

IMPACT PROPERTIES OF GRAPHENE MODIFIED POLYMERS AND COMPRESSION AFTER IMPACT (CAI) PROPERTIES OF GRAPHENE MODIFIED CFRPs

C. Kostagiannakopoulou, S. Tsantzalis, G. Sotiriadis, V. Kostopoulos
Department of Mechanical Engineering and Aeronautics, University of Patras,
University Campus, GR-26504 Rio Patras, Greece
Email: kostagia@mech.upatras.gr, Web Page: <http://aml.mech.upatras.gr>

Keywords: Graphene, CFRP, Impact resistance, Damage tolerance

Abstract

The aim of the present study is to investigate the influence of Graphene Nano-Platelets (GNPs) on the impact behavior of nano-reinforced polymers and carbon fiber reinforced polymer (CFRP) laminates. The incorporation of GNPs into the epoxy B-stage system was succeeded by using a three-roll mill technique. The prepared mixtures were used for the impregnation of unidirectional fabrics with a purpose of producing nano-modified pre-impregnated fabrics according to the manual prepregging technique. For the production of composite panels a combination of compression molding method with vacuum bag technique was used in a curing and pressure profile according to manufacturer. Finally, Charpy, impact and Compression after Impact (CAI) tests were performed in order to study the impact properties of developed graphene based nanocomposites. Charpy tests reveal that the introduction of GNPs into the polymer improved 50% the impact resistance of the reference material. However, the addition of GNPs into the CFRP laminates at a level of 0.5% wt. did not cause any further improvement in the reference material.

1. Introduction

Fiber reinforced polymers (FRP) are increasingly in demand as structural materials in the aero-space, automotive and transportation industries due to their higher specific strength and stiffness properties compared with conventional materials such as aluminium. These characteristics are the key in designing sustainable transportation systems with composite material systems as constituents. However, the poor out-of-plane performance of composites has acted as a hindrance for extending their use in a variety of application and widely adopting them in the design process. The key properties for enabling such use of composite materials are the fracture toughness and damage tolerance of CFRPs and further on the possibility to tailor multifunctional properties.

A considerable amount of research has been conducted regarding the improvement of interlaminar fracture toughness fracture toughness and damage tolerance of CFRPs. In the last decade, the incorporation of nanoparticles into the epoxy matrices offers new possibilities toward this direction. The nano-phase controls the fracture behavior of the composites, by introducing additional energy dissipation mechanisms during fracture. Specifically, the addition of nano-inclusions contributes to the toughening of polymer matrix and to the improvement of fiber-matrix adhesion both of which lead to the enhancement of interlaminar fracture toughness [1-5].

The last few years, derivatives of graphite have been under investigation toward this direction. The most widely used graphene nano-species (GNSs) are graphene nanoplatelets (GNPs). According to

literature, GNSs seems to be very promising fillers for the production of nano-reinforced polymers with enhanced fracture and impact properties [6-8].

This study focuses on the development of graphene based nano-reinforced polymers and CFRPs with improved damage tolerance properties.

2. Experimental Section

2.1 Materials

For the purposes of the present study, a four component epoxy B-stage system was supplied by Huntsman Advanced Materials, Switzerland. This system contains the low-viscosity epoxy resin Araldite LY1556, the hardener paste Aradur 1571, the accelerator paste 1573 and the polyamine hardener Aradur XB 3403. The few-layered Graphene NanoPlatelets (GNPs) were provided by Cheap Tubes Inc., USA. GNPs are consisted of ~4 graphene layers with an average thickness of 4 nm and a typical diameter of 1-2 microns while their specific surface area is about 750m²/g. The reinforcement phase, comes from TORAYCA, Japan, and it is a 200 g/m² UD non-crimp fabric, 300 mm wide T700SC, having a 75 dtex PET binding yarn on both sides.

2.2 Preparation of nano-reinforced polymers

Nano-modified suspension was produced using 3 roll-mill, also known as calender. The sequential gap settings were 120, 40, 13, 5 μ m respectively, and five cycles of milling were repeated for each gap setting in order to process the graphene based blends. The speed of the apron roll was maintained at 270 rpm. The matrix system was activated by the addition of the other three parts of the B-stage system in the graphene based suspension. After that the mixture was placed in a vacuum chamber to avoid air inclusion. The it was poured in silicon rubber moulds and cured, according to manufacturer recommended curing cycle, 120°C for 2h and 6 bars pressure. Following the aforementioned process, nano-reinforced polymers including 0.5%wt. GNPs were produced while neat epoxy samples were also manufactured for reference.

