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MECHANICAL PROPERTIES OF CARBON NANOTUBES-MODIFIED EPOXY GROUT FOR PIPELINE REPAIR SYSTEM

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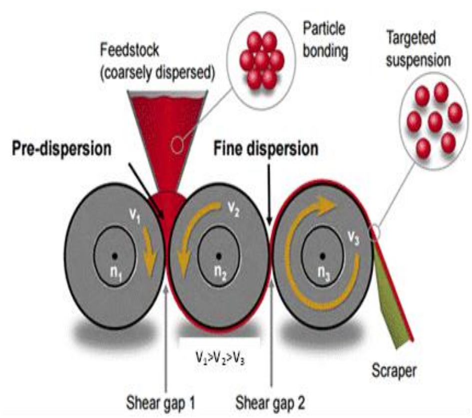
Graphical abstract



Abstract

Epoxy grout properties are theoretically important in predicting the behaviour of the composite pipeline repair system. Usually, it is used as an infill material to fill the gap or irregularity on the surface caused by pipe corrosion and ensures a smooth bed before fibre wrapper can be applied to recover the pipeline strength. In this research, the existing commercially available epoxy resin grout has been strengthened by using Carbon Nanotubes (CNTs) at the amount 0.1% of weight fractional to evaluate their apropos behaviour to the neat epoxy grout. The various mechanical tests were performed on this modified grout to identify its compression, tensile, flexural and lap shear strength. In addition, the dispersion process of CNTs was carried out by using ultrasonication and three-roll mill technique to ensure an optimum enhancement in the properties of the polymer matrix. By comparing the strength, 0.1% of CNTs filler has significantly improved the strength of grout in flexural, tensile and shear bonding but not in compression. In addition, the results also indicate that CNTs filler has increased the modulus of elasticity of the infill material. Therefore, it demonstrates the intrinsic potential of the CNTs in modifying the properties of the epoxy grout.

Keywords: Pipeline repair, epoxy grout, infill material, carbon nanotubes, three-roll mill

Abstrak

Ciri-ciri grout epoksi secara teorinya penting dalam meramal tingkah laku sistem pembaikan saluran paip komposit. Biasanya, ia digunakan sebagai bahan infill untuk mengisi jurang atau ketidakteraturan pada permukaan yang disebabkan oleh kakisan paip dan memastikan tempat tidur yang licin sebelum pembungkus serat boleh digunakan untuk memulihkan kekuatan saluran paip. Dalam kajian ini, resin grout epoksi yang sedia ada secara komersial telah diperkuatkan dengan menggunakan tiubnano karbon (*CNTs*) pada jumlah 0.1% daripada berat pecahan untuk menilai tingkah laku ke atas grout epoksi yang tidak terubah. Pelbagai ujian mekanikal telah dilakukan ke atas grout diubahsuai ini untuk mengenal pasti kekuatan mampatan, tegangan, lenturan dan pusingan ricihnya. Di samping itu, proses penyebaran CNTs telah dijalankan dengan menggunakan teknik *ultrasonication* dan *three-roll mill* untuk memastikan peningkatan optimum dalam sifat-sifat matriks polimer. Dengan membandingkan kekuatan, 0.1% pengisian *CNTs* berupaya meningkatkan kekuatan grout dalam ikatan lenturan, tegangan dan ricih tetapi tidak dalam mampatan. Di samping itu, hasilnya juga menunjukkan bahawa pengisi CNTs telah meningkatkan modulus keanjalan bahan infill. Oleh itu, ia menunjukkan potensi intrinsik CNTs dalam mengubah sifat-sifat resin grout epoksi.

Kata kunci: Pembaikan saluran paip, resin grout epoksi, bahan isian, tiubnano karbon, *three-roll mill*

1.0 INTRODUCTION

Corrosion has been found as a major cause of failure in both onshore and offshore pipelines [1], [2]. Consequently, their repair techniques are one of the major interest of the researchers [3]. In general, a wide range of rehabilitation techniques are available for onshore and offshore pipelines. Removing and localizing the damaged section of pipelines are the traditional solutions for repairing the damaged pipes. This conventional repair often involves inspection, installation and maintenance service that are bulky, costly and time-consuming especially for underground pipelines [4].

