Experimental study of UV/water-spray coupled hydrothermal accelerated aging on fire resistive of intumescent coatings for steel structures

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Abstract. In this paper, the durability of fire resistive performance of two types of water-borne intumescent coatings was studied using UV/water-spray and hydrothermal accelerated aging. Exposure conditions Type X intended for all environmental conditions with reference to ETAG-018-2 was used as the aging method. The aging effects and mechanism were preliminary analysed using fire test, adhesive test and X-ray Fluorescence spectrometer (XRF). The results show that after 2 stages of aging (for the intended use of 10 years), type-B coating shows better durability while type-A coating losses its basic performance of fire resistive and adhesion. Visually assessment and XRF results show that the aging is mainly caused by surface cracking and the precipitation of fire resistive components.

1. Introduction

In recent years, steel structures have been widely used in commercial centres, stations, airports, exhibition halls, sports venues, immersed tunnels, large factories and other super high-rise or large-span public buildings. However, the strength of the steel structure drops sharply at 450 to 650 °C, so scientific and reliable fire protection is needed for steel structures. Among various forms of fire protection method, intumescent coating is particularly favoured as an effective way to provide passive fire protection for steel structure[1].

However, long-term exposure to environmental conditions can lead to a severe decline in the performance of intumescent coatings, which is generally caused by the precipitation of reactive materials or the degradation of polymers. Since fire safety requirement runs through the entire life cycle of a building structure, which may last for decades, it is of great significance to study the long-term protective performance of intumescent coatings under environmental conditions[2].

At present, there are very few reported researches on the aging performance of intumescent coatings for steel structures, especially on the fire resistive after accelerated aging. Jimenez et al. studied the fire resistance loss of an epoxy-based intumescent coating after a series of hydrothermal, immersion and salt bath aging[3, 4]. The results showed that the fire resistance of the coating decreased most seriously after salt bath aging, and the intumescent coating almost lost its expansion ability. Sakamoto et al. used Xenon-arc lamps to simulate the outdoor high-humidity climate conditions in Japan, constructed a new aging test method, and used this method to study the durability of several commercial intumescent coatings[5]. Wang et al. studied the effect of UV/water-spray accelerated aging on the performance of APP-PER-MEL nano-expansion fireproof coating[6, 7]. The results showed that the fire resistance and thermal insulation of the coating decreased significantly after 500 h accelerated aging test. By adding
nano-TiO₂ into the coating, the UV aging resistance of the material was improved obviously. However, aging conditions of most previous works were relatively simple, by which the comprehensive aging performance of coatings cannot be proved.

In this paper, the durability focus on fire protection performance of two intumescent coatings for steel structures were studied and the aging effects were analysed. Two stages of aging method have been conducted including UV/spray and hydrothermal intended for all environmental conditions. Fire tests and adhesive tests were conducted to characterize the effect of basic performance during the aging progress. In addition, the aged intumescent coatings were subjected to XRF to see the surface element composition. The test results were associated with performance changes and the degradation processes were preliminary analysed.

2. Experimental

2.1. Materials

Primer: GZH 208 zinc rich primer, supplied by Tianhehongye Science Technology Develop CO., LTD, China.

Intermediate coating: GZH 206 epoxy micaceous iron oxide intermediate coating, supplied by Tianhehongye Science Technology Develop CO., Ltd, China.

Intumescent coatings: type-A, water-borne fire resistive coating for steel structure, supplied by an international famous manufacturer; type-B, water-borne fire resistive coating for steel structure, supplied by a domestic manufacturer.

Topcoat: Interthane 870 acrylic polyurethane topcoat, supplied by AkzoNobel Paint (China) Co., Ltd.

