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COMPRESSIVE, TENSILE AND THERMAL PROPERTIES OF EPOXY GROUTS SUBJECTED TO UNDERWATER CONDITIONING AT ELEVATED TEMPERATURE

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ABSTRACT

Oil and gas pipes are susceptible to failure initiated by corrosion due to their operating pressure under adverse atmospheric conditions. Repairs, comprising a composite shell assembled around the pipe with a small gap, which is then infilled with grout, are considered a suitable option for corroded pipelines. This paper presents the investigation on the mechanical (compression, tension) properties and glass transition temperatures of two infill grouts, after 1000 hour of hot/wet conditioning. An extended investigation on the moisture absorption behaviour was also carried out, revealing the highest absorption to be about 6% after 2520 hours of immersion. The glass transition temperatures of the grouts are reduced by approximately 20ºC. The results suggest that the grouts underwent significant reduction of strength and stiffness due to hot/wet conditioning when tested at an elevated temperature, compared to room temperature. This reduced strength and stiffness is the result of the grouts being tested in close proximity to their glass transition temperatures.

KEYWORDS

Epoxy grout, repair, infill, hot/wet conditioning, properties.

INTRODUCTION

Oil and gas pipes are susceptible to corrosion due to harsh atmospheric conditions. Fibre-reinforced polymer composites have been recognised as suitable materials for repairing tubular metallic pipes (Shamsuddoha et al. 2013a), and providing infill to the composite repair is considered a suitable option for pipeline with metal loss (Palmer-Jones et al. 2011). In such a repair system, the properties of an infill are critical for its overall performance. The structural grouts derived from epoxy thermoset resins, hardeners and fillers serve as a protective layer and effectively transfer load in a repair system. Elevated temperatures found to affect the physical properties of epoxy polymers (Carbas et al. 2013). The effect of hot/wet conditioning on infill epoxy grout for composite repair of a pipeline should be studied.

In this paper, two commercially available grouts were selected for mechanical and thermal property characterisation. These properties were determined for as-manufactured grouts specimens tested at 23ºC in a previous study (Shamsuddoha et al. 2013b). The work was extended to observe the effect of hot/wet conditioning on the mechanical and thermal properties of grout materials that can be used in grouted repairs, resembling elevated temperature in underwater conditions. The compressive, tensile, and thermal properties of these grouts were determined. The results of the study also provide justification of serviceability criteria according to ISO/TS 24817 (2006), which is a standard used for qualification of polymer matrix composite repair materials.

METHODOLOGY

Materials and Preparation

Two epoxy grouts with different compositions of neat resin, hardener and aggregate were selected based on their mechanical and thermal properties at ambient temperature as presented in Shamsuddoha et al. (2013b). Table 1 shows the proportions of various ingredients of the grouts. The first grout had two parts: high viscosity resin with fine filler particles already included, and low viscosity hardener, whereas the second grout was a modification of the first grout, with added coarse filler. Due to confidentiality, the grouts were investigated in this article are named as grouts C and E. For ease of comparison, these names are kept similar to the previous study (Shamsuddoha et al. 2013b).

Table 1. Composition of the grouts

ID	Components	Ingredients (% Weight)		
		Part A[3]	Hardener	Aggregate filler
C	Resin with fine filler[1] and Hardener	90.48	9.52	-
E	Resin with fine filler[1], Hardener and Coarse filler[3]	45.24	4.76	50.00

[1]0.05 – 300.0 µm, [2]45 µm – 2.36 mm, [3]Bisphenol A and/or F epoxy resin and fine filler

A hand held electric drill mixer was used to mix the batch in a plastic container. Freshly mixed grouts were poured into moulds at 23ºC. The specimens were cut and polished to the required dimensions. Table 2 shows details of the specimen size. The specimens were removed from the moulds after 24 hours and cured in a controlled environment at 23ºC for 7 days prior to hot-wet conditioning.

Conditioning and Testing

1000 hours of hot/wet conditioning was selected to conform to long-term durability test criteria suggested by ISO/TS 24817 (2006). ASTM E1640 provides a number of methods to determine glass transition temperature. According to ISO/TS 24817, the service temperature of a repair component for non-leaking (Type A) and leaking pipes (Type B) should not be 20ºC and 30ºC less respectively than the glass transition temperature (T_g). An investigation on the thermal properties of these unconditioned grouts suggested T_g values of 53ºC and 60ºC, and T_t of 83ºC and 90ºC for the grouts C and E, respectively (Shamsuddoha et al. 2013b). It is to be noted that the mentioned article presents only T_t values as glass transition temperature. The T_g values are retrieved from the grouts used in that study as reference. In this study, T_t is taken as the glass transition temperature ceiling for leaking and non-leaking conditions to determine whether this less conservative measure of the glass transition temperature is acceptable, in comparison to the conservative T_g measure prescribed in ASTM D1640. Therefore, 70ºC is considered for hot-wet conditioning and 65ºC is considered for mechanical testing, which is around the range of 20 – 30ºC less than the T_t of the unconditioned grouts.

