 10, 20 and 30 J impact energy levels. The results show that the contact forces and energy absorption capacities are improved by adding SiO_2 nanoparticles into the epoxy matrix. The absorbed energy at cryogenic temperatures is increased by 24.87% from 18.1 J of pure epoxy resin to 22.7 J of modified epoxy. For the purpose of comparison, the LVI properties of composites at room temperature (RT) are also investigated. It is noted that the energy absorption capacity is not higher at cryogenic temperatures than that at RT for the modified and neat epoxy composites. Moreover, the peak contact forces are reduced in low-temperature conditions.

1. Introduction

With the progress in science and technology, the use of polymer composites has become increasingly widespread for both scientific research and engineering applications [1]. Especially, glass and carbon fiber/epoxy composites are increasingly demanding for structural applications in aerospace, automotive and marine industries due to their excellent mechanical performance and design advantages over conventional materials [2]. At the same time, the dynamic behavior of composites under impact loading is one of the major concerns in the use of the industry as it is highly susceptible to impact loads which cause significant damage such as cracking of the matrix, delamination, and breakage of the fibers [3].

Fiber composites have the potential for extensive use in space applications, such as solar arrays, antennas, optical platforms and supports for cryogenic tanks [4]. However, the composites utilization and material selection for low-temperature applications are often

*Corresponding author: hasanulus@selcuk.edu.tr
[a] orcid.org/0000-0001-5112-6170; [b] orcid.org/0000-0002-2312-7106; [c] orcid.org/0000-0001-8591-8993; [d]orcid.org/0000-0001-9411-775X; [e] orcid.org/0000-0002-3105-7849
DOI: http://dx.doi.org/10.17515/resm2018.55is0704

obstructed by the inconsistency of material properties of its components [5]. Because thermal contractions of fiber and matrix due to the cool conditions on the composite structure the give rise to thermal residual stresses and strains which influence most of the mechanical properties. The general of used matrices are brittle and do not allow the release of residual stresses at low temperatures [6] and the toughness degradation induced by low temperatures can lead to structural damages in the form of microcracks or delamination [7].

Dramatic changes in the properties of composites can occur when they are exposed to cryogenic temperatures [8]. Low-velocity impact exposures of the composite structure (such as the drop of an object or the impact to a harsh ground) are commonly encountered a situation in the industry. In order to develop such impact-resistant material to cryogenic applications, LVI behavior investigation is very important to optimize composite systems components. Some studies performed to investigate the impact properties of laminated composites at low temperature but the reports about the lowest temperature (down to –60 °C) is scarce. Laminated composites become rigid with high stiffness at low temperatures (−50 °C to 120 °C) so as their deflections in impact tests were small [9]. Damage areas also smaller and higher perforation threshold resulted for laminates subjected to a low-velocity impact at low temperatures (−60 °C to 20 °C) condition [10]. However, comparatively little work has been done to understand the cryogenic temperatures of laminated composites. Therefore, in this work, the cryogenic behaviors of glass/epoxy composites are reported in terms of the LVI impact properties.

2. The Effect of Factors on Shear Force

In this study, the low-velocity impact (LVI) behaviors of glass/epoxy nanocomposite laminates were investigated using a drop weight impact test. Having been used for LVI tests, neat and nano SiO_2 (silica) added glass/epoxy laminates were produced as 10 layers. The SiO_2 nanoparticles have a specific surface area of 650 m^2/g and the average primary particle diameter of 15 nm. Adding 4 wt% SiO_2 nanoparticles were preferred as nano-reinforcements according to literature survey to perform matrix modification. It is known that best mechanical properties have been determined while using 4%wt SiO_2 nanoparticle addition [11-14]. The epoxy resin system used was a bisphenol-A (DGEBA) from Momentive Hexion, Inc. Fibers were supplied from Dost Kimya Company in Istanbul, respectively. The SEM images of the used nano SiO_2 powder is given in Fig. 1.a and it is seen that the nanoparticles form agglomeration due to attractive forces (van der Waals and others). However, it is important to avoid this situation during production because these agglomerations will cause many defects and stress concentrations in laminates [15]. SEM image of the nanoparticle distribution of 4 wt% is presented in Fig. 1.b The SiO_2 nanoparticles are seen mostly dispersed uniformly. The LVI tests were repeated three times under 10, 20, 30 Joule (J) impact energy levels and at the RT, 0 °C, -50 °C, -150 °C temperatures. In order to reach cryogenic temperature, the composite specimens were immersed in liquid nitrogen and bargain for 10 min to reach the liquid nitrogen temperature [16]. Details of experimental procedures are available in our previous studies [17-21].

