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# Intumescent Thermoplastic Elastomer Fire Shield Material

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## ABSTRACT

The intumescent material described in this paper expands under high heat or fire conditions to form an insulating sponge. Its composition is based on a blend of high-density polyethylene and chlorinated polyethylene. The material is processed using normal plastic techniques. Tensile properties, heat aging, fluid aging, and thermal properties have been evaluated and are presented. Fire testing using a 1000 °C Bunsen burner as a source, showed that the material does not drip or burn through, even after 30 minutes of exposure. Prototype fire shields made of the intumescent material have been tested on fuel tanks, bulkheads, and wheel well covers. In all cases the intumescent material provided excellent protection. Similar applications are identified for airplanes, motorcycles, industrial and residential buildings. The material is recyclable and can be made from recycled polymers.

## INTRODUCTION

Thermoplastic elastomers are a family of materials that have the properties of elastomers, but can be processed as plastics. Being elastomeric they have the desirable properties of flexibility, impact resistance, shock absorption, and sound and vibration reduction. They can be formed by compression molding, injection molding, extrusion, vacuum forming and blow molding. Thermoplastic elastomers are recyclable, allowing in-plant scrap generated during processing to be chopped up and used to make parts. An intumescent material made of thermoplastic elastomer would then provide fire shielding and would be easily made into parts for automotive and other industrial and residential applications.

Intumescent materials expand and char when exposed to fire and form a heat resistant spongy barrier that will reduce the transfer of heat to neighboring objects and reduce the progress of fires. It is desirable that this thermally stable spongy structure have some

mechanical strength so that it will not break or crumble by disturbances that sometimes occur during fires. Some degree of mechanical strength will insure physical integrity during fire and effectiveness as a barrier.

The intumescent material presented in this report could be used to replace existing plastic parts in cars, or as a cover layer or shield to protect other plastic parts. Similarly, it can be used to manufacture components for other industries, such as transportation, shipping, industrial buildings, residential buildings, office and home furniture. In the market place most commercial grades of intumescent materials are available either in the form of coatings [1, 2] or paper-like composite products [3].

## EXPERIMENTAL

### A - OVERALL COMPOSITION OF THE INTUMESCENT MATERIAL

The intumescent material is based on high-density polyethylene as the hard phase of the thermoplastic elastomer and chlorinated polyethylene and/or silicone rubber as the soft phase. The composition also contains fillers, thermal stabilizers, fire retardant additives, blowing agents and char forming additives [4]. All ingredients were used as received except that some were dried in an oven before processing.

### B - MIXING THE COMPOSITION

In the laboratory, the melt mixing of compositions was conducted using two techniques: a two-roll heated mill, and a Brabender extruder having a mixing screw with a length to diameter ratio of 20 to 1. In addition to laboratory mixing, 8000 lb. batches of the material were compounded in the plant using a 3.5-inch diameter single screw extruder having an L/D ratio of 11.

## C - PROCESSING OF THE COMPOSITION

Extrusion, compression molding, injection molding, and vacuum forming were used to process the intumescent material in order to make samples for laboratory testing and to form prototypes for in-service field testing.

## D – MECHANICAL PROPERTIES TEST PROCEDURES

The tensile properties of compression molded and injection molded samples of intumescent material were obtained using ASTM D638 procedures. The tear properties of the samples were obtained using procedures outlined in ASTM D624. The flexural properties were determined per ASTM D790 procedures.

## E – CHEMICAL RESISTANCE TEST PROCEDURES

The chemical resistance of the intumescent material to automotive fluids was investigated per GM engineering test specification 2217M. In separate experiments, samples were immersed in water, 5% by weight salt water, engine coolant, premium diesel fuel, hydraulic brake fluid, windshield washer solution, and automatic transmission fluid, for four hours at 25°C. The degree of swell by the fluids and the tear strength of the material, before and after exposure were measured.

## F – THERMAL ANALYSIS

Thermal gravimetric analysis (TGA), differential scanning calorimetry (DSC), and thermal conductivity measurements were carried out using procedures previously developed for investigating thermal and flammability properties of automotive polymers [5-8]. High-resolution TGA measurements were conducted using the TA Instrument 2100 module. These analyses were carried out using 13 to 15 mg samples. The samples were heated from 25 to 1000°C using a linear heating rate of 50°C /min., and a resolution factor of four. All runs were conducted in nitrogen or air atmospheres at a flow rate of 50 ml /min. Decomposition temperature, weight loss percent and weight loss rate were determined for each decomposition peak. The amount of residue left after heating the sample to 900°C was also determined.

DSC measurements were used to quantitatively determine thermal events. The apparatus used was the modulated DSC manufactured by TA Instrument (MDSC 2920). The technique is relatively new. Conventional DSC is a device for measuring the heat flow into and out of a sample as it is being heated isothermally or in a linearly rising temperature mode. From these measurements, thermodynamic properties of materials such as heat capacity, melting points, heats of fusion, and glass transition temperatures are quantified. The

modulation is achieved by imposing a sine wave input on the underlying constant rate. Applying Fourier transform analysis on the output signal from the MDSC, one is able to discern between thermodynamically reversible changes and non-reversible kinetic changes as the polymer is being heated. The reversing heat flow is used as a direct measurement for heat capacity. This is of primary interest to the work in this report since heat capacity and thermal conductivity are related properties [6].