At present, Fibre Reinforced Polymer (FRP) composite repair has become a preferred technique over conventional repairs and proven effective in rehabilitating the damaged pipelines [3], [5]–[8]. It is perfectly fit for quick structural repairs due to their lightweight, high strength and high corrosion resistance [3], [8]. Apart from replacing the whole segment, the composite repair can be used to minimize the cost of pipe replacement with the capability of recovering pipe remaining strength. This can be done by applying the wrapper over the defective area accompanied by epoxy grout or "putty" as infill material that is used to fill the irregularities or dents on pipe surface and cylindrical sections [9]. However, the world of pipeline repair industry is now keen to minimize the use of composite wrapping layers as it is expensive and difficult to handle especially for damaged pipes that are located in congested areas; thus it has a limited working area for the wrapping process [10]. To do so, two options can be accomplished in order to optimize the thickness of composite wrapping layer through layer minimisation. The first option is by introducing new material for epoxy grout with superior performance and the alternative option is by enhancing the performance of the existing epoxy grout.

Even though the idea of minimising composite wrapper for pipeline repair through reinforced putty is well acknowledged by researchers [10], [11], yet current efforts are still rather insufficient. Research on pipeline composite repair is still highly dominated by composite wrapper performance instead of putty. Lack of attention to putty performance is due to wrong perception among researchers and industry whereby putty is normally perceived as material for patching of the damaged area and providing a smooth bed for the composite wrapper only with a minimal contribution towards repair system capability. Several initiatives have been made to produce new material for wrapper with minimal thickness yet superior in terms of strength [12], [13]. However, this technology is still in the stage of infancy and it will be a while for the technology to become feasible. Thus, the modification of an existing epoxy grout for performance improvement and increasing its contribution to the composite repair system is one of the possible ways of achieving this goal, as the grout performance is also a contributing factor for the effectiveness of the composite repair system [3]. Therefore, the most realistic initiative that can be taken in order to optimize the current fibre reinforced repair with the potential to reduce the wrapper thickness is by improving the strength of the infill material. Hence, improving the load-bearing function of infill material apart from its original intended function related to load-transfer mechanism.

As infill material can only be used in small quantity, the addition of additives into the epoxy grout is somehow limited. Thus, additives that can react effectively with the polymer in a very small quantity are vitally required. For that reason, additives (filler) with nanoparticles size such as Carbon Nanotube (CNT) is considered as one of the most practical candidates. In the past few years, studies that involved the combination of CNTs with various polymer matrices have greatly intensified [14]–[17]. These combinations have been proven effective in improving the mechanical and electrical properties of many polymers and it has been widely used in different industries [18]–[20]. Moreover, several factors need to be considered to effectively improve the infill material properties by incorporating nanomaterial as filler, which are the dispersion of nanomaterial in the polymer matrix and the optimum amount of nanomaterial for the highest performance by the infill material. Therefore, it is important to characterize the mechanical properties of the epoxy grout as a stand-alone material in order to determine the level of contribution by CNTs towards strength improvement of infill material. Hence, this paper will study the potential of carbon nanotubes (CNTs);

nanomaterial additives; in strengthening the existing infill material of epoxy grout as a step forward to transfer part of load bearing function from wrapper to infill material. The efficiency of the pipeline repair may be improved by the presence of high-performance infill materials as secondary layer protection in the repair system in the event of composite layer failure so that the thickness of wrapper can be potentially minimized with greater confidence.

2.0 EXPERIMENTAL WORK

2.1 Materials

The material used in this study is the existing commercially available epoxy resin grout that is generally used for grouting and fixation in a structure application. It is a combination of modified epoxy resins, mainly Bisphenol A diglycidyl ether, which are crosslinked using aliphatic amine hardener as a curing agent. When mixed together, the cured resin produces a hard-thermoset cross-bracing with high solvent resistance and produces a relatively high impact strength.

