The Interfacial Properties of a Novel Concrete Damping and Protective Material

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ABSTRACT: One of the most important factors for the application of coatings on infrastructure is their interfacial performance. The paper presents an experimental study on the influence of adverse environmental circumstances in the interfacial performance of a coated mortar. For the purpose, a damping and protective material was applied a mortar substrate and the saturated Ca(OH)₂ solution, the salt-spray and the static bending load were independently and collectively applied on the coated mortar specimens. The adhesion strength and the interfacial micro-deformation of the coating are determined. SEM and FTIR were employed to characterize the interfacial morphology and analyze the cohesive mechanism between the coating and the mortar.

Test results lead to the conclusion that the adhesion strength of the coating is not significantly influenced by the corrosive environment. The adhesion strengths of specimens are above 1MPa under 20d of the immersion of saturated Ca(OH)₂ solution, the co-action of bending load and saturated Ca(OH)₂ solution and the co-action of bending load and salt-spray, respectively. Meanwhile, the interfacial strain of the coating-substrate varies according to different experimental circumstances and it was affected most by the coaction of bending load and saturated Ca(OH)₂ solution. Under the coaction of bending load and saturated Ca(OH)₂ solution, the ultimate interfacial strain in the center decreases by at least 11 compared to controlled specimens, while the ultimate interfacial strain decreases by at least 7 under the co-action of bending load and salt-spray.

KEYWORDS: Interfacial properties, adhesion strength, interfacial strain, damping and protective coating

INTRODUCTION
Polyurea material is widely used for concrete and steel protection in civil engineering structures because it offers high resistance against chemical and physical aggressions, good anti-aging ability and even effective containment of fragmentation during concrete failure. (Chi, Li, Zhou, Zhao, Chao and Wang, 2015; Toutanji, Choi, Wong, Gilbert and Alldredge, 2013). The latest development for this material is to apply it for both structural protection and vibration or noise reduction because it is demonstrated to have high damping properties as well (Lu, Liu, Ma and Huang, 2011).
However, to take full advantage of this multi-functional material, the adhesive ability of the coating with substrates and the adhesion loss under environmental influence should be taken into account. If the coating bonds badly to the substrate, it could result in the reduction in mechanical properties and service life and the coating failure (Bajat, Milošev, Jovanović and Mišković-Stanković, 2010).

Many factors would influence the adhesive properties of the coating. Material data or parameters, such as the chemical structure and the degradation of the coating, the adhesive layer thickness and the intrinsic porosity and roughness of substrates, are of great importance to the adhesive ability (Ali, Duan, Jiang, Goh, Lamb, Tadich, Poinern, Fawcett, Chapman and Singh, 2014; Alia, Arenas, Suárez and Pinilla, 2016; Baltazar, Santana, Lopes, Correia and Rodrigues, 2014; Baltazar, Santana, Lopes, Paula Rodrigues and Correia, 2014; Dong, Wang and Su, 2014). Exterior environment, such as temperature, relative humidity and load, is also a huge factor for the bonding between the coating and the substrate, and the influence of the environment varies according to the dependence of materials on temperature and humidity (Banea and da Silva, 2009; Baldan, 2012; Cambier, Posner and Frankel, 2014; Zheng, Lin, Wang, Zhu and Wu, 2015).

As regard to the bond characteristics of the coating, many studies were focused on interfacial conditions and their effects on the adhesive ability.

There are also a number of studies focused on the bonding laws between the coating/adhesive and the substrate. For a thermal spray coating, such as polyurea, the origin of adhesion strength could be classified into (a) mechanical interlocking effect in the coating–substrate interface; (b) adhesive effect of intermediate or bonding layer; (c) inter-diffusion effect in a thicker interfacial region between the coating and the substrate and (d) interfacial adhesion in which the adhesive forces are centered around a well-defined thin interface (Hass, Wittel and Niemz, 2013). The mechanical interlocking is the primary mode for coating adhesion, inter-diffusion and interfacial adhesion is also important to the coating adhesion (Chen, Zhou, Lu and Lam, 2014). And the use of intermediate layer also has a significant effect on the bonding. Ping Lu et al. used an epoxy primer and a polyurethane primer on concrete surfaces before applying polyurea, investigated their thermal stresses and found that the polyurethane primer had a lower thermal stress than the epoxy primer at low temperature, and the cohesive property would be more difficult to be influenced by temperature variation. (Lu, Zhu, Zhang and Huang, 2012).

