

# Characterization of Mechanical Properties of Graphene-Modified Epoxy Resin for Pipeline Repair

S. N. A. Azraai, K. S. Lim, N. Yahaya, N. M. Noor

**Abstract**—This experimental study consists of a characterization of epoxy grout where an amount of 2% of graphene nanoplatelets particles were added to commercial epoxy resin to evaluate their behavior regarding neat epoxy resin. Compressive tests, tensile tests and flexural tests were conducted to study the effect of graphene nanoplatelets on neat epoxy resin. By comparing graphene-based and neat epoxy grout, there is no significant increase of strength due to weak interface in the graphene nanoplatelets/epoxy composites. From this experiment, the tension and flexural strength of graphene-based epoxy grouts is slightly lower than ones of neat epoxy grout. Nevertheless, the addition of graphene has produced more consistent results according to a smaller standard deviation of strength. Furthermore, the graphene has also improved the ductility of the grout, hence reducing its brittle behaviour. This shows that the performance of graphene-based grout is reliably predictable and able to minimise sudden rupture. This is important since repair design of damaged pipeline is of deterministic nature.

**Keywords**—Composite, epoxy resin, graphene nanoplatelets.

## I. INTRODUCTION

THE oil and gas industry uses steel pipelines as a basic element to transport oil and natural gas. These pipelines are subjected to deterioration due to several factors, including third party damage, material and construction defects, natural forces and corrosion [1], [2]. The deterioration of steel pipelines is a common and serious problem scenario experienced by the industry and may lead to reduced life span or a loss of structural integrity. In order to extend the durability of these pipelines, methods to repair the damages have been developed. Recently, polymeric composite repaired pipelines have been proven to be a viable pipeline technique as an alternative repair system. It has been shown that it can perform sufficiently under different environments and industrial projects [3].

In repairing damaged pipes, epoxy resins are widely used as matrices of composites due to their unique characteristics such as high stiffness, high adhesion strength, and low shrinkage in cure [4], [5]. Epoxy grouts are usually used as infill material to ensure a smooth bed for the composite layer. More

Siti Nur Afifah Azraai and Nordin Yahaya are with Faculty of Civil Engineering, Universiti Teknologi Malaysia, UTM Skudai, Johor 81310, Malaysia (e-mail: snaffah2@live.utm.my, nordiny@utm.my).

Kar Sing, Lim is with Faculty of Civil Engineering and Earth Resources, Universiti Malaysia Pahang, Lebuhraya Tun Razak, Gambang, Kuantan, Pahang 26300, Malaysia and Faculty of Civil Engineering, Universiti Teknologi Malaysia, UTM Skudai, Johor 81310, Malaysia. (e-mail: limkarsing@ump.edu.my).

Norhazilan Md Noor is with Faculty of Civil Engineering, Universiti Teknologi Malaysia, UTM Skudai, Johor 81310, Malaysia. (phone: +607-5538704; e-mail: norhazilan@utm.my).

importantly, the infill grout fills the damaged profile caused by corrosion and provides a continuous support to minimise the outward distortion. Epoxy grouts play a key role of transferring the load from pipe to the composite repair and to increase the load resistance of the structure. This means that when the infill material fails to transfer the load, the attached fibres fail to reinforce the structure [4]. Recently, there is tendency in reducing the usage of composite wrapping layer due to several reasons. These reasons include composite wrapping layer being more expensive as compared to infill material. In addition, some damaged pipes are located in congested areas such as piping on offshore platforms, piping of boiler tank and underground pipelines that have limited working area for the wrapping process. This makes the replacement of the damaged pipes the only possible solution to maintain its service life. Therefore, researchers are looking for potential infill material to gradually reduce the usage of composite wrapping layer, hence the thickness. Ultimately, it is hoped that one day the repair can be done without composite wrapping. One of the possible ways of achieving this goal is by increasing the contribution and performance of infill material as part of the repair system.

The properties of the infill material are significant parameters which are required in order to predict the behavior of a repair system for an optimum design. High performance infill material may increase the repair efficiency and serve as second protection layer if failure of composite layer occurs. Numerous research works have been carried out concerning the properties enhancement of infill material. The enhancement of mechanical properties has been done through reinforcing fillers such as carbon nanotube, nanofibers, various particles, and so on. Owing to their properties, carbon nanotubes are considered ideal reinforcing agents for polymers and they have been widely used to enhance the mechanical, thermal and electrical properties of epoxy polymers [6]–[8]. The recent discovery of graphene nanoplatelets for use as nanofillers is being studied but their effect on the mechanical properties is not yet clear. For this reason, it is essential to characterize the mechanical properties of graphene-based grouts to determine their efficiency as infill materials in the repair. Hence, this paper has taken the initial step to investigate the mechanical properties of graphene-based epoxy grout to be used as infill material in composite repair system of pipeline.