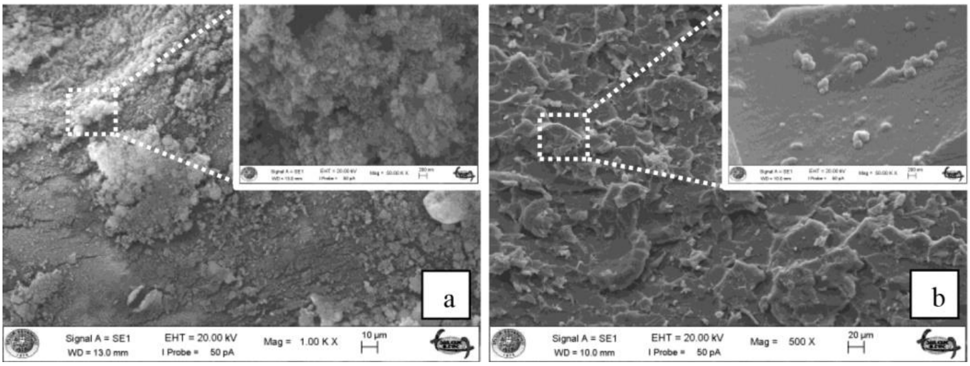


Fig. 1 a) SEM images of used nano-SiO2 powders, b) Homogeneous distribution of 4 wt% SiO2 nanoparticles in epoxy

4. Results and Discussions

Fig. 2 compares the contact force-time and force-displacement curves of the neat composite specimens with the different impact energy levels. The results show that the contact force increases significantly with the decrease of the temperatures. Meanwhile, we also compared the displacements of different temperatures in Fig. 2. It can be seen that the displacements at liquid nitrogen temperature are reduced significantly than that of at RT. Here, diminish of displacement values occur relate to the decreasing contact duration at low temperatures than that at RT. This behavior can attributed to a reduction of ductility and increase of the rigidity in laminate.

The characteristic impact parameters: peak contact force and absorbed energy for composite laminate are presented in Table 1. The peak contact force at liquid nitrogen temperature is the highest for all impact energy levels. Under the 10 J impact energy, comparing at RT to 0 °C, -50 °C, -150 °C and -196 °C the energy absorption capacity are decreased by 0.3%, 13.46%, 17.53% and 25.74% respectively. On the other hand, under the impact energy of 30 J comparing at room temperature, energy absorption capacity are decreased by 23.68%, 27.09%, 28.81% and 30.42% respectively. These results show that low velocity impact behaviors of the composite materials affected between cryogenic and room temperatures. Also clearly seen, the absorbed energy decreases with the decreasing temperature for all impact energy levels. Laminated composites exhibit relatively ductile behaviors at room temperature compared to that cryogenic temperatures because of the epoxy resins' show obviously brittle behaviors at cryogenic temperature for all compositions [22, 23]. Components of composites can create significant thermal mismatch and internal stress at low temperature applications. The internal stress within composites can greatly affect the mechanical performance by creating micro-cracks and voids [23]. On the other hand, the formed hydrogen bonds in the epoxy become stronger at cryogenic temperatures due to the shorter chemical-bond length. Free volumes in the composites would be reduced at cryogenic temperatures due to thermal shrinkage and it promotes to a higher intermolecular force then also leads to enhanced strength compared to the RT [24].