Typically, a polymer sample in pellet form weighing approximately 10 mg was placed in an aluminum pan and hermetically sealed. The pan containing the sample was then placed in the MDSC nitrogen-purged cell. The sample was allowed to equilibrate at -60°C for five minutes, before starting programmed heating at 5°C per minute to 350°C. During the run, modulation of +/- 0.5°C was programmed at time intervals of 40 seconds. MDSC measurements were used to determine the melting temperature ( $T_m$ ), the glass transition temperature ( $T_g$ ), heat of fusion ( $\Delta H_f$ ) and the heat capacity ( $C_p$ ) of the different Intumescent material formulations.

## G – FLAMMABILITY APPARATUS FOR MEASURING INTUMESCENCE

The intumescent flammability test apparatus is shown in Figure 1. It consists of a three walled chamber having a left side wall, a back wall, and a right side wall. Each wall is a steel plate 229 mm high, 127 mm wide, and 1 mm thick. A 152 mm x 152 mm x 1mm steel plate was employed as a roof member. The intumescent material test sample is affixed to the lower surface of the roof plate for exposure to flame. Six thermocouples were welded on the topside of the plate to monitor temperatures during the flammability test. Flame temperatures were measured using a thermocouple placed through the back wall at a location corresponding to the blue flame region of the burner. A 165-mm tall Bunsen burner was used as the flame source. The flame height above the burner was 60 mm, and was adjusted so that the tip of the inner blue cone of the flame touched the surface of the intumescent material. Burning tests were carried out for 30 minute.

## RESULTS AND DISCUSSION

### DEVELOPMENT OF THE INTUMESCENT MATERIAL:

The base resin is a thermoplastic elastomer consisting of polyethylene and chlorinated polyethylene and/or silicone rubber. It can be processed as a plastic. Many grades of high-density polyethylene (HDPE) can be used depending on the application and the method of processing. High molecular weight/high melt viscosity grades are used for blow molding applications. Low melt viscosity grades are preferred for injection molding.

Extrusion is normally performed using intermediate melt viscosities. The presence of a plastic and an elastomer in the base resin allows adjustments of mechanical properties to meet part performance requirements.

Additives were incorporated in the polymer base resin to achieve desired mechanical properties, thermal properties and flammability characteristics. These include plasticizers, heat stabilizers, dehydrochlorination stabilizers, gas generating compounds to effect intumescence, flame retardants and char formers. Using the right combination of ingredients we were able to design intumescent materials for specific applications.

#### CHARACTERIZATION OF INTUMESCENCE EFFECTIVENESS:

The main function of the intumescent material is in resisting the spread of flame from a fire source, and shielding articles protected by the material from reaching high temperatures. The efficiency of intumescence is measured using the apparatus shown in Figure 1. As described in the experimental section, the sample is attached to a steel plate that is placed on top of the steel housing to form the roof. The sample is placed face down for direct flame exposure from the Bunsen burner. Figure 2 is a graph of temperature versus time of exposure to the Bunsen burner flame. The temperature is measured by thermocouples placed at four locations on the top surface of a steel plate protected by the intumescent material. The flame temperature of the Bunsen burner is set at over 1000 °C. The flame thermocouple reading does not reach this expected temperature because of the cooling effect of the gases from the intumescent material and because in some cases the sample comes in contact with the flame thermocouple during intumescence and also cools it. As seen in Figure 2, the maximum temperature reached on the top surface of the steel plate is 299°C, indicating that the intumescent material was very effective in decreasing the heat flow to the steel plate. Similar results on the efficiency of injection molded samples of intumescent materials were obtained.

The temperature of the steel plate rises rapidly at the start of the run, reaching its maximum at about five minutes of Bunsen burner exposure. By that time the sample had intumesced and most of the organic binder is consumed. Very little temperature rise is observed there after. No plastic dripping or burn through of the shield are observed. The spongy residue composed of char, fillers and phosphorous compounds stays strong and continues to provide protection. A micrograph of the cross-section of the sample after terminating the experiment at 30 minutes is shown in Figure 3. The porous structure that forms during fire exposure is the result of intumescenting by evolved gases.

The effectiveness of the intumescent material in shielding fires depends on its thickness. Figure 4 shows the effect of thickness on the maximum temperature reached for a steel plate protected by the intumescent material. An unprotected plate will reach a temperature of 853°C. A film of 0.12 mm of the intumescent material placed on the steel plate provides thermal protection by reducing the maximum temperature to 525°C. Further reduction in temperature is observed with increased thickness of the intumescent material as seen in Figure 4. The desired thickness used for any application depends on the maximum allowable temperature the part can tolerate.

#### MECHANICAL PROPERTIES:

Measurements of tensile properties tear strength, and flexural properties of materials are needed for choosing the right materials for a part. The tensile and tear properties of compression molded and injection molded samples of intumescent material are shown in Table I.

The properties of the injection molded and compression molded samples are quite different reflecting the orientation of glass fiber in the machine direction during injection molding. Thus, although the tensile strength, elongation, and tear strength of the compression molded sample are similar in the machine and cross machine directions, these properties are quite different for the injection molded sample. Tensile strength is increased from 7.3 MPa for the compression molded sample to 18.4 MPa for the injection molded sample when measured in the machine or melt flow direction. Ultimate elongation on the other hand decreased from 653% to 16.6%, respectively, for the same two samples. The properties in the cross machine direction are closer to each other for the injection and compression molded samples.