Table 2. Summary of specimen and test details

Tests	Standards/Methods	N	Dimensions	Geometry	Loading rate
Compressive	ASTM C579 (2001)	5	25 x 25 mm	Cylinder	1.3 mm/min
Tensile	ASTM D638 (2010)	5	10 x 10 mm	Dog bone	1.0 mm/min
DMA	ASTM E1640 (2009)	3	60 x 12 x 5 mm	Prismatic	1°C/min

The compressive, tensile, and thermal properties of the grouts were determined after 1000 hours of hot/wet conditioning. Table 2 provides details of the tests. Relevant standards and practices are also shown in the table. All mechanical tests were carried out using a 100 kN MTS hydraulic testing machine, with the exception of the 50 mm diameter cylindrical compressive specimens, which were tested using a 2000 kN SANS servo-hydraulic compression testing machine. Figure 1 shows a typical test at elevated temperature. The specimens were preheated at 65ºC inside a water-bath for at least 30 minutes prior to being placed into the temperature chamber.

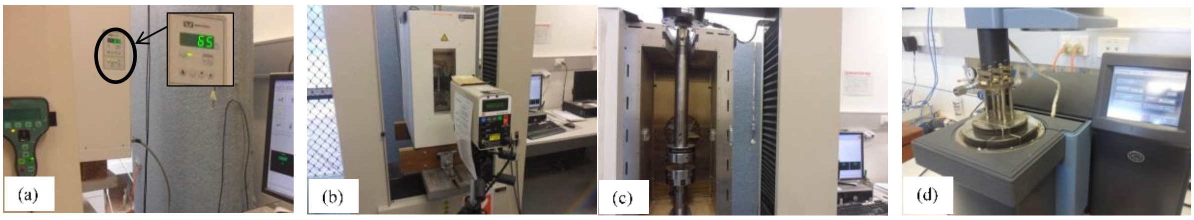


Figure 1. Testing of the grouts; (a) elevated temperature, (b) tensile, (c) compressive, and (d) DMA

RESULTS

The mechanical and thermal properties of the grouts are given in Table 3. The details of the results are discussed later in this section.

Compressive Properties

The strengths are found to be 93 MPa and 45 MPa for grouts C and E, respectively. Compressive moduli are found to be 0.42 GPa and 0.56 GPa for grouts C and E, respectively. From the plots, the yield strain of the specimens of grouts C and E are about 32% and 11% respectively. The compressive strengths and moduli of these two epoxy grouts at room temperature without condition were found to be 120 and 106 MPa, and 5.6 and 11.0 GPa, respectively for the grouts C and E, respectively (Shamsuddoha et al. 2013b).

Figure 2 shows the typical compressive stress-strain curve and failure pattern of the tested specimens. The compressive specimens of the grouts show an elastic behaviour followed by yield stresses. The stress-strain behaviour of the compression specimens of the grouts shows that subsequent to yield, the stress drops. Under compression, grout C exhibits circumferential bulging after the initial elastic behaviour. The bulging continues until failure which is initiated by vertical cracks. The bulging starts after the linear elastic zone of the stress-strain curve. There is minor lateral expansion in grout E. For the 25 mm grout C specimen, following yield and the accompanying reduction in stress, there is an incremental increase in stress, but not beyond the peak stress seen at yield. This is due to the meeting of the failure wedges causing prolonged strain. The cracks in the specimens of grout E are randomly oriented with no visible wedge.

Table 3. Summary of mechanical and thermal properties of grouts after hot/wet conditioning

	Properties	C 25 mm	E 25 mm
Compressive	Compressive strength (MPa)	93.10 (2.47)	45.23 (1.85)
	Compressive modulus (GPa)	0.418 (0.030)	0.559 (0.024)
	Strain at peak stress (mm/mm)	0.318 (0.004)	0.111 (0.004)
Tensile	Tensile strength (MPa)	12.22 (1.26)	1.29 (0.111)
	Tensile modulus (GPa)	0.381 (0.075)	0.029 (0.004)
	Strain at peak stress (%)	3.05 (0.71)	6.44 (0.62)
Glass transition	Glass transition, T_g (°C)	39	42
	Tan δ peak, T_t (°C)	67	69

Values in the parenthesis are standard deviations.