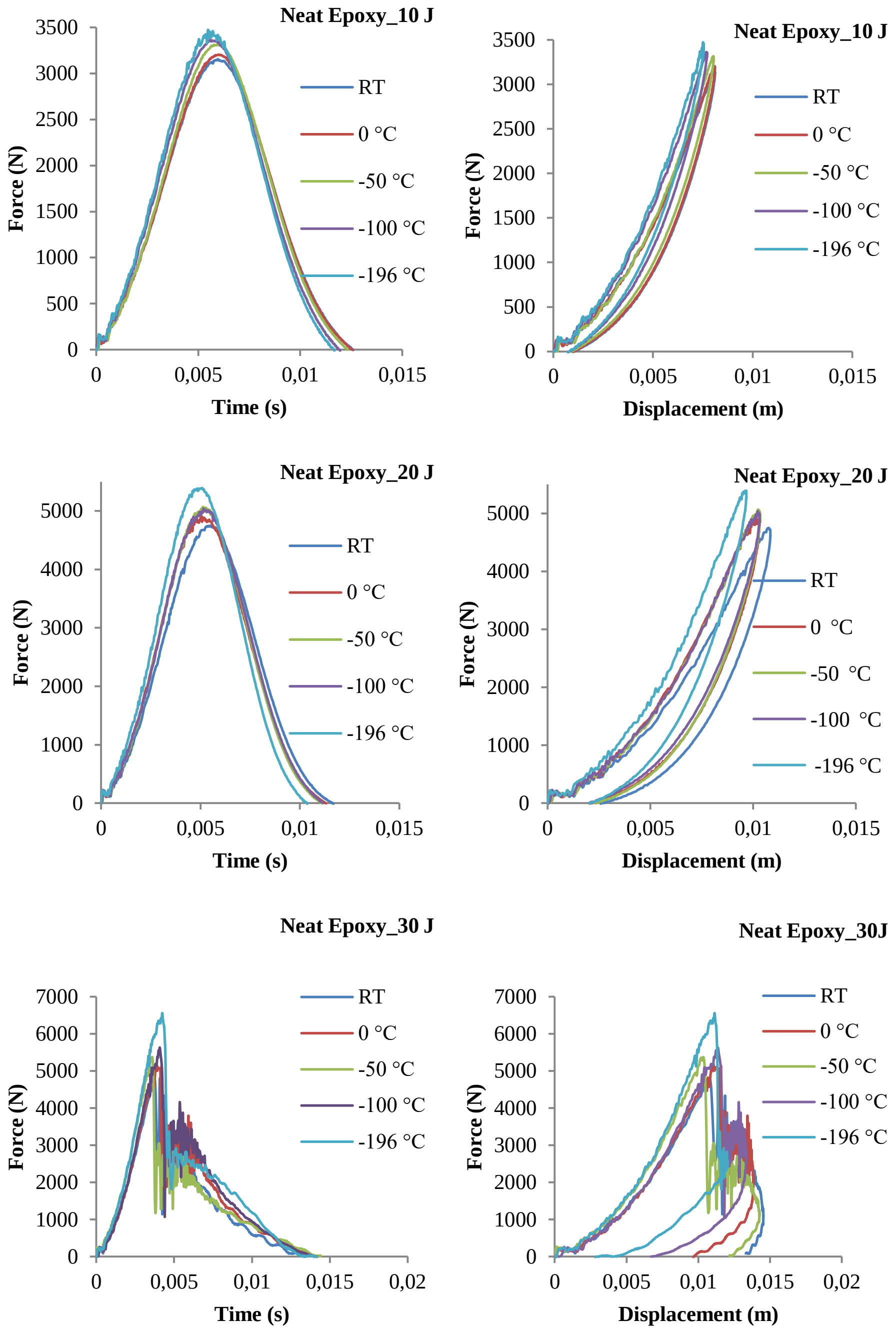


Fig. 2 Force-time and force-displacement curves at different impact energy levels

Fig. 3 shows the force-time and force-displacement histories of neat and 4 wt% nano SiO_2 reinforced for different energy levels. In the graphs, no significant change was observed with the addition of SiO_2 nanoparticles for 10 and 20- joule energy levels while average contact force value increased significantly depending on the addition of SiO_2 nanoparticles for 30-joule energy level.