The flexural properties of the injection molded sample were also determined. The flexural strength at yield was found to be 14.5 MPa, and the strain at yield 5.5%. The notched Izod impact strength of the sample was also measured per ASTM D256 procedure and found to be 284 J/m.

#### THERMAL PROPERTIES:

Thermal gravimetric analysis (TGA), differential scanning calorimetry (DSC), and thermal conductivity measurements were performed on the intumescent material. TGA behavior is shown in Figure 5. The curves show weight loss and derivative of weight loss with temperature rise. The run was conducted in air using the procedure described in the experimental section. Decomposition starts at about 200°C. Three large decomposition peaks are observed at 306°C, 460°C, and 715°C. Each of these peaks represents weight loss from more than one ingredient as indicated

by shoulders observed on the derivative plot. Inorganic and organic ingredients contribute to the weight loss all through the temperature range. Gas analysis was conducted on the effluent coming out of the TGA furnace as the sample is heated in an air atmosphere. At low temperatures, up to 300°C low concentrations of decomposition products are observed, the main decomposition product being ammonia (see Figure 6). This is the decomposition product of ammonium dihydrogen phosphate placed in the formulation to cause intumescence. When the temperature of the sample reaches 400°C an appreciable amount of water, and carbon dioxide and a small amount of carbon monoxide and hydrocarbons are observed (Figure 7). The water is the result of decomposition of pentaerythritol, and the loss of water of hydration from magnesia or alumina. The presence of carbon dioxide, carbon monoxide and hydrocarbon indicates the decomposition of the polymer portion of the material. When the sample is heated to 700°C, carbon dioxide becomes the major decomposition product. Low concentrations of carbon monoxide, hydrocarbons and water are observed indicating that intumescence is complete and most of the organic portion of the composition has been consumed (see Figure 8). It was surprising to note that hydrogen chloride (HCl) which is the decomposition product of chlorinated polyethylene was not observed. Most likely, the evolved HCl reacts with the magnesia or alumina and gets trapped in the solid phase as a metal chloride.

Differential scanning calorimetry (DSC), which measures heat flow versus temperature, was conducted in the temperature range of -55°C to 175°C. In this region a weak glass transition temperature is observed at -18°C, assigned to the chlorinated polyethylene matrix present in the composite, and a pronounced melting point at 133°C assigned to HDPE. At higher temperatures melting peaks of the other ingredients, present in the formulation, were also observed.

The thermal conductivity of the intumescent material was measured at three temperatures and found to be 0.43 watts per meter per degree Kelvin at 58°C, 0.41 W/m-°K at 77°C, and 0.35 W/m-°K at 97°C. Corresponding values for HDPE were 0.27, 0.27 and 0.26 W/m-°K respectively. The measurements show that the addition of fillers and intumescent ingredients did not compromise the thermal insulation characteristics of the HDPE to any significant degree. After intumescence the thermal conductivity for one of the samples was measured at 0.23 W/m-°K at 100°C

#### SOLVENT RESISTANCE:

The effects of exposure of the intumescent material to different automotive fluids were measured per GM 2217M and are shown in Table II. Degree of swell and changes in tear strength after exposure to automotive fuels were used to assess fluid resistance.

Compression molded samples were immersed in the fluids for four hours at 25 °C. After the exposure the degree of swell was less than 1% in water, salt water, engine coolant, hydraulic brake fluid, windshield washer and automatic transmission fluid. The sample swelled 1.3 % in diesel fuel. The tear strength of the sample before fluid exposure was 55 kN/m and as seen from Table II the tear strength was maintained at about this value after exposure to the fluids.

#### OVEN AGING OF INTUMESCENT MATERIAL:

Oven aging of the intumescent material was conducted at 120 °C for periods of time ranging between 24 hours to 1008 hours as seen in Table III. The effects of heat aging on tensile strength, elongation, and tear strength were evaluated. In all cases no indication of degradation was observed even after 1008 hours of aging at 120 °C.

#### SOUND TRANSMISSION LOSS PROPERTIES:

The sound transmission versus frequency curve was measured for the intumescent material per SAE J1400 procedure. The frequency range covered was 100 to 10000 Hz. As seen in Figure 9 the curve was very similar to a thermoplastic polyolefin material (TPO) currently used in the automotive industry as a sound barrier.

#### OTHER FORMULATIONS:

Formulations based on the addition of commercial or novel silicone elastomers to the intumescent material in order to partially or totally replace chlorinated polyethylene have been successfully developed and reported [9]. Formulations based on polymers other than polyethylene, such as polyethylene vinyl acetate, polypropylene, and polyvinyl chloride were also successfully developed [10].

#### RECYCLING

Three approaches were used to evaluate recycling issues for the intumescent material. The first was to determine if regrinds could be used to make parts. We conducted physical property and flammability measurements on parts made of 100% regrind and found them to be no different from those made of non-recycled material. The excellent heat aging properties shown in Table III explain why no changes in properties were observed for the regrind.