In this study, the adhesion strength and interfacial strain of polyurea coated mortars in three different experimental circumstances were investigated, the interfacial morphology and the chemical bonds related to bonding ability of the coating were observed with the scanning electron microscope (SEM) and the Fourier Transform Infrared Spectroscopy (FTIR), and finally the influence of these circumstances and the cohesive mechanism were analyzed.
**EXPERIMENTAL**

**Materials and sample preparation**

The interfacial properties between polyurea coating and concrete substrate were investigated in this study. The two components of polyurea coating, which has been employed for the protection of actual marine structure, were provided by Qingdao Shamu International CO., Ltd.. Prior to coating application, mortar specimens were made and used as substrate. Mortar specimens with the size of 40mm×40mm×160mm were fabricated and then maintained for 28d according to GB/T 17671-1999. The composition is shown in Table 1. After curing, strain gages adhered to the surface of one side of the mortar blocks at specific positions and one wire was attached to each of them. The locations and directions of strain gauges are shown in Fig.1. Afterwards, a homogeneous polyurea mixed with component A and component B was brushed on each surface of the mortar specimen to form uniform coatings with the thickness of 2mm. The coated specimens were placed in the ambient environment for 24h. 16 coated specimens were fabricated and uniformly divided into 4 groups (A, B, C, D).

![Figure 1. The positions and directions of strain gauges.](image)

(Point 1, point 2, point 3 and point 4 refer to strain gages in marginal area, and point 5 and point 6 refer to strain gages in the center; Point 1, point 3 and point 5 refer to strain gauges along axis x, and point 2, point 4 and point 6 refer to strain gauges along axis y).

<table>
<thead>
<tr>
<th>Items</th>
<th>P·O 42.5 cement</th>
<th>ISO standard sand</th>
<th>Water</th>
<th>Component A of polyurea</th>
<th>Component B of polyurea</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mortar blocks</td>
<td>20</td>
<td>11</td>
<td>18</td>
<td>/</td>
<td>/</td>
</tr>
<tr>
<td>Coating</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>11</td>
<td>10</td>
</tr>
</tbody>
</table>

**Methods for Preparing Environmental Conditions**

To investigate the effects of different environmental conditions on the interfacial properties of coated mortars, three conditions were studied: the saturated Ca(OH)$_2$ solution immersion, the co-action of bending load and saturated Ca(OH)$_2$ solution immersion (hereinafter referred to the load-immersion) and the co-action of bending load and salt-spray (hereinafter referred to the load-salt-spray). The loading method is to impose a static and
vertical force on specimens by loading devices, which is made of thick steel panels, springs and threaded rods. The structure of the loading device is shown in FIG.2. The force, which can be adjusted by the deformation of the springs, was set as 30% of the compressive strength for the mortar specimens.

To make comparisons, group A was used as the controlled group and placed in the ambient environment. Group B was placed in the saturated Ca(OH)\(_2\) solution. Group C and D were both equipped with loading devices, and then were put into the saturated Ca(OH)\(_2\) solution and the salt-spray environment respectively. The salt-spray corrosion tester equipment YFX-750 (Longyue Instrument Equipment Co. Ltd., China) was used and the salty solution was prepared with 5.0 wt.% analytically pure NaCl and 95.0 wt.% distilled water to provide the neutral salt spray environment.

**Measurements for Adhesion Strength and Interfacial Strains**

After 20d, group B, C and D were taken out of and transferred to the same ambient environment as group A experienced for 2h. All the specimens are dried and cleaned with cloths. The interfacial strain of group A, C and D was monitored with the static strain tester model DH3816N (Donghua Testing Technology Co., Ltd., China) for 2h and the collected data was processed with Origin Software. The adhesion strength between the coating and the mortar substrate of group A, B, C and D was tested according to ISO 4624:2002 (Paints and varnishes — Pull-off test for adhesion), and the automobile portable adhesion tester model PosiTestAT (DeFelsko Corporation, America) was used.

