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Comparison study of dicyandiamide-cured epoxy bonded steel joints and polyamidoamine-cured epoxy bonded steel joints

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Abstract

There are instances where efficiency and safety may be compromised as a result of deteriorating fluid transport systems. Thus, it is worth evaluating other methods that can repair the damage for a temporary period without shutting down the operation. The objective was to evaluate the durability of an epoxy-bonded steel in aqueous environments that would represent such a repair. $EPON^{\circ}$ 828 was chosen as the epoxy resin, and dicyandiamide and polyamidoamine were two types of curing agents evaluated in this study. The epoxy-bonded steel joints were exposed in either distilled water or 3.4% NaCl solution for various times. The mechanical strength of the bonded joints was evaluated using a three-point flexure test. The interfacial shear strength of unaged samples ranged from 0.93 to 0.32 MPa. It was found that the interfacial shear strength decreased with aging time for both epoxy-bonded systems. Scanning electron microscopy (SEM), optical microscopy, and X-ray photoelectron spectroscopy (XPS) were used to determine the locus of failure of the bonded joints. It was concluded that failure occurred cohesively within the oxide layer if oxides were present on the substrate surface prior to the bonding procedure. \odot 2000 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Metals are often the materials of choice because of their excellent mechanical properties and durability. However, the performance of metals can be greatly compromised by corrosion. Corrosion is defined as the deterioration of metals by environmental factors [1,2]. The service lifetime of most structures, from bicycles to bridges, from buckets to battleships, is limited by wet corrosion [3]. It has been documented that heat exchangers tubed with nickel alloys and low-carbon steel have been seriously damaged as a result of corrosion [4]. When a pipe corrodes in a fluid transport system, the plant is shut down to inspect and repair the damage. If the damage is not severe, welding is the preferred repair method, and the defective part will have to be eventually replaced. While the plant is shut down, there is a loss of production time and labor and materials costs associated with the repair [2]. In addition to the expense, there is a certain level of danger involved with welding. The pipe

might contain reactive fluid that could escape the pipe through the damage site. The complexity and difficulty of welding increase dramatically when the defective pipe is part of an underwater fluid transport network. The costs of materials and labor involved in repairing the damage also escalate as welding is performed underwater. As more fluid transport systems are constructed offshore, there is an urgent need for an alternative method to repair defective piping networks.

An alternative repair method that uses an adhesive could be a logical choice because of the simplified application procedure. Although adhesive bonded joints may not be as durable as welded ones, the use of an adhesive is an excellent temporary repair method to address this problem without shutting down the plant while a new part is being constructed. There is considerably less risk connected with using an adhesive than with welding. There is also no need for special training with an adhesive because of the convenience in bonding procedures. Some research has been performed by the Admiralty Research Establishment (UK) using adhesives to repair steel structures offshore in the North Sea [5]. Before adhesives can be used in such a repair, the effect of

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environment on the mechanical properties of the bonded joints needs to be studied extensively to understand how adhesively bonded joints behave in an underwater environment. The objective of this work was to study how environment affected the mechanical properties of epoxy-bonded joints for possible use in underwater pipe repair.

We made use of a three-point flexure configuration incorporating a cast resin rib on a substrate for our testing mode. The overall goal was to evaluate the strength of the interface as a function of environmental conditioning. It was felt that this configuration, while not leading to a pure shear mode, would be sensitive to structural changes associated with the softening of the rib and interphase region with water absorption. Other studies have made use of this type of testing configuration to determine the influence of surface chemistry on adhesion $[6-8]$.

2. Experimental

2.1. Materials

 $EPON^{\circledast}$ resin 828, an undiluted difunctional bisphenol A/epichlorohydrin derived liquid epoxy resin was obtained from Shell Chemical Company. Two curing agents were used in this study. One was dicyandiamide (DICY), which was used in conjunction with *N*, *N*-dimethyl formamide (DMF) and 2-methylimidazole. The other was EPI-CURE® 3090, a modified polyamidoamine adduct and blush-resistant epoxy curing agent, capable of curing epoxy resin at and below room temperature on damp or underwater substrates, including concrete and steel [9]. Two types of low-carbon alloy steel were studied, with chemical compositions listed in Table 1. The steel used with the EPON® 828/DICY system was supplied by the U.S. Navy Coastal Systems Command, Panama City, FL, and the steel used with the EPON® 828/EPICURE® 3090 system was supplied by McMaster-Carr (New Brunswick, NJ).