## II. EXPERIMENTAL WORK

### A. Materials

The epoxy resin used in this study is a commercially

available three-part pourable grout based on a combination of modified epoxy resins, hardener and fine silica sand. This epoxy resin is the most commonly used resin for grouting and filling in construction application. The resin used for this experiment had a tensile strength of 14 N/mm<sup>2</sup>, a compressive strength of 100 N/mm<sup>2</sup> and a flexural strength of 20 N/mm<sup>2</sup> with mixing ratio of 2:1:12 parts by weight recommended by manufacturers' data sheet.

The graphene nanoplatelets were selected as a filler material to improve the various properties of the epoxy resin. These graphene nanoplatelets are unique nanoplatelets that have an average thickness of approximately 0.68-3.41 nm and particle diameter is 1-4 μm with >99.5 wt% carbon content.

### B. Sample Preparations

The preparation of graphene-based epoxy grouts was carried out as per manufacturer's guideline. First, epoxy resin, hardener and silica filler were weighted based on ratio as recommended in manufacturer's datasheet. Graphene-based epoxy grouts were prepared by dispersing specified weight percentage of graphene nanoplatelets (2%) in hardener and thoroughly mixed using high speed electrical mixer for 15-20 min to get a homogeneous suspension. The next step was the addition of epoxy resin to the mixture and a continuous mixing process until a smooth consistency paste is obtained. After homogeneously mixing graphene suspension with the resin, silica filler was added and all parts were mixed until a homogeneous grout was produced. When the mixing of epoxy resin with the graphene nanoplatelets and hardener was completed, the resin mixture was poured into the designated molds and cured at room temperature for one day. Fig. 1 shows the mixing process of graphene-based epoxy grouts.

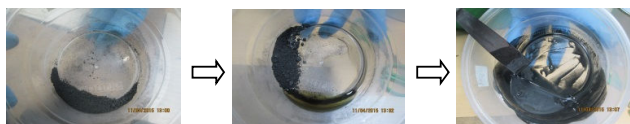


Fig. 1 Mixing process of graphene-based epoxy grout

## III. CHARACTERIZATION

### A. Compressive Tests

All the tests were carried out using a 25 kN universal testing machine (Instron). The compressive tests were performed in accordance to ASTM: D695, using five specimens with dimension of 12.7 mm x 12.7 mm x 50.8 mm for each specimen. The tests were performed at a constant cross-speed of 1.3 mm/min. The specimens were tested at room temperature. The compressive strength results were obtained by using (1):

$$C.S = \frac{P}{b.d} \quad (1)$$

where: C. S= compressive strength (N/mm<sup>2</sup>), P= maximum load (N), b = width of specimen (mm), d = thickness of specimen (mm).



Fig. 2 Compressive test

### B. Tensile Tests

To conduct the tensile tests, the specimens were made according to ASTM: D638, using five specimens with dimensions of 13.0 mm x 3.2 mm. Specimens were pulled apart at crosshead speed of 5 mm/min and carried out at room temperature. Tensile strength and tensile modulus were achieved using the tensile curves. The area under the tensile curves was also determined as the material toughness.



Fig. 3 Tensile test

### C. Flexural Tests

Five specimens with dimensions of 127 mm x 12.7 mm x 3.2 mm were used for the flexural tests, according to the ASTM D790 recommendations. The mechanical properties of maximum bending strength, bending yield stress and bending elastic modulus were evaluated by flexural tests. These tests were performed at constant cross-speed of 1.365 mm/min, at room temperature, using an appropriate device for flexural test. For a test sample, the bending strength ( $\sigma_f$ ) and modulus ( $E_B$ ) are according to (2) and (3), respectively.

$$\sigma_f = \frac{3.P.L}{2.b.d^2} \quad (2)$$

$$E_B = \frac{m.L^3}{4.b.d^3} \quad (3)$$

where:  $\sigma_f$  = flexural strength (N/mm<sup>2</sup>), P= load at a given point on the load-deflection curve (N), L= support span (mm), b= width of beam tested (mm), d= depth of beam tested (mm), m = the slope of the tangent.



Fig. 4 Flexural test

#### IV. RESULTS AND DISCUSSION

##### A. Compressive Properties

Table I shows the summary of compressive properties of graphene-based and neat epoxy grouts. The compressive strength for the graphene-based is nearly 90 MPa which is comparable to ultra-high strength concrete (80MPa) and compressive modulus is found to be 14 GPa. Also, it can be seen from the table that compressive strength for the neat epoxy grout is approximately 87 MPa.

