THERMAL MODELLING OF EPOXY BASED INTUMESCENT COATING IN FIRE

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Abstract

Steel is the commonly used material for structural framework especially in industrial and civil buildings. Although it offer numerous advantages but it suffer in fire, where it softens or loses its rigidity and compromises the integrity of the structure. Fire retardant intumescent coatings are extensively used as passive fire protection system for structural protection which will be degraded and expanded forming into a char layer with relatively high resistance to heat transfer and consequently improve the fire resistance as well as survivability of structures. Most of intumescent coatings are polymer based compound contains ammonium polyphosphate (APP) as acid source, melamine (Mel) as the blowing agent and pentaerythritol (PER) as carbon agent. Current procedures for evaluation of structural fire protection systems involve large-scale burner and jet-fire tests which are expensive and often inhibit products development. Many works were developed for mathematical modelling of heat transfer process and obtaining thermal information like time-temperature curve of substrate protected by intumescent coating.

In this research, in-house and commercially prepared epoxy based intumescent coating were selected to evaluate the accuracy of the developed model in different intumescent coating system. The fire protection of the intumescent coatings were simulated by bonding them to a 10 mm thick steel plate, exposing them to a constant heat flux and recording the temperature rise of the plate respectively. A model which includes heat conduction, heat evolved due to reaction of active compounds and convective heat flow of volatiles gases was applied to predict the time-temperature of substrate protected by these two intumescent coatings. Kinetic and thermal properties of these two intumescent coatings were analysed using TGA and DSC. This data will be the kinetic parameters for the thermal modelling. The coefficient of determination R2 of TGA curve fitting is more than 99.5%. The modelling predictions and the experimental results were show a good agreement with each other.

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1. Introduction

Steel is the commonly used material for structural framework especially in in industrial and civil buildings. Although it offer numerous advantages but it suffer in fire, where it softens or loses its rigidity and compromises the integrity of the structure. Steel starts to reduce its rigidity and strength around 450oC. Passive fire protection materials with low thermal conductivity protect the steel or other structural materials from the effect of elevated temperature that may be generated during a fire. For the structural fire protection in industrial and civil buildings, fire retardant intumescent coatings are extensively used as passive fire protection system which will be degraded and expanded to form a carbon char layer with relatively high resistance to heat transfer and consequently improve the fire resistance and survivability of the structures. Most of intumescent coatings are polymer based compound contains ammonium polyphosphate (APP) as acid source, melamine (Mel) as the blowing agent and pentaerythritol (PER) as carbon agent when subjected to a flame [2,3].

Current procedures for evaluation structural fire protection systems involve large-scale burner and jet-fire tests which are expensive and often prevent products development. A lot works has been established for mathematical modelling of heat transfer process and obtaining thermal information like time-temperature curve of substrate protected by intumescent coating [1,4-6]. Employing a modelling approach based on thermal properties of intumescent coatings may not only helpful to understanding the reaction mechanism of intumescent but also beneficial to the design of heat resistance structure.

In this paper, in-house and commercially epoxy based intumescent coating were prepared to evaluate the heat resistance for fire protection system. The fire protection of the intumescent coatings were simulated by bonding them to a 10 mm thick steel plate, exposing them to heat flux and recording the temperature rise of the plate respectively. A model which included the effects of heat conduction, heat evolved due to reaction of active compounds and convective heat flow of volatiles gases was applied to predict the time-temperature of substrate protected by these two intumescent coatings. The modelling predictions are compared with the experimental results.

2. Theory

Intumescent coatings decompose and expand to a porous and charred insulating layers when they subjected to fire or high temperature due to its chemical and physical reaction together with accompanying effects of the endothermic and exothermic reactions, convective heat transfer, radiation heat transfer, weight loss and volume expansion during the whole process. A model presented by Griffin [5] was selected here for modelling the heat transfer through epoxy based intumescent coatings and for calculation of the temperature rise of the intumescent coatings.

$$\left[\frac{\partial T}{\partial t}\right]_{ic} = \frac{\left\{\left[\frac{\partial}{\partial x}\left(k_c^{ic}(T)\frac{\partial T}{\partial x}\right)\right] + \Delta h_{ic}\right\}}{\left[\rho_{ic}(T)\cdot C_p^{ic}(T)\right]} \quad (1)$$