The second approach was undertaken to examine whether or not regrind from fuel tanks can be used to make the intumescent material. To address this question we prepared three formulations of intumescent material using fuel tank regrind to replace the virgin polyethylene. The first formulation was made using fuel

tank regrind un-exposed to fuel. In the second formulation the regrind was soaked in gasoline for 168 hours before compounding. For the third formulation the regrind was exposed to 85/15 gasoline/methanol (M15 gasohol) before use. The physical properties and flammability performance of these formulations are compared to a formulation using virgin HDPE as shown in Table IV. The flammability performance was measured by the degree of intumescence (% increase in thickness after fire exposure), and by the fire shielding efficiency (the maximum temperature reached on a steel plate protected by the intumescent material). The data was obtained using compression molded samples. For samples made of the unexposed regrind, and regrind exposed to gasoline the properties are similar to that made of virgin HDPE. Only ultimate elongation shows significant drop from 653% for the virgin based material to 225-326 for the other two formulations. However elongation above 200% are acceptable for most applications. The formulation based on regrind exposed to gasohol showed more significant decrease in tensile strength, elongation and tear strength.

The third approach was to determine whether or not fuel tank regrind containing intumescent material can be used back for the making of the fuel tank. To answer this we added known concentrations of intumescent material to high density polyethylene and determined the physical properties. All measurements were conducted using injection molded samples. The results are shown in Table V. The presence of up to 20% intumescent material in HDPE leads to a loss of only 10% in tensile strength, 10% in ultimate elongation and 4% in modulus. Even at 40% intumescent material in HDPE the tensile strength is reduced from 40 MPa to 31 MPa, the elongation from 56% to 45%, and the modulus from 24 MPa to 21 MPa. Hence the presence of intumescent material in the fuel tank regrind does not present a problem.

## **OTHER ENVIRONMENTAL ADVANTAGES**

The intumescent material is an enabler for plastic fuel tanks. Fire shields can be designed, using this material, to be placed on specific area of the tank to prevent leaks during fire per ECE 34 flammability test.

Case studies have shown that replacing a metal fire shield with an intumescent metal shield would lead to 25 to 50% reduction in weight in addition to saving of 40 to 60% in tooling cost, 50% reduction in lead time, and 15 to 30% reduction in cost. Weight saving is of course a way of fuel saving.

In addition to replacing the metal shield, the fire protection provided by the intumescent material will encourage more use of plastic fuel tanks to replace metal tanks again leading to appreciable weight savings.

## **CONCLUSION**

An intumescent material has been developed and characterized. Normal thermoplastic techniques can be used for processing the material. Large-scale production technology has been developed for mixing, sheet extrusion, and forming into a fire shield. Laboratory tests show that the material has good mechanical properties, chemical resistance, heat aging, and sound isolation properties. It can be used for automotive applications including under hood, cockpit or exterior. The material is also a candidate for use in other transportation vehicles, shipping pallets and containers, and in residential and industrial buildings. Field testing involving large fuel pool fires, engine compartment fires and other tests for various applications proved that the material is very effective as a fire shield. The intumescent material is recyclable, and can be formulated using recycled plastics. Its use will facilitate wider use of plastic fuel tanks, or as a replacement of metal shields currently used for fire protection of plastic fuel tank and lead to appreciable weight saving.

## **ACKNOWLEDGMENTS**

The author wishes to acknowledge the help of Craig Jaynes, Joe D'Angelo and Sabiha Khalid for conducting most of the experimental work on this project.

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**TABLES:****Table I: Mechanical Properties Of Compression And Injection Molded Intumescent Composition**

<u>Property</u>	<u>Compression Molded</u>		<u>Injection Molded</u>	
	<u>Machine Direction</u>	<u>Cross Machine Direction</u>	<u>Machine Direction</u>	<u>Cross Machine Direction</u>
<b>Tensile Strength (MPa)</b>	<b>7.3</b>	<b>6.4</b>	<b>18.4</b>	<b>11.0</b>
<b>Elongation (%)</b>	<b>653</b>	<b>550</b>	<b>16.6</b>	<b>481</b>
<b>Tear Strength (kN/m)</b>	<b>61</b>	<b>56</b>	<b>60</b>	<b>-</b>

**Table II: Automotive Fluid Resistance Of the Intumescent Material**

<u>Fluid</u>	<u>% Swell (by weight)</u>	<u>Tear Strength (kN/m)</u>
<b>None</b>	<b>--</b>	<b>55.0</b>
<b>Water</b>	<b>0.1</b>	<b>54.7</b>
<b>Salt Water (5%)</b>	<b>0.1</b>	<b>56.7</b>
<b>Engine Coolant 50/50 (v/v) water/glycol</b>	<b>0.1</b>	<b>54.9</b>
<b>Premium Diesel Fuel</b>	<b>1.3</b>	<b>52.4</b>
<b>Hydraulic Brake Fluid (GM 9985013)</b>	<b>0.3</b>	<b>54.9</b>
<b>Windshield Washer Solution (GM9985129)</b>	<b>0.1</b>	<b>57.7</b>
<b>Automatic Transmission Fluid</b>	<b>0.2</b>	<b>56.1</b>

