

Mechanical Properties of Graphene-Modified Epoxy Grout for Pipeline Composite Repair

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Abstract: In general, when the pipeline experiences metal loss on the external surface, epoxy grout has always be used to fill the gap before fibre reinforced composite can be applied to recover the pipeline strength. In this research, the existing commercially available epoxy grout has been strengthened using graphene nanoplatelets (GNPs) at the amount of 0.1wt% to enhance its mechanical properties. Various mechanical tests were conducted on this modified epoxy grout to identify the compression, tensile, flexural and shear properties and were compared to the neat epoxy grout to observe its potential improvement. GNPs were dispersed using a sonication process followed by three-roll milling technique to ensure a uniform and homogeneous dispersion within the epoxy matrix can be well achieved. The experimental results clearly show an improvement in the strength and Young's modulus especially for tensile, flexural and lap shear test by incorporating GNPs as additives. The presence of GNPs has a significant reinforcement effect and has succeeded in increasing the ductility of the grout, thus reducing its brittle behaviour. This gives an indication that the performance of modified epoxy grout is expected to be reliable and capable to minimize sudden rupture of the pipeline due to bursting.

Keywords: Pipeline repair, infill material, epoxy grout, graphene nanoplatelets (GNPs), three-roll mill

1. Introduction

Pipeline subsea or underground are exposed to metal loses in the forms of pitting or general corrosion. As pipeline ages, the corrosion that results in metal loss either internally or externally will continue to progress due to aggressive environments [1]. This can cause serious hazard which may lead to structural failure, loss of life, loss of capital investment, and environmental damage [2]. Therefore, repair and maintenance of these damaged pipelines are critical for the prevention of such accidents.

Nowadays, the selection of FRP composite material is the best solution as it has been proven effective for structural repair and rehabilitation [3-4]. Fibre Reinforced Polymer (FRP) has emerged as an alternative to the conventional pipeline repair practices due to their lightweight, high strength, stiffness, and corrosion resistance [5-7]. Besides that, composite repair has additional advantages of avoiding pipeline operation interruptions and eliminating welding and cutting processes thereby preventing potential hazards [5-6]. Apart from replacing the whole segment, the Fibre-Reinforced Polymer (FRP) composite can be used to minimize the cost of pipeline repair with the capability of recovering its strength effectively. This practice must be

accompanied with epoxy grout that is used to flatten the damaged surface of steel pipe. A composite wrap will then be used to wrap around the repair segment of damaged pipe with adhesive applications on each layer as a bonding agent [8]. Despite many advantages offered, there are some drawbacks associated with FRP repair method. These include composite wrapping layers are significantly more expensive than infill material and if the damaged pipes are located in the congested area, there is difficulty in wrapping process due to the limited working area [9-10].

Through improved innovations and technology, the pipeline industry benefited from the continued development of composite materials. The future trend will likely to focus on optimizing the design of the composite repair system and therefore efforts are undertaken by reducing the layers of composite wrap used in the pipeline composite repair. There are few initiatives to reduce the thickness used in composite wrap, and this initiatives includes either through the invention of new material for wrapper with a minimal thickness yet stronger than the current wrapper or use an existing composite wrapper that has proved to be excellent but enhances the properties of the infill material. Even though there are few efforts in producing new wrapper for repair [11-12], but this initiative has not

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yet materialize as it is still in its early stages and will take some time before it can be adopted in the industry. Therefore, the most realistic initiative that can be taken to optimize the current FRP repair system with the potential to reduce the thickness of wrapper is by improving the strength of infill material. Hence, improving the load-bearing function of infill material apart from its original intended function related to the load-transfer mechanism.