2.2. Sample preparation

The steel bars had visible corrosion products on the surface prior to any surface pretreatment, and three surface pretreatments were performed before the bonding procedure with the EPON® $828/DICY$ mixture. Method I scrubbed the surface with soap and water, method II sanded the surface with 220 grit sandpaper, and method III sandblasted the surface with glass beads to remove the visible corrosion products. A silicone mold with cutouts for adhesive was constructed and used to fabricate the epoxy-bonded joints. Fig. 1 illustrates the top view of the mold with the shaded area representing the cutouts of the mold. The surface pretreated steel bars Table 1

Elemental composition of steel (Steel 1: used with EPON® resin 828/DICY, Steel 2: used with EPON® resin 828/EPI-CURE® 3090)

Element	Steel 1 $(\%)$	Steel 2 (%)	Element	Steel 1 (%)	Steel 2 $(\%)$
C	0.043	0.17	Co	0.003	0.014
Mn	0.25	0.79	Cu	0.03	0.17
P	0.011	0.022	Al	0.05	0.035
S	0.006	0.030	B	0.001	0.01
Si	0.02	0.15	W	0.001	< 0.01
Ni	0.02	0.17	As	0.007	0.023
Cr	0.04	0.20	Sn	0.004	0.014
Mo	0.01	0.05	Zr	0.001	0.002
V	0.001	0.002	N	0.005	0.013
Nb	0.002	N/A	O	0.002	0.001
Ti	0.001	0.002			

Fig. 1. Top view of the silicone mold.

were sealed onto the bottom of the mold with a silicone sealant to prevent resin leakage. DICY of 4 wt% was dissolved in DMF in a warmed beaker. The solution was mixed until it was free of any visible solid DICY particles. After the curing agent was completely dissolved in the solvent, 0.5 wt\% of 2-methylimidazole was added as a catalyst to the solution. The resin was then poured into the mixture of curing agent, solvent and catalyst. The mixture was poured into the cutouts of the mold. The mold was placed in an oven at 50° C for 12 h to evaporate the DMF. Teflon films and aluminum weights were placed on top of the mold to insure uniform sample thickness. The oven temperature was raised to 180° C at 1° C/min and the temperature was held at 180° C for 1 h to cure the epoxy resin. The oven temperature was then lowered to room temperature at a cooling rate of 1° C/min to avoid thermal shock of the samples. After the epoxy was cured, coupons were extracted from the mold. The epoxy on the steel had average dimensions of 77 mm in length, 5 mm in thickness, and 5 mm in width.

There was no visible corrosion on the steel surfaces prior to the bonding procedure with the EPON® 828/EPI-CURE® 3090 mixture. Two surface pretreatment methods were performed. Method I wiped the steel surface with acetone to remove contamination off the

Fig. 2. Three-point flexure geometry for epoxy-bonded steel.

surface. Method II submerged the steel bars in a 3.4% NaCl solution bath at 50° C for one month to yield corrosion products on the surface. The surface-pretreated steel bars were sealed onto the bottom of the mold with a silicone sealant to prevent resin leakage. $EPON^@$ 828 and EPI-CURE® 3090 were mixed in a 1 : 1 weight-mix ratio with a mechanical stirrer to insure the complete mixing of the two components. The mixture was then degassed in a centrifuge and was poured into the cutouts of the silicone mold. Teflon films and aluminum weights were placed on top of the mold to insure uniform thickness of the samples. The mold was then immersed in a water bath, and the adhesive was cured at 65° C for 24 h. The bonded steel bars were then removed from the mold. The epoxy rib had average dimensions of 70 mm in length, 8 mm in thickness, and 4 mm in width.

2.3. Aging experiment

The aging program consisted of several sample types: neat epoxy bars and epoxy- bonded steel with different surface treatments. The sample groups were placed in separate plastic bottles filled with the aging solution, and they were set in a water bath at 50° C. The aging environments were distilled water and 3.4% NaCl solution, and the aging time ranged from 1 to 4 weeks. Sea water contains approximately 3.4% salt, predominantly sodium chloride [10]. Following the aging exposure, the samples were blotted dry and tested.

