Thermal Conductivity Measurements of Intumescent Coatings

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A solvent-borne organic intumescent paint and a water-borne inorganic intumescent paint, both are commercially available, were investigated for their thermal characteristics including thermal reactions and weight loss rate as a function of temperature. These were conducted by DSC and TGA thermal analysis techniques. The results indicate that the initial thermal reactions of the water-borne inorganic paint starts earlier and the reactions appear wider range of temperature than the solvent –borne organic intumescent paint. Applying a laser flash method at thermally expanded state, thermal diffusivity of the intumescent paints was measured. With this information, in conjunction with the experimentally acquired specific heat and densities values, theoretical insulation effectiveness, which is expressed as the temperature differences between the surface of the expanded intumescent paints and the backside, was calculated. These calculated values were compared with the experimental values and the incoherence was explained.

1 Introduction

Intumescent coatings are widely used to increase thermal resistance of steel building structures under high temperature fire conditions. By measuring thermal conductivity that is the product of thermal diffusivity, density and specific heat capacity under the assumption of homogeneity of the interested material, the characteristic insulation properties of intumescent coatings can be defined at an interested temperature. With this information time-dependant temperature increases of the interested steel structure under the protection of intumescent coatings can be known, providing the heat flux can be measured or calculated. Therefore, thermal diffusivity of intumescent coatings and thermal conductivity at fully expanded state of intumescent paints were measured and compared with the experimental values. The information obtained would be applied in designing economic intumescent coating applications in construction industry in the future.

2 Experimental

2.1 Sample preparation

Intumescent coatings used for this paper are solvent-borne organic intumescent paint (SBOIP) and water-borne inorganic intumescent paint (WBIIP). SBOIP contains polyvinylacetate as the main polymer resin, ammonium polyphosphate and pentaerythritol derivates as the expanding agents and melamine derivatives as the carbon source. WBIIP is known to contain sodium silicate as the expanding agent and thermally stable alumina silicate and titanium dioxide compounds as fillers.

Intumescent coatings were prepared using an automatic film applicator, PA2101 Byk Gardner, to achieve uniform sample thickness of wet coated film. For differential scanning calorimetry (DSC) and thermo gravimetric analysis (TGA) experiments, samples were prepared on acetate film and dried for 4 days in circulating oven at 23 ℃ and 50 % relative humidity [1]. After 4 days of drying, there was no significant weight change was shown.

2.2 Sample characterization

To understand thermal behavior of intumescent paints, DSC experiments were performed using a Shimadzu DSC-60 thermal analyzer operated with a TA 60WS analyzer. Samples were contained in standard aluminum pans under normal atmosphere and nitrogen gas atmosphere when inert conditions were required for DSC experiments. Also DSC experiments were performed to measure specific heat capacity(C_p) of intumescent paints, which were conducted after ASTM 1296 [2]. Due to the reactive characteristics of intumescent paints, both SBOIP and WBIIP dried films were heated at 400 ℃ for 20 minutes prior to DSC experiments. TGA experiments were conducted with a Netzch QMS 403C model. Both DSC and TGA experiments were carried out at 10 degree per minute of elevating temperature rate.

Thermal diffusivity(α) of intumescent coatings was measured using a laser flash apparatus, a Netzch LFA 417. Due to the fragile characteristics of intumescent coatings after thermal expansion, samples were contained in sapphire crucible to measure thermal diffusivity constants at each measured temperature and the thermal conductivity values was calculated with the achieved thermal diffusivity and specific heat values.

Thermal insulation experiments were conducted with a Dual Cone Calorimeter, from Fire Testing Technology Ltd. Basic experimental set up was after ISO 5660-1 [3]. SBOIP and WBIIP were coated on one side of 70x70x3 mm^3 standard steel plate and 5 thermocouples were attached at the other side to measure temperature variations as a function of time. Samples were located 57 mm away from the radiating source for one hour at the radiating heat flux of 35.5+/-0.2 kW/m^2.