Typical stress-strain curves for graphene-based and neat epoxy grouts are depicted in Fig. 5. Only one curve for each case is presented to ease the comparison of the results. As can be seen, the behavior of graphene-based grouts are found to be different compared to neat epoxy grouts. The graphene-based exhibited a linear response in the initial loading stage, followed by nonlinear behavior up to the yielding point followed by a strain softening. On the other hand, the behavior of neat epoxy is different to some extent. It appears that the stress-strain compression neat epoxy plummets after the yielding point. The yield stresses of both grouts are the maximum strengths which are indicated through the decline of stress beyond the yield stress.

Fig. 6 demonstrates the failure patterns of the tested grouts. Under compression, graphene-based grout exhibit noticeable deformation after the initial elastic behavior. Initial cracks were observed at top and bottom part of the sample where the maximum stress occurred. It was then followed by gradual reduction in stress prior to failure. The neat epoxy grout displayed split inclined crack without any deformation prior to yield stress. The neat epoxy grout also exhibits sudden rupture as compared to graphene-based grout.

TABLE I  
SUMMARY OF THE COMPRESSIVE PROPERTIES

Grout	Compressive strength (MPa)	Compressive Modulus (GPa)
Graphene-based	88.41 ± 1.58	14.10 ± 1.54
Neat epoxy	87.52 ± 1.95	18.93 ± 4.78

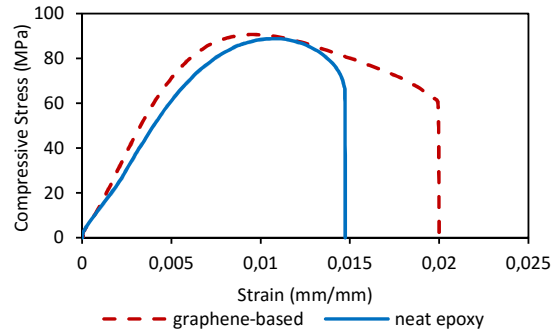


Fig. 5 Typical stress-strain curves for compressive stress

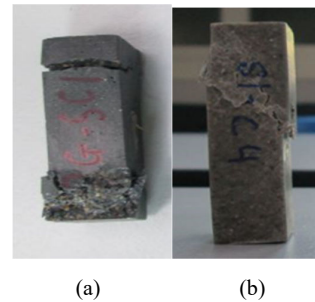


Fig. 6 Failure patterns of grouts under compression 0(a) graphene-based grout (b) neat epoxy grout

##### B. Tensile Properties

Table II provides a summary of the strength and modulus of the investigated grouts in tension. It can be seen from the table that the tensile strength of the investigated grouts is between 15 and 19 MPa and tensile modulus for both tested grouts are approximately 18 GPa. Neat epoxy grout exhibited the highest tensile strength, which is 19MPa.

Typical stress-strain curves for each group of grouts obtained under tensile loading are illustrated in Fig. 7. Two distinct stress-strain behavior are observed. All grouts exhibit much lower ductility under tension compared with their compression response. In addition, the ultimate strength in tension is much lower than those exhibited under compression. It can be seen that the graphene-based grout shows relatively prolonged ductile deformation under tensile load compared to neat epoxy grout. All the grouts failed due to splitting, which are perpendicular to the length. The failure of tensile specimens occurred without noticeable deformation. Fig. 8 shows the failure pattern of the specimens under tension.

TABLE II  
SUMMARY OF THE TENSILE PROPERTIES

Grout	Tensile strength (MPa)	Tensile Modulus (GPa)
Graphene-based	15.18 ± 0.32	17.35 ± 1.67
Neat epoxy	18.82 ± 4.62	18.82 ± 4.42

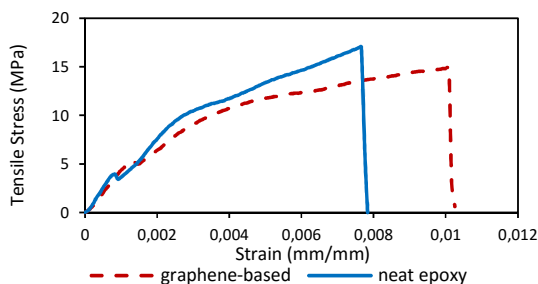


Fig. 7 Typical stress-strain behavior of tensile specimens

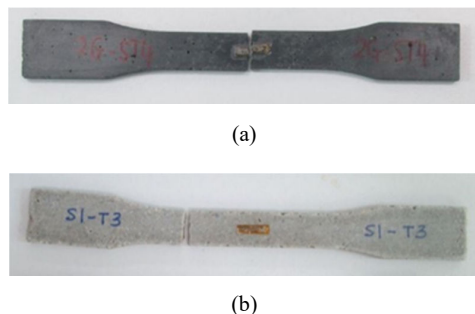


Fig. 8 Failure patterns of grouts under tension (a) graphene-based grout (b) neat epoxy grout