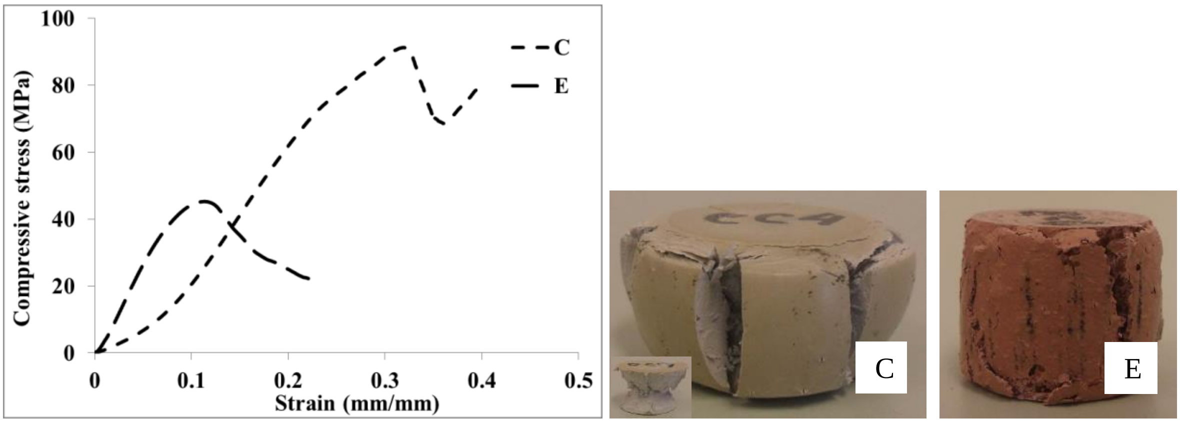


Figure 2. Typical compressive behavior; (a) stress-strain, and (b) failure pattern of the specimens

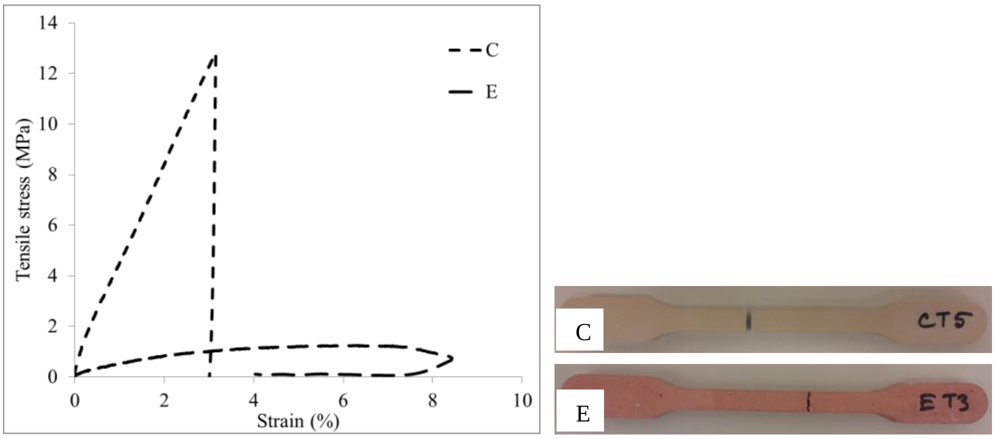
Tensile Properties

Table 3 provides a summary of the tensile strengths and elastic moduli of the investigated grouts. From the table it can be seen the highest tensile strength and stiffness of the investigated grouts for grout C are 12 MPa and 0.38 GPa, respectively. The lowest tensile strength and modulus for grout E are 1.29 MPa and 0.03 GPa, respectively. For a comparison, the tensile strengths and moduli at room temperature without conditioning were 32 and 19 MPa, and 4.9 and 16.5 GPa, respectively for the grouts C and E, respectively (Shamsuddoha et al. 2013b).

The comparison of the typical stress-strain behaviour for each type of grout is shown in Figure 3. Two distinct stress-strain relations are observed. Grout C exhibited a linear stress-strain relation and the highest strength. Grouts E transition to non-linear behaviour almost immediately upon loading, and exhibit very low yield strengths compared to grout C. The highest failure strain is observed in grout E. The formation of crack in grout C is sudden and a splitting sound is heard. Failures in grout E progress slowly compared to grout C and are not perfectly perpendicular to the length or straight along the thickness. The failure surfaces of grout C have a smooth appearance. The failure surfaces of grout E specimens appear jagged and the coarse aggregate fillers are visible.

Glass Transition Temperature

Table 3 provides a summary of the glass transition temperatures of the grouts. The T_g and T_t are found to be 39ºC and 42ºC for grout C and 59ºC and 69ºC for grout E, respectively. The onset of a rapid decline in the storage modulus provides a lower glass transition temperature measure than the Tan δ peak. Separate plots of the storage modulus and Tan δ vs. temperature are shown in Figure 4. The highest value of Tan δ peak was observed in grout E with a value of 69ºC which is about 2ºC higher than that of grout C.