Previously, there were attempts to improve infill material performance so that it can contribute to the overall strength of composite repair [19,13]. This is an important fact since industrial practice treats infill material with limited function; to fill the damaged section and provide a smooth surface for the composite wrap only. However, from an engineering point of view, infill material is important in transferring the load from the pipe to the composite repair and increases the stress resistance of the structure. Thus, if infill material failed to convey the load, the structure would not be reinforced effectively by the composite [14]. This means that the aforementioned goal can be achieved by strengthening the infill material of pipeline composite repair in the first place since grout performance; hypothetically; can influence the effectiveness of pipeline repair system [5,15]. Since infill material is only used in small quantity, the addition of additives into the epoxy grout is limited. Thus, it is crucial to use the additives that can react effectively with the polymer in a very small quantity. Therefore, the use of additives (filler) with nanoparticle size is the best way to improve the properties of infill material in the pipeline composite repair.

In recent years, studies conducted by several researchers have proved that the enhancement of mechanical properties can be done through the incorporation of nanomaterials; such as graphene nanoplatelets, carbon nanotubes and nanoclay; as a filler in the polymer matrix [16–18]. In this study, graphene nanoplatelets was selected as nanofiller due to its superior properties that capable to improve the mechanical, thermal and electrical properties of epoxy polymers [19–20]. It is known as the most suitable reinforcing agents for polymers and they have been widely used in different industrial areas such as area of sensors, energy and multifunctional material [21–23]. These outstanding performance could be attributed to its large specific area and packed carbon atom aligned in the hexagonal structure [24]. The recent discovery of graphene nanoplatelets as nanofiller in epoxy grout is being studied but its effect on the mechanical properties is not very clear yet due to dispersion issue [9,18]. Moreover, several factors should be considered in attaining an ideal improvement of infill material properties that include the ideal nanomaterial dispersion in the epoxy matrix, and the optimum amount of nanomaterial required to successfully enhance material properties. Therefore, the stand-alone material characterization of epoxy grout through mechanical testing is important to determine the possible contribution of graphene nanoplatelets toward the strength improvement of infill material. Hence, this paper will focus on the potential of graphene nanoplatelets in

strengthening the epoxy grout used as infill material in pipeline composite repair system so that the contribution of infill material is not limited to load transfer mechanism only but can be extended as a secondary load bearing component.

2. Experimental Work

2.1 Materials

In this research, commercially available epoxy grout was used with a combination of epoxy resins and hardener, later denoted as neat epoxy grout. Commonly, this epoxy grout was applied for grouting and filling in a construction application. The existing epoxy grout has been modified and referred as modified epoxy grout by incorporating graphene nanoplatelets (GNPs) at the amount of 0.1wt% to enhance its mechanical properties. The GNPs comes with the appearance of black/ grey powder with an average thickness of approximately 0.68–3.41 nm while its particle diameter is 1– 4 μm with >99.5 wt% carbon content.

2.2 Graphene Nanoplatelets Dispersion

A weighted amount of GNPs was prepared at the desired concentrations. First, the GNPs were pre-dispersed in an acetone solution for 45 minutes using Hielscher ultrasound sonicator and were left to evaporate for 24 hours at room temperature. Then, GNPs was mixed with resin until it was homogeneously distributed. The epoxy/GnP mixture was further dispersed using a three-roll mill machine (EXAKT 80E Advanced Technologies GmbH) to achieve homogeneous dispersion as shown in Fig. 1. The epoxy/GnP mixture was poured into the gap between the feed roller and centre roller and transported to the third roller as shown in Fig. 2. The dwell time of graphene suspension on the roll was approximately 1 minute while graphene was dispersed in the resin by enormous shear forces resulting from the rollers turning at a speed ratio of 9:3:1.



Fig. 1 Dispersion using three-roll mill machine.

The calendaring process was repeated for four consecutive times for each batch and the time required for each cycle was approximately 10 minutes. Details of the parameters of three-roll mill process such as the gap size between the roller and the speed (represent the lowest speed) are tabulated in Table 1. Fig. 3 shows the outcome of the dispersion process using three-roll mill machine. A homogeneous and well-dispersed mixture is a product obtained after the calendaring process was completed.