![Figure 2. The schematic diagram of the static loading device.](image)

**Microstructure Observation**

The SEM model JSM-5600 (JEOL instrument, Japan) is used to examine the interfacial morphology of the cut section of specimens. Therefore, the specimens are placed on top of a circular platform, plated with gold and then examined at 100× and 2000× magnifications using an accelerating voltage of 25 kV.

FTIR measurements were performed using a Perkin Elmer Spectrum 100 instrument with an accessory for attenuated total reflection (ATR) (Perkin
Elmer Precisely, USA). FTIR spectra were then recorded with the resolution of 4 cm\(^{-1}\).

RESULTS AND DISCUSSION

The Adhesion Strength of Coated Mortars

For controlled specimen (group A) and specimen going through the Ca(OH)\(_2\) solution (group B), the load-immersion (group C) and the load-salt-spray (group D) respectively, the bonding ability of the interface between the coating and the mortar is characterized by the adhesion strength. The data is shown in Table 2.

Table 2. The Pull-off test results of specimens in various experimental circumstances for 20d.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Adhesion strength</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average value (MPa)</td>
<td>Standard deviation (%)</td>
<td></td>
</tr>
<tr>
<td>Group A</td>
<td>1.36</td>
<td>5.62</td>
<td></td>
</tr>
<tr>
<td>Group B</td>
<td>1.24</td>
<td>6.11</td>
<td></td>
</tr>
<tr>
<td>Group C</td>
<td>1.21</td>
<td>4.28</td>
<td></td>
</tr>
<tr>
<td>Group D</td>
<td>1.26</td>
<td>5.62</td>
<td></td>
</tr>
</tbody>
</table>

The adhesion strengths of group B, C and D for 20d are 1.24, 1.21 and 1.26 respectively, decreasing by 8.8%, 11.0% and 7.4% compared to controlled specimens. The results indicate that the bonding of the coating-substrate is influenced by Ca(OH)\(_2\) solution, bending load and salt-spray to some extent. Most polymeric materials are vulnerable to aqueous alkali. OH- ions migrate and gather to the interfacial area through holes in the coating and weaken the bonding force. Also, because of the bending load, micro-cracks occur and expanding on the surface of the mortar substrate and the interfacial area suffers from both tensile stresses and shear stresses, leading to a decline in adhesion strength. (Cui et al. 2014) It can be found from Table 2, the load-immersion coaction plays the most important role in deteriorating the adhesion strength. Salt-spray also influences the bonding property because the salty moisture can transfer to the coating-substrate interface as well. However, from the result, the effect of salt-spray is weaker than that of Ca(OH)\(_2\) solution.

Strain Analyses of Specimens in Various Experimental Circumstances

From the macroscopic level, the change in the interface can be shown in term of the variation of adhesion strength, while the micro-deformation in the interface can be detected by the strain variation in microscope level. The interfacial strains of group A are monitored and the results are shown in FIG.3. It can be found that strain gages are under shrinkage stresses and the shrinkage stresses in marginal area and in the center fluctuate from 34 to 41 and from 26 to 30, respectively. The strain curve of point 1 is not quite different from point 25 and that of point 5 is not quite different from point 6 as well, indicating that strains
led by extender environment are similar in different directions. In the following analyses of this study, the strains in axis x and in axis y are considered to be isotropic and point 1 and point 5 are selected to present the strain in all directions of the point.

The results show that the stains in the center keep stable and are slightly less than that in marginal area. This is because in the marginal area there is stress concentration which makes the flexible coating stretched or compressed, leading to fluctuation.

![Graph showing strain over time](image)

**Figure 3. Interfacial strain of group A.**

After 20d of the bending-immersion and the bending-salt-spray, group C and group D are unloaded and taken out above experimental environment, and then, they are moved to ambient environment. The strains of them are monitored and shown in FIG.4 and FIG.5 respectively.