2.4. Three-point flexure

The mechanical properties of the epoxy-bonded joints were evaluated with a three-point flexure configuration at room temperature. The testing geometry is shown in Fig. 2. An epoxy-bonded specimen was set on the two supports with the adhesive facing down, and a load was applied midway between the supports. The distance between the supports was 90 mm for the EPON[®] $828/DICY$ -bonded steels and 89 mm for the EPON® 828/EPI-CURE® 3090-bonded samples. The cross-head speed was set at 2.5 mm/min for the EPON® 828/DICYbonded steels and 4.5 mm/min for the EPON® 828/EPI- $CURE^{\circledast}$ 3090-bonded samples. Under the loading condi-

Fig. 3. A typical force-deflection curve from a three-point flexure test of an epoxy-bonded joint.

tions, the epoxy-bonded joint can be viewed as a beam with a stiffening rib. This composite structure bends as a stiffer member than the substrate alone, when the epoxy is bound to the substrate. Stress increases at the bond line with continued bending, and eventually, delamination of the epoxy from the substrate is observed. After delamination, the applied load decreases and approaches that of the substrate deflection alone. The interfacial shear strength, τ_{xy} based on this configuration, can be determined using simple composite beam theory having the form

$$
\tau_{xy} = \frac{F[h_a^2 - 2y_2h_a]}{4[(E_s/E_a)I_s + I_a]}.
$$

In this equation, F is the applied force, h_a is the thickness of the adhesive stiffening rib, Y_2 is the distance from the neutral axis to the interface, E_s , and E_a are the elastic moduli of the substrate and adhesive as a function of environmental conditioning and I_s and I_a are the moments of inertia of the substrate and adhesive, respectively.

The goal of this test was to identify the interfacial shear strength of a bonded joint; therefore, the test was performed until the epoxy delaminated from the substrate, recording the force at delamination. A force-deflection curve was obtained for each specimen and a representative one is shown in Fig. 3. The force at delamination and sample geometry were used in this analysis.

2.5. Failure analysis

The failure mode of the EPON® $828/DICY$ -bonded steels was determined by optical and scanning electron microscopy on the failure surfaces. The failure surfaces of the EPON® $828/EPI-CURE@ 3090-bonded samples$ were examined with XPS. The samples were attached to the sample mount with double-stick tape and were degassed in vacuum at 115° C overnight prior to the analysis. The XPS spectra were obtained with a Perkin-Elmer PHI model 5400 photoelectron spectrometer, equipped with a Mg K_{α} source. The spot size was 1 mm \times 1 mm. Each sample was irradiated at an angle of 45° with

Fig. 4. Average interfacial shear strengths of EPON® 828/DICYbonded joints in distilled water (the error bars represent \pm one standard deviation).

Fig. 5. Average interfacial shear strengths of EPON® 828/DICYbonded joints in 3.4% NaCl solution (the error bars represent \pm one standard deviation).

respect to the entrance to the electron analyzer. Areas under the peaks were used to determine the atomic concentrations.

3. Results

The shear strength results for the EPON® 828/DICYbonded joints that were aged in distilled water and in 3.4% NaCl solution are shown in Figs. 4 and 5, respectively and are summarized in Table 2. The maximum value calculated for axial stress of epoxy at failure was 34 MPa. The reported value for the tensile strength of the cured epoxy resin is 70 MPa [11]. Thus, the axial stress in the resin when the interface failed, was less than its tensile strength. This calculation confirmed the observation that the failure mechanism of the epoxy-bonded joints resulted from delamination of the epoxy from the Table 2

Shear strength results for EPON® resin 828/DICY-bonded joints

Environment	Exposure time (weeks)	Shear strength (MPa)			
surface treatment: soap and water scrubbed					
Control	0	0.93			
Distilled water	1	0.46			
Distilled water	2	0.05			
3.4% NaCl solution	1	0.08			
3.4% NaCl solution	\overline{c}	0.03			
Surface treatment: 220 grit sandpaper					
Control	$_{0}$	0.37			
Distilled water	1	0.14			
Distilled water	\mathfrak{D}	0.06			
3.4% NaCl solution	1	0.11			
3.4% NaCl solution	\overline{c}	0.03			
Surface treatment: sand blasting					
Control	$_{0}$	0.32			
Distilled water	1	0.12			
Distilled water	\overline{c}	0.08			
3.4% NaCl solution	1	0.12			
3.4% NaCl solution	2	0.03			

substrate. If the axial stress had exceeded the tensile strength, the failure would have resulted from the epoxy fracture in tension.