2.3 Preparation of nano-modified CFRPs

The uncured nano-modified system was also used for the impregnation of unidirectional fabrics with a view to produce prepregs. The fabric impregnation was carried out manually, at room temperature, while the B-staging was succeeded by keeping the prepreg for 48h at 20°C and 55% to 65% R.H. Next, they were placed in the freezer at -18°C. Then, quasi-isotropic carbon fiber reinforced plastics (CFRPs) were manufactured by using 16 plies of the aforementioned B-stage material. Their curing was carried out combining the compression molding method with the vacuum bag technique in appropriate temperature and pressure profile, (120°C for 2h and 6 bars pressure). Reference composites plates were also manufactured using the same procedure.

2.4 Testing Campaign

Charpy impact tests were performed on a universal testing machine (Karl Frank GMBH), Figure 1a, according to ASTM D 6110 standard by using V-notched samples. This test method is used to determine the resistance of plastics to breakage by flexural shock. Specifically a notched specimen is supported as a horizontal simple beam, Figure 2b, and is broken by a single swing of the pendulum with the impact line midway between the supports and directly opposite the notch. The notch produces a stress concentration which promotes a brittle, rather than a ductile, fracture. All the reported values were calculated as averages over five specimens for each composition.



Figure 1: a) Universal testing machine Karl Frank GMBH, b) Presentation of the supported V-notched specimen.

Low velocity impact and CAI tests were performed according to Airbus Standard: AITM 1-0010. Figure 2 presents the testing machine which was used for the performance of the low velocity tests. A manual operated drop tower was utilized for the low velocity impact tests. Equipped with bound and rebound velocity sensor and a force transducer at the tip of the impactor, it can deliver impacts up to 100 J with velocities up to 5 m/s. Four toggle clamps hold the specimen against the support during the test as presented in the figure. The dimensions of the subjected samples were: 150x100x3mm. Three samples of each material configuration were tested and the applied impact energy was 35J. Furthermore, ultrasonic inspection (C-Scan) was used in order to observe the damage of the impacted specimens.



Figure 2: Images of the testing machine used for the performance of the low velocity tests.

Subsequently, the samples were subjected to compression after impact (CAI) tests according to the aforementioned standard. A purpose built anti-buckling jig was employed and all test were performed at an Instron 8872 \pm 250kN electromechanical universal testing machine, as presented in Figure 3.

C. Kostagiannakopoulou, S. Tsantzalis, G. Sotiriadis and V. Kostopoulos



Figure 3: Depiction of a snapshot during the CAI test.

3. Results and Discussion

3.1 Charpy Tests

The next figure presents the results obtained from Charpy Tests which are reported in terms of energy absorbed per unit of specimen area. As it can be seen from the bar diagram, the introduction of GNPs improved the impact properties of the reference material. Particularly, the addition of GNPs into the polymer caused a 50% enhancement in impact resistance of final products. Considering the above, it is concluded that the use of 4-layered GNPs as filler in content of 0.5%wt. into the specific polymer system could act positively for the toughening of the developed materials. This is due to the fact that the addition of nano-fillers into the polymer introduces additional toughening /energy dissipation mechanisms during the crack propagation. According to literature [6], crack tip bifurcation upon its meeting with the nano-particles, separation between the graphitic layers, shear failure of the matrix (due to the difference in height observed on fracture surfaces) and pull out of GNPs are the basic failure mechanisms that have been observed in the case of graphene based polymers.