Carbon nanotubes were selected as nanofiller at the amount of 0.1% weight fraction to enhance the properties of the epoxy grout. These CNTs was categorized as multi-walled CNTs that have an average diameter of approximately 12.0~15.0 nm and particle length of 12.0~15.0 µm with >97% of carbon purity with the appearance of black powder. Other material used in this study is acetone as a solvent to isolate the CNTs nanoparticles.

2.2 Sample Preparations

An appropriate dispersion technique was applied to CNTs in order to totally utilize the unique properties of this special material. Before the mixing process, these carbon nanoparticles were pre-dispersed in acetone using Hielscher Ultrasonic Homogenizers UP200s (Hielscher Ultrasonics GmbH, Germany) according to the prepared weight percentage and were left to evaporate for 24 hours at room temperature. It then underwent another dispersion using Three-roll mill EXAKT 80E (EXAKT Technologies, Inc.) with resin as shown in Figure 2. The calendering process was applied four consecutive times. The resin with CNTs mixture was poured directly into the first roll, sheared in Gap 1, and followed by Gap 2. The sheared mixture was then manually poured back into the machine for the next process. The rollers turned at a speed ratio of 9:3:1 and the time taken for each calendering was approximately 10 minutes. The parameter details of the three-roll mill process such as gap size between the rollers and the speed (represent the lowest speed) are tabulated in Table 1. Figure 1 and Figure 2 show the schematics and process of dispersion using the three-roll mill machine.

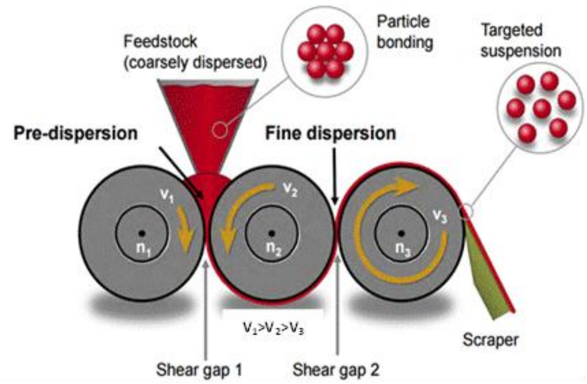


Figure 1 Schematic of the calendaring process (Source: EXAKT Advanced Technologies GmbH [21])

Figure 2 Dispersion process using three-roll mill machine

The specified composition of resin and hardener in the ratio of 2:1 was mixed together at the room temperature. The mixing process of CNTs/epoxy nanocomposite with hardener was done thoroughly in a clean dry container; denoted as modified grout; by using an electrical hand mixer at low speeds until a smooth consistency specimen was achieved as shown in Figure 3. Then, the mixture was transferred into the designated moulds and left for 24 hours at room temperature for curing process. Prior to testing, all cured specimens were polished to eliminate any impurities and defects on the surface of the specimen. Later, the modified epoxy grout will be compared with the existing commercial epoxy resin grout, denoted as neat epoxy grout. The above mentioned steps were also used in preparing the neat epoxy grout.

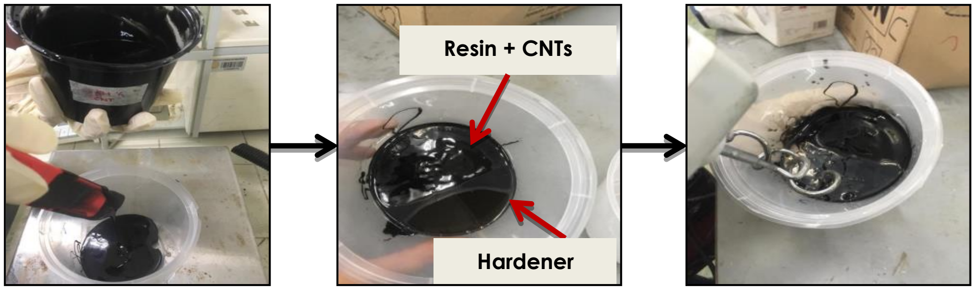


Figure 3 Mixing process of modified epoxy grout

Table 1 Details of the calendering process

Pass number	Gap 1 (µm)	Gap 2 (µm)	Rotational speed (rpm)
1	90	30	350
2	45	15	350
3	15	5	350
4	15	5	350