3 Results and discussion

3.1. Thermal behavior of intumescent paints

Figure1 shows DSC results of SBOIP, WBIIP and ammonium polyphosphate (APP) as a comparison purpose. APP is a known agent of ammonia gas production for intumescent coating products at elevated temperature. DSC results of **Figure1** can explain the thermal exothermic or endothermic reaction of intumescent coatings as a function of temperature. As is shown on the figure, SBOIP shows distinctive endothermic peaks around 200, 320~440 ℃. These indicate that around 200 ℃ intumescent behavior starts and the partial unreacted intumescing agents activates later on at temperature range between 320~440 ℃. Due to the nature or organic coatings at this temperature range all the organic materials would decompose and contribute the generation of endothermic peaks as well. Therefore, it would be known that SBOIP initiates the intumescent behavior at 200 ℃ and terminates around 440 ℃. On the contrary, WBIIC shows relatively small scale of broad endothermic behavior around 100 and 400 ℃. This may be due to the physically and chemically adsorbed complex water molecules within WBIIP.

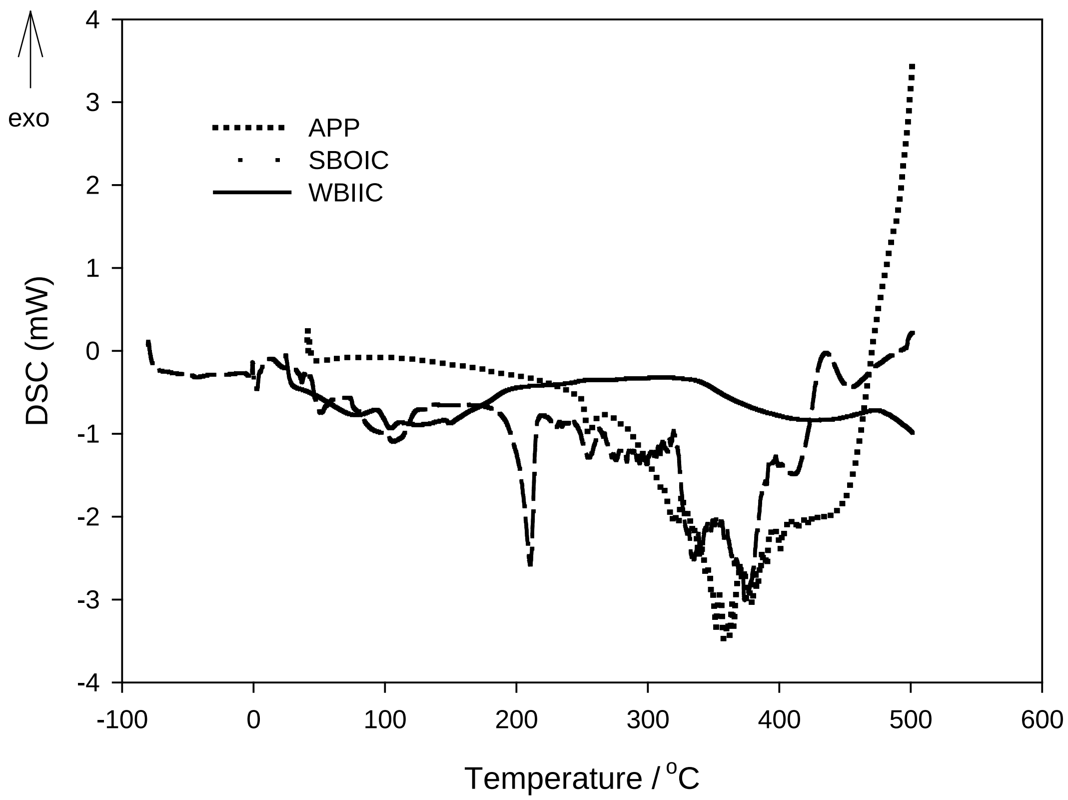

Figure1 DSC results of ammonium polyphosphate, SBOIP and WBIIP

Figure2 shows TGA results of SBOIP and WBIIP. This figure shows the mass reduction rate of intumescent coatings due to increasing temperature. Derivate weights of SBOIC and WBIIC are shown on the figure as well. These show more clearly the mass reduction rate at specific temperature ranges. It also can be shown that SBOIC shows ca 70% of weight decrease but WBIIC only shows 20 % decreases. The differences are originated the nature of WBIIC and WBIIC as organic and inorganic materials.