The average shear strength of the control group with steel scrubbed with soap and water was calculated to be 0.93 MPa. This value decreased to 0.46 and 0.05 MPa after one and two weeks of exposure in distilled water, respectively. After similar aging times in 3.4% NaCl solution, the average shear strength decreased more precipitously to 0.08 and 0.03 MPa. After just one week of aging in either environment, the bonded joints had lost most of their initial shear strength. It was observed that the bonded joints performed better in distilled water than in 3.4% NaCl solution.

The average shear strength of the control group with the sanded steel at the interface was 0.37 MPa. The average shear strength decreased to 0.14 and 0.06 MPa after one and two weeks of exposure in distilled water. With similar aging in saltwater, shear strength was reduced to 0.11 and 0.03 MPa. The bonded joints had again lost most of their original strength with the residual strength of the bonded joints still higher in distilled water.

The average shear strength of the control group with the sand-blasted substrate was 0.32 MPa. The average shear strength decreased to 0.12 and 0.08 MPa after one and two weeks of aging in distilled water. In saltwater shear strength was reduced to 0.12 and 0.03 MPa. It was again observed that the bonded joints lost most of their initial shear strength during the aging cycle and the saltwater was more detrimental to joint strength.

Fig. 6. Average interfacial shear strengths of EPON® 828/ EPI- $CURE^{\circ}$ 3090-bonded joints in distilled water at 500C (the error bars represent \pm one standard deviation).

Fig. 7. Average interfacial shear strengths of EPON® 828/ EPI-CURE® 3090-bonded joints in 3.4% NaCl solution at 500C (the error bars represent \pm one standard deviation).

The shear strength results for the EPON® 828/EPI- $CURE^{\circ}$ 3090-bonded joints, aged in distilled water and in 3.4% NaCl solution, are shown in Figs. 6 and 7, respectively, and are summarized in Table 3. The maximum axial stress in the resin as the sample failed was 14 MPa, less than the resin tensile strength of 70 MPa $[11]$. This further confirmed the observation that failure resulted from delamination of epoxy from the substrate. If the axial stress exceeded the tensile strength, the epoxy would have failed in tension instead of delaminating from the substrate.

The average shear strength of the control group whose substrate was acetone-wiped was 0.43 MPa. The average shear strength decreased to 0.14, 0.11, and 0.09 MPa after one, two, and four weeks in distilled water. Shear strength was reduced to 0.15, 0.14 and 0.07 MPa after one, two and four weeks of aging in 3.4% NaCl solution. There is no significant difference in the joint shear strength between the two environments.

Table 3

Shear strength results for EPON® 828/ EPI-CURE® 3090-bonded joints

Environment	Exposure time (weeks)	Shear strength (MPa)
Surface treatment: acetone wiped		
Control	0	0.43
Distilled water	1	0.14
Distilled water	2	0.11
Distilled water	4	0.09
3.4% NaCl solution	1	0.15
3.4% NaCl solution	\overline{c}	0.14
3.4% NaCl solution	4	0.07
Surface treatment: corroded		
Control	θ	0.47
Distilled water	1	0.19
Distilled water	\mathfrak{D}	0.15
Distilled water	4	0.12
3.4% NaCl solution	1	0.20
3.4% NaCl solution	\mathfrak{D}	0.18
3.4% NaCl solution	4	0.10

The average shear strength of the control group for the corroded substrate was calculated to be 0.47 MPa. The average shear strength decreased to 0.19, 0.15 and 0.12 MPa after one, two and four weeks of aging in distilled water and declined to 0.20, 0.18 and 0.10 MPa after similar times in saltwater. The difference in shear strength loss was not statistically significant between the environments.

The failed surfaces of the EPON® 828/DICY-bonded joints were examined by optical microscopy. It was observed that the failure occurred cohesively within the oxide layers. The failure surfaces of the $EPON^{\circledast}$ 828/EPI-CURE® 3090-bonded samples were also analyzed by XPS. The results are shown in Fig. 8 for the epoxy-bonded acetone-wiped substrate and Fig. 9 for the epoxy-bonded corroded substrate.