Table 2 Summary of testing details

Tests	Compressive	Tensile	Flexural	Lap Shear
Standards	ASTM: D695	ASTM: D638	ASTM: D790	ASTM: D1002
Number of samples	5	5	5	5
Dimensions (mm)	12.7 x 12.7 x 50.8	13.0 x 3.2	127 x 12.7 x 3.2	25.4 x 12.7
Geometry	Prismatic	Dog bone	Prismatic	-
Loading rate (mm/min)	1.3	5.0	1.367	1.27

3.0 CHARACTERIZATION OF EPOXY GROUT

3.1 Mechanical Analysis

To evaluate the effects of adding CNTs into the infill material, laboratory testing on the infill properties was carried out by referring to the industry standards. Table 2 shows the summary of mechanical properties tests for all grouts. Relevant standards and practices are also shown in Table 2. All the tests were carried out using a Universal Testing Machine (INSTRON 5567) with the capacity of 25 KN as shown in Figure 4. The strain gauge was used in the compressive and tensile test to determine Young's modulus of tested grouts. A Low Voltage Displacement Transducer (LVDT) was used in the flexural test to measure the deflection at the midspan. Data logger (TDS-530) was used to record the strain and LVDT value throughout the test. A minimum of five specimens of each grout were tested for each property.

Figure 4 Universal Testing Machine (INSTRON 5567)

4.0 RESULTS AND DISCUSSION

4.1 Compressive Properties

Summaries of the compressive test results for the tested grouts are given in Table 3. The highest compressive strength and stiffness are found in the modified epoxy grout. As can be seen from the table, the ultimate compressive strength of neat epoxy grout and modified epoxy grout are found to be 64.29 MPa and 65.63 MPa, respectively. However, based on the analysis of the data, there is no significant improvement on the ultimate compressive strength of the modified epoxy grout. The marginal increment in compressive strength can be explained as CNTs are not stronger under compression as they tend to undergo buckling due to their hollow structure and high aspect ratio [22]. Besides, the results obtained also correlated with the previous study by Loos *et al*. [23], where the increment on the compressive strength of CNT/epoxy composite was below than 10 % at 0.1% of CNTs content. Even so, the addition of nanofiller in this study has increased the stiffness of the materials by 12% from 2.52 GPa to 2.82 GPa, as compared to the neat epoxy grout.

Figure 5 shows the typical stress-strain curves behaviour of the neat and modified epoxy grout. Both of the tested grouts show similar behaviour to each other. Based on the graph, both tested grouts exhibited a linear response during the initial loading stage, followed by strain softening behaviour beyond its ultimate compressive strength. The tested grouts demonstrated ductile behaviour with noticeable deformation after the initial elastic behaviour and no sudden failure had occurred throughout the testing. The plots illustrate that the modified epoxy grout shows the stiffer slope at the elastic region.

The failure patterns of the tested grout under compression loading are shown in Figure 6. Both tested grouts exhibited a noticeable deformation as they started to buckle after the initial elastic zone. The top and bottom of the specimens experienced maximum stress as the initial cracks were spotted in that area. It was then followed by gradual reduction in stress before failure. Through observation, no lateral expansion occurred on the tested grout specimens.