**Table III: Aging Data at 120°C for Injection Molded Intumescent Material**

<u>Aging Time (Hr)</u>	<u>Tensile Strength (MPa)</u>	<u>Elongation (%)</u>	<u>Tear Strength (N/mm)</u>
<b>0</b>	<b>18.4</b>	<b>16.6</b>	<b>60</b>
<b>24</b>	<b>20.6</b>	<b>21.7</b>	<b>68</b>
<b>72</b>	<b>21.7</b>	<b>22.0</b>	<b>74</b>
<b>168</b>	<b>22.0</b>	<b>23.6</b>	<b>72</b>
<b>336</b>	<b>23.8</b>	<b>22.6</b>	<b>75</b>
<b>504</b>	<b>23.2</b>	<b>21.6</b>	<b>75</b>
<b>840</b>	<b>22.6</b>	<b>22.9</b>	<b>74</b>
<b>1008</b>	<b>23.1</b>	<b>22.9</b>	<b>72</b>

**Table IV: Mechanical and flammability characteristics of intumescent material made using virgin HDPE, fuel regrind, fuel regrind pre-exposed to gasoline, and fuel regrind pre-exposed to M15 gasohol**

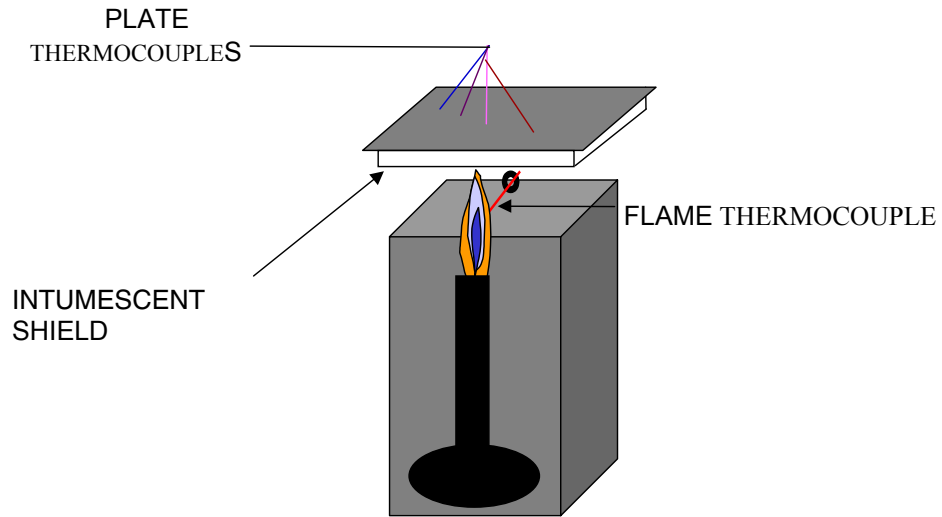
<u>Property</u>	<u>Virgin HDPE</u>	<u>Intumescent Material Formulated with</u>		<u>Gasohol Soaked Regrind</u>
		<u>Tank Regrind</u>	<u>Gasoline Soaked Regrind</u>	
Tensile Strength (MPa)	7.3	6.2	6.2	5.4
Elongation (%)	653	225	326	225
Tear Strength (N/mm)	61	65	60	52
Degree of Intumescence (%)	97	82	115	86
Maximum Temperature (°C )	299	272	251	246

**Table V: Mechanical properties of blends of intumescent material with high density polyethylene**

<u>Property</u>	<u>% Intumescent in Blow Molding Grade of High Density Polyethylene</u>				
	<u>0</u>	<u>5</u>	<u>10</u>	<u>20</u>	<u>40</u>
Tensile Strength (MPa)	40	36	38	36	31
Modulus @ 10% Elongation (MPa)	24	23	23	23	21
Ultimate Elongation (%)	56	50	39	45	45