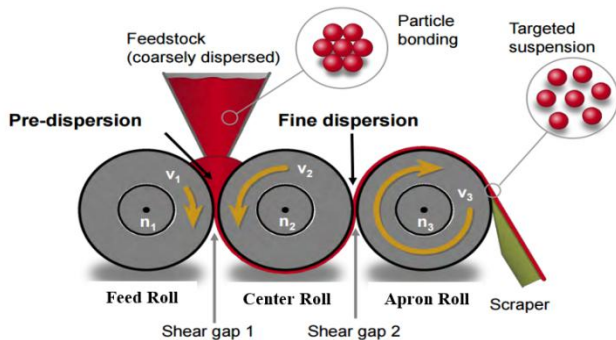


Fig. 2 Three-roll mill process (Source: EXAKT Advanced Technologies GmbH [20]).

Table 1 Details of three-roll mill process.

Pass cycle	1st gap (μm)	2nd gap (μm)	Rotational speed (rpm)
1	15	5	250
2	15	5	250
3	15	5	350
4	15	5	350

2.3 Preparation of Composites

The preparation of graphene-based epoxy grouts was carried out as per manufacturer’s guideline [28]. The GNPs/epoxy nanocomposite was mixed with the hardener at the ratio of 2:1 and thoroughly mixed using an electric hand mixer at a lower speed for one minute to assure the mixture was well blended. The mixtures were cast into the designated mould and were cured at room temperature for 24 hours. The neat epoxy grout was prepared by the same procedure without adding nanomaterial. Prior to testing, all specimens were polished to eliminate any impurities and surface defects. Fig. 4 shows the sample preparation of the modified epoxy grout.

3. Characterization

3.1 Mechanical Analysis

Mechanical tests need to be carried out to determine the properties of modified epoxy grout and to investigate the contribution of graphene as nanofiller. INSTRON 5567 Universal Testing Machine with 25KN of capacity is used to test the specimens until failed as shown in Fig. 5.

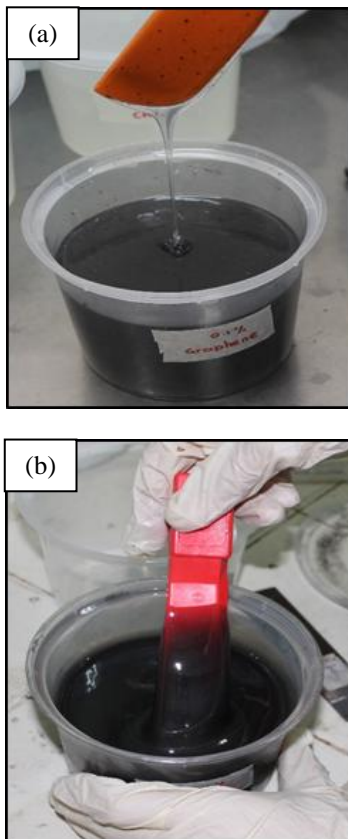


Fig. 3 (a) Before dispersion and (b) After dispersion process



Fig. 5 Universal Testing Machine (INSTRON 5567)

Table 2 Details of mechanical properties test

Tests	Standards	N	Dimensions (mm)	Geometry	Loading rate (mm/min)
Compressive	ASTM: D695	5	12.7 x 12.7 x 50.8	Prismatic	1.3
Tensile	ASTM: D638	5	13.0 x 3.2	Dog bone	5.0
Flexure	ASTM: D790	5	127 x 12.7 x 3.2	Prismatic	1.365
Shear	ASTM: D1002	5	25.4 x 12.7	-	1.27