Two terms on the right hand side are heat conduction and the net thermal energy respectively. The subscript/superscript ic refers to intumescent coating. The thermal conductivity (k_c^{ic}) , specific heat capacity (C_p^{ic}) and the density (ρ_{ic})of the intumescent coating are assumed to be isotropic and temperature-dependent, and these properties must be experimentally measured. Both exothermic (or endothermic) energy from the chemical reactions and the mass flow energy of reaction volatiles from the coating to the surface are considered in the net thermal energy (Δh_{ic}) which is calculated using:

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$$\Delta h_{ic} = \rho_0 \left(1-\omega\right) \sum_k \gamma_k r_k \Delta h_k \quad (2)$$

The net thermal energy(Δh_{ic}) relates to the initial density ρ_0 , the void fraction of the intumescent coating ω , the mass fraction γ_k , reaction rate r_k and specific enthalpy of the active compound h_k (designated type k) in the intumescent reaction process.

The voids are developed during the melt and decomposition of the intumescent under high temperature . The void fraction of the intumescent coating ω is determined by:

$$\omega = \frac{a_{ex} - \sum_k \gamma_k m_k}{a_{ex}} \quad (3)$$

where a_{ex} is the expansion factor of the intumescent coating, which must be determined experimentally.

The reaction rate of the active compound in the intumescent coating is determined using the first-order Arrhenius rate equation:

$$r_k = \frac{\partial m_k}{\partial t} = -z_k \exp\left(\frac{-E_k}{RT}\right) m_k \quad (4)$$

where z_k the pre-exponential constant, E_k is the activation energy of the reaction, and m_k is the normalised mass fraction of the active compound converted into gas for reaction. These three must be determined by TGA test. R is the universal gas constant.

3. Materials and Experiments

3.1. The intumescent coatings

3.1.1 In-house intumescent coatings

In-house intumescent coatings was prepared using a non-fired retardant epoxy resin which are epoxy resin RS-L135 and hardener RS-H137 from PRF Composite Material while three main fire retardant additives, Exolit AP 422 a fine grain Ammonium polyphosphate (APP), by Clariant Produkte, Deutshland, Melamine (Mel) and Pentaerythritol (PER) are supplied by Sigma-Aldrich, United Kindom.

Fire retardant additives powders mixture were added to resin without hardener and hand mixed until uniform. Hardener was added at the end of the process. 100×100×10mm steel plate was roughly grinded, clean (using acetone) and dry before the coating application. Sample was left to dry for 24h at room temperature with dry coating thickness is within 6±1mm.

3.1.2 Commercial intumescent coating

Commercial intumescent coating was prepared by external party according the standard procedure of the coating manufacturer. Dry coating thickness for commercial intumescent coating is 16mm, 20mm and 32mm respectively.

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3.2 Fire Test

Samples were placed on a metal frame with all the sides covered with ceramic blanket leaving the rear face open to air. A Bullfinch propane burner placed 350mm in front of the sample to provide a constant heat flux of 116kW/m2. Propane burner will give a one-sided heating to the sample and simulate an actual fire as illustrated in Figure 1.

Figure 1. Fire test set up.

Temperature profile is recorded using type K thermocouple placed 1cm from the sample hot surface and another type K thermocouple is bonded halfway through the back of the steel. Data was collected by IO data logger acquisition brand IO. Tech DAQ Shuttle 55 which is connected to a computer.

3.3 TGA/DSC Experiments

Mass loss kinetical parameters of intumescent coatings, determine through TGA experiment using Perkin Elmer instrument STA 6000. The temperature range of experiments was from ambient up to 900°C. The experiment was performed in a nitrogen atmosphere with a flow of 40.0 ml/min and at a 10°C/min heating rate. The endothermic and exothermic reaction during the experiments was also recorded through the differential scanning calorimetry (DSC) using the same instrument.