The derivative weight results indicates that WBIIP initiates the intumescent behavior at 100 ℃ but SBOIP just over 200 ℃. This would be explained that WBIIC contains hydrated inorganic materials and these start to decompose around this temperature but the intumescent behavior of SBOIP starts by the decomposition of polyphosphate derivate compounds, which starts over 200 ℃, which agrees with DSC results shown at **Figure1** as well.

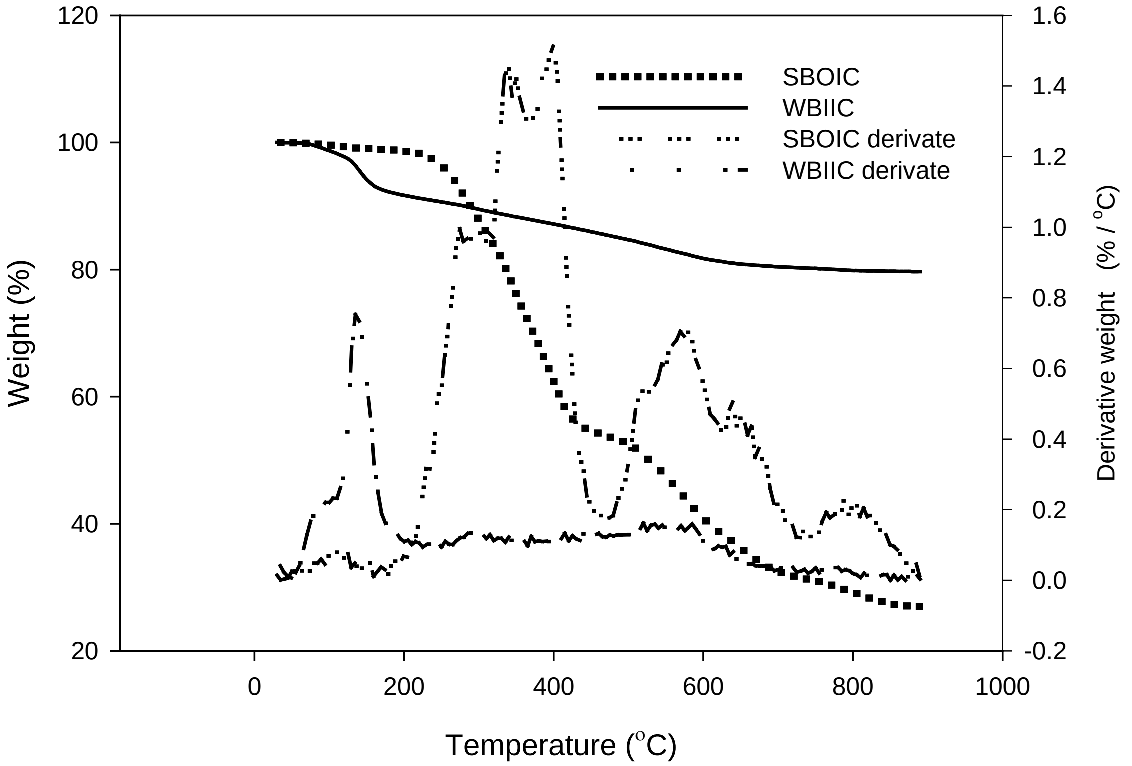

Figure2 Mass reduction rate of SBOIP and WBIIP as a function of temperature.

3.2 Thermal insulation characteristics of intumescent paints

Specific heat capacity is a variable against temperature. Following ASTM 1269, specific heat capacity of expanded SBOIP and WBIIP samples were measured. During experiments, sapphire was used as an reference material. The measured data are shown at the following table.

Table 1 Specific heat capacity of intumescent paints at 512 ℃

	SBOIP	WBIIP
Specific heat capacity ($Jg^{-1}K^{-1}$)	1.73	1.31

Thermal diffusivity (α) is the speed with which heat transfers through a material. α values can be calculated using Parker's formula [4]

$$\alpha = \frac{1.38\, d^2}{t_{1/2}} \qquad \textbf{(1)}$$

where d is the sample thickness and $t_{1/2}$ is the time necessary for the signal to reach 50 % of its maximum value. As an example **Figure3** represents the actual measurement of heat transfer and a mathematical fit of SBOIP sample at 512 ℃.