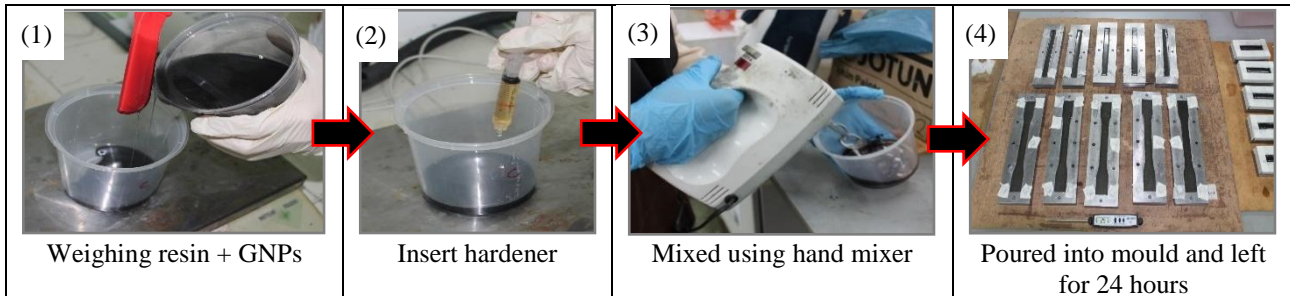


Fig. 4 Sample preparation of modified epoxy grout.

In order to determine Young’s modulus and Poisson’s ratio, strain gauges were mounted on the surface of the compression and tensile specimens while Low Voltage Displacement Transducer (LVDT) was used in the flexural test to determine the deflection of the flexural specimen. Both strain gauge and LVDT were connected to a data logger (TDS-530) to record the strain and LVDT values throughout the testing. The reported test results are an average of five repetitive samples to ensure the consistent and reliable results. The details for tests conducted are given in Table 2. All mechanical tests were performed at room temperature and as per ASTM (American Society for Testing and Materials) standards as shown in Table 2.

4. Results and Discussion

4.1 Compression Properties

Table 3 shows a summary of the compressive test results for the neat and modified epoxy grout. Based on the table, the highest compressive strength and stiffness are found in the modified epoxy grout. The ultimate compressive strength of the neat and modified epoxy grout are recorded at 64.29 MPa and 64.81 MPa, respectively. As can be seen, the inclusion of 0.1wt% graphene does not give any significant improvement on the ultimate compressive strength. However, the stiffness

Table 3 Summary of compressive test results.

Grouts	Compressive Strength (MPa)	Young’s Modulus (GPa)
Neat epoxy	64.29 ± 0.78	2.52 ± 0.24
Modified epoxy	64.81 ± 1.02	2.82 ± 0.26

in modified epoxy grout shows 12% increment from 2.52 GPa to 2.82 GPa, as compared to the neat epoxy grout.

The stress-strain curve under a unidirectional compression load was depicted in Fig. 6. All grouts showed comparable strain value with similar behaviour to one another starting with linear elastic behaviour during the initial loading stage until it reaches ultimate compressive strength, followed by a strain softening and plastic deformation. Based on the graph, both grouts demonstrated ductile behaviour and no sudden failure occurs. Fig. 7 presents the failure patterns of the tested grouts. After the initial elastic behaviour, both grouts exhibit ductile behaviour with visible deformation. The specimen exhibit buckling and initial crack when the maximum stress occurred, and then the stress is gradually reduced prior to failure. This failure pattern can be seen at the top and bottom of the specimens for both grouts.

In narrow confinements and under high pressure, this infill material is expected to experience compressive stress in the radial direction that leads to pipeline failure. Therefore, adequate compressive strength is required to minimize radial deformation by transferring the stress from damage pipe to the composite wrap [27]. As mentioned previously, both tested grouts demonstrated ductile behaviour with visible deformation under compression load. This behaviour of epoxy grout is suitable to be utilized in pressurize pipeline as it capable to minimize sudden rupture of the pipeline due to bursting. According to Duell *et al.* [1], grouts with higher compressive modulus can increase the overall repair performance. Therefore, the modified epoxy grout with higher compressive strength and modulus provide better load-transfer mechanism, thus potentially enhance the

overall repair performance in pipeline composite repair system.