**FIGURES:**

**Figure 1: Flammability Apparatus**



**Figure 2: Temperature of Flame and Temperatures of Steel Plate Protected by the Intumescent Material**

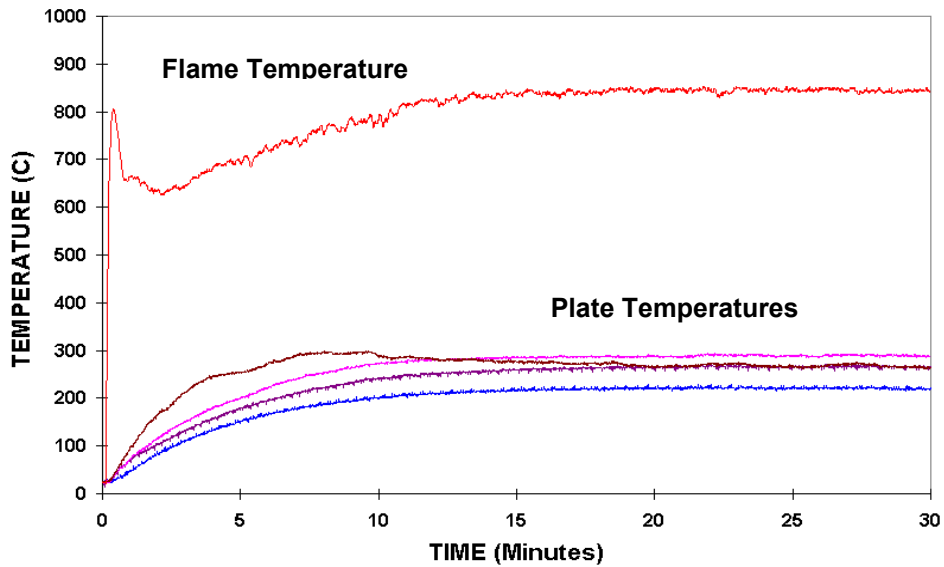
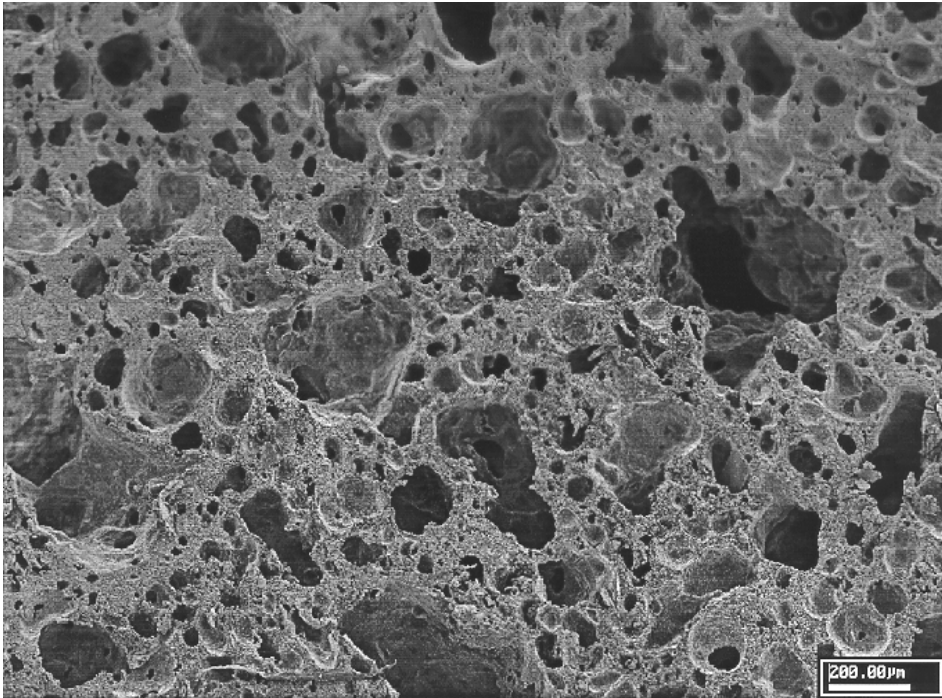
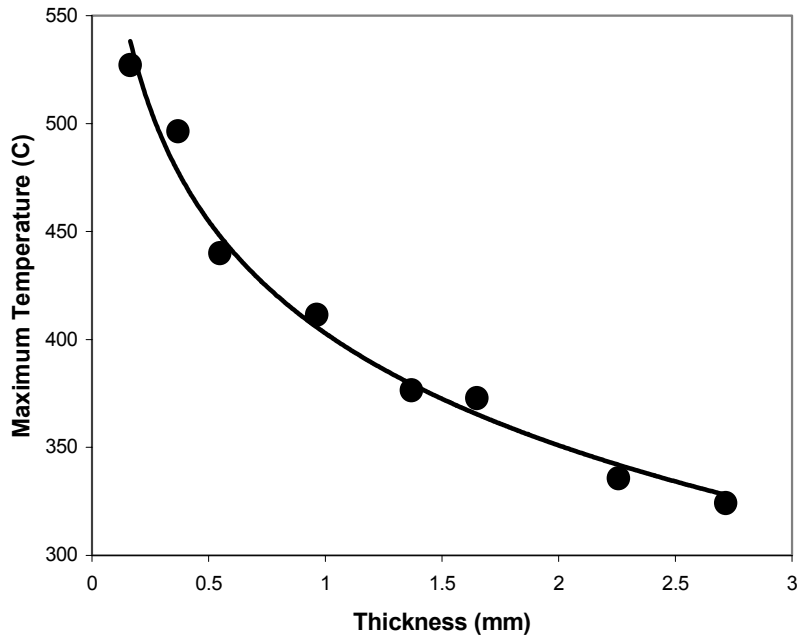


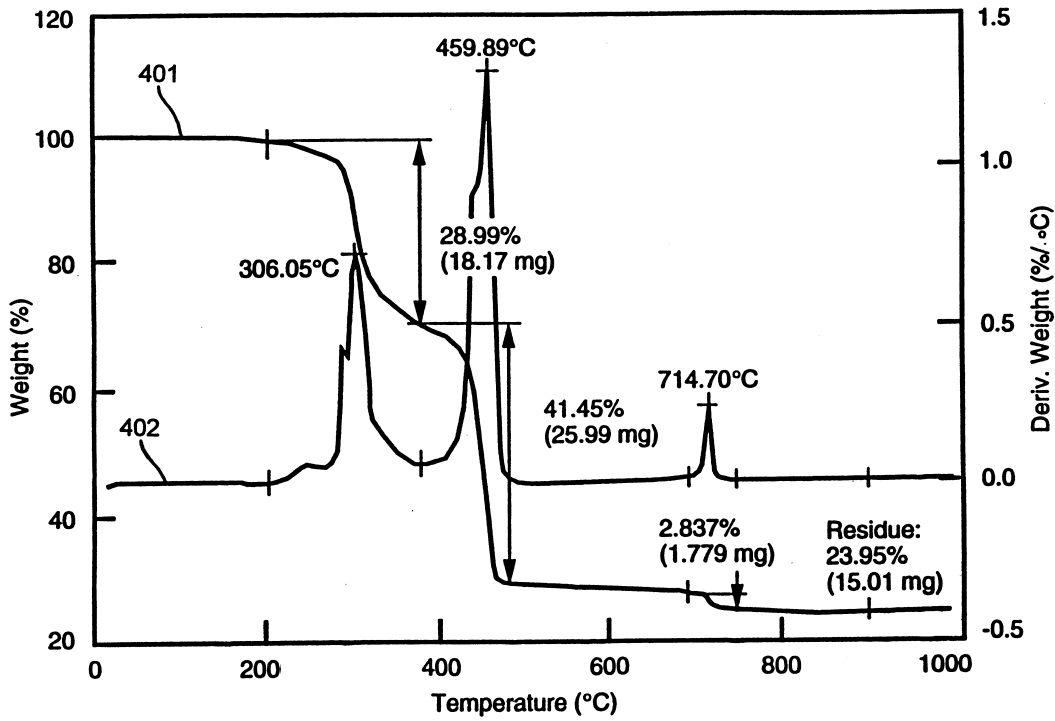
Figure 3. Micrograph of Intumescent Material After Fire