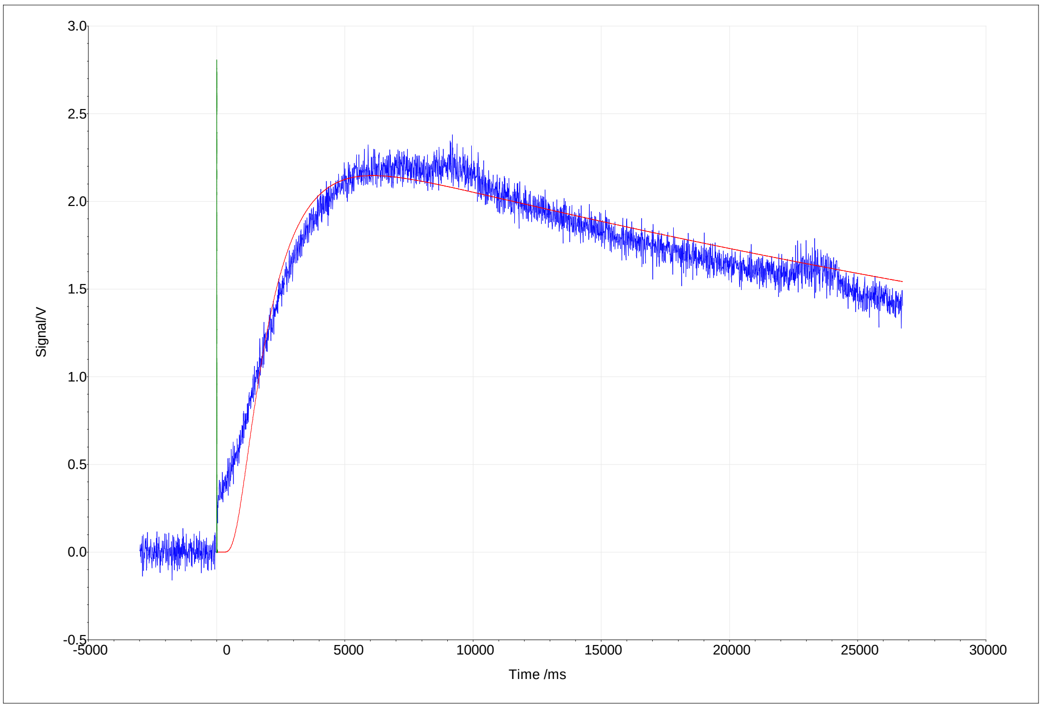

Figure3 Signal intensity changes and its mathematical fit of SBOIP at 512 ℃

With the achieved α values for SBOIP and WBIIP, thermal conductivity (λ) can be calculated by the following equation.

$$\lambda = \alpha\, \rho\, C_p \qquad \textbf{(2)}$$

where λ is thermal conductivity, α is thermal diffusivity and ρ is density. Here α and ρ are variables against temperature. ρ would be an temperature dependant variable but within our experimental range the change of density was ignorable so it was regarded as an constant throughout this work.

The calculated λ values at 512 ℃ for SBOIP and WBIIP are shown at **Table 2**

Table 2 Thermal conductivity of intumescent paints at 512 ℃

	SBOIP	**WBIIP**
Thermal Conductivity ($Wm^{-1}K^{-1}$)	7.05x10-$^{-3}$	3.06x10-$^{-2}$

To characterize the effective thermal insulation due to intumescent coatings, 70x70x3 mm^3 standard steel plates attached with thermocouples were prepared and performed using a cone calorimeter. Applying a constant heat flux of 35.5+/-0.2 ㎾/m^2, the increase of temperature was an exponential growth for bear steel plate and intumescent coated steel plate samples. The temperature changes against elapsed time of irradiation were shown at **Figure4 and 5**.