Concerning pipeline repair, infill that is confined in between the damaged pipe and the composite wrap is expected to undergo compressive stress. As stated in [24], the compressive strength of the infill material is important as it will reduce deformation of the pipe wall under composite wrap due to radial stress. Aforementioned, both tested grout has demonstrated a ductile behavioural in which there is no sudden failure occurred throughout the testing. This behaviour of epoxy grout might prevent sudden rupture of the grout in the repair of composite pipes and suitable for high pressurize pipelines.

Table 3 Compressive properties of epoxy grouts

Grout	Compressive strength (MPa)	Compressive Modulus (GPa)
Neat epoxy	64.29 ± 0.78	2.52 ± 0.24
Modified epoxy	65.63 ± 1.57	2.82 ± 0.50

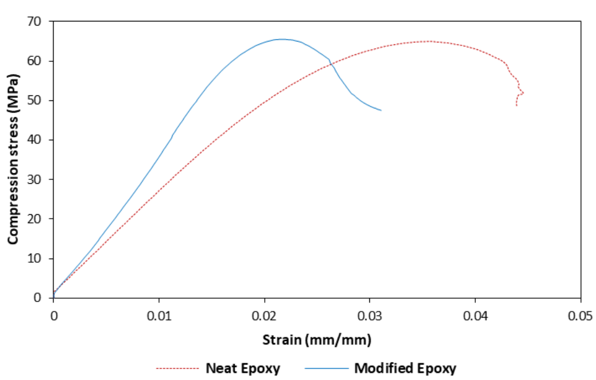
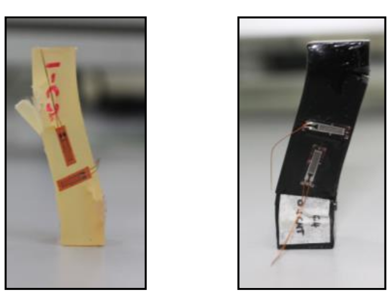


Figure 5 Stress-strain curves for compression specimens



(a) Neat epoxy grout (b) Modified epoxy grout

Figure 6 Specimens failure under compression

4.2 Tensile Properties

According to the results, there is an enhancement shown on the tensile strength and tensile modulus of the tested grouts. It can be seen from Table 4 that the tensile strength of the investigated grouts are found to be 26.42 and 34.61 MPa while the tensile modulus are found to be 2.21and 2.36 GPa. The modified epoxy grout with additional filler showcase higher tensile strength of 34.61 MPa with substantial increment of 31% as compared to neat epoxy grout. This result agreed with the results reported by Xiao *et al*. [25], which stated the increment in the tensile strength and modulus induced by the CNTs. Despite significant increase in tensile strength of the modified epoxy grout, however, it only contributes to a marginal change of 7% in its tensile modulus. Even though CNTs possess high tensile strength and elastic modulus [26]–[28], however, these outstanding properties of CNTs are still depending on the size and structure of nanotubes in the matrix [29].

Figure 7 and Figure 8 show a typical stress-strain curve and failure pattern for the tested grouts under tensile loading. Both grouts exhibit lower strength as well as lower ductility under tension as compared to its respective compressive strength. Based on the plotted graph, the tested grouts show linear elastic behaviour at the initial region, which exhibit ductility stage before its failure and there is no plastic deformation detected during the test. Under tensile load, the failure of specimens has occurred without noticeable deformation and sudden failure occurred as the material reaches its ultimate tensile strength. All grouts have undergone fracture due to splitting which is perpendicular to the length of the specimen.

The pipelines, when subjected to internal pressure, will cause the pipe to fail in tension mode predominantly in the hoop direction. Therefore, grout with higher tensile strength is required in providing additional load bearing capacity for repaired pipe [30]; hence, a better load-sharing mechanism can be achieved in composite repair system. Based on the results, modified epoxy grout has the potential to be used in structural rehabilitation based on its tensile strength when compared to the properties recommended by Mendis [31]; tensile strength range from 28 to 43 MPa. The grout with a higher tensile strength provides additional requirements by sharing the substantial stress from the high operational pressure instead of just transferring it from the pipeline to the composite wrap. This condition will improve the overall capacity of the repaired pipe, thus minimises the risk of failure if the composite wrap fails first by acting as secondary layer protection.