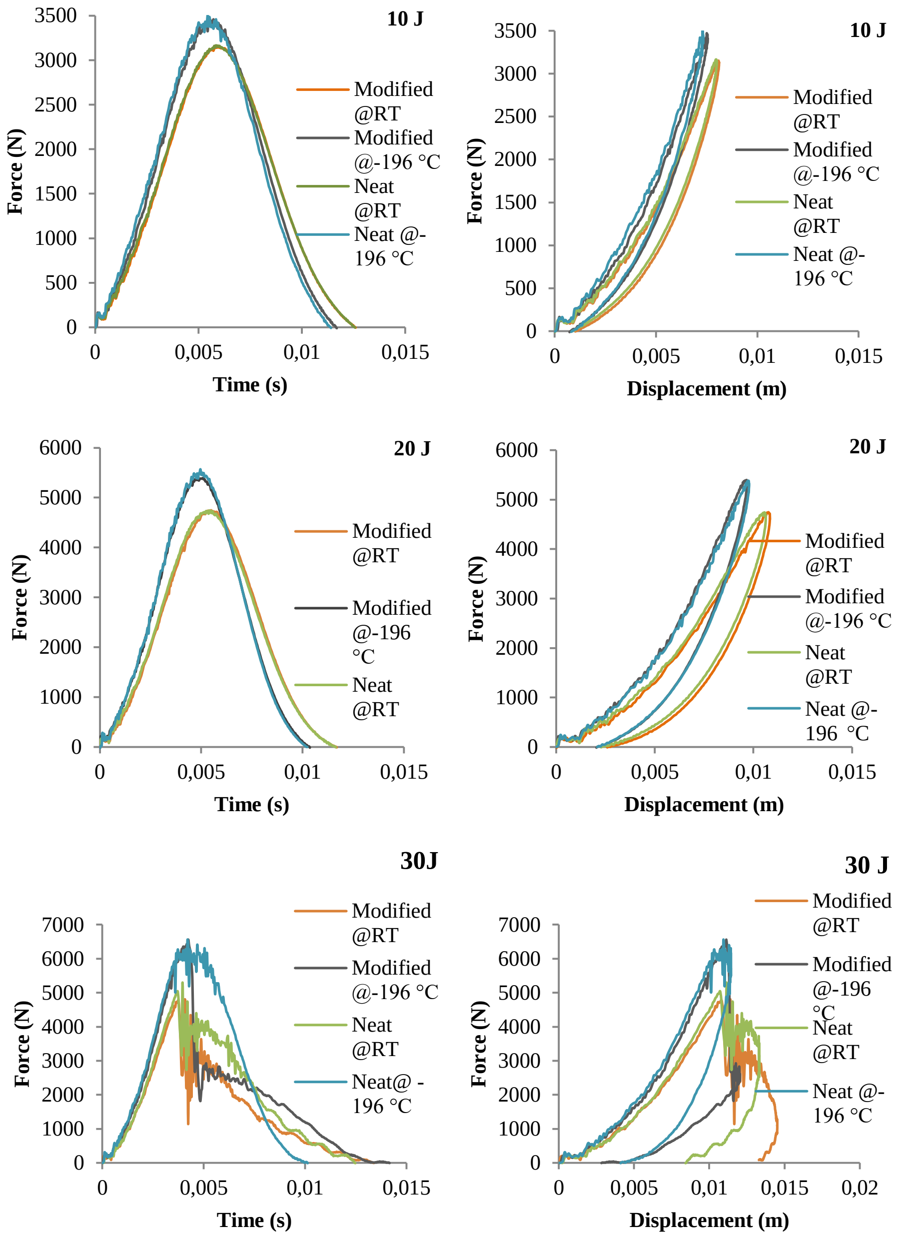


Fig. 3 Force-time and force-displacement curves of neat and modified composites at different impact energy levels

The change in the impact properties of the nanoparticle-added specimens is given in table 2. It is seen that peak forces are not affected much by the temperature under the low impact energy while it affected depending on temperature. The ratio of percentage change of peak contact force under the 30-joule energy level are observed 13% and 24.8% for SiO_2 nanoparticle added composites, respectively.

Nano reinforcements have been extensively used to develop of the fracture toughness of matrix material and restrict of crack propagation. Therefore, to increase the mechanical performance of composite materials, researchers have been carried out to modify the matrix materials by SiO_2 nanoparticle. Many advantages of SiO_2 nanoparticles have been reported in literature to improve fracture toughness [25]. Especially, for the tests which under the 30 j impact energy level, the laminates absorbed energy is drastically increased than that of the neat epoxy matrix. This is because the laminates weren't severe destruct under the impact energy levels of 10 and 20-joule. However, it is understood that SiO_2 nanoparticles activate the toughening mechanisms for 30-joule energy levels effectively.

For the sample with the 4 wt% SiO_2 content, the specimens' absorbed energy and peak contact forces have slightly higher than that of the neat matrix. This is because the improvement of nanocomposites strongly depends on the toughening mechanisms of SiO_2 nanoparticles. When the probable crack in the matrix encounter with SiO_2 nanoparticles, it can be pinned/branched/deflected shown as in Fig. 4. The paths of crack propagation into matrix causes more fracture energy absorption. Moreover, microcracks formations in epoxy matrix are observed in Fig. 4. The microcracks formation and its propagation are also important factor for increasing fracture energy [14].