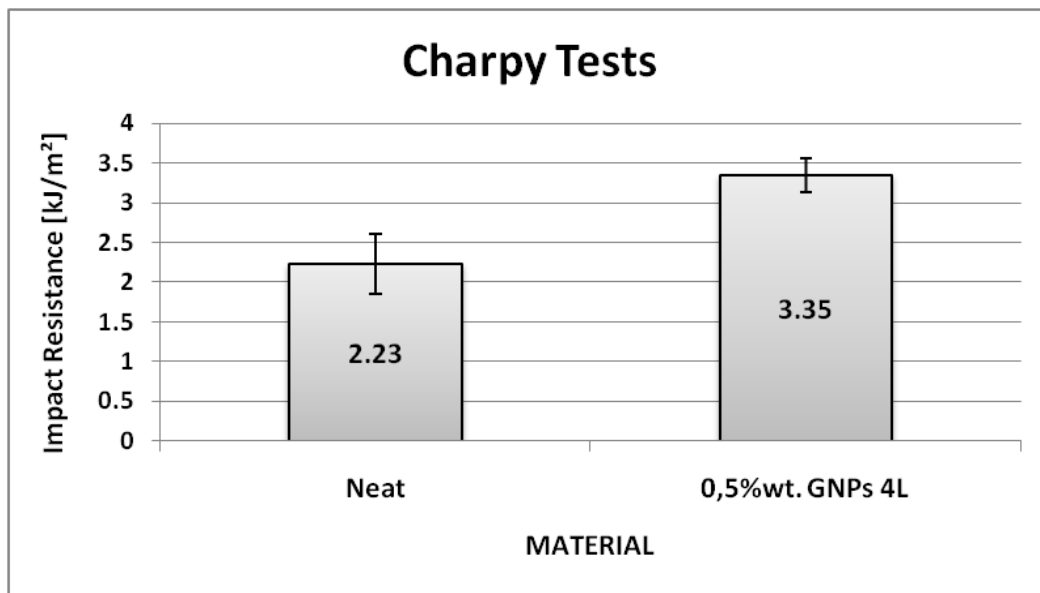


Figure 4: Charpy Impact resistance of produced polymers

3.2 Impact and CAI tests

Firstly, ultrasonic testing by utilizing the C-scan technique was performed in order to assess the quality of produced samples before the impact and identify the delaminations in the impacted samples. Figures 5 and 6 present the C-Scan images of samples before and after impact tests for each material configuration. The color gradient that represents the amplitude of the reflected ultrasonic signal is depicted on the right side of both figures.

In particular, Figure 5 corresponds to the neat material while Figure 6 presents the C-Scan results for doped material. It was observed that samples before impact were of similar quality in both configurations. In addition, no significant flaws were found and the homogeneity of the samples was considered satisfactory. In the case of impacted samples, the developed delamination damage is depicted for each specimen. It can be noted that no major differences are evident between the developed materials. Specifically, the incorporation of GNPs into the composites did not lead to an obvious reduction of the delamination damage area.

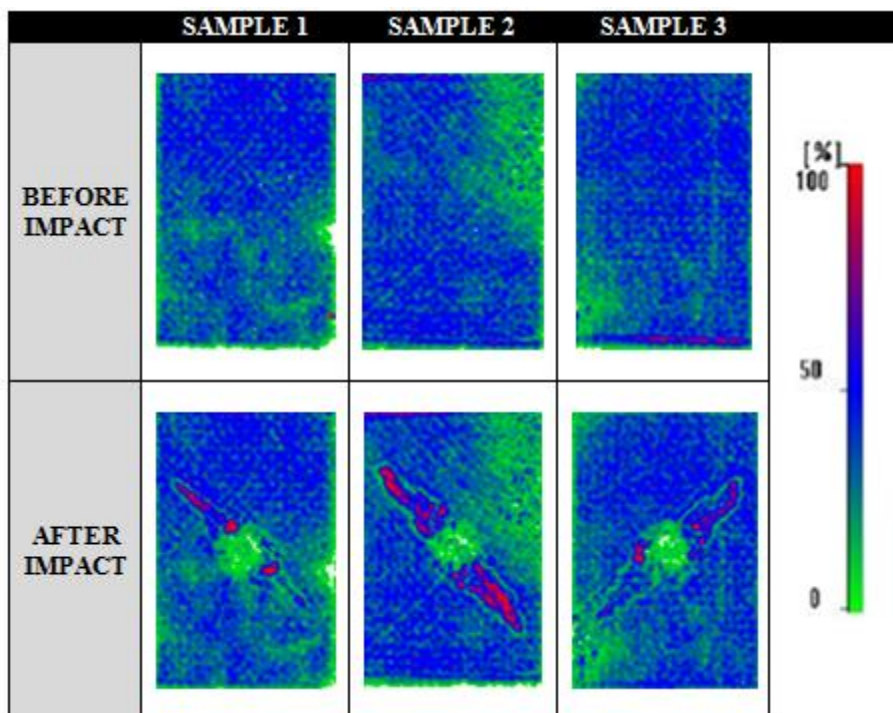


Figure 5: Presentation of C- Scan images of subjected samples before and after impact for the reference material.