4. Discussion

Shear strength decreased with exposure time for all the epoxy-bonded joints. There is a corresponding reduction in the axial stress in the resin at failure, primarily due to the reduction in the interfacial shear strength at the edge with further aging. For the EPON® 828/DICY-bonded joints, the bonded joints formed from the soap-and water-scrubbed substrate had the highest shear strength initially and after one week of exposure in distilled water. However, the difference in shear strength between the surface pretreatment methods was not statistically significant after two weeks of exposure in distilled water or after one or two weeks in saline. The higher initial shear strength of the scrubbed substrates that were bonded resulted from the rougher surface morphology of the

Fig. 8. XPS results for EPON® 828/ EPI-CURE® 3090 bonded on acetone-wiped steel.

Atomic Concentration

Fig. 9. XPS results for EPON® 828/ EPI-CURE® 3090 bonded on corroded steel.

oxide layer on the steel. Minford has reported that the loose oxide layers are susceptible to interfacial failure when the bonded joints are under stress, impact or weathering conditions [12]. If the oxide layers were loosely bound, this would explain why there was no significant difference in the shear strength between the surface pretreatment methods after exposure in distilled water and saline solution.

For the EPON® 828/EPI-CURE® 3090-bonded joints that were aged at 50° C, the bonded joints with the corroded substrate surface had the higher shear strength initially and throughout the aging cycle in both aging environments. The oxide layer of the corroded surface was formed by immersing the steel with no visible corrosion products in a 3.4% NaCl solution immediately before the bonding procedure. The newly formed oxide layer contributed to the higher strength of the bonded joints because of its high surface roughness and chemical reactivity. Minford also reported this observation [12]. The SEM micrographs of the two steel surfaces in Figs. 10 and 11 showed that the corroded surface appeared to be rougher than the substrate that was wiped with acetone. It is shown that the scrubbed joints that

were subsequently bonded experienced the greatest loss in the shear strength in saline. The scrubbed steel surface had loose oxide layers on the steel surface. Although the oxide layer contributed to the high shear strength initially, the NaCl solution caused the interfacial failure of the bonded joints. Minford suggested that the loose oxide layer on the substrate surface can lead to bonded joint failure under severe weathering conditions [12]. The sand blasted steel that was subsequently had the least loss in the shear strength in distilled water and saline. Leidheiser said that oxide layer removal may be necessary for longer service lifetime of bonded joints [13]. The elimination of the oxide layer by sand blasting stabilized the bonded joints in the aqueous environments and prolonged the service lifetime.

The bonded joints formed from the corroded steel substrate had the least loss in the shear strength in distilled water and in 3.4% NaCI solution. The oxide layer on the substrate surface was formed immediately before the adhesive bonding and was higher in roughness and chemical reactivity [12]. The oxide layer on the substrate surface led to the greater mechanical integrity of the bonded joints.

Fig. 10. SEM of acetone-wiped steel before bonding.

Fig. 11. SEM of corroded steel before bonding.

One additional consideration for the lower shear strength of the stiffened construction is softening of the rib due to the water absorption. The reduction in the stiffness of the rib would reduce its capacity as a stiffener and this would be observed in the force-deflection curve. The stress at failure would incorporate the reduced stiffness of the saturated resin and would not change the failure mode of the rib during deformation.

SEM and optical microscopy were used to examine the failure surfaces of the $EPON^{\circledast} 828/DICY$ -bonded joints. Oxide particles were observed by optical microscopy on the epoxy failure surface, and no epoxy was found on the steel failure surface according to the SEM micrographs. It was also observed that oxide was present uniformly on the epoxy failure surfaces by visual inspection. Thus, it is concluded that the failure occurred cohesively within the metal oxide layer. XPS was used to analyze the failure surfaces of the EPON® 828/EPI-CURE® 3090-bonded joints. It was found that oxide particles, such as $Fe₂O₃$ and FeO, were found on the epoxy failure surface that was bonded to corroded steel. On the corroded steel failure surface, $Fe₂O₃$ were present. The epoxy-bonded joints with the corroded steel had failed cohesively within the metal oxide layer because of the oxides present on the epoxy failure surface. Gledhill and Kinloch had also observed adhesive failure of the iron-epoxy system that was exposed under wet conditions [14]. For the bonded joints containing steel wiped with acetone, no oxides were seen on the epoxy failure surface, and no nitrogen which is unique in the epoxy system, was seen on the steel side. It was concluded that failure also occurred adhesively.