2.2. Specimen preparations

A total of 48 specimens containing two size of 200*300*5mm and 70*70*5mm were prepared for each type of intumescent coating. Q 215 steel plate was selected as substrate using sandblasting treatment to reach clean grade of Sa 2.5 and roughness of 80 μm. Anticorrosion system adopts a combination of spraying epoxy zinc-rich primer and epoxy micaceous iron oxide intermediate coating. Primer was sprayed twice to reach a Dry Film Thickness (DFT) of about 80 μm and intermediate paint was sprayed once to reach a DFT of about 40 μm. The intumescent coating and topcoat were applied by brushing to reach a DFT of 1.5 mm and 80 μm, respectively. The fire protection and corrosion protection supporting system meets the requirements of GB/T 28699–2012: Appendix A, Table A. 8. In order to prevent side intrusion and other factors from affecting the aging behaviour, paraffin wax: rosin=1:1 was used to seal the edge of each specimen with a depth of at least 5 mm, as shown in Figure. 1. After construction, the samples should be cured at a constant environment of 23±2 °C and 50±5 RH% for at least 10 days.

2.3. Aging method

The accelerated aging method was performed with reference of European Standard EN 16623 and European Technical Approval Guideline (ETAG) ETAG 018-2. Exposure conditions for Type X for reactive coating system intended for all conditions was selected as the aging method based on the
assumed working life of the product for the intended use of 10 years. Aging method is divided into two stages: UV/water-spray stage and hydrothermal stage. In the first stage, UV/water-spray accelerated aging was carried out by UVTest II (Atlas Material Testing Technology LLC) with UVTest DI Water Recirculation System (DIWRS) for water-spray with reference to EN ISO 16474-3 as shown in Table 1. The specimens were subjected 112 cycles (28 days) without interruption as shown in Figure. 2. The sampling frequency was set as every 8 cycles (2 days).

| Exposure period | Lamp type | Irradiance | Black-panel temperature | Relative humidity |
|-----------------|-----------|-------------|-------------------------|------------------|
| 5 h dry         | UVA-340   | 0.83 W/m²/nm(@340 nm) | 50±3 °C                 | not controlled   |
| 1 h water spray | -         | -           | 25±3 °C                 | not controlled   |

Table 1. UV/water-spray aging test parameters

In the second stage, the specimens were assessed and then exposed for 2 weeks (2 cycles) according to Table 2. Hydrothermal aging method was carried out by Sartec SS-7123X (SARTEC TESTING INSTRUMENTS Co., Ltd) as shown in Figure. 3. The sampling frequency was set as every 1 week (1 cycle).

| Day       | 6 h       | 6 h       | 6 h       | 6 h       |
|-----------|-----------|-----------|-----------|-----------|
| 1+2       | 20±3 °C   | 70±3 °C   | 20±3 °C   | 70±3 °C   |
|           | 95±5 %RH  | 95±5 %RH  | 20±5 %RH  | 20±5 %RH  |
| 3+4       | 20±3 °C   | 30±3 °C   | 40±3 °C   | 30±3 °C   |
|           | 95±5 %RH  | 95±5 %RH  | 45±5 %RH  | 45±5 %RH  |
| 5+6+7     | -20±3 °C  | 95±5 %RH  | -20±3 °C  | 95±5 %RH  |

Table 2. Hydrothermal aging test parameters

2.4. Characterizations
Fire test was carried out by a self-made test furnace and the heating curve was set as ISO 834 ($T=345 \lg (8t+1)+20$) as shown in Figure. 4. Two K-type thermocouples were used to monitor the
backside temperature and the fire resistant time was defined as the time that the mean backside temperature rise reaches 500 °C.

Adhesion test were carried out using PosiTest AT-A Pull-off Adhesion Testers (DeFelsko, USA) with reference to ISO 4624.

X-ray fluorescence (XRF) was conducted by an Olympus VANTA-VES Analyzer to characterize the surface element composition of each specimen.

3. Results and discussion

3.1. Surface appearance

Visual of each coating was assessed every 8 cycles (2 days) in the first aging stage of UV/water-spray. In the second aging stage of hydrothermal aging, each coating was visually assessed every 1 cycle (7days). Digital pictures of type-A coatings during two stages of accelerated aging methods are shown in Figure 5. We can see that with the progress of aging, the surface of the coating has always maintained high integrity and gloss without obvious cracks or blisters. In contrast, type-B coating shows poor durability as shown in Figure 6. With the aging process, a series of defects such as wrinkling, foaming, cracking and precipitation gradually appeared on the coating surface. After 10 days of aging, slight wrinkling appeared on the coating surface. After 18 days of aging, the coating surface wrinkle is more serious. At the end of first aging stage, the coating surface cracks and precipitates severely.
3.2. Fire resistant performance of the coatings