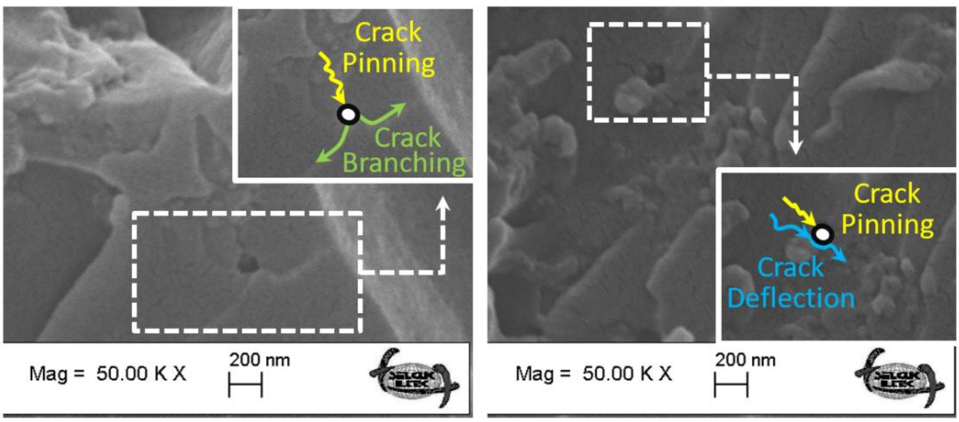


Fig. 4 Toughening mechanisms of SiO_2 nanoparticles

The cross-sectional view of LVI exposed samples at the damage site is given in Fig. 5. In the tests performed at room temperature, it is clear that the neat matrix samples failure is seen developed by delamination (shown by yellow dashed lines). In contrary less delamination is observed in the modified matrix samples, while the damage is predominantly seen as a fiber break (shown by red dashed lines). This is a clear indication that the interlaminar strength increases due to matrix modification. Moreover, in high magnification observations, a significant roughness is noticed on the fiber surfaces of modified matrix composites. This increase in fibers' roughness is evidence that fiber-matrix interface adhesion has been developed and the modified matrix supports the reduction of delamination in samples [26]. Reduction of the residual deflection which performed tests under cryogenic temperatures occurs primarily due to the show low ductility of the materials under cryogenic temperatures [11]. Similarly, nanoparticles modified composites show fewer delamination damages and better fiber-matrix interface adhesion.

Table 1 LVI properties of 10, 20 and 30 J impacted composites at RT, 0 °C, -50 °C, -150 °C and -196 °C.

Impact Energy	Temperature Condition	Peak Contact Force (N)	%↑	Absorbed Energy (J)	%↓
	RT	3143		2,95	
	-50 °C	3198	1,75	2,94	-0,34
10 J	-100 °C	3334	6,08	2,6	-13,46
	-150 °C	3375	7,38	2,51	-17,53
	-196 °C	3452	9,83	2,35	-25,74
	RT	4700		9,27	
	-50 °C	4885	3,93	9,26	-0,10
20 J	-100 °C	4971	5,76	8,71	-6,04
	-150 °C	5043	7,29	8,1	-12,62
	-196 °C	5381	14,48	7,9	-14,77
	RT	4980		26,13	
	-50 °C	5185	4,12	19,94	-23,68
30 J	-100 °C	5359	7,61	19,05	-27,09
	-150 °C	5635	13,15	18,6	-28,81
	-196 °C	6357	27,65	18,18	-30,42

Table 2 LVI properties of 10, 20 and 30 J impacted composites at different temperatures

Temperature (°C)	Neat Force (N)	SiO_2 Force (N)	Change (%)	Neat Energy (J)	SiO_2 Energy (J)	Change (%)
			10 Joule			
RT	3143	3159	0,51	2,93	2,95	0,68
-196 °C	3452	3502	1,45	2,35	2,56	0,94
	9,83%	10,86%		-19,80%	-13,22%	
			20 Joule			
RT	4700	4756	1,19	9,08	9,27	2,05
-196 °C	5381	5566	3,44	7,85	7,94	1,14
	14,49%	17,03%		-13,54%	-12,56%	
			30 Joule			
RT	4980	5295	6,33	26,13	29,54	13,05
-196 °C	6257	6741	7,71	18,18	22,7	24,86
	25,64%	27,30%		-30,42%	-23,16%	