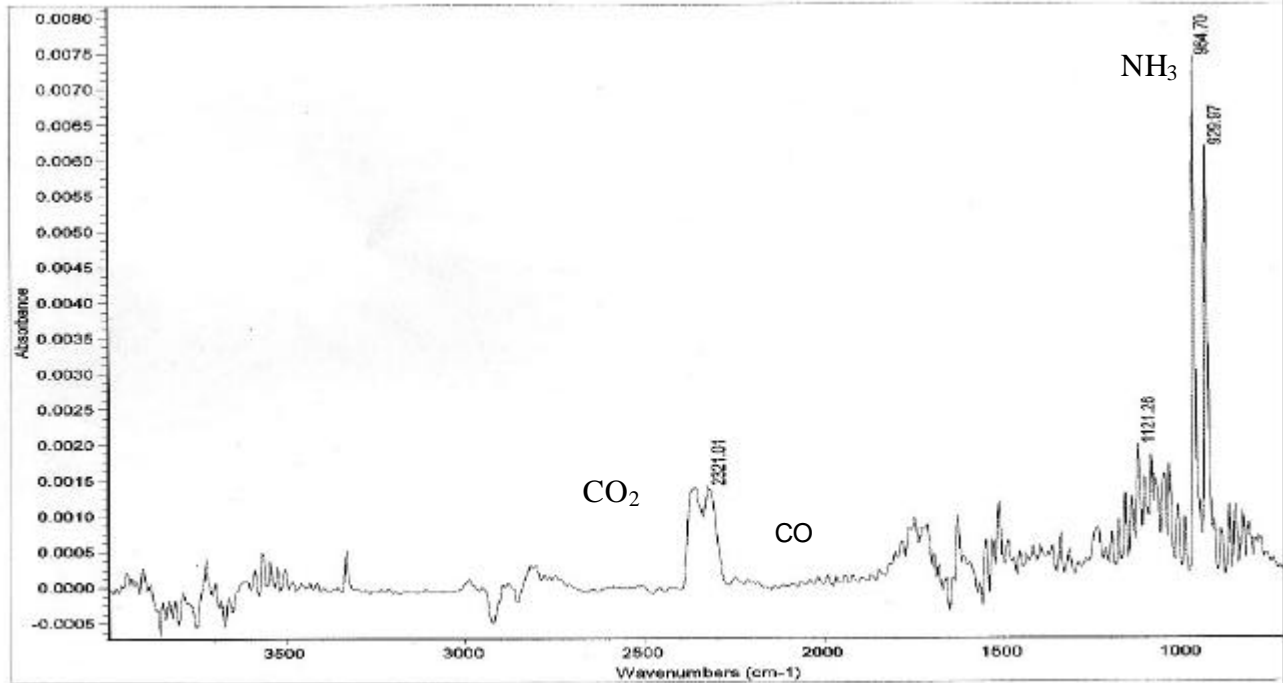
**Figure 4: Effect of Intumescent Material Thickness on Protected Plate Maximum Temperature**