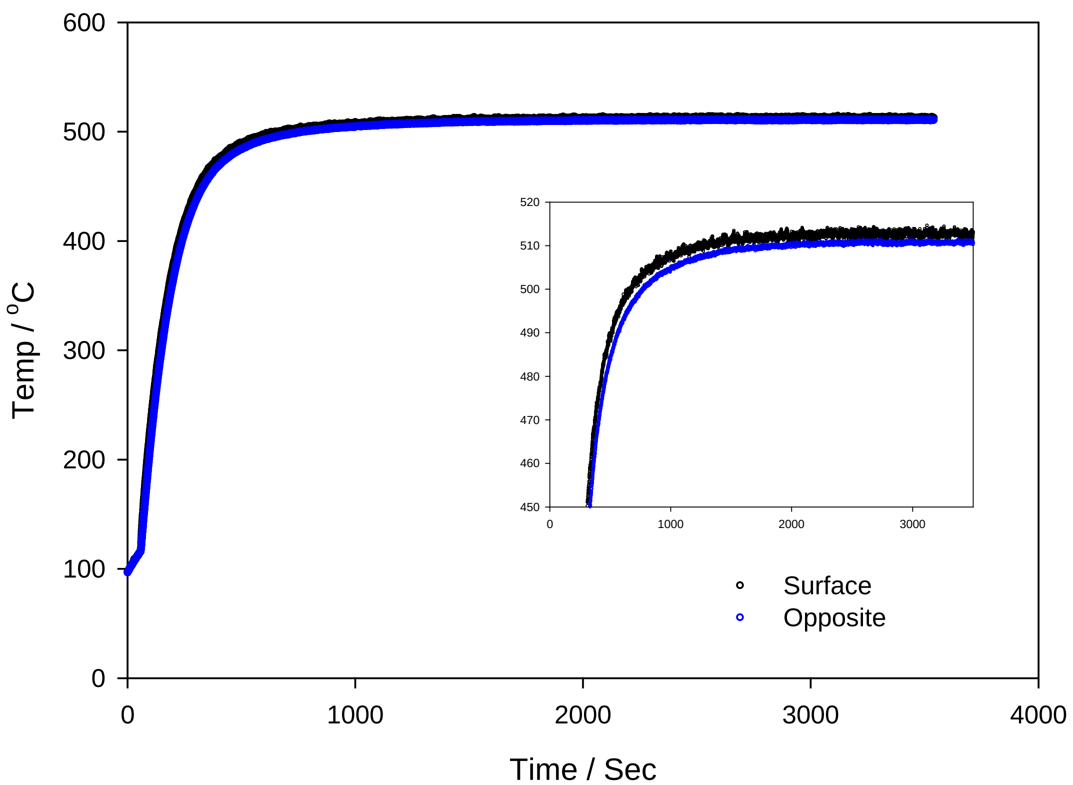

Figure4 Surface temperature changes of bare 3mm thick steel plate irradiated at an constant heat flux of 35.5 ㎾/m^2

Figure4 indicates bare 3mm steel plate temperature profile. After 2000 seconds the temperature reaches a steady state and the average temperature is 512.6 and 510.6 ℃ respectively for heat radiated surface and opposite side surface temperature of steel plate. As can be seen, at a fixed heat flux, temperature reaches at steady state after certain period time. At this state, the following equation can be applied to calculate the temperature differences between both sides of sample surface.

$$q'' = \lambda \frac{\Delta T}{L} \qquad \textbf{(3)}$$

Where q″ is heat flux, λ is thermal conductivity, ΔT is the temperature difference at heat radiated surface against opposite surface and L is sample thickness. This cone calorimetry experiment allowed having a constant heat flux and actual temperature changes. Also by calculation, the temperature difference can be achieved. As would be shown at **Figure4** the measured temperature difference was 8 ℃ but by calculation it was between 1.9 and 2.1 ℃. The difference is expected due to the heat loss through horizontal directions.