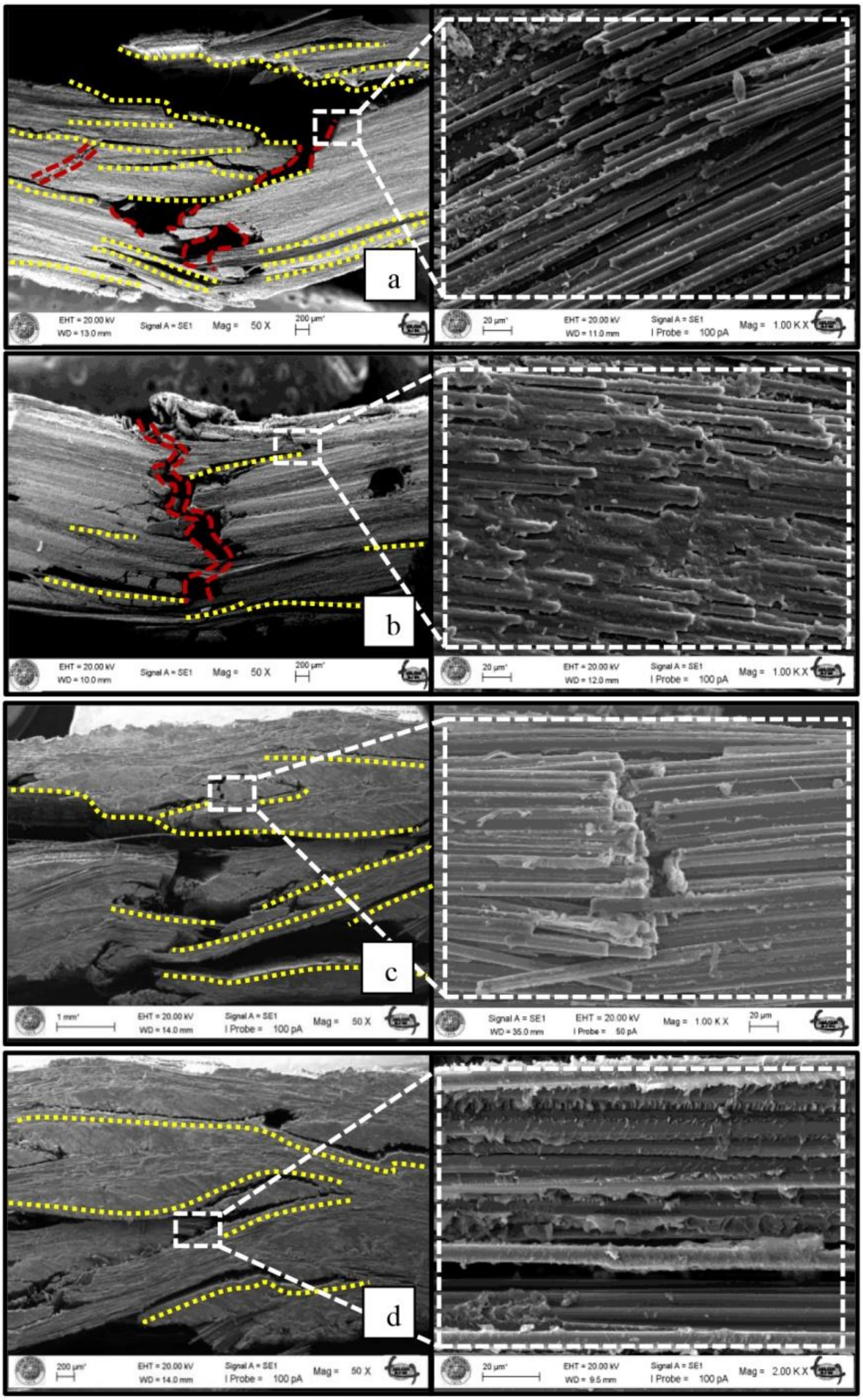


Fig. 5. Cross-sectional SEM images of impacted area: a) neat epoxy matrix_RT, b) modified epoxy matrix_RT, c) neat epoxy matrix_-196 °C, d) modified matrix_-196 °C

5. Conclusions

In this study; low-velocity impact responses of glass fiber reinforced neat epoxy composite and 4% (wt) SiO_2 modified epoxy composite laminates were investigated at different temperatures. From the results, the following conclusions can be drawn:

