



## Laboratory and gas-fired furnace performance tests of epoxy primers for intumescent coatings



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### ABSTRACT

Protection of steel structures, using so-called intumescent coatings, is an efficient and space saving way to prolong the time before a building, with load bearing steel constructions, collapses in the event of a fire. In addition to the intumescent coating, application of a primer may be required, either to ensure adhesion of the intumescent coating to the steel or to provide corrosion resistance. It is essential to document the performance of the intumescent coating together with the primer to ensure the overall quality of coating system. In the present work, two epoxy primers were used to investigate the potential failure mechanism of a primer applied prior to an intumescent coating. The analysis was carried out using; (1) gas-fired test furnace, (2) a specially designed electrically heated oven, and (3) thermo gravimetric analysis. When tested below an acrylic intumescent coating, exposed to a gas-fired furnace following the ISO834 fire curve (a so-called cellulosic fire), one of the primers selected performed well and the other poorly. From tests in the electrically heated oven, it was found that both primers were sensitive to the film thickness employed and the presence of oxygen. At oxygen-rich conditions, higher primer thicknesses gave weaker performance. In addition, a color change from red to black was observed in nitrogen, while the color remained red in the oxygen–nitrogen mixture. In summary, the results suggest that an adequate choice of primer, primer thickness, and intumescent coating is essential for a good performance of an intumescent coating system.

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### 1. Introduction

In the event of fire, or merely temperatures in the range of 400–550 °C, the load bearing ability of steel is reduced significantly and this has implications for buildings based on steel parts [1,2]. An efficient way to protect the building structure and prolong the time before the problematic steel temperature is reached is by intumescent coatings. At elevated temperatures, the latter swells to a thermally insulating char with a thermal conductivity from 0.1 to 1 W m<sup>-1</sup> K<sup>-1</sup>, depending on coating composition and temperature [2–4]. For the intumescent coatings suited for so-called cellulosic fires, the coatings are applied at thicknesses up to 1.5 mm [5]. An important target in ongoing intumescent research is to reduce the required dry film thickness [6].

Intumescent coatings are comprised of five basic compound groups: blowing agent, acid- and carbon sources, a binder, and pigments. All compounds are important for the char formation process [5,7]. The interaction between these compounds is complex, and various reaction sequences are reported in the literature.

Bourbigot and Duquesne [8] stated that the intumescent process consists of six steps, the first being the temperature-triggered release of an inorganic acid, which esterify with the carbon source. Following this, the binder melts, and the ester dehydrates and forms a carbon-inorganic residue. Furthermore, gasses are simultaneously released, blowing the melted structure into a foam, and finally gelation and solidification occurs. However, in addition to these reaction mechanisms, intumescent coatings are also subject to interaction with other coating layers, such as an underlying primer. The latter ensures good adhesion to the metal substrate and provides anticorrosive properties [9]. Primer detachment from the substrate will cause irreversible damage to the intumescent coating performance, yet very few scientific studies on primers in intumescent coating systems are available. A study of a zinc primer, protected by an intumescent coating consisting of expandable graphite, ammonium polyphosphate, melamine, boric acid, bisphenol-A epoxy resin, and ACR hardener polyamide was presented by Ullah et al. [10]. Effects of variations in the intumescent coating formulation on steel-primer-intumescent coating performance was studied by heating in a muffle furnace (a type of furnace where the heating source is separated from the furnace room) to 500 °C and following the development by analysis of SEM pictures before and after heating. The investigation showed that with an

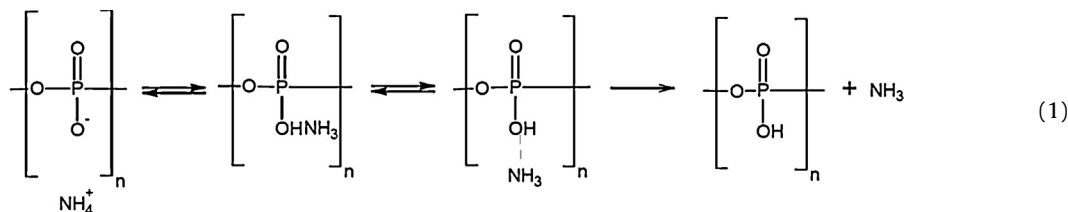
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### Explanatory notes

Gas-fired furnace	Gas-fired furnace for testing steel panels according to ISO834 fire curve.
Horizontal oven	Electrically heated oven with variation in the gas composition.
Primer A	Solvent based polyhydroxyether epoxy/polyamide. The primer is a shop primer pigmented with zinc aluminum phosphate.
Primer B	Solvent based bisphenol-A epichlorhydrin/phenalkamine. The primer is pigmented with zinc phosphate and micaceous iron oxide.
Reduced oxygen	Mixture of 10% oxygen and 90% nitrogen.
SEM	Scanning Electron Microscope.
TGA	Thermo gravimetric analysis.

optimal intumescent coating formulation, i.e. weight percentages of ammonium polyphosphate and boric acid of 25 and 15%, respectively, the strongest structure of the coating-primer-metal after heating was obtained. An example of a SEM picture of a coating-primer-metal interface before and after heating is provided in Fig. 1. Subsequent exposure, it can be seen that the primer and intumescent coating are still attached to the substrate and primer, respectively, although minor cracks are present.