It was observed that failure always occurred cohesively within the metal oxide layers if oxides were present on the substrate prior to the bonding procedure for both resin systems under all experimental variables. The oxide layers are thought to be the weakest component of the bonded joints; therefore, failure should take place there. This suggests that optimum substrate surface morphology is essential in order for the service lifetime of a formed joint. The substrate surface characteristics are the limiting factors in the performance of the bonded joints. Therefore, a proper surface pretreatment procedure is crucial for a bonded joint to maintain the appropriate mechanical strength throughout its service lifetime.

5. Conclusions

Water diffuses through the interface between the epoxy and the steel substrate resulting in a weaker bond. It is easier for moisture to diffuse along the interface than through the bulk of the epoxy because of the thickness of the epoxy rib. Since the oxide layers are commonly the weakest region in a bonded joint, the oxide layers would experience the greatest loss in strength in a severe environment. In a bonded joint, the epoxy is bonded to the oxide layer. If the oxide layer is compromised by the environment, so is the interfacial strength of a bonded joint. It has been shown that the interfacial shear strength of the bonded joints decreases with exposure time.

The saline solution was harsher in affecting the shear strength of the 828/DICY-bonded joints. When NaCl

solution diffuses into the bondline, redox reactions occur dissolving the oxides, resulting in less bonded area.

It would be important to predict the service lifetime of the bonded repair. It was found that the $EPON^{\circledast}$ 828/DICY-bonded joints with the soap-and water-scrubbed steel substrate suffered the greatest loss in shear strength, up to 97% after two weeks of exposure in the NaCl solution. This was due to the presence of loosely bound oxide layers, that were susceptible to environmental attack, prior to the bonding procedure. The EPON 828/ DICY-bonded joints had lost at least 50% and up to 97% of their original strength after only one week of exposure. The realistic lifetime of an EPON 828/DICY-bonded joint is about one week, and sand blasting would be recommended as preferred method for surface treatment.

It was observed for the EPON® 828/EPI-CURE® 3090-bonded joints with the corroded substrate that there was a 57% loss in the shear strength after one week of exposure in a NaCl solution. The bonded joints containing the acetone-wiped substrate had lost 65% of their original shear strength after one week of exposure in a NaCl solution. Therefore, it is suggested that protective oxide layers should be formed just before the bonding procedure to ensure longer lifetime of a bonded joint. The $EPON^@ 828/EPI-CURE^@ 3090-bonded joints with the$ corroded steel lost 60 and 77% of their original shear strength after two weeks and four weeks of exposure in the NaCl solution, respectively. It is estimated that the service lifetime of an EPON[®] 828/EPI-CURE[®] 3090bonded joint with corroded substrate is up to two weeks.

The surface analysis results show that failure always occurred cohesively within the oxide layers if oxides were present on the substrate surface prior to bonding. This observation can be made for both resin systems and for all experimental variables, such as exposure time and environments. Thus, the oxide layers are the weak components in the bonded joints, and an optimum substrate surface morphology is critical to the performance of the bonded joints. If the loosely bound oxides are not removed before bonding, the lifetime and the strength of these bonded joints is more limited. However, some mechanical interlocking between the adhesive and the substrate is essential to optimize the mechanical strength of the bonded joints. It has been shown that a substrate with a rougher surface morphology allows for better mechanical interlocking with an adhesive than one that is relatively featureless in its surface morphology. It has

also been shown that the steel substrate with oxide layers formed shortly before the bonding procedure has rougher surface morphology than one that was polished. Therefore, loosely bound oxides should be removed and that a protective oxide layer should be formed prior to bonding to ensure maximum lifetime and performance of the bonded joints.

The use of an adhesive as a temporary underwater pipe repair method is a promising possibility. However, more studies are needed to understand the failure mechanisms of the bonded joints in more depth, to prolong the lifetime of the bonded joints, and to predict the lifetime of the bonded joints more accurately.

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