Table 4 Tensile Properties of epoxy grouts

Grout	Tensile strength (MPa)	Tensile Modulus (GPa)
Neat epoxy	26.42 ± 2.83	2.21 ± 0.20
Modified epoxy	34.61 ± 2.85	2.36 ± 0.20

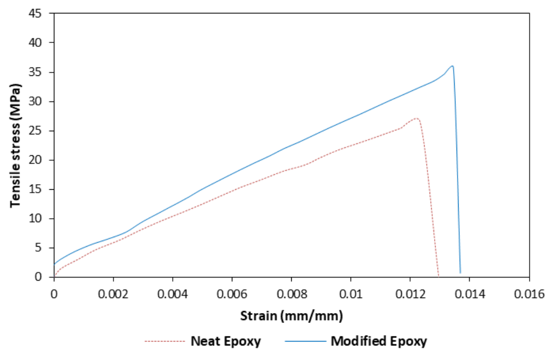


Figure 7 Stress-strain curves of tensile specimens

(a) Neat epoxy grout

(b) Modified epoxy grout

Figure 8 Specimens failure under tension

4.3 Flexural Properties

Table 5 represents the summary of the strength and modulus of tested grout under flexural. From the table, the modified epoxy grout has higher flexural strength and stiffness compared to neat epoxy grout with 78.41MPa and 2.16 GPa respectively. This is an increment by 29% and 27% for flexural strength and stiffness respectively. The comparison of the load-deflection behaviour of the grouts in flexural testing can be seen in Figure 9. Both grouts demonstrated linear elastic load-deflection behaviour prior to failure. The load-deflection behaviour of neat epoxy grout shows lower strength than the modified epoxy grout, but not much difference in deflection. Through observation on the crack formations on the surface of the grouts, the visible wedge was formed at the middle of the specimen indicated that the crack occurred from the bottom part (tension) and propagate to the top part (compression) as shown in Figure 10. The enhancement in strength for the modified epoxy grout can be explained by taking into account the orientation of the CNTs in the matrix, which might be in line with the axis and almost perpendicular to the loading. Due to that orientation, the load transfer from the matrix to the CNTs particles needs to be large enough for it to be broken under flexural loading.

Due to the nature of design and operational conditions, pipelines sometimes experience bending force. Localized corrosion and axisymmetric type of defect geometry can be proven critical in the steel pipe when significant bending occurs in the defect transitions zone under internal pressure [32]. Thus, the flexural deformation of the steel surface needs to be supported with higher flexibility grout as infill system. Therefore, modified epoxy grout may be applicable in situations that require higher bending forces to prevent the failure of the pipe and providing additional strength during bending moment.

Table 5 Flexural properties of epoxy grouts

Grout	Flexural strength (MPa)	Flexural Modulus (GPa)
Neat epoxy	61.01 ± 2.95	1.70 ± 0.11
Modified epoxy	78.41 ± 2.35	2.16 ± 0.08

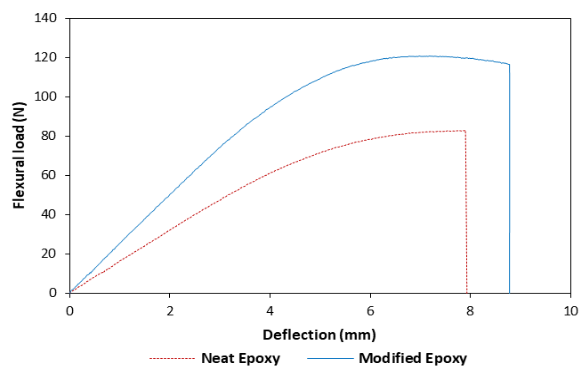
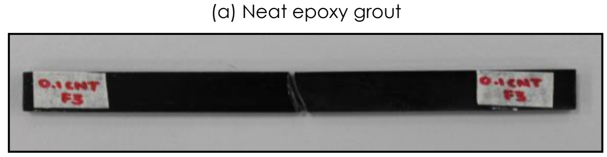