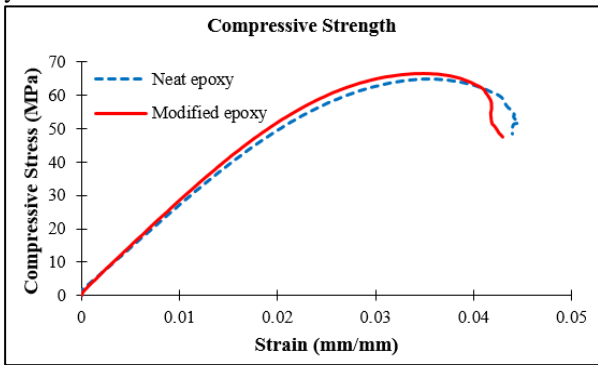


Fig. 6 Stress-strain curve for compression test

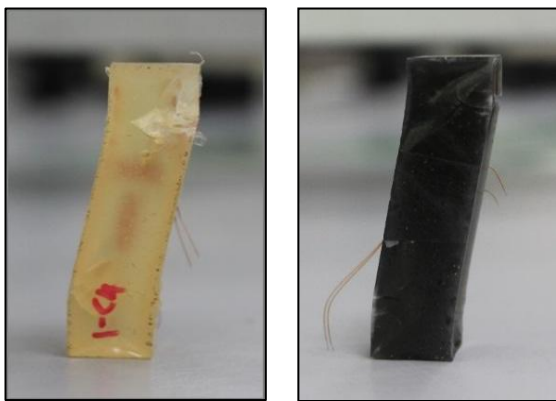


Fig. 7 Compression specimens after failure

4.2 Tensile Properties

Tensile properties of the test are summarized in Table 4 and Figure 8 illustrates the stress-strain curve under tensile loading conditions. Based on the results, the tensile strength of the modified epoxy grout is found to increase with the inclusion of 0.1wt% graphene nanoplatelets. A maximum increment of 24% in the tensile strength of modified epoxy grout is observed with 32.64 MPa as compared to neat epoxy grout with 26.42 MPa. It can also be noticed that graphene nanoplatelets has improved the tensile modulus of the neat epoxy grout. A gain of 14% in the tensile modulus is observed in the modified epoxy grout from 2.21 GPa to 2.52 GPa. As depicted in Fig. 8, the stress-strain curve for all grouts demonstrated comparable behaviour where linear elastic behaviour was observed from the beginning of the testing until the specimens reach ultimate tensile strength up to failure, indicating the brittleness of the grouts.

Table 4 Summary of tensile test results.

Grouts	Tensile Strength (Mpa)	Young's Modulus (GPa)
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Neat epoxy	26.42 ± 2.83	2.21 ± 0.20
Modified epoxy	32.64 ± 2.69	2.52 ± 0.04

Besides that, it shows that the strain value of the neat epoxy grout is slightly lower than the modified epoxy grout. Fig. 9 shows failure pattern for the tensile test. All the specimens failed due to fractures that break the specimen into two part without any noticeable deformation or necking.