### C. Flexural Properties

Table III presents the flexural strength values for both tested grouts. As shown in Table III, graphene-based grout has the higher flexural strength compared to neat epoxy grout. Fig. 9 shows a typical comparison of the load-deflection behavior of the grouts in flexure. All the grouts show linear elastic load-deflection behavior prior to failure. The load-deflection behavior of neat epoxy shows lower strength as well as lower deflection than graphene-based grout. The typical failure patterns of the flexural specimens of the grouts are shown in Fig. 10. All tested grouts fail in a brittle manner. The crack formations are almost vertical and perpendicular to the length for both specimens.

TABLE III  
SUMMARY OF THE FLEXURAL PROPERTIES

Grout	Flexural strength (MPa)	Flexural Modulus (GPa)
Graphene-based	32.94 ± 2.12	13.60 ± 0.99
Neat epoxy	34.57 ± 2.39	11.87 ± 5.24

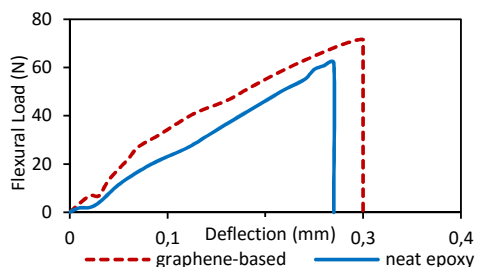


Fig. 9 Typical flexural load-deflection behavior

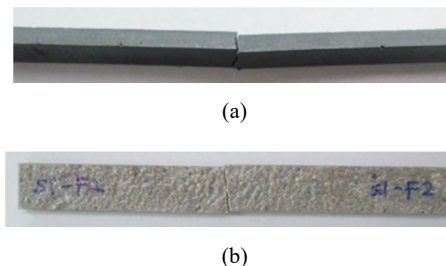


Fig. 10 Failure patterns of grouts under flexural (a) graphene-based grout (b) neat epoxy grout

### V. CONCLUSION

Mendis [9] suggested the typical properties of epoxy grouts used for repair and rehabilitate damaged structures. Compressive and tensile strength greater than 40MPa and 14MPa was reported suitable for repairing concrete crack. The author also suggests that for structural rehabilitation, compressive and tensile strength is suggested to be more than 80MPa and 28MPa, respectively. In composite repair of externally corroded pipeline, the infill material serves as medium to transfer the stresses on internal surface of pipeline generated by internal pressure (without sharing the load) requires high compressive strength. Therefore, the tested can serve in high compressive condition and has the potential in reducing the wrapping thickness.

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### REFERENCES

- [1] M.N. Noor, N. Yahaya, and S.R. Othman, "The Effect of Extreme Corrosion Defect on Pipeline Remaining Life-time," *Malaysia Journal of Civil Engineering*, vol. 20 (1), 45-577, 2008.
- [2] M. Shamsuddoha, M.M. Islam, T. Aravinhan, A. Manalo, and K.T. Lau, "Characterization of mechanical and thermal properties of epoxy grouts for composite repair of steel pipelines," *Material and Design*, 52, 315-327, 2013a.
- [3] J.M. Duell, J.M. Wilson, and M.R. Kessler, "Analysis of a carbon composite overwrap pipeline repair system," *Int J Press Vess Piping*; 85:782-8B, 2008.
- [4] O. Sindt, "Molecular architecture mechanical behavior relationships in epoxy network," *Polymer*, vol 37(14), pp. 2989-2997, 1996.
- [5] O. Buyukokturk, "Progress on understanding debonding problems in reinforced concrete and steel members strengthened using FRP composite." *Construction and Building Materials*, vol. 18(1), pp.9-19, 2004.
- [6] J.D. Fidelus, "Thermo-mechanical properties of randomly oriented carbon/epoxy nanocomposite," *Composites Part a-Applied Science and Manufacturing*, vol. 36(11), pp.15501561, 2005.
- [7] J.F. Shen, "The reinforcement role of different amino-functionalized multi-walled carbon nanotubes in epoxy nanocomposites," *Composites Science and Technology*, vol. 67(15-16), pp. 3041-3050, 2007.
- [8] E. Ivanov, "Effects of Processing Conditions on Rheological, Thermal, and Electrical Properties of Multiwall Carbon Nanotube/Epoxy Resin Composites," *Journal of Polymer Science Part B-Polymer Physics*, vol. 49(6), pp. 431-442, 2011.
- [9] P. Mendis, "Commercial Applications and Property Requirements for Epoxies in Construction," *SP.ACI Special*, 127-40, 1985.