(a) (b)

Figure 3. Typical tensile behavior; (a) stress-strain, and (b) failure pattern of the specimens

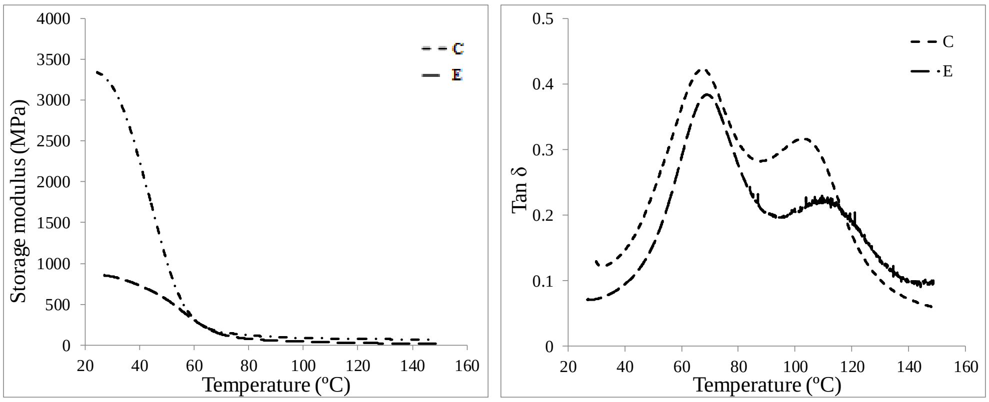


Figure 4. Thermal plots; (a) storage modulus vs temperature, and (b) Tan δ vs temperature

DISCUSSIONS

The comparison between the as-manufactured and hot/wet conditioned states suggests a considerable reduction of properties among the hot-wet conditioned specimens. The comparison is drawn by considering the mechanical and thermal properties for the grouts cured at 23ºC for 7 days, and tested at room temperature, in a previous publication (Shamsuddoha et al. 2013b), where tensile moduli were found to be 4.9 GPa and 16.5 GPa for grouts C and E, respectively. The dominant reduction occurs in the stiffness of the grouts. Tensile moduli of the hot-wet conditioned grout decrease by more than 90% compared to unconditioned grouts. The tensile strength of grouts is also reduced by more than 90%. The reduction in compressive strength is higher in the coarse aggregate filled grout E than that of the fine filled system. Hence, a coarse filled system is more susceptible to degradation under hot-wet conditioning. It can also be seen that debonding of aggregate and matrix is smooth, indicating that interface bonding between aggregate and matrix is weaker than the particle strength of the aggregate. Again, since the matrix itself splits, the resin matrix is weaker than the aggregate particle strength. This implies that hot-wet conditioning reduced the strength of the matrix-grout interface in each of the grouts.

The T_g of the conditioned grouts C and E is found to be about 39ºC and 42ºC, respectively. In contrast, T_t is measured at 67ºC, and 69ºC, for grouts C and E, respectively. The investigation by the authors on the thermal properties of these grouts without conditioning suggested that the T_g of these grouts ranged from 50 – 60ºC (Shamsuddoha et al. 2013b), indicating that hot-wet conditioning reduced the glass transition temperatures. According to ISO/TS 24817 (2006), the service temperature of a repair component for non-leaking (Type A) and leaking (Type B) pipes should not be 20ºC and 30ºC less than the as manufactured glass transition temperature, respectively. It is evident that the conservative glass transition assignment approach used here, taking the onset of decline in the storage modulus, is a more appropriate approach than taking the peak of the Tan δ curve (T_t). Hence, for the purposes of pipeline repair and rehabilitation, it is wiser to consider the conservative approach for taking the onset of decline in the storage modulus for glass transition temperature.

CONCLUSIONS

Two grouts (C and E) were tested for mechanical and thermal properties. Grout E was added with additional filler to grout C. The grouts were hot-wet conditioned for 1000 hours at 70ºC. Compressive, tensile, and glass transition properties were determined.

Aggregate filled grout experiences higher reduction in compressive properties than that of fine filled grout due to hot-wet conditioning. More than 90% of the compressive modulus is found to be reduced when tested elevated temperature after hot/wet conditioning, compared to the unconditioned specimens tested at room temperature. Tensile strength and stiffness decrease by more than 90% compared to the unconditioned specimens, except the strength of grout C which reduced by 62%.

The T_g values provide a conservative, however appropriate, measure of the glass transition temperature the investigated grouts. The large reduction of strength and stiffness is due in part to the tests being performed in close proximity to the glass transition temperatures. Hence, for the purposes of pipeline repair and rehabilitation, it is more appropriate to consider the conservative approach for taking the onset of decline in the storage modulus for glass transition temperature.

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