In the ETAG 018 approval guideline for intumescent coating testing, the importance of a primer is emphasized. When an intumescent coating is approved, it should be specified, which primers can be used in combination with the coating [11]. In addition, due to the expensive and time consuming approval requirements of intumescent coating systems, fast screening tests for intumescent



coating systems are of interest. Jimenez et al. [12,13] correlated the following input parameters, (1) the insulating properties of the intumescent coatings below a radiant heater, (2) mass loss, (3) char expansion, and (4) rheology of heated intumescent coatings, to the output parameter, fire test performance in a gas-fired furnace. Another aspect of a potential fast screening test is the mechanical stability of intumescent chars using shock heating, which is investigated in our earlier work [14]. In the present work, the

focus is on testing of the primer below the intumescent coating. An empirical correlation relating results of gas-fired furnace tests of a primer-intumescent coating system to the primer thickness and gaseous environment (oxygen content) below an intumescent coating is developed. An important reason to study the effects of oxygen content is that in a well-tuned test furnace chamber, the oxygen concentration will be around 4 mol% [15]. At other conditions, the oxygen concentration may vary significantly from well ventilated conditions below a radiant heater to very low concentrations in an impinging flame [15]. In addition to the variation in the oxygen content of the gas phase, the oxygen concentration close to the primer may vary even more because of the intumescent char present between the fire and the primer. The motivation to study primer thickness is that the performance of many anticorrosive coatings (e.g. barrier coatings) requires a high thickness to work well [9].

## 2. Brief overview of intumescent mechanisms

In this paragraph, a brief overview of the intumescent chemistry, comprised of seven steps, is given. The steps occur as the temperature increases and the exact sequence will be dependent on the chemical coating composition. Common intumescent coating compounds are: ammonium polyphosphate, melamine, pentaerythritol, melamine, SiO<sub>2</sub>, and TiO<sub>2</sub>.

1. At coating temperatures between 150 and 215 °C, an inorganic acid is released from a salt [8,16]. For instance, the acid source (e.g. ammonium polyphosphate, (NH<sub>4</sub>PO<sub>3</sub>)<sub>n</sub>) can thermally degrade into NH<sub>3</sub>, water and acidic phosphoric groups [17]. Based on information in references [18,19] an example of the degradation with release of NH<sub>3</sub> is shown in reaction (1).

The repeating unit, *n*, in the ammonium polyphosphate may take a value of 700 up to more than 1000 [20,21].

2. At coating temperatures slightly higher than those of the acid formation, the acid and hydroxyl groups on the carbon source react and form a phosphoric ester [8,22]. The formation of the phosphoric ester can happen according to esterification or phosphorylation (alcoholysis) [21,23,24], which can lead to many different structures [23]. An example of the esterification

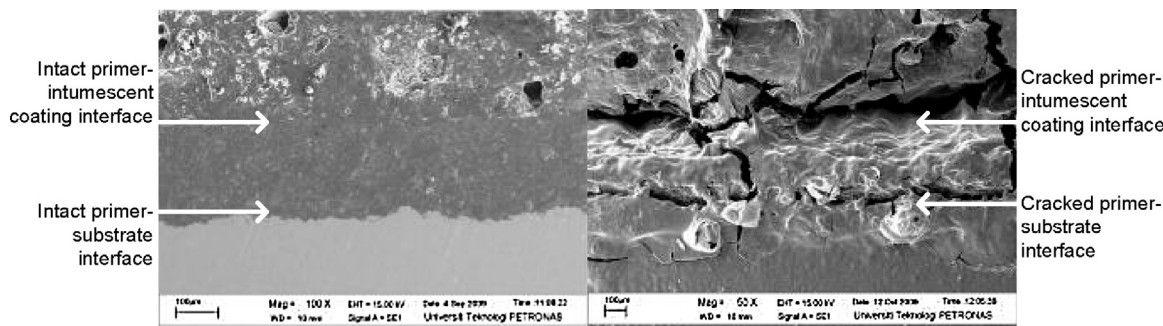


Fig. 1. SEM picture of a metal-primer-coating interface. The picture to the left shows the sample before heating and the figure to the right the sample after heating to 500 °C. After [10] with explanatory notes added.

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