Introducing Novel Flame Retardant Materials to Produce Exceptionally Low Viscosity, High Temperature Resistant Epoxy Encapsulation Compounds

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ABSTRACT

The most common epoxy encapsulation compounds available on the market utilise specialised fillers, such as Alumina trihydrate (ATH), to provide a high level of flame retardancy. Such fillers decompose endothermically at 200˚C producing water which cools the substrate. This inhibits the effects of the ignition source and reduces the substrates' ability to sustain a flame. Such fillers are therefore extremely efficient and as such are utilised in many applications where high operating temperatures and viscosity are not crucial requirements for the user. Due to the decomposition temperature being relatively low, the stability of encapsulation compounds which incorporate ATH in their formulation are limited above 150-200˚C. In addition, the use of such fillers dramatically increases the viscosity, making the resins difficult to work with when encapsulating complicated geometries or where space is limited. To overcome these limitations, a novel flame retardant system has been investigated. Although still a filler, approximately 10 times less material is required to produce a flame retardant system, therefore making it possible to formulate a flame retardant encapsulation resin with viscosities of less than 700mPa s, whilst still meeting UL94 V-0. In addition, this novel system does not decompose at temperatures around 200˚C and exhibits excellent stability at very high temperatures, including those seen in typical reflow profiles. This paper details the advantages of this novel flame retardant system, highlighting the performance advantage over standard metal hydroxide fillers and concludes with possible applications when formulated into an encapsulation resin.

INTRODUCTION

Epoxy resins are used for a vast array of applications, including the encapsulation of electronics. Encapsulation resins are designed to protect the electronics from their environment and as such the desired properties can differ depending on the particular application. One key requirement for many applications is that the resin is flame retardant. Traditionally, epoxy systems would incorporate halogenated, in particular, brominated flame retardants into the resin formulation; approximately, 15-20% by weight of bromine would be incorporated into the formulation in order to meet the highest level of flame retardancy, V-0, as classified by the Underwriters Laboratory UL94 specification. (1). Halogen compounds, including bromine, provide excellent flame retardancy but also generate toxic gases and in some cases produce carcinogenic substances, such as dioxin (2). As a result, other flame retardant additives such as phosphorus and/or nitrogen containing compounds and inorganic flame retardant fillers have all been investigated and in some cases utilised as suitable replacements for brominated flame retardants.

Inorganic flame retardants, such as alumina trihydrate (ATH), are widely used in many encapsulation products available on the market, providing a high level of flame retardancy. ATH is a flame retardant filler which decomposes endothermically at about 200˚C producing water which cools the substrate. This inhibits the effects of the ignition source and reduces the substrates' ability to sustain a flame. The addition of such fillers also dramatically increases the viscosity, making the resins difficult to work with when encapsulating complicated geometries or where space is limited. Therefore, these inorganic materials are extremely efficient but also limited to applications where high operating temperatures and viscosity are not crucial requirements for the user. Another alternative to brominated-flame retardants are phosphorus-based materials, typically available in liquid form for encapsulation resins. As these materials usually also function as plasticizer, the hardness of the encapsulation resin produced is often compromised. In addition, such resins also drip readily when subjected to a heat source (3), preventing them from achieving UL94 V-0 level of flame retardancy and highlighting a major limitation of their use.

To overcome all the above limitations, a novel, non-halogenated flame retardant system has been investigated. Although still a filler, approximately 10 times less material than ATH for example, is required to produce the same level of flame retardancy, therefore making it possible to formulate a flame retardant encapsulation resin with viscosities of less than 700mPa s, whilst still meeting UL94 V-0. In addition, this novel system does not soften the epoxy resin and exhibits excellent stability at very high temperatures, including those seen in typical reflow profiles, due to a much higher decomposition temperature of approximately 300˚C. This paper details the advantages of this novel flame retardant system, highlighting the performance advantage over standard metal hydroxide fillers and concludes with possible applications when formulated into an encapsulation resin.

BACKGROUND

Conventional flame retardants are additives that can be mixed or applied as a treatment to organic materials such as plastics, textiles and timbers. They can also be a chemical modification of a plastic material. Flame retardants function by their interaction or interference with one of the three required components for a fire: **fuel, heat and oxygen**.

Halogenated flame retardant systems function mainly by reducing or eliminating fuel in the vapour phase. Combustion of hydrocarbons generates highly active fragments in the solid or condensed phase. These fragments vaporise, react with oxygen, and form free radicals. Free radical formation is highly exothermic, resulting in volatilization of additional fragments from the condensed phase. The process continues unless free radical formation is interrupted and stable compounds are being produced. When subjected to a flame, brominated flame retardants generate hydrogen bromide which is very effective in deactivating free radicals in the vapour phase. Antimony trioxide is not a flame retardant in its own right but is used in combination with halogenated materials where it exhibits a profound synergism of flame retardancy (4).