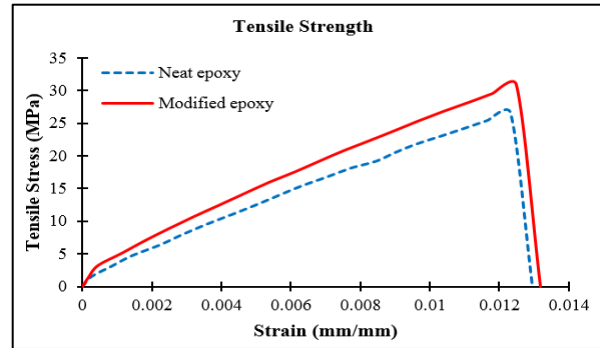


Fig. 8 Stress-strain curve for tensile test

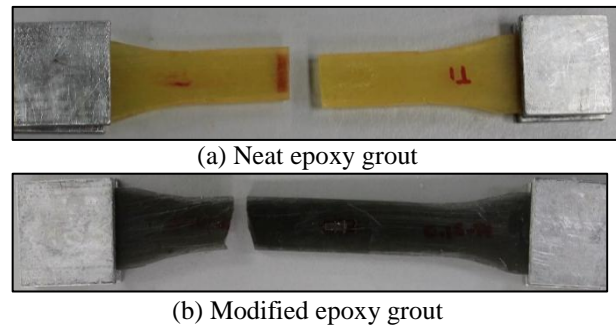


Fig. 9 Tensile specimens after failure

In the pressurized pipe, hoop stress is the most dominant and critical stress in the circumferential direction and will cause the pipe fail in tension mode. Therefore, high performance of grout in terms of tensile strength is indispensable to provide the additional load bearing capacity and thus a better load-sharing mechanism in pipeline repair [28]. According to Mendis [29], Lim *et al.* [30] and Shamsuddoha *et al.* [31], the tensile strength within the range of 19 to 48 MPa has the potential to be used in structural rehabilitation and it has been employed as a benchmark to assess the suitability of epoxy grout for pipeline repair. Hence, the tensile strength of the modified epoxy grout is sufficient for structural repair with a value of 32 MPa. In repairing pipeline with higher operating pressure, the modified epoxy grout may be suitable as it can serve as secondary-layer protection by sharing the stress from the high operational pressure instead of just transferring it from the pipeline to composite wrapping layer. Therefore, as aforementioned, the higher tensile strength provides

better performance thereby improving the overall capacity of the repaired pipe.

4.3 Flexural Properties

Table 5 shows the summary of flexural test results. As shown in the table, modified epoxy grout with the inclusion of 0.1wt% graphene nanoplatelets results in higher flexural strength and stiffness compared to the neat epoxy grout with a difference of 22% and 41% of the increment, respectively.

Table 5 Summary of flexural test results.

Grouts	Flexural Strength (Mpa)	Young's Modulus (GPa)
Neat epoxy	61.01 ± 2.95	1.70 ± 0.11
Modified epoxy	74.34 ± 3.12	2.40 ± 0.29

Fig. 10 presents a comparison of the load-displacement curve for flexural specimens. Both tested grouts show linear elastic behaviour prior to failure. The modified epoxy grout exhibited higher flexural load but slightly lower in deflection and this behaviour is contrary to the neat epoxy grout with lower flexural load but has higher deflection value. Under flexural load, the incorporation of GNPs into the epoxy grout increases the surface area to volume ratio. GNPs has the capability of high endurance to deform during loading, thus enhances more loading ability and increasing the stiffness of the epoxy grout while decreased its deflection. The failure pattern of the neat and modified epoxy grout was presented in Fig. 11. It is observed that all grouts show similar failure pattern under flexural test with relatively prolonged deformation. The specimens were observed from the beginning of the test until the failure occurred and based on the observation, it showed that the crack formation was initiated at the middle of the specimen and forms a visible wedge.

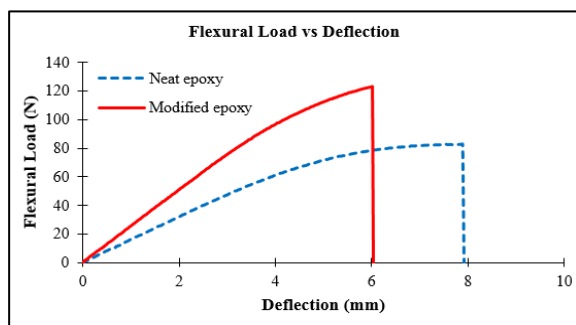


Fig. 10 Load-deflection curve for flexural test

Pipeline may also be vulnerable to failure when subjected to bending forces caused by the nature of design and operational conditions. The maximum bending stress is usually generated at the mid-span location of the pipe which results in flexural deformation

[32]. Under these circumstances, high flexural strength and stiffness are necessary to restrain the bending force effectively. Therefore, the modified epoxy grout may be appropriate to provide additional strength in conditions that require higher bending forces thus preventing the pipe failure.