Figure5 shows the temperature differences of intumescent paint coated samples. As was shown at Figure4 intumescent coated specimen shows thermal stable region, even though, the steady state initiates later than shown at **Figure4**. The thickness of SBOIP and steady state temperature shows linear correlation (see **Figure6**) as well. **Eq. 3** can be rearranged as the following equation

$$\Delta T = \frac{q''}{\lambda} L \qquad \textbf{(4)}$$

Using **Eq. 4** and theoretical ΔT can be calculated and the calculated values and actual measured values are shown at **Figure 6**. **Figure 6** temperature differences between surface and backside of samples against dry film thickness of intumescent coatings. The temperature difference at vertical axis represents the temperature differences between coated samples and bare steel plate.

As would be seen, measured data show higher temperature differences than calculated ones. These differences may be originated from thermal energy loss. For actual measurements, the prepared samples were radiated vertically from the heat source of a cone calorimeter but the edges of the samples were not insulated. Although it was known that the cone calorimeter irradiate 100 x 100 mm^2 area uniformly, still the surface temperature of the prepared samples and the surrounding atmosphere would have some temperature differences and therefore heat horizontal transfer would have happened during the temperature measurements. Moreover, with increasing thickness of the coatings, the gap between calculated predictions and the actual measurements goes wider. This would have happened because of the increasing side area of the samples with coating thickness increases.

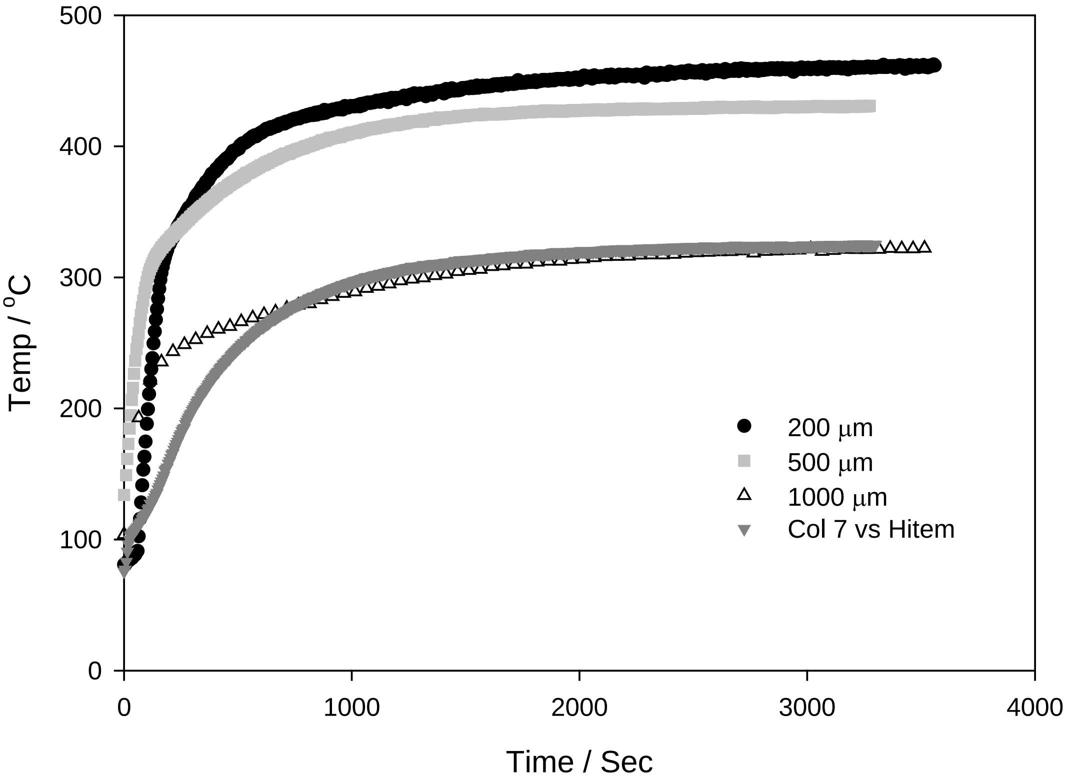

Figure5 Surface temperature changes of SBOIP and WBIIP coated steel plate irradiated at an constant heat flux of 35.5 kW/m^2