In contrast to halogenated flame retardants, phosphorus containing flame retardants function by reducing or eliminating fuel in the condense phase (5). These flame retardants seek to place chemical barriers between the polymer and the fire, and form insulating or minimally combustible chars on polymer surfaces exposed to external heat sources. This char reduces volatilization of active fragments and absorbs and dissipates heat. It is generally accepted that nitrogen synergises with phosphorus to enhance flame retardancy.

Metal hydroxide flame retardants such as Alumina trihydrate (ATH), function not only by removing heat (caloric absorption) but also by diluting or eliminating oxygen from the flame/material interface (smothering effect). These flame retardants decompose to produce water vapour as the non-combustible gas. Heat is absorbed because of decomposition as well as vaporization of liquid water. Water vapour formed also reduces oxygen concentration or even eliminates oxygen at the ignition point. ATH flame retardant begins to decompose at about 200ºC releasing approximately one-third of its original mass as water vapour. Magnesium hydroxide functions in this manner but at a higher temperature (340ºC). Typically, 50 to 100 parts by weight of these compounds are added per 100 parts of resin to achieve adequate flame retardation, which would have a far greater effect on the viscosity of the resin, making potting applications more difficult, however.

Flame retardant efficiency varies from product to product even within each flame retardant category. However, it is generally accepted that halogenated and phosphorus containing flame retardants are superior to metal hydroxide flame retardants in this aspect, i.e. the latter would require a much higher percentage flame retardant loading in the resin formulation in order to achieve a same level of flame retardancy than the other two. As already discussed, phosphorus containing flame retardants can be further enhanced by incorporating nitrogen into the flame retardant system and therefore this investigation was focused on phosphorus and nitrogen containing flame retardants.

PROJECT OUTLINE

Miniaturisation of electronic devices is a common trend in today's electronics industry and as such, the space available for potting is becoming increasingly more limited. Epoxy resin systems based on ATH flame retardants have high viscosities due to the high proportion of ATH required in the formulation to obtain the desired flame retardancy characteristics. In addition, the requirement for materials which are capable of withstanding a reflow profile had been identified through various customer enquiries. Therefore, a clear gap in the market was highlighted for a low viscosity flame retardant epoxy resin that can withstand high temperatures for short periods of time. The following requirements were detailed as a starting point for this development:

- The resin must withstand reflow oven conditions for at least 2 minutes at 245˚C.
- The viscosity of the resin should be low enough to allow it to flow underneath some of the components on the PCB (underfilling).
- The resin must achieve the maximum flammability rating UL 94 V-0.

Further requirements were also detailed for some customer specific applications:

- The cured resin must exhibit a Shore hardness of D45-55
- The coefficient of thermal expansion (CTE) must be less than 100ppm

An essential requirement for encapsulation resins used in the electronics industry is electrical insulation. This again becomes more prominent with the miniaturisation trend, especially when combined with the increasing complexity of devices. Therefore the flame retardants employed in the resin formulation must not introduce any ionic contamination to the system and thus, both electrical and flame retardant properties of the proposed formulations were reviewed.

EXPERIMENTAL DETAILS

Flammability test: The test is conducted in an in-house test facility conforming to ANSI/UL 94 - 2009 specification. The test specimens were prepared as detailed in the standard producing cured resin strips of 130 x 13 x 6mm (1ength x width x thickness). The flammability test was conducted according to UL94 Vertical Burning Tests with a 20mm flame height.

Water absorption test and Ionic materials leach test: A disc of 51mm diameter and 9.7mm thickness was prepared and the resin cured at 23°C for 24 hours. The cured disc was weighed to 4 decimal places and its weight recorded as WS1. 150 g of demineralised water was placed into a clean 250 ml beaker with weight of WC. The sample was then placed in the beaker which was heated and left boiling for 1 hour. More demineralised water was added throughout to ensure the sample was fully immersed in water during the boiling period. On completion, the flask containing the sample was cooled down to 20°C. The sample was then taken out and rinsed with more demineralised water and dried using paper tissues. The sample was weighed immediately to 4 decimal places and recorded as WS2.

Water Absorption % = [(WS2/WS1) -1] x 100

More demineralised water was now added to bring the total weight of the beaker plus the residual water to WC + 200 g. Conductivity of the residual liquor was then measured using a HI 9033 multi-range conductivity meter (HANNA Instruments).

Viscosity Measurement: All viscosity of liquid or paste was measured using Brookfield DV-E viscometer Model RV DE-230.

Shore hardness measurement: Coats Durometer (Coats Machine Tool Co. Limited) was used for all hardness measurements.

Coefficient of Thermal Expansion (CTE) measurement: A fully cured specimen with a dimension of 130mm x 13mm x 6mm was placed in a thermal cycling oven (TAS LT 600 fs.) and heated/cooled to the required temperature and then kept at this temperature for 60 minutes before the length of the specimen was measured. Linear Coefficient of Thermal Expansion was calculated using the following equation:

CTE = (1/L) (Length change/temperature change)

Where L is the length of the specimen at 20 ºC

Thermal Conductivity Measurement: Thermal conductivity of the cured resin was measured using LaserComp FOX 50 heat flow meter instrument.