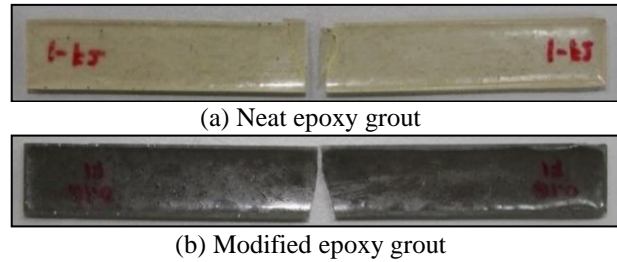


Fig. 11 Flexural specimens after failure

4.4 Lap Shear Properties

Table 6 summarizes the shear strength based on a single lap-joint test. Based on the results, the modified epoxy grout exhibits higher shear strength as compared to the neat epoxy grout with a recorded value 6.47 MPa and 5.83 MPa, respectively. As can be seen, shear strength for the modified epoxy grout has increased up to 11%.

Table 6 Summary of lap-shear test results.

Grouts	Shear Strength (MPa)	Load (kN)
Neat epoxy	5.83 ± 1.53	1.86
Modified epoxy	6.47 ± 0.66	2.06

It shows that the inclusion of graphene nanofiller gives a higher shear bonding compared to grout without nanofiller. In addition, the modified epoxy grout shows the highest load during failure recorded at 2.06 kN while the neat epoxy grout failed at load 1.86 kN. Figure 12 presents the failure pattern of the tested grouts. As depicted in the figure below, both grouts show similar failure pattern as some parts of the matrix remains attached to both surfaces of the steel coupons, indicating cohesive shear failure. This behaviour implies that the bonding between the matrix and the steel coupon is much stronger than the strength of the matrix itself [33].



(a) Neat epoxy grout

(b) Modified epoxy

grout

Fig. 12 Failure pattern of shear test

The issue related to the bonding strength in the composite pipeline repair have been studied by several researchers and they have emphasized that one of the important factors affecting the performance of composite repair system is the bonding strength of damaged pipeline with infill material or composite wrap [30,31]. As stated in ISO/TS 24817 [36] and ASME-PCC2 [37], the requirement for shear strength of the adhesive composite bond to the substrate should at least 4 MPa and 5 MPa. Besides that, both standards also stated that at least 30% of the composite material should remain at the bonded area in which the failure occurred. Although both standards do not specifically mention the minimum required shear strength for the infill material, these standards can be adopted as the requirement for the shear strength of infill material. In this study, both tested grouts are considered suitable as part of the composite repair system as it complies a minimum requirement of shear strength mentioned previously.

5. Conclusion

This study investigated the influence of graphene nanoplatelets (GNPs) on the mechanical properties of epoxy grout. The results obtained had confirmed the improvement in strength of modified epoxy grout by 20% for a tensile and flexural test, while 11% for the shear test. This indicates the significant reinforcement effect given by 0.1wt% GNPs. Apart from that, 10% up to 40% improvement in Young's modulus was also achieved for modified epoxy grout. The increase in modulus indicates considerable load transfer from the matrix to the GNPs fillers when stress is being applied. The addition of GNPs in epoxy grout shows comparable strain reading without significant difference for compression and tensile test except for flexural. In the flexural test, GNPs did not improve the toughness of epoxy grout as it was observed that there is a substantial increase in stiffness and the ultimate strength of modified epoxy grout but also resulted in a decrease in ductility of modified epoxy grout. Notwithstanding the fact that graphene-based materials have shown attractive mechanical properties, this nanomaterial prone to form agglomerates due to its high surface area and strong van der Waals attraction that cause the deterioration of a final nanocomposites properties. Considering that fact, dispersion of GNPs in epoxy matrix using sonication and calendaring processes has successfully contributed to the enhancement of mechanical properties in epoxy grout. If the performance of epoxy grout used as infill material in composite pipeline repair can be improved then it may increase repair efficiency and provide secondary protection to the composite repair. The reduction of wrapping thickness in pipeline repair can be made possible by strengthening the epoxy gout using nanomaterials, hence may reduce the overall cost of repair and time to completion of repair activity.

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