Figure 9 Load-deflection curves of flexural specimens

(a) Neat epoxy grout



(b) Modified epoxy grout

Figure 10 Specimens failure under flexural

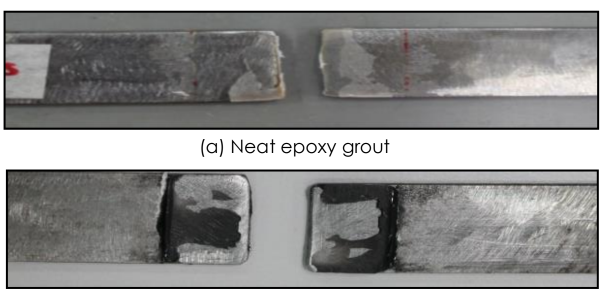
4.4 Shear Bonding

The individual average and maximum shear stress values of tested grouts are shown in Table 6. The results indicate that the shear strength of grout is increased with the inclusion of CNTs filler. The modified epoxy grouts with 0.1% of CNTs content has the highest shear bond strength compared to neat epoxy grouts of about 6.62 MPa and 5.83 MPa, respectively. On the other hand, the modified epoxy grouts showed the highest load failure at 2.11 kN, as compared to the neat epoxy grouts of 1.86 kN. The improvements in shear strength have also been reported in several literatures [14], [33]. Close examination of the failure surface of the tested grouts can be seen in Figure 11. The shear surface failure of both tested grouts showed that parts of the matrix were still attached to both of the substrate plate surfaces, indicating cohesive failure. This behaviour indicates that the matrix bonding between the applied grout with substrate plate (steel) is stronger than the strength between the matrixes.

The bonding strength between damaged pipe and repair component; infill grout and composite wrap; is also one of the factors contributing to the effectiveness of the composite repair system [34], [35]. Referring to ISO/TS 24817 [36] and ASME-PCC2 [37], the lap shear strength between the pipe and composite wrap should not be less than 5MPa and 4MPa, respectively. Above-mentioned standards also stated that at least 30% of the composite wrap (repair laminate) retain on the interfacial surface of the bonded area where the failure occurs during the short-term lap shear test. Although both standards do not specifically mention the required shear strength for infill material, this condition can be adapted as the minimum requirement for the infill material. Based on the shear strength and failure pattern, both tested grout were considered suitable as part of the composite repair system.

Table 6 Shear properties of epoxy grouts

Grout	Shear strength (MPa)	Load (kN)
Neat epoxy	5.83 ± 1.53	1.86
Modified epoxy	6.62 ± 0.81	2.11



(a) Neat epoxy grout

(b) Modified epoxy grout

Figure 11 Specimens failure under shear

4.0 CONCLUSION

This study has demonstrated the potential of the CNTs in enhancing the modified materials. Through observation, a small inclusion of CNTs showed a significant influence on the mechanical properties of infill material, especially on material strength. The superior properties of CNTs such as high aspect ratio, high flexibility, high tensile strength and high elasticity have improved matrix performance. However, the level of increment in the matrix properties may be different if the level of dispersion can be improved by considering the size and structure of nanotubes in the matrix [29]. In conclusion, when CNTs are used as filler in the pipeline repair component, it is found to be very promising to increase (i) strength (ii) stiffness and (iii) adhesion efficiency as stand-alone material. Hence, this may be an indication that the addition of higher CNTs content may further enhance the strength of the material, provided that the dispersion of CNTs within the resin matrix is sufficient. In that way, the excellent properties of individual CNTs will be fully maximized in order to achieve more significant increase in properties of final materials. Thus, with the enhancement in the properties of infill material, the efficiency of the composite repair system may be increased and infill material may act as secondary layer protection in the event of composite layer failure.

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