4 RESULTS AND DISCUSSION

4.1 TGA/DSC Experiments

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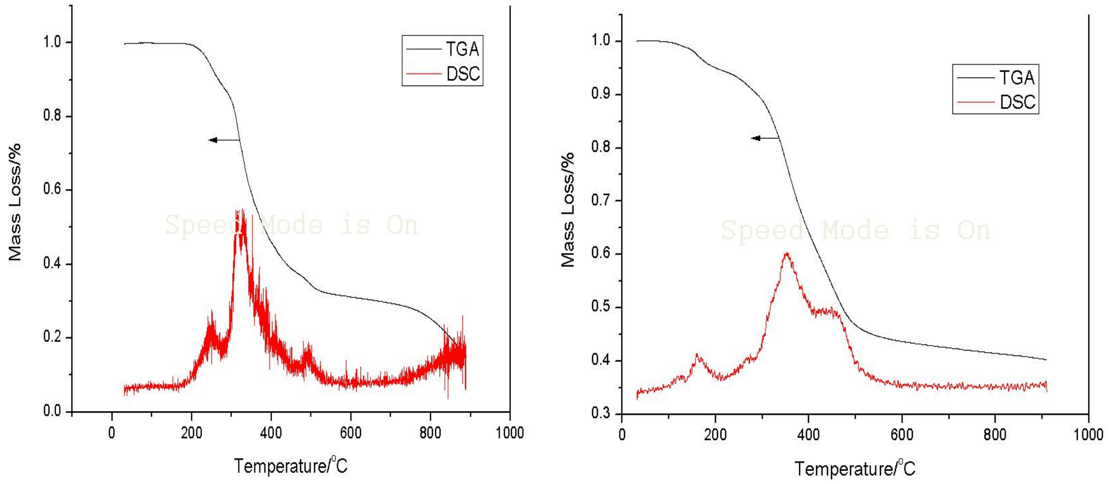

Figure 2. TGA and DSC curves for in-house intumescent coating (left) and commercial intumescent coating (right).

The TGA/DSC data (Figures 2) for the intumescent coatings show the mass loss and the relative reaction with increasing temperature.

The in-house intumescent coating and commercial intumescent coating demonstrate different pattern due to different ingredients due to the differences between TGA/DSC data. The mass loss of commercial intumescent coating was relatively stable above 500°C, while the char degradation was still continuing above 800°C for in-house intumescent coating.

The residual mass fraction of intumescent as a function of temperature can be described based on first order reaction according the [5]:

$$mf = \sum_{k=1}^{n}\left(\gamma_k \exp\left[-\frac{z_k}{\beta E_{a,k}/R}T^2\exp\left(-\frac{E_{a,k}}{RT}\right)\right]\right)+\gamma_{inert} . \tag{5}$$

where mf is the residual mass fraction of intumescent materials, γ_k is the mass fraction of the active compound, z_k is the pre-exponential constant, $E_{a,k}$ is the activation energy of the reaction, R is the universal gas constant.

The thermal kinetical parameters of intumescent coating during the TGA process under fire test were obtained by non-linear curve fitting. The coefficient of determination R2 of TGA curve fitting for both in-house intumescent coating and commercial intumescent coating are more than 99.5% as shown in Figure 3.

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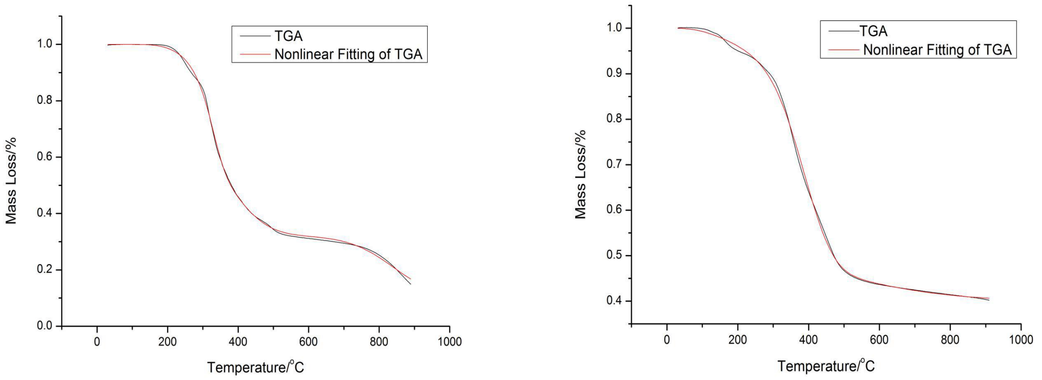

Figure 3. Kinetical parameters obtained by TGA data fitting for in-house intumescent coating and commercial intumescent coating.

4.2 Thermal modelling

This section compares the temperature profiles of intumescent coating surface and back face, as well as the thermal modeling results for the back face temperature of intumescent. Equation (1) was used to calculate the temperatures of the intumescent coatings for in-house one and commercial sample.
For these two types intumescent coatings, the hot surface temperature recorded by thermocouple reached around 1000°C in a short time which can be seen as constant temperature as shown in Figure 4 and Figure 5.

Fire tests were conducted for 1000s for both samples. The modelling temperature profiles were compared with experimental results for three coatings with different thickness and shows good agreement.

