



Effect of Multi-Walled Carbon Nanotubes on Infill Material for Pipeline Composite Repair

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Abstract: The properties of the infill material are an important parameter in predicting the performance and behaviour of Fibre Reinforced Polymer (FRP) composite for effective design. This paper identifies the potential of Multi-walled Carbon Nanotubes (MWCNTs) as nanofiller in enhancing the performance of infill material. Two compositions of MWNTs at 0.1% and 0.5% of weight fractional were evaluated toward neat epoxy on tensile and lap shear test. By comparing MWCNTs-based modified epoxy grouts and neat epoxy grout, a significant increase in tensile strength was observed, especially with 0.5% of MWCNTs by almost 53.3%. While the inclusion of MWCNTs showed a comparable increment in shear strength in both 0.1% and 0.5% weight fractional by 13%. The image of morphologies showed that MWCNTs were well incorporated into the matrix, making the cross-section of the fracture rougher by sharing stress. This shows the potential of the MWCNTs in changing the properties of the modified epoxy grout, provided that the MWCNTs are appropriately dispersed throughout the resin matrix.

Keywords: Pipeline repair, fibre reinforced polymer (FRP) composite, epoxy grout, carbon nanotubes, nanofiller

1. Introduction

Oil and gas pipeline networks are normally made of steel. Like any other structure in engineering, the pipeline can experience structural failure due to integrity degradation. Although steel pipes are technically strong, they have low corrosion resistance and have become one of the pipeline system's significant flaws, reducing its operational integrity. This deterioration is likely to contribute to a bigger problem later if not treated. Therefore, the effective method of pipeline repair has become a leading concern among industry players.

Removing or replacing the damaged pipe section using a relatively new pipe segment is a conventional method. However, this technique is costly. Then comes the repair method using Fibre Reinforced Polymer (FRP) composite, which has been regarded as a preferred technique to replace this conventional method ever since [1]–[4]. This repair method is perfectly fit for structural repair that requires fast installation because of its lightweight, excellent strength and resistance to corrosion [5], [6]. Besides, it can eliminate the risk of explosion since no hot works like welding are conducted during the installation. Apart from removing the entire defective section, this composite repair can be used on the operating pipeline by utilising the wrapper over the defect region with the epoxy grout as an infill material on the

pipe surface and the cylindrical section [7]. This could mitigate the cost of fixing the pipeline with the potential to recover the remaining strength of the pipe without the need to shut down the operation.

The recent research findings show that trends are changing from FRP wrapper to infill material [8]–[13]. The world of the pipeline repair industry is now in progress to produce high-performance infill material [12], [14]–[16]. The goal is to reduce the usage of wrapper layers since it is costly and challenging to manage, particularly for damaged pipes situated in dense areas, the complexity of possible laminar failure and conservativeness in the existing design code for composite repair thickness [15], [17], [18]. The primary role of infill material is to serve as a means of transferring load from the pipe to the wrapping system. It is not designed to contribute to the system's strength but only provides a smooth surface in imperfect areas. This is essential to ensure that the outward bulging of the rusty sector is continuously reduced. Consequently, the efficiency of the infill material has been one of the criteria to determine the efficacy of the composite pipeline repair system [8], [19]–[21].

Although previous researchers have made efforts to improve the properties of infill with nanomaterial, there are some shortcomings in the study. There are dispersion issues, and the studies focus more on bonding performance [10]–[12]. Modifying the existing epoxy grout can improve infill performance and increase its contribution to the repair system. This material is commonly used in small amounts, so adding additional material on epoxy grout is limited. Therefore, nanofiller, which can react efficiently with polymers in minimal quantities, are preferable. Hence, nanoparticle-sized additives such as Carbon Nanotubes (CNTs) are receiving more attention to improve the performance of infill material. In recent years, studies involving the integration of CNTs into various polymer matrices have greatly intensified. The combination aims to produce a functional composite material that will improve its mechanical and electrical properties. Researchers have studied the characterisation of CNTs-epoxy nanocomposites with different concentrations as low as 0.01 wt% by correlating different dynamic analysis techniques including dynamic mechanical analysis (DMA), impedance and dielectric analysis (DEA) to find the optimum percentage. The studies have found that the critical concentration of carbon nanotubes is between 0.1 % to 0.5 % of the nanocomposite composition [22]–[25]. The results showed an initial increase in the mechanical properties of nanocomposites at low proportion and a decrease in higher proportion. The homogeneity of the CNTs in matrix composition was confirmed as a critical point for the mechanical behaviour of the nanocomposite.

Therefore, it is essential to characterise the mechanical properties of the modified epoxy grout to determine the degree to which CNTs contribute to the reinforcement of the infill material. The information on infill properties can be used to make an early prediction on the behaviour of the system for an optimum repair design using CNTs as nanofiller. Therefore, this study investigates the ability of the nanofiller-type Multi-walled Carbon Nanotubes (MWCNTs) to enhance the tensile and shears bonding properties of the epoxy grout.

2. Methodology

2.1 Materials

Liquid Bisphenol A Diglycidyl Ether (DGEBA) epoxy resin type crosslinked with an aliphatic amine hardener was used in this study. The cured resin creates a hard-thermoset cross-bracing with a high solvent resistance and a relatively high impact strength. The MWCNTs with a purity of more than 97% carbon obtained from the local manufacturer were used as particulate nanofillers for epoxy enhancement with particle thickness around 12.0–15.0 nm and a length of 12.0–15.0 μm .

2.2 Sample Preparations

Fig. 1 shows the pristine MWCNTs with strongly entangled networks prior to the dispersion process. An appropriate dispersion technique was applied to detangle the agglomeration using mechanical and chemical approaches. The MWCNTs were pre-dispersed first using the acetone-ultrasonication method for 15 minutes according to the prepared weight percentage as shown in Fig. 2. The MWCNTs/acetone suspension was then left at room temperature for 24 hours until it had fully evaporated. It is then followed by the calendaring process of pre-dispersed MWCNTs/acetone with resin as shown in Fig. 3 using a three-roll mill (EXAKT 80E) machine. The mixture was fed into the first gap between feed roll and centre roll before being moved into the second gap between centre roll and the apron roll linked by a 9:3:1 speed roller. The process was applied at four consecutive cycles for 0.1 wt% and five cycles for 0.5 wt% of MWCNTs. The time required is about 10-15 minutes for each calendar cycle.

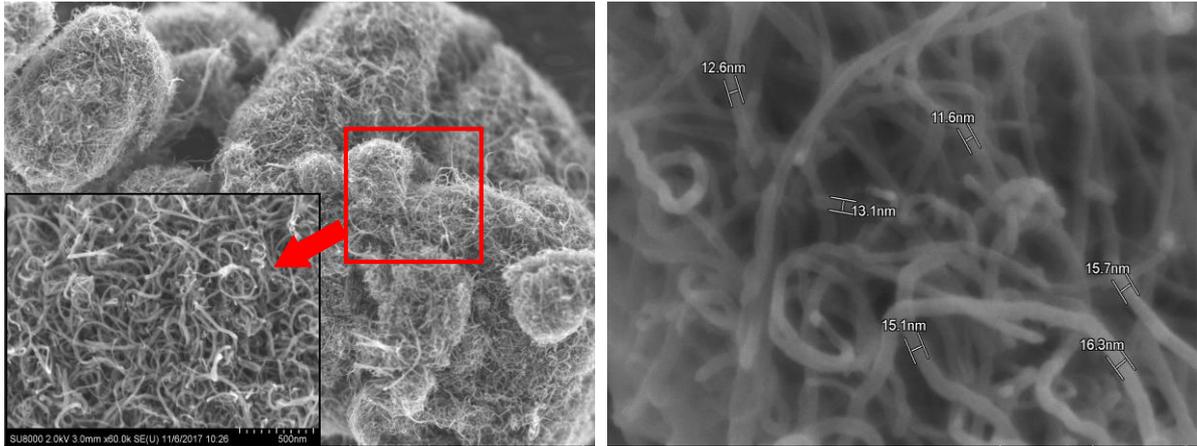


Fig. 1 - Entangled network of pristine MWCNTs



Fig. 2 - Pre-dispersion process using Hielscher Ultrasonic Homogenisers UP200s

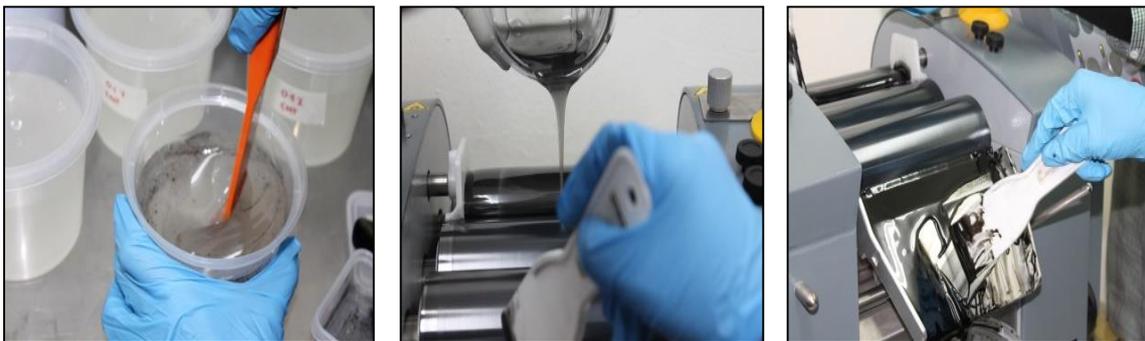


Fig. 3 - Process of dispersion using the calendaring method

Table 1 - The composition of epoxy grout

Specimens	Composition (ratio)			Remarks
	DGEBA Epoxy Resin	Hardener	MWCNTs	
Neat epoxy grout	2	1	-	Control
0.1 wt% MWCNTs-based	2	1	0.1%	Modified
0.5 wt% MWCNTs-based	2	1	0.5%	Modified



Fig. 4 - Preparation of modified epoxy grout

2.3 Tensile and Lap Shear Characterisation

Five specimens were prepared for tension and lap shear tests referring to appropriate industry standards. The 25kN INSTRON 5567 universal test machine, as seen in Fig. 5, was used to conduct both tests. Dumbbell or dogbone specimens with the size of 13.0 mm x 3.2 mm were used for tensile testing, i.e., Type I of the ASTM: D638. The crosshead speed used for the traction specimens was 5.0 mm/min. The specimens were marked and clamped at the top and bottom using the end tab to prevent premature failure of the grip length. All specimens are equipped with strain gauges and attached to a data logger at a specific INSTRON 5567 grip separation before being pulled up until failure.

A single lap joint test was conducted in accordance with ASTM D1002. ASTM D1002 determines the shear strength of adhesives for bonding metals by applying tension loads to the specimens. Two pieces of carbon steel coupons measuring 25.4 mm x 100 mm (width x length) were bonded together with a sample at each end where the two coupons overlapped. The specimen was pulled in opposite directions to produce shear stress at the specified load rate of 1.27 mm/min.

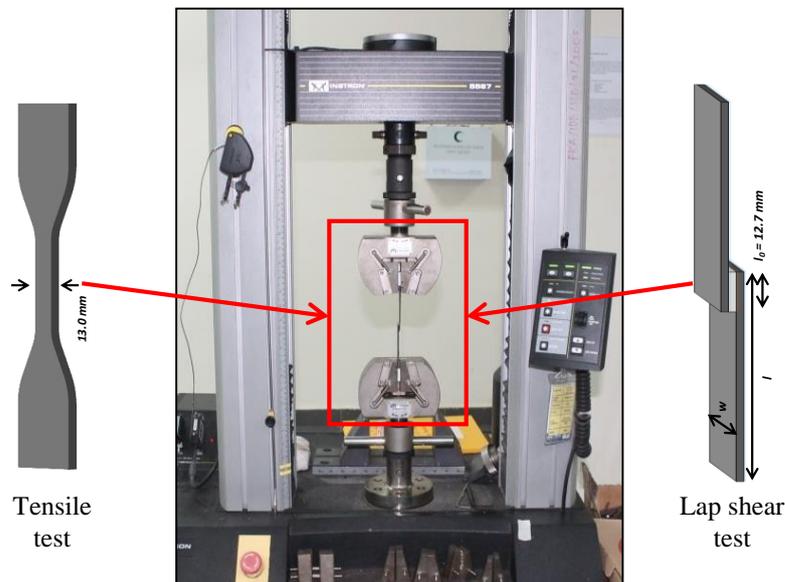


Fig. 5 - Universal testing machine-INSTRON 5567

3. Results and Discussion

3.1 Tensile Properties

The pressurised pipeline will experience three stresses under circumferential (hoop), longitudinal (axial) and radial directions [26], [27]. However, hoop stress is the most critical stress for the pipe under internal pressure. This stress will cause the failure of the pipe in tension. Therefore, the tensile properties of repair material are considered as the most important properties to be understood. The representative tensile stress-strain curves for each specimen are shown in Fig. 6. As can be seen, both control and modified epoxy grout displayed a linear stress-strain relationship in the initial elastic region, and no plastic deformation has occurred before fail. Fig. 7 shows the fractured specimen for modified epoxy grouts in the tensile test. All specimens failed due to splitting into two parts perpendicular to the direction of loading during peak stress. No significant lateral dimensional differences were found in all specimens before failure.