Fire resistant times of type-A and type-B coatings after two stages of accelerated aging methods are shown in Figure 7 (a) and each specimen’s backside temperature of type-A and type-B coatings are shown in Figure 7 (b), (c), respectively. We can see that the initial fire resistant time (107 min) of type B coating shows much better than type-A coating (61 min). However, as the aging progresses, fire resistant time of type-A coating dropped sharply to 56 min after 20 days and then dropped to 21 min at the end of aging stage 1. While fire resistant time of type-B coating almost constant till the end of aging stage 2. The results of fire resistant time of two coatings show the same trend with the visual performance in Figure 5&6.
For type-A coating, digital pictures of char layers after fire test are shown in Figure 8. It can be seen that the type-A coating maintains good expansion ability after two stages of aging and can form complete and dense carbon layer. For type-B coating, digital pictures of char layers after fire test are shown in Figure 9. It can be seen that there are obvious defects in the intumescent char layer of the coating after 20 days of aging, the defect position is consistent with the obvious wrinkle and cracking position of the surface, resulting in a serious decline in the fire resistant time. After 2 stages of aging, type-B coating has completely lost its expansion capacity and fire resistant performance.

3.3. Adhesion strength of the coatings
Adhesive strength of type-A and type-B coatings after two stages of accelerated aging methods are shown in Figure 10. We can see that the initial adhesive strength (1.94 MPa) of type-A coating shows better than type-A coating (1.21 MPa). Similar to fire resistance time, as the aging progresses, adhesive
strength of type-A coating dropped sharply to 0.38 MPa after 20 days and then dropped to 0.2 MPa at the end of aging stage 1. While adhesive strength of type-B coating almost constant (about 1.9 MPa) till the end of aging stage 2.

![Figure 10 Adhesive strength of two coatings during aging progress.](image)

3.4. Elemental analysis of the intumescent coating
Surface element composition of each specimen for type-A and type-B coatings are shown in Figure.11 (a), (b), respectively. Since elements such as C, N and O are difficult to be detected by XRF, the content of P as the significant element in fire resistant components has been paid special attention as shown in Table 3. For type-A coatings, since the topcoat remains intact during the aging process, the distribution of the main elements on the surface remains basically stable. Except a small amount of P element appeared on the surface at the end of the second aging stage. For type-B coatings, we can see an obvious appearance of P element at the aging time of day 16 and the fraction of P element increase with the aging progress. This may be due to the surface cracking after aging 16 days, and the fire components in the coating precipitate due to the water-spray. This is also reflected in the change of fire resistance time and adhesive strength. Then we can preliminarily judge that the aging mechanism is the precipitation of filler.

![Figure 11 (a), (b). Surface element composition of type-A and type-B, respectively.](image)

| Aging time (day) | 0-16 | 18  | 20  | 22  | 24  | 26  | 28  | 35  | 42  |
|------------------|------|-----|-----|-----|-----|-----|-----|-----|-----|
| **P (%)**        |      |     |     |     |     |     |     |     |     |
| Type-A           | —    | —   | —   | —   | —   | —   | —   | —   | —   |
| Type-B           | 2.78 | 2.36| 2.38| 3.46| 4.53| 6.32| 7.22| 11.41|

4. Conclusion
In this paper, the durability focus on fire protection performance of two intumescent coatings for steel structures were studied and the aging effects were analysed. The following conclusions can be drawn:
1. After two stages of aging including UV/water-spray and hydrothermal, type-A coating maintained excellent surface appearance. While type-B coating began to crack after 10 days of aging and the cracking deteriorated with the continuous of aging.

2. After two stages of aging including UV/water-spray and hydrothermal, type-A coating maintained excellent fire resistant time. While the fire resistant time of type-B decreased by 47.66% from 107 min to 56 min after 20 days of aging and further reduce to 21 min at the end of aging stage 2. Meanwhile, the instrument char after fire test and the adhesive strength of two coatings show the similar trend.

3. For type-B, the appearance of P element on surface proved that the fire resistive components precipitated during the aging progress. And the decrease of performance attenuation may be caused by this precipitation. We can preliminarily judge that the aging mechanism is the precipitation of filler.

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