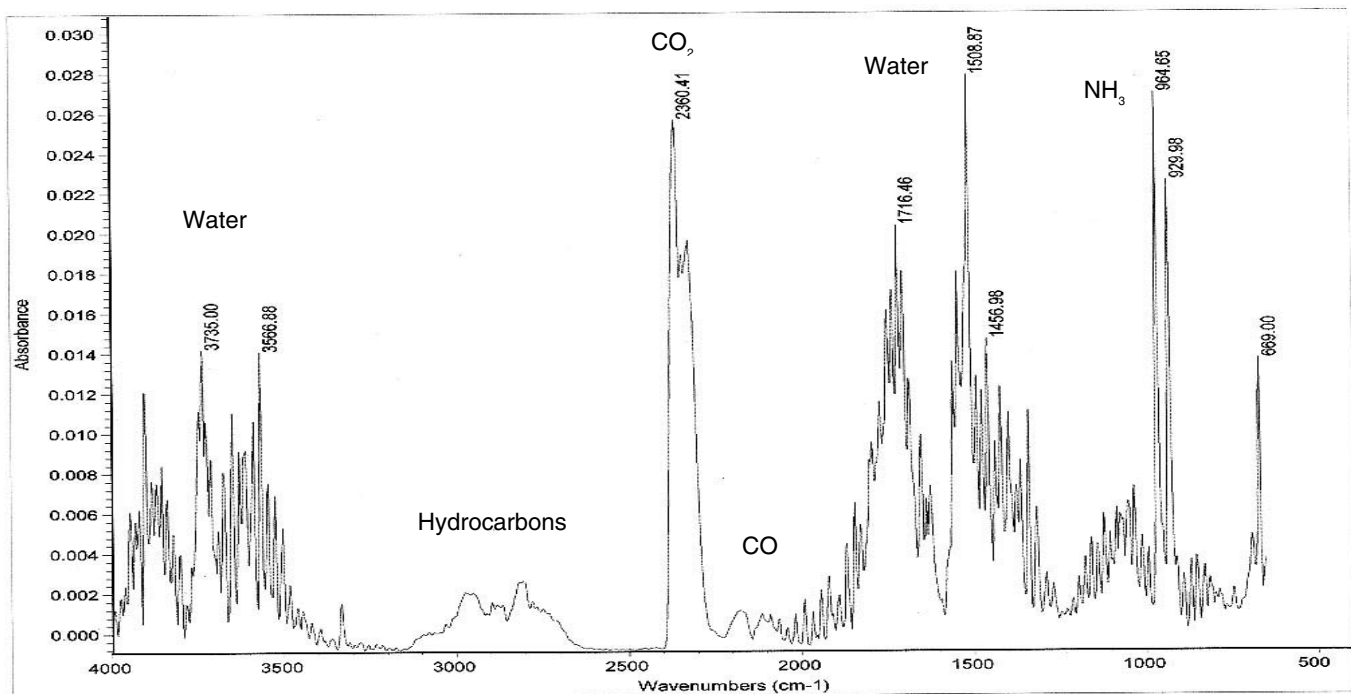
**Figure 5: Thermal Gravimetric Analysis (TGA) Scan of Intumescent Material**



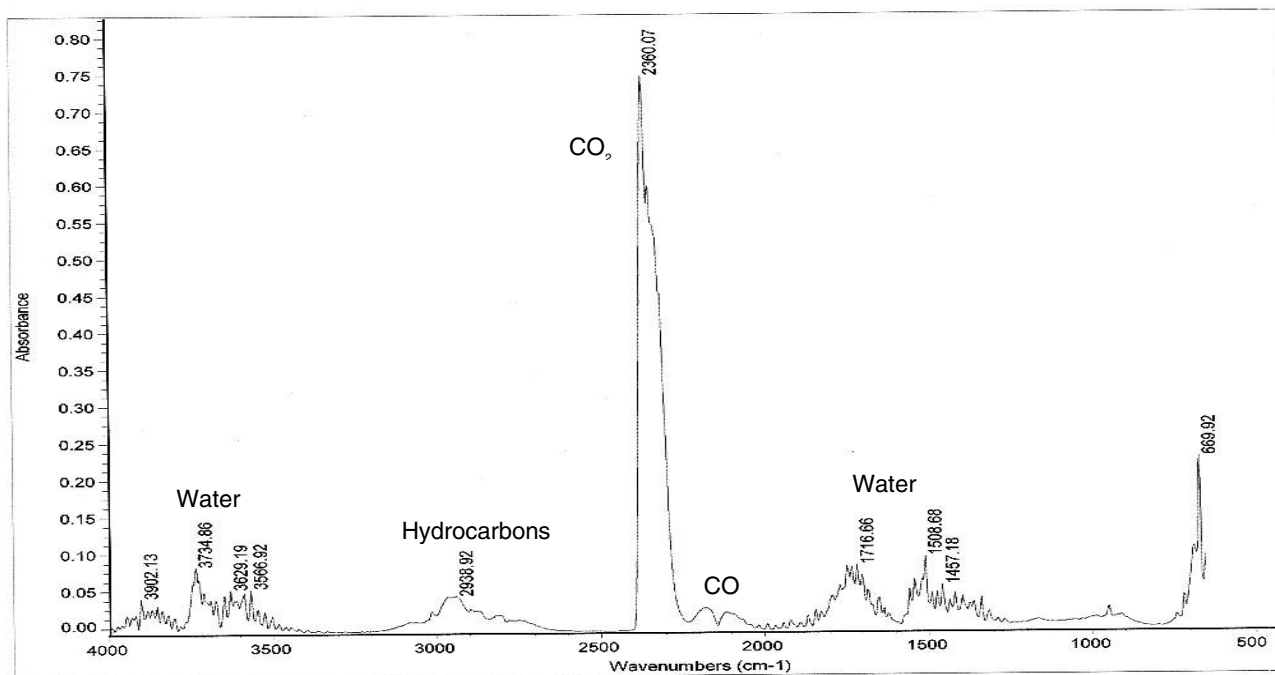
**Figure 6: Infrared Spectrum of the Effluent from an Intumescent Material Sample Heated in a Thermogravimetric Analyzer to 300°C**



**Figure 7: Infrared Spectrum of the Effluent from an Intumescent Material Sample Heated in a Thermogravimetric Analyzer to 400°C**



**Figure 8: Infrared Spectrum of the Effluent from an Intumescent Material Sample Heated in a Thermogravimetric Analyzer to 700°C**



**Figure 9: Sound Transmission Loss of Intumescent Material Barrier and a Thermoplastic Elastomer Polyolefin (TPO) Barrier**