Electrical Test: Dielectric Strength was tested according to ASTM D149. Volume Resistivity was tested according to (ASTM D991). Both were conducted by an accredited external laboratory.

RESULTS AND DISCUSSIONS

Flammability and Mixed System Viscosity

The new proprietary flame retardant (FR001) which has a phosphorus and nitrogen containing nature, a commercially available ATH (ATH01) and a commercially available phosphorus containing flame retardant (PFR01) were used in some development epoxy formulations. Table 1 gives the mixing resin viscosity and the flammability rating of each formulation. It can been seen that although a desirable viscosity was achieved, the cured formulation C which partially used a phosphorus containing flame retardant (PFR01) drips badly when subjected to a heat source during the burning test, which makes the highest achievable flammability rate to be UL94 V-2. Although the required flame retardancy is achieved the viscosity of formulation B which used an ATH (ATH01) flame retardant is too high to be used for underfilling or for encapsulation of complicated or space-restricted components. In contrast to these two conventional flame retardants, the novel flame retardant system (FR001) achieved both a desirable viscosity and the UL94 V-0 flame retardant rating.

Table 1: Viscosity and flammability of various formulations

	Formulation A	**Formulation B**	**Formulation C**
Part A (resin)			
FR001	23		
ATH01		300	50
PFR01			22
Epoxy base resin	85	85	85
The rest of diluent, filler and additives	60	60	60
Part B (hardener)			
Hardener	21	21	21
Other additives	19	19	19
Flammability and Viscosity			
Mixed System Viscosity (mPa s @20-23ºC)	520	9300	500
Flammability: UL94	V-0	V-0	V-2

Hydrolytic Stability

The cured resins of formulation A and B were boiled in demineralised water for 1 hour and the weight gain of the test specimens from treatment was recorded and water absorption calculated. The residual demineralised water from the test was carefully collected and its conductivity was measured. Table 2 gives the results which indicate that the novel flame retardant system gives a comparable level of water/moisture resistance and residual ionic concentration. The latter is important with regard to electrical insulation property of an encapsulation resin, as ionic contamination would contribute significantly to the conduction of current in the presence of condensed water.

Table 2: Water absorption and ionic leaching of various formulations

	Formulation A	Formulation B
Water absorption (%)	0.13	0.12
Conductivity of water bath before test (µS/cm)	10.9	10.9
Conductivity of residual water bath (µS/cm)	20.5	20.3

Thermal Stability

Thermal stability of the encapsulation resin formulations were initially assessed by the measurement of coefficient of thermal expansion (CTE). In addition, the hardness change before and after a 2 minute exposure to 245ºC was also measured. When tested, formulation A failed to meet the target CTE value of less than 100ppm. For this particular requirement, the hardness is reasonably low for an epoxy resin system; D45-55 in comparison to standard epoxy resin systems at ~D80-90. Therefore, the combination of low CTE and a slightly softer resin are somewhat contradictory requirements and a careful balance between the two properties had to be obtained by further modifying the resin formulation. The formulation A was thus further adjusted to give a new formulation (Formulation D) producing a slightly harder resin. Table 3 shows the CTE and hardness results on the original and adjusted formulations.

Table 3: Thermal stability of resin formulations

	Formulation A	Formulation D
Flame retardant	FR001	FR001
CTE (ppm)	117	92
Hardness before	D45	D50
Hardness after	D42	D50

When subjected to the maximum reflow oven conditions (5 minutes at 245ºC) Formulation D, made using the novel flame retardant system, performed exceptionally well as it was unaffected by the treatment, whilst the conventional formulation B failed badly due to vapour being released after 5 minutes, and eventually cracking after 10 minutes. This can be explained by water vapour formation inside the resin as a result of decomposition of the ATH flame retardant at 245ºC (table 4).

Electrical and other performance properties

The development epoxy encapsulation resin listed as formulation D has been extensively tested and compared to a standard epoxy encapsulation resin, Formulation B, which utilised ATH as the flame retardant. These results are collated in table 4. It is evident that in addition to excellent flame retardancy, a significant reduction in viscosity and density of the resin is achieved using Formulation D, incorporating the novel flame retardant. These properties are extremely desirable as it provides application benefits for the end user as well as the benefit of cost saving and reduction in weight of an assembly. Furthermore, dielectric strength, volume resistivity and other properties of the formulations are either at the same level or comparable. Formulation D is now commercially available as Electrolube ER2218.

CONCLUSIONS

A novel, non-halogenated, highly effective flame retardant has been evaluated alongside conventional flame retardants commonly used in epoxy encapsulation resin formulations. The proