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Effective Dispersion of Carbon Nanotube in Epoxy Grout for Structural Rehabilitation

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Abstract. The industry nowadays is incorporating the composite repair system for repairing pipelines rather than the conventional steel repair. The mechanism of this repair method usually consists of three components which are the composite wrapping, infill material and the adhesive. However, there has been very little research on the function of the infill in the repair mechanism. This work is concerning the enhancement of the performance or the strength properties of the infill material in pipeline repair by reinforcing the putty with carbon nanotubes (CNT). The enhancement of the performance of the infill has been carried out by dispersing the CNT into epoxy resin with a three roll mill. In the mechanical properties testing, it is found that the CNT is an effective material to improve the tensile strength of the epoxy grout. However, the CNT-modified samples in the compressive property test show a contrast to the tensile test. All the CNT-modified samples exhibit a lower compressive strength than the control sample and the milled down sample. In conclusion, CNT shows the potential to be a very good material to enhance the mechanical properties of epoxy grout, however, with this specific brand of epoxy grout that contains steel filler in the resin, the CNT only improve the tensile properties but the compressive properties of the epoxy grout has been compromised as compared to the control sample.

1 Introduction

Despite the fact that steel pipelines are the most effective and safe ways for oil and gas transportation over a long distance, they are prone to adverse deterioration in the form of corrosion, crack, dents, wearing, buckling, and gouging that may potentially lead to leaking and rupture [1-4]. Repair methods have since then being developed to retrofit damaged pipelines and composite repair method has become more popular in recent years [5-7]. Composite pipeline repair consists of three components which are the composite wrapping, infill material and the adhesive [8-9]. The infill material of composite repair system is neglected in current design codes. However, these past few years the number of pipeline operators using fibre reinforced polymer composite repair system to repair pipelines has

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been swelling. This has caused the recent development of design codes for the design of the repairs of pipelines such as ASME PCC-2 [10] and ISO/TS 24817 [11]. These codes were developed to standardize the design method of composite pipeline repair. This has also made quality control in this field a reality as pipeline operators has increasingly utilizing this method of repair. However, the codes only take into consideration the remaining strength of damaged pipe and the composite wrapping strength in the design without involving the strength of the infill material. This study is only aimed to modifying the commercially available epoxy grout by adding different percentages of carbon nanotubes (CNT).

Theoretically, the CNT will be able to enhance the mechanical properties of the epoxy grout as it exhibits a very high aspect ratio which results in a high specific surface area (SSA) [12]. The issue with CNT is that the interfacial bonding of the nanoparticles is strong and to achieve a proper dispersion of the individual CNTs in the epoxy grout can be difficult. The advantage of reinforcing epoxy grout with CNT can be limited if the linkage between CNTs and the epoxy grout is not sufficient. A three roll mill right now is the most popular dispersion machine out there. The dispersion of the CNT was done by the three roll mill through a calendaring process. The calendaring process of the three roll mill utilized the shear force created between the roller to separate the agglomeration of CNT and dispersing it as evenly as possible. After the dispersion process is completed, the effectiveness of nano-particle CNT as reinforcement in an epoxy grout will be evaluated by determining the mechanical properties of the epoxy grout samples.

2 Methodology

The putty used in this study is commercially available steel-filled epoxy grout. The samples are prepared in 5 different variables which is the control sample, a milled down sample, 0.01% CNT sample, 0.05% CNT sample and 0.1% CNT samples. The modification of putty starts with mixing epoxy resin and CNT using planetary centrifugal mixer, the Kakuhunter SK-350TII. CNT was added at different percentage into the epoxy resin and transfer into the Kakuhunter SK-350TII machine for 120 seconds for mixing and degassing purposes. The mixer is capable to accommodate mixing and degassing for various materials regardless of any viscosity to achieve a homogeneous mixing. The mixing process is shown in Fig. 1. The CNT-epoxy resin mixture was then undergoing calendaring process using a three-roll mill, the EXACKT 80E machine to disperse the CNT. A milled down sample where the resin is put through the three roll mill without any CNT added was done because size of the existing filler is bigger than the smallest gap size of the rollers which is 15 µm. Fig. 2 shows the calendaring process of CNT into epoxy resin and Table 1 summarizes the configuration used for the calendaring process. The samples are prepared for two different mechanical properties tests which are the tensile and compression test. The tensile and compressive strength tests were done in accordance to the ASTM D638 and ASTM D695, respectively. A Shimadzu 50kN Universal Testing Machine was utilized for the tests. A Field Emission Scanning Electron Microscopy (FESEM) test is also conducted to find out the nature of the failure of selected samples.

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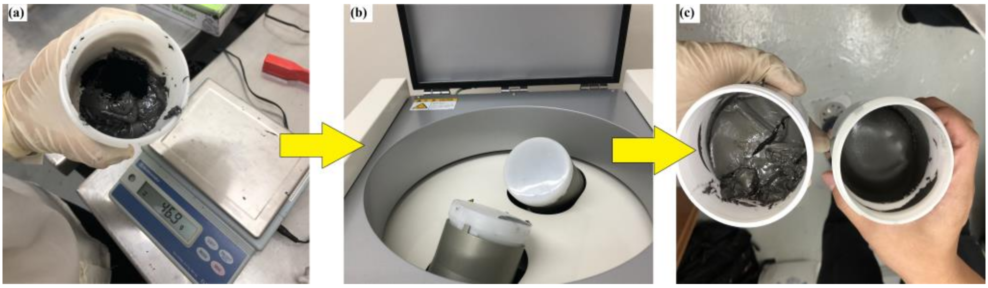

Fig. 1. (a) neat resin with CNT particle; (b) mixing process; (c) neat resin (left) and mixed resin-CNT (right).


Fig. 2. Calendaring process of CNT-resin mixture.

Table 1. Three roll mill configuration.

No of passes	Gap 1 (μm)	Gap 2 (μm)	Roller Speed (rpm)
1	100	60	200
2	60	30	200
3	45	15	350

3 Results and discussions

The results of tensile test are tabulated in Table 2. The plus and minus sign (±) after average value represents standard deviation of the sample. The tensile test result of the samples in Table 2 shows that the 0.01% CNT samples recorded the highest average tensile strength and the milled down sample has the lowest average tensile strength. Samples with more CNT added has a lower average tensile strength than the 0.01% CNT which is 14.33MPa for the 0.05% CNT sample and 15.56MPa for the 0.1% CNT sample. There is an increase of 62% and 13% of tensile strength from the milled down samples and control sample to the 0.01% CNT added sample, respectively. This shows that CNT is a very effective material to enhance the tensile strength of the epoxy grout. The decrease of the tensile strength for the 0.05% CNT and the 0.1% CNT added can be explained by the optimum amount added to enhance the performance of the epoxy grout. The drop happens after the percentage of weight content added exceeds the optimum percentage [13].

Table 2. Tensile test result.

Sample	Label	Tensile Strength (MPa)	Young's Modulus (GPa)
Control	TC	15.93 ± 0.36	8.65 ± 0.91
Milled Down	TM	11.09 ± 2.32	8.83 ± 2.52
0.01% CNT	T01C	17.99 ± 2.60	9.91 ± 3.85
0.05% CNT	T05C	14.33 ± 1.16	7.88 ± 1.90
0.1% CNT	T10C	15.56 ± 0.71	9.12 ± 0.38

Compression strength of the infill material is brought onto action as the infill material act as the load transfer from the pipeline and the composite wrapping. The compression test result are summarised in Table 3. The compression test result in Table 3 shows that the sample with the highest compressive strength is the milled down sample with 71.12MPa and the sample with lowest compressive strength at 61.79MPa is the 0.05% CNT samples. In contrast to the strength, the young's modulus of the milled down sample exhibits the lowest Young's modulus in all the samples at 7.21GPa. The 0.1% CNT sample has the highest compressive strength and Young's Modulus in all the CNT-modified samples at 65.90MPa and 9.24GPa, respectively. Despite expected rise in mechanical properties, the compressive strength of the CNT-modified samples all decrease from the milled down sample where it drops 8.77% from the milled down sample to the 0.01% CNT added, 13.12% for the 0.05% CNT added and 7.33% for the 0.1% CNT added.

Table 3. Compression test result.

Sample	Label	Compressive Strength (MPa)	Young's Modulus (GPa)
Control	CC	69.19 ± 3.56	8.30 ± 1.59
Milled Down	CM	71.12 ± 5.56	7.21 ± 1.89
0.01% CNT	C01C	64.88 ± 4.64	7.36 ± 1.38
0.05% CNT	C05C	61.79 ± 2.49	8.62 ± 0.53
0.1% CNT	C10C	65.90 ± 8.46	9.24 ± 0.88

In both the mechanical properties test, the result of the milled down sample differ from the other. This situation is most likely caused by the phase separation of the existing epoxy grout filler in the resin from the resin by the three roll mill (refer Fig. 3). The sizes of the existing filler in the resin are too big to go through the designated gap of the three roll mill. The milled down sample has the highest compressive strength but the lowest tensile strength. The existing steel filler in the resin are suspected to act as the main tensile strength contributor in the unmodified matrix of the epoxy grout. Hence, with the steel filler which is the tensile strength contributor in the matrix separated from the resin in the milled down sample, the tensile strength drops dramatically from the control sample to the milled down sample. On the other hand, with the tensile strength contributor separated, the compressive strength of the matrix can be improved because the matrix will be denser and without any filler.

Fig. 3. Phase separation of the existing filler on the three roll mill.

Fig. 4 shows the result of the FESEM test on the failure surface of selected samples. The FESEM test conducted shows that the CNT in the 0.1% CNT (T10C-03) is abundant on the failure surface. On the other hand, there is nothing but the epoxy grout on the failure surface of the milled down sample (TM-05). In contrast to the abundant of CNT on the 0.1% CNT, the 0.05% CNT added has very little CNT on the failure surface, hence, the tensile strength of the 0.05% CNT is the lowest. In the control sample (TC), the existing steel filler is present as evident in the FESEM images but absent in the milled down sample which proves the phase separation on the existing steel filler.

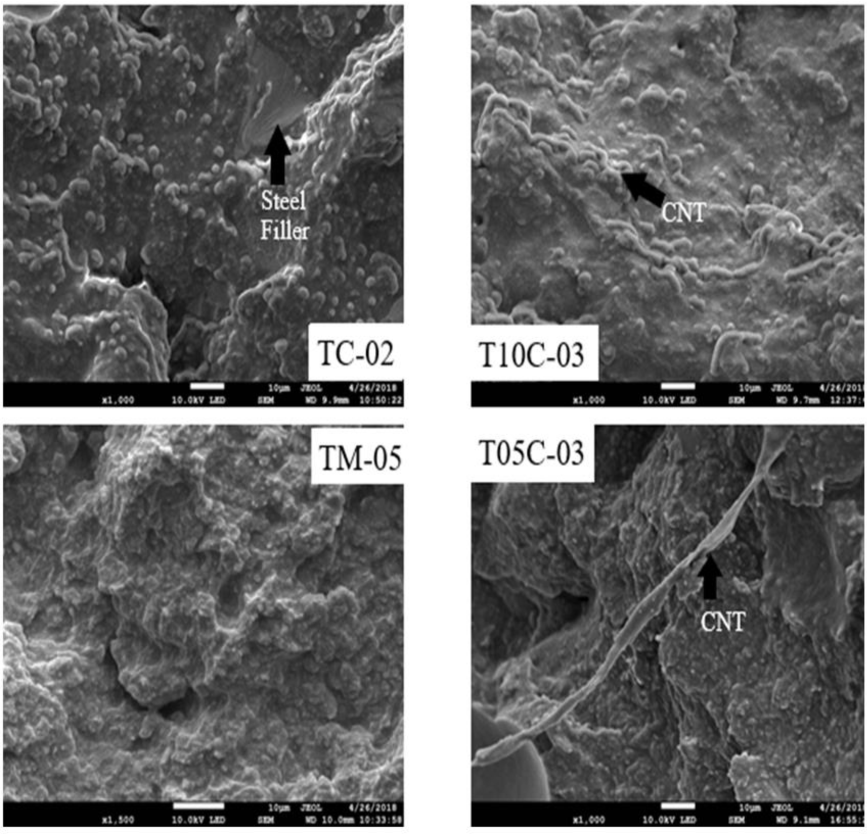

Fig. 4. The failure surface of selected samples.

4 Conclusions

The tensile strength of the epoxy grout has improved with the addition of CNT into the epoxy matrix but it is the opposite for the compressive strength of the epoxy grout where it decreases with the addition of CNT. In conclusion, CNT can be a very good material to enhance the mechanical properties of epoxy grout, however, with this specific brand of epoxy grout that contains steel filler in the resin, the CNT only improve the tensile properties but the compressive properties of the epoxy grout decrease as compared to the control sample. More percentage variables can be done to get the optimum percentage of CNT to be added. The optimum percentage is important to optimize the amount of CNT used for the enhancement of the performance of the infill material.

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