- It is clear that nanoparticle addition has an effect on slightly higher energy levels as opposed to low energy levels. The absorbed energy of 4%wt SiO_2 nanoparticles reinforced and neat epoxy composites for 30 J impact energy have been found an average value of 22.7 J and an average value of 18.1 J respectively for cryogenic temperature tests. SiO_2 nanoparticle addition into the epoxy matrix has provided increment by 24.8% for the absorbed energy. Besides, peak contact forces for 4%wt SiO_2 nanoparticle reinforced and neat composites, have been obtained an average of 6741 N and an average value of 6257 N respectively. Small energy absorptions were showed under cryogenic temperatures.
- It has been considered that toughening mechanisms (deflection of cracks, crack pinning and crack branching) causes enhance absorption capacities with energy dissipation. The toughness mechanisms have been observed in SEM images. The more delamination failures have also been observed on the impacted cross-sections of neat epoxy composites than SiO_2 nanoparticle reinforced specimens.
- This study revealed that cryogenic temperatures influence LVI responses of epoxy based glass fiber composites. The influence of the matrix modification by adding SiO_2 on the low-temperature behavior of composites was also investigated. Experimental results showed that the cryogenic temperatures have a direct and significant impact on the LVI behaviors. Therefore, we report that evaluate service conditions is needed especially in low-temperature applications.

Acknowledgments

This project was supported by the Selcuk University Scientific Research Projects under grant number 18101001.

References

[1] Kagitci, Y.C. and N. Tarakcioglu, The effect of weld line on tensile strength in a polymer composite part. The International Journal of Advanced Manufacturing Technology, 2016. 85(5-8): p. 1125-1135. https://doi.org/10.1007/s00170-015-8007-0 [2] Guermazi, N., et al., Investigations on the fabrication and the characterization of glass/epoxy, carbon/epoxy and hybrid composites used in the reinforcement and the repair of aeronautic structures. Materials & Design (1980-2015), 2014. 56: p. 714-724. https://doi.org/10.1016/j.matdes.2013.11.043

[3] El Moumen, A., et al., Dynamic properties of carbon nanotubes reinforced carbon fibers/epoxy textile composites under low velocity impact. Composites Part B: Engineering, 2017. 125: p. 1-8. https://doi.org/10.1016/j.compositesb.2017.05.065

[4] Bansemir, H. and O. Haider, Fibre composite structures for space applications—recent and future developments. Cryogenics, 1998. 38(1): p. 51-59. https://doi.org/10.1016/S0011-2275(97)00110- 0

[5] Praveen, R., et al., Hybridization of carbon–glass epoxy composites: An approach to achieve low coefficient of thermal expansion at cryogenic temperatures. Cryogenics, 2011. 51(2): p. 95-104. https://doi.org/10.1016/j.cryogenics.2010.12.003

[6] Hartwig, G. and S. Knaak, Fibre-epoxy composites at low temperatures. Cryogenics, 1984. 24(11): p. 639-647. https://doi.org/10.1016/0011-2275(84)90083-3