It can be found from FIG.4 the strains in both marginal area and the center experience obvious changes. The marginal point suffers from a tensile stress, which decreases from 15 to 1 between 0s and 1430s and then increases to 15 again in 7000s. To the contrary, the central point undergoes a shrinkage stress, which rises from 0 to 23 before 2640s and then drops down to 15 in 7000s. When specimens are under the action of the load-immersion, both bending load and Ca(OH)$_2$ solution contribute to stresses in the coating, which leads to strains in the interface of the coating-substrate. After the specimens are taken out of above circumstance, the specimens are away from the bending load and the Ca(OH)$_2$ solution immersion and get into a relaxation process. In the process, stresses redistribute and result in strain fluctuation. The contrary trend of strains in the marginal area and in the center may be attributed to redistribution of stresses in the whole interfacial layer.
Figure 4. The strains of group C after 20d of the bending-immersion.

The strains in both marginal area and the center show a rising trend. The strains in the two areas start from about 5 to around -36 and -19 respectively between 0s and 5000s, indicating that the coating is recovering from the stretched state in this period. It can be deduced that the circumstance of the bending-salt-spray contributes to stretched stresses and strains in the interface. After 5000s, the strains in the two areas keep steady, indicating that the coating in a stable constrained state and the strains in this period should be intrinsic strains of the coated mortar after the bending-salt-spray. The strain in marginal area is larger than that in the center and the result coincides with controlled specimens. In addition, compared to controlled specimens, the interfacial strains in the center of specimens under the coaction of the bending-salt-spray decreased by at least 7. This may be because the coaction of the bending-salt-spray neutralizes part of the intrinsic strains in the central interface.

Analyses on Morphologies and Chemical Bonding

The SEM images of group A are shown in FIG.6. It can be seen from the
cross-section of the adhesive joint that the interface between the coating-substrate is bonded. However, from FIG. 6 a), holes in various sizes can be found in the internal structure of polyurea coating. These holes were mainly formed because of the introducing of air during casting. Component A reacted with component B to form a film on the surface of the mortar. When specimens were immersed in Ca(OH)$_2$ solution or in salt-spray, ions such as OH$, Cl^-$, Ca$^{2+}$ and Na$^+$ immigrated through holes to the interior of the coating and even to the interface between the coating-substrate. They could not only damage the chemical structure of the coating, but also weaken the adhesion between these two materials.

![Image](image1.png)  
**Figure 6.** The cross-section morphologies of the coating-substrate interface of controlled specimens.

To detect the chemical structure of polyurea coating, FTIR was employed and the infrared spectrogram is shown in FIG. 7. The spectrogram depicts the presence of vibrational bands in amidogen (~3360cm$^{-1}$), carbonyl (1600~1701cm$^{-1}$) and C-N or N-R (~1531cm$^{-1}$) stretching regions as well as bands between 2870 and 2966cm$^{-1}$ associated with the symmetric and asymmetric CH stretching vibrations of CH$_2$ and CH$_3$ groups. Above bands illustrate the presence of the functional group -NHCONH- or -NHCONR- in the prepared coating. The vibrational band near 1100cm$^{-1}$ relates the stretching vibration of the C-O-C group. Because of the presence of groups such as -NHCONH-, -NHCONR- and C-O-C, it is possible for hydrogen bonds to form by the reaction between them in the interfacial area, which contribute to the bonding between the coating-substrate.
Figure 7. The infrared spectrogram of the coating.

CONCLUSION

In this study, interfacial adhesion strengths between polyurea coatings and mortar substrates and strains in their interfaces in three experimental circumstances—the Ca(OH)$_2$ solution immersion, the load-immersion and the load-salt-spray—are investigated. SEM and FTIR are employed to characterize the interfacial morphology and analyze the cohesive mechanism between the coating and the substrate. The adhesion strengths are above 1.00MPa in all these three experimental circumstances, indicating they are not significantly influenced by the above circumstances. The load-immersion has the largest influence in the bonding between the coating-substrate. After taken out of the experimental circumstances, the strains in the interface may experience a relaxation process. After 20d action of the load-immersion the strains in marginal and center area experience obvious and contrary changes, which are deduced to be the result of redistribution of stresses in the interfacial layer. After 20d action of the load-salt-spray, the intrinsic strains in marginal area and in the center are around -36 and -19 respectively, and after above effects the strains in the center decreased by at least 7 compared to controlled group. The SEM results show the interface between the coating-substrate is bonded and the holes in various sizes provide tubes for OH$^-$, Cl$^-$, Ca$^{2+}$ and Na$^+$ immigration.

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