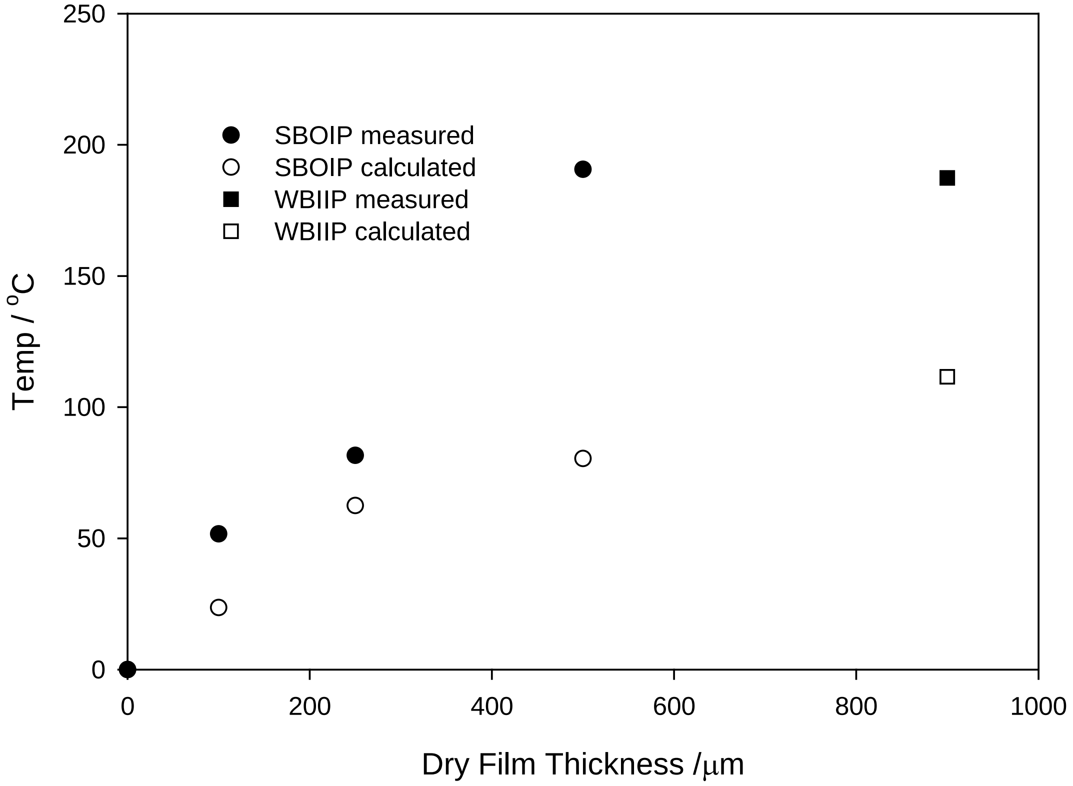

Figure6 Temperature differences between surface and backside of SBOIP and WBIIP coated steel plate at heat flux of 35.5 ㎾/m^2

4. Conclusions

A solvent-borne organic intumescent paint(SBOIP) and an water-borne inorganic intumescent paint(WBIIP) were prepared and thermal characteristics as a function of temperature were investigated using DSC and TGA thermal analysis techniques. Initial intumescent characteristics starts earlier for WBIIP and the reacting temperature ranges and weight changes are widely extended. On the contrary, SBOIP reacts around 200 ℃ and around 60 % (w/w) of its components reacts at the first reaction between 200 and 400 ℃.

At 512 ℃, which is the steady state surface temperature of bare steel plate of 35.5 ㎾/m^2 heat radiation, SBOIP and WBIIP thermal conductivities were measured 7.05 $x10^{-3}$ and 3.06 x 10^{-2}-$w^{-1}K^{-1}$ respectively. The measured values with the information of expanded SBOIP and WBIIP thickness and density values, theoretical temperature differences and measured temperature ones between the surface and backside samples were compared. Although some differences are shown, the values are within the same orders. These differences may be due to the imperfect thermal isolation conditions during the experiments.

5 References

[1] KSM5000 Testing method for organic coatings and their related materials

[2] ASTM 1296-01 Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry

[3] ISO 5660-1 Reaction to fire tests; heat release, smoke production and mass loss

[4] Parker, W. J. Jenkins R. J., Butler C. P. and Abbott G. L., J. Appl. Phys., 32 (1961) 1679