[7] Kim, M.-G., J.-B. Moon, and C.-G. Kim, Effect of CNT functionalization on crack resistance of a carbon/epoxy composite at a cryogenic temperature. Composites Part A: Applied Science and Manufacturing, 2012. 43(9): p. 1620-1627. https://doi.org/10.1016/j.compositesa.2012.04.001
[8] Timmerman, J.F., et al., Matrix and fiber influences on the cryogenic microcracking of carbon fiber/epoxy composites. Composites Part A: Applied Science and Manufacturing, 2002. 33(3): p. 323-329. https://doi.org/10.1016/S1359-835X(01)00126-9
[9] Salehi-Khojin, A., et al., The role of temperature on impact properties of Kevlar/fiberglass composite laminates. Composites Part B: Engineering, 2006. 37(7-8): p. 593-602. https://doi.org/10.1016/j.compositesb.2006.03.009
[10] Icten, B.M., et al., Low temperature effect on impact response of quasi-isotropic glass/epoxy laminated plates. Composite Structures, 2009. 91(3): p. 318-323. https://doi.org/10.1016/j.compstruct.2009.05.010
[11] Deng, S., L. Ye, and K. Friedrich, Fracture behaviours of epoxy nanocomposites with nano-silica at low and elevated temperatures. Journal of materials science, 2007. 42(8): p. 2766-2774. https://doi.org/10.1007/s10853-006-1420-x
[12] Phonthammachai, N., H. Chia, and C. He, One-Step Synthesis of Oval Shaped Silica/Epoxy Nanocomposite: Process, Formation Mechanism and Properties, in The Delivery of Nanoparticles. 2012, InTech. https://doi.org/10.5772/34800
[13] Sadej-Bajerlain, M., H. Gojzewski, and E. Andrzejewska, Monomer/modified nanosilica systems: photopolymerization kinetics and composite characterization. Polymer, 2011. 52(7): p. 1495-1503. https://doi.org/10.1016/j.polymer.2011.01.058
[14] Demirci, M.T., et al., Fracture toughness (Mode I) characterization of SiO2 nanoparticle filled basalt/epoxy filament wound composite ring with split-disk test method. Composites Part B: Engineering, 2017. 119: p. 114-124. https://doi.org/10.1016/j.compositesb.2017.03.045
[15] Zare, Y., Study of nanoparticles aggregation/agglomeration in polymer particulate nanocomposites by mechanical properties. Composites Part A: Applied Science and Manufacturing, 2016. 84: p. 158-164. https://doi.org/10.1016/j.compositesa.2016.01.020
[16] Kara, M., et al., Impact behavior of carbon fiber/epoxy composite tubes reinforced with multi-walled carbon nanotubes at cryogenic environment. Composites Part B: Engineering, 2018. 145: p. 145-154. https://doi.org/10.1016/j.compositesb.2018.03.027
[17] Kaybal, H.B., et al., Effects of alumina nanoparticles on dynamic impact responses of carbon fiber reinforced epoxy matrix nanocomposites. Engineering Science and Technology, an International Journal, 2018.
[18] Eskizeybek, V., et al., Static and dynamic mechanical responses of CaCO3 nanoparticle modified epoxy/carbon fiber nanocomposites. Composites Part B: Engineering, 2018. 140: p. 223-231. https://doi.org/10.1016/j.compositesb.2017.12.013
[19] Üstün, T., et al., Evaluating the effectiveness of nanofillers in filament wound carbon/epoxy multiscale composite pipes. Composites Part B: Engineering, 2016. 96: p. 1-6. https://doi.org/10.1016/j.compositesb.2016.04.031
[20] Ulus, H., et al., Low-velocity impact behavior of carbon fiber/epoxy multiscale hybrid nanocomposites reinforced with multiwalled carbon nanotubes and boron nitride nanoplates. Journal of Composite Materials, 2016. 50(6): p. 761-770. https://doi.org/10.1177/0021998315580835
[21] Ulus, H., Ö.S. Şahin, and A. Avcı, Enhancement of flexural and shear properties of carbon fiber/epoxy hybrid nanocomposites by boron nitride nano particles and carbon nano tube modification. Fibers and Polymers, 2015. 16(12): p. 2627-2635. https://doi.org/10.1007/s12221-015-5603-4

[22]Chen, Z.-K., et al., Reinforcement of epoxy resins with multi-walled carbon nanotubes for enhancing cryogenic mechanical properties. Polymer, 2009. 50(19): p. 4753-4759. https://doi.org/10.1016/j.polymer.2009.08.001

[23]Yang, G., S.-Y. Fu, and J.-P. Yang, Preparation and mechanical properties of modified epoxy resins with flexible diamines. Polymer, 2007. 48(1): p. 302-310. https://doi.org/10.1016/j.polymer.2006.11.031

[24]Yang, J.-P., et al., Simultaneous improvements in the cryogenic tensile strength, ductility and impact strength of epoxy resins by a hyperbranched polymer. Polymer, 2008. 49(13-14): p. 3168-3175. https://doi.org/10.1016/j.polymer.2008.05.008

[25]Demirci, M.T., et al., Fracture toughness (Mode I) characterization of SiO 2 nanoparticle filled basalt/epoxy filament wound composite ring with split-disk test method. Composites Part B: Engineering, 2017. 119: p. 114-124. https://doi.org/10.1016/j.compositesb.2017.03.045

[26]Eskizeybek, V., A. Avci, and A. Gülce, The Mode I interlaminar fracture toughness of chemically carbon nanotube grafted glass fabric/epoxy multi-scale composite structures. Composites Part A: Applied Science and Manufacturing, 2014. 63: p. 94-102. https://doi.org/10.1016/j.compositesa.2014.04.013