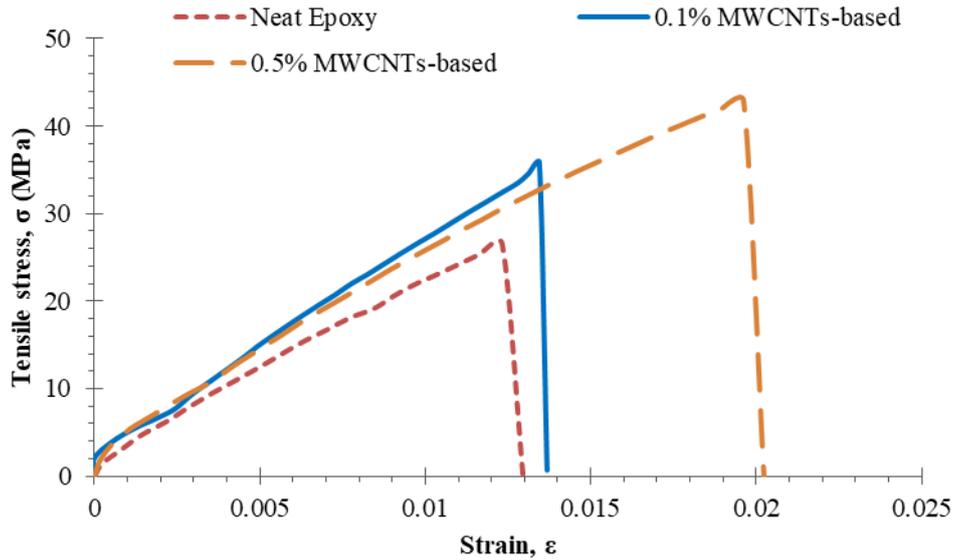


Fig. 6 - Specimens' stress-strain curves

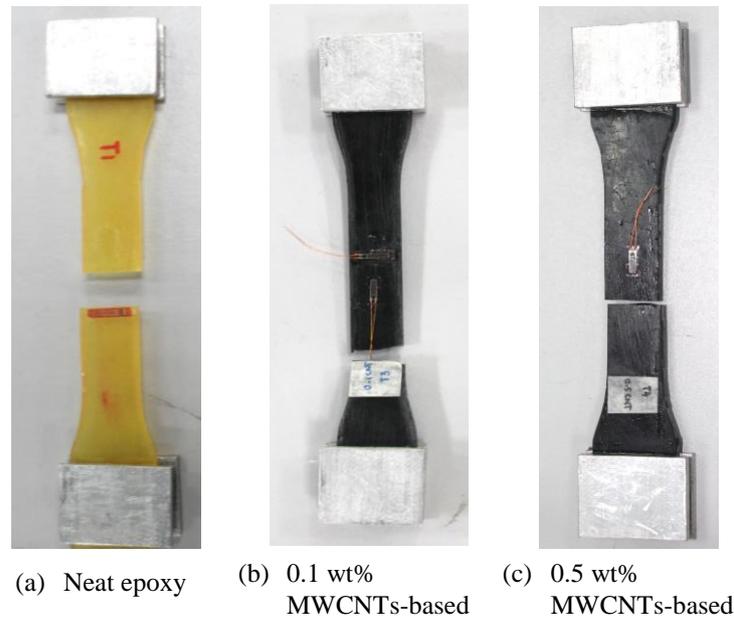


Fig. 7 - The failure of tension specimens

Fig. 8 shows the effects of MWCNTs addition on epoxy grout under tensile loading were compared. The tensile strength and Young's modulus of control grout (neat epoxy grout) and modified epoxy grout were found between 26 MPa to 40 MPa and 2.2 GPa to 2.4 GPa, respectively. The inclusion of MWCNTs in epoxy grout has significantly increased the strength and modulus under tensile loading. Fig. 8 shows that the tensile strength of epoxy grouts with 0.1 wt% and 0.5 wt% of MWCNTs nanofiller was 31% and 53.3 % better than the control grout, respectively. The increase of the tensile strength is believed to be due to the MWCNTs' nano-sized dimension. MWCNTs have large reactive surface areas, which contributed to effective stress distribution and reduced interfacial stress concentration [28–30]. However, there is a slight reduction in tensile modulus at a higher percentage of the MWCNTs filler.

Under tension, the bonding interaction is more important than the bridging effects. The strong interface between MWCNTs and matrix has led to higher strength and strain at break under tensile loading. This strong interface allowed the load to be transferred from the matrix to the MWCNTs filler. This enhancement is supported by the Field Emission Scanning Electron Microscope (FESEM) image as presented in Fig. 9. The image indicates that the MWCNTs were evenly distributed within the matrix. From the observation, most of the MWCNTs nanofiller were dispersed into smaller broken particles and ranging out of the epoxy resin, showing a rough profile compared to control grout. This reflects the strong interfacial strength between MWCNTs and the resin matrix, which prevents MWCNTs from being pulled out [31]. Thus, it effectually improves the mechanical properties of modified epoxy grout. However, some MWCNTs bundles

were observed at higher content of MWCNTs, which is referred to as re-agglomeration that might have occurred during the curing process. This could be a reason for the modulus reduction on the higher MWCNTs content.