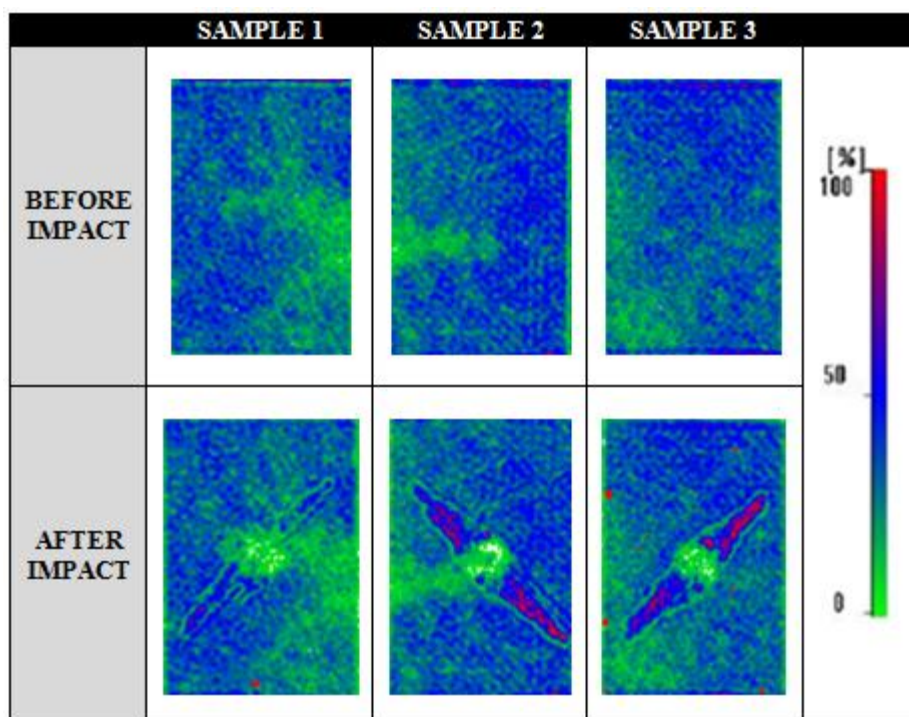


Figure 6: Presentation of C- Scan images of subjected samples before and after impact for the graphene based material.

Excerpt from ISBN 978-3-00-053387-7

Afterwards, the residual compression after impact (CAI) strength of both doped and un-doped specimens were calculated according to the aforementioned standard. It is evident from the following figure that the inclusion of the GNPs did not enhance the CAI strength of the developed materials. Specifically, taking into consideration presented standard deviation for each material system, the graphene based materials did not show any significant improvement in CAI strength. These results are in accordance with the observations for the developed damage in the above C-Scan images.