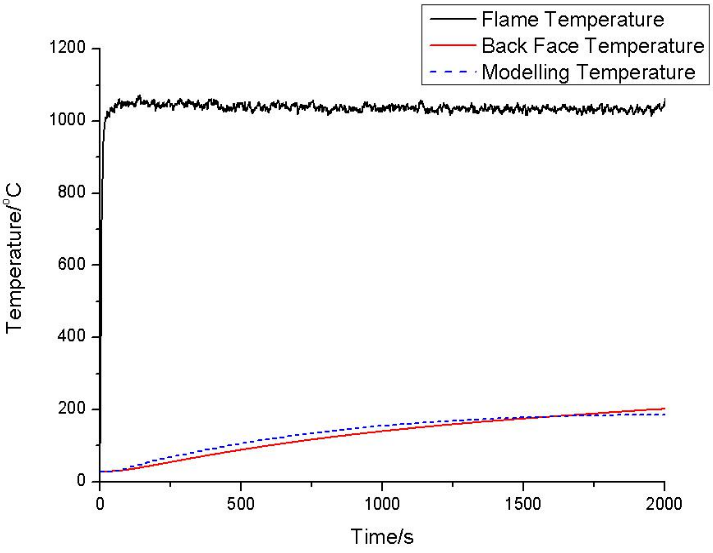

Figure 4. Modelling temperature and experimental temperature for in-house intumescent coating.

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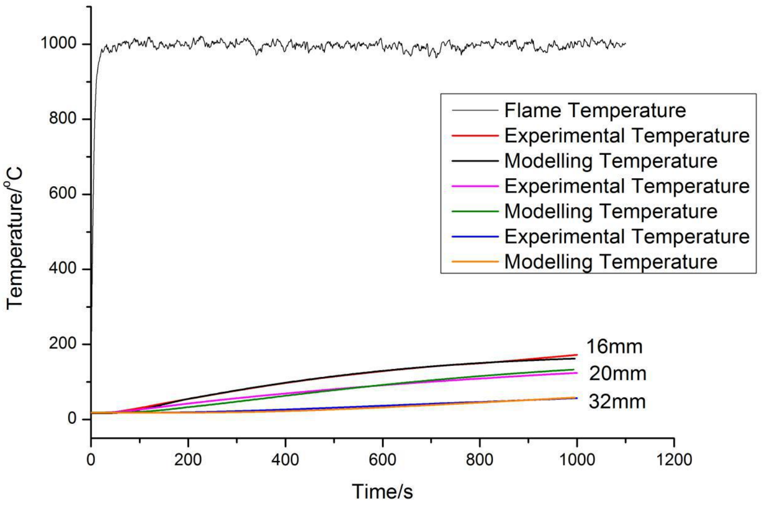

Figure 5. Modelling temperature and experimental temperature for commercial intumescent coating.

5. Conclusions

Comparison between in-house and commercial intumescent coating according to the TGA/DSC test, show they have different ingredients and thermal properties. The modelling time-temperature of these two intumescent coatings using a mathematical model show good agreement with the experimental results. The results show this method can be used to evaluate the thermal resistance of intumescent in the future work.

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References

[1] A.P. Mouritz, S. Feih, E. Kandare, A.G. Gibson. Thermal–mechanical modelling of laminates with fire protection coating. *Composites: Part B,* 48: 68–78, 2013.

[2] S. Duquesne, S. Magnet, C. Jama, R. Delobel. Intumescent paints: fire protective coatings for metallic substrates, *Surface and Coatings Technology,* 180 –181, 302–307, 2004.

[3] Jun-wei Gu, Guang-cheng Zhang, Shan-lai Dong, Qiu-yu Zhang, Jie Kong. Study on preparation and fire-retardant mechanism analysis of intumescent flame-retardant coatings, *Surface & Coatings Technology,* 201: 7835–7841, 2007.

[4] Robert J. Asaro, Brian Lattimer, Chris Mealy, George Steele. Thermo-physical performance of a fire protective coating for naval ship structures. *Composites: Part A,* 40: 11–18, 2009.

[5] Griffin GJ. The modeling of heat transfer across intumescent polymer coatings. *J Fire Sci,* 28:249–77, 2010.

Excerpt from ISBN 978-3-00-053387-7

[6] Y. Zhang, Y.C.Wanga, C.G.Bailey, A.P.Taylor. Global modelling of fire protection performance of intumescent coating under different cone calorimeter heating conditions. *Fire Safety Journal,*50: 51–62, 2012.

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