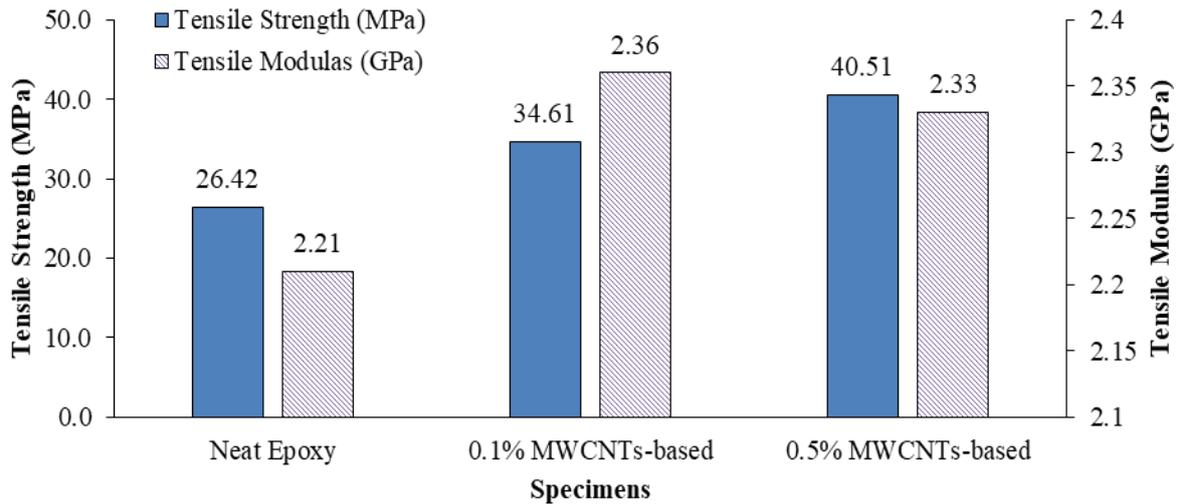


Fig. 8 - Specimen tensile strength and tensile modulus

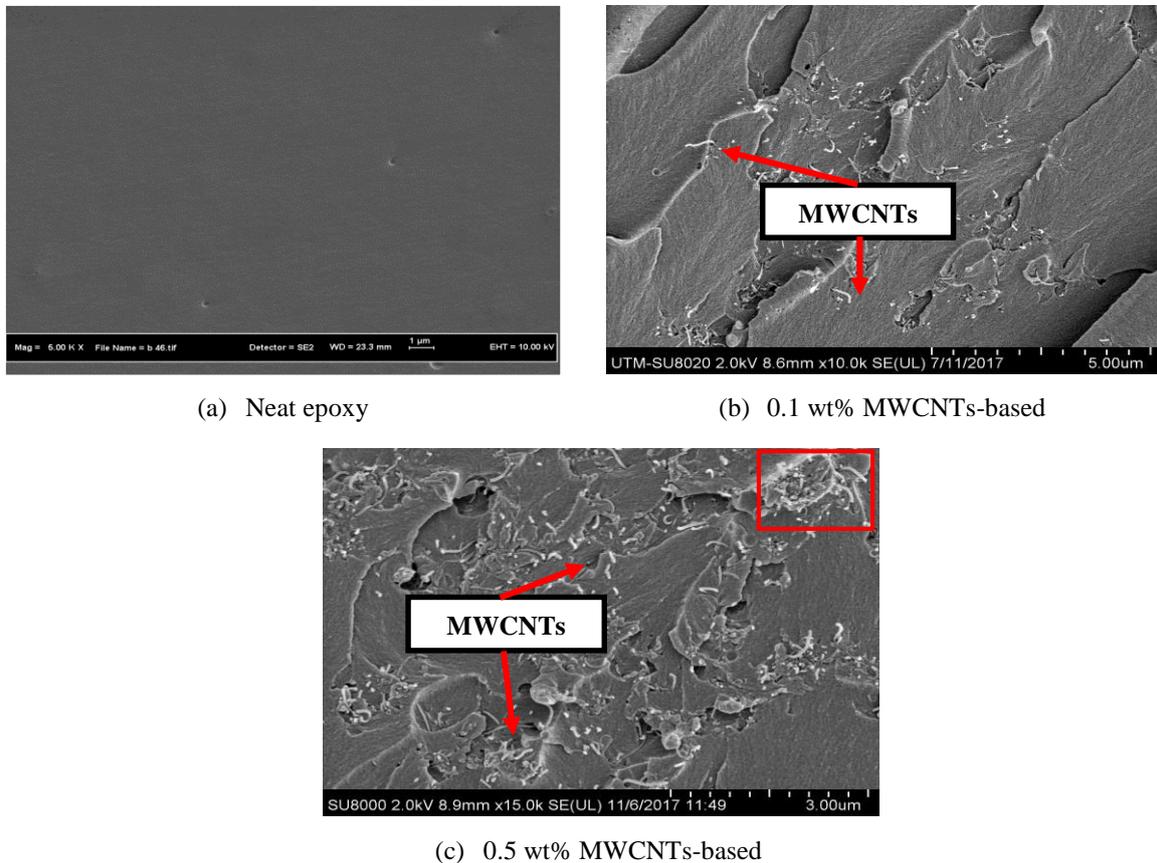


Fig. 9 - FESEM image of the fracture surface of control and modified epoxy grout

The dispersion of CNTs using ultrasonication and a three-roll mill has shown good results that correlate with previous studies [32–34]. The effect of shearing in the three-roll mill surely has succeeded in tearing the MWCNTs agglomeration. The material’s high strength is attributed to an exceptionally high surface area of MWCNTs, which in turn exhibits a very high interfacial area [35]. Hence, the stress transferability across the interface is easily facilitated. The surface area provided, which was combined with the specific mechanical nature of the nanofiller and the aspect ratio is a determining factor for strengthening the epoxy mechanical matrix by nanoparticles. Therefore, a good nanofiller impregnation is a prerequisite before the potential of MWCNTs as structural reinforcement.

3.2 Shear Bonding

Another aspect that contributes to the efficiency of the composite repair system is the bonding strength between the damaged pipe and the repair component [19], [20]. If the bond between the repair components is not sufficient, the load will not be effectively transferred within the repair system.