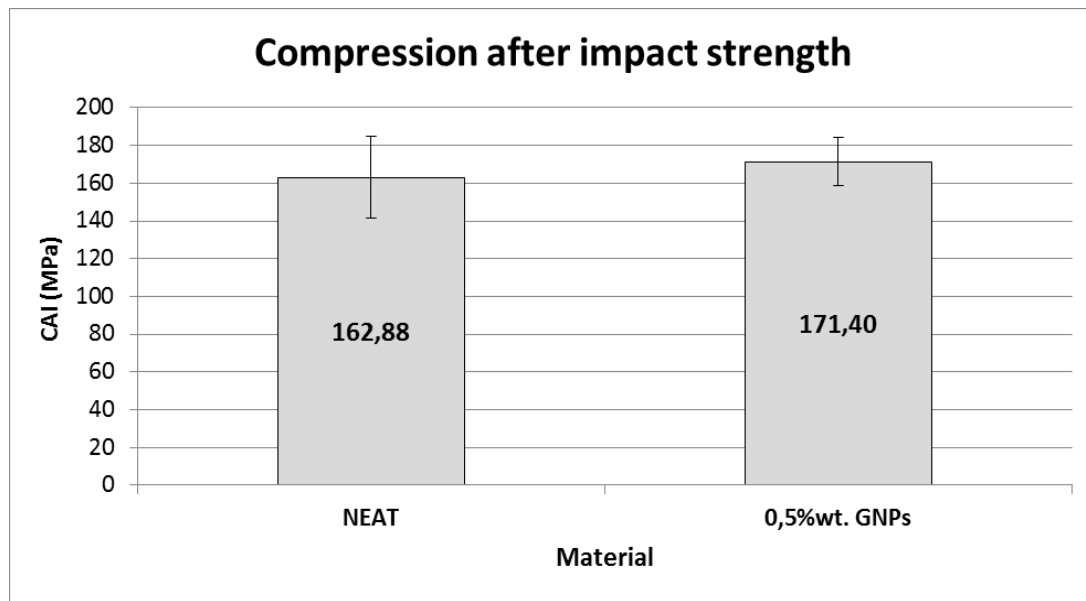


Figure 7: Compression after impact strength of produced CFRP laminas.

4. Conclusions

Charpy tests reveal that the introduction of GNPs into the polymer improved 50% the impact resistance of the reference material. In addition, the inclusion of GNPs into the composites did not cause any significant difference in developed delamination damage area. Simultaneously, the graphene based nano-reinforced polymers did not show any significant enhancement in CAI strength in comparison with the reference material.

Acknowledgements

The authors gratefully acknowledge the support of the present work by the European Union (European Social Fund - ESF) and the Greek national funds executed by the Greek Secretariat for Research & Technology within the frame of bilateral cooperation between Greece and Germany under the activity with the acronym GRACE.

References

- [1] F.H. Gojny, M.H.G. Wichmann, U. Köpke, B. Fiedler and K. Schulte. Carbon nanotube reinforced epoxy composites enhanced stiffness and fracture toughness at low nanotube content. Developments in carbon nanotube and nanofibre reinforced polymers. *Composites Science and Technology*, 64: 2363-2371, 2004.
- [2] Z. Fan, H.M. Santare and G.S. Advani. Interlaminar shear strength of glass fiber reinforced epoxy composites enhanced with multi-walled carbon nanotubes. *Composites part A: Applied Science and Manufacturing*, 39:540-554, 2008.
- [3] L. K. Kepple, P. G. Sanborn, A. P. Lacasse, M. K. Gruenberg and J. W. Ready. Improved fracture toughness of carbon fiber composite functionalized with multi walled carbon nanotubes. *Carbon*, 46:2026-2033, 2008.
- [4] G. M. Kim, S. J. Hong, G. S. Kang and G. C. Kim. Enhancement of the crack growth resistance of a carbon/epoxy composite by adding multi-walled carbon nanotubes at a cryogenic temperature. *Composites part A: Applied Science and Manufacturing*, 39:647-654, (2008).
- [5] V. Kostopoulos, A. Baltopoulos, P. Karapappas, A. Vavouliotis and A. Paipetis. Impact and after-impact properties of carbon fibre reinforced composites enhanced with multi-wall carbon nanotubes. *Composites Science and Technology*, 70: 553-563, 2010
- [6] S. Chandrasekaran, N.Sato, F. Tölle, R. Mülhaupt, B. Fiedler and K. Schulte: Fracture toughness and failure mechanism of graphene based epoxy composites. *Composites Science and Technology*, 97: 90-99, 2014.
- [7] E. Mannov, H. Schmutzler, S. Chandrasekaran, C. Viets, S. Buschhorn, F. Tölle, R. Mülhaupt and K. Schulte. Improvement of compressive strength after impact in fibre reinforced polymer composites by matrix modification with thermally reduced graphene oxide. *Composites Science and Technology*, 87:39-41, 2013.
- [8] C. Kostagiannakopoulou, T.H. Loutas, G. Sotiriadis, A. Markou and V. Kostopoulos. On the interlaminar fracture toughness of carbon fiber composites enhanced with graphene nano-species. *Composites Science and Technology*, 118:217-225, 2015.