The findings show that the addition of the MWCNTs improves the adhesive strength of the epoxy grout. For both modified epoxy grout, the bonding strength increases by around 13% compared to the control grout, from 5.83 MPa to over 6.62 MPa and 6.63 MPa. On average, modified epoxy grout with 0.1 wt% and 0.5 wt% of MWCNTs show comparable shear strength despite differences in nanofiller content. The improvements in shear strength using this nanofiller have also been reported in the literature [10], [36]. The failure surface of the tested grouts is shown in Fig. 10. As can be seen, parts of the matrix remain on both surfaces of the steel coupons, signifying the cohesive failure pattern for all tested grout. This shows that the chemical bond between the matrix and the steel coupon (steel) exceeds the solidity of the matrix.

A strong bond at the adhesive/substrate interface indicated that the interaction between MWCNTs and the matrix was achieved, which improved the adhesion performance of the MWCNTs/epoxy grout material to the substrate. MWCNTs nanofiller was also found to increase the roughness surface of the matrix as captured in the FESEM image. Hence, the mixture of an epoxy grout with MWCNTs increased the bonding strength of particles with the material in contact. This indicates that the component will be durable throughout its service life. Other than that, it also can be explained that MWCNTs might have infiltrated properly within an epoxy matrix. However, no further improvement was observed at the higher content of MWCNTs. Since infill material is only used in small quantities, further addition of MWCNTs content might not be appropriate. The FESEM image also shows a bundle of MWCNTs in the matrix, which restricts load transfer from the matrix to the nanofiller.

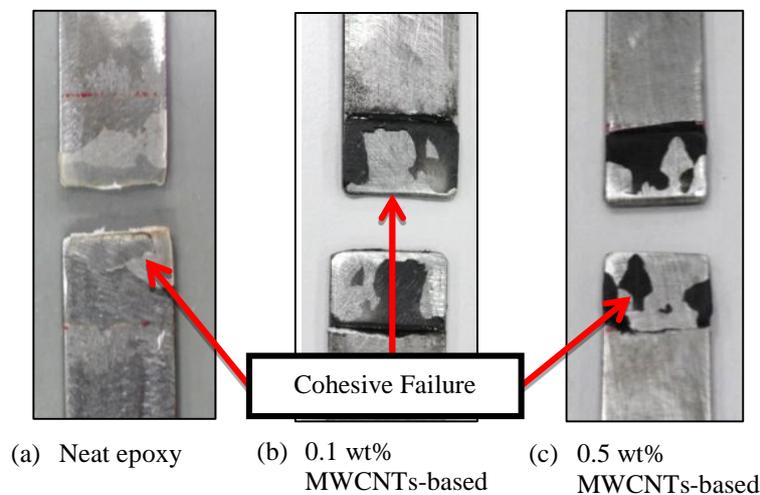


Fig. 10 - The failure of shear specimens

4. Conclusion

This study identified the potential of MWCNTs in enhancing the tensile and bonding properties of epoxy nanocomposite material. The deagglomeration of MWCNTs was carried out through ultrasonication and three-roll mill techniques. The experimental results demonstrated that the modified epoxy grout with 0.1 wt% and 0.5 wt% of MWCNTs improved tensile strength and shear bonding better than the neat epoxy grout. An increase of 31% and 53.3% were recorded for tensile strength of 0.1 wt% and 0.5 wt% of MWCNTs, respectively, and bonding strength increased by 13%. The addition of MWCNTs has also increased the stiffness of the epoxy grout. The random orientation of the MWCNTs in the matrix accounts for the significant improvement in the performance of the epoxy grout. This demonstrates that the effect of shearing in a three-roll mill was successful in tearing the MWCNT agglomeration. As a result, MWCNTs have been discovered to be very promising in terms of improving the strength, stiffness, and adhesion performance of epoxy grout for pipeline repair applications. This suggests that the addition of higher MWCNTs content may further improve the material's strength, provided good dispersion of MWCNTs in the resin matrix to unlock its remarkable properties. Therefore, the improvement in the properties of the infill material is seen as a significant step toward boosting the effectiveness of the composite repair system by functioning as secondary layer protection in the system in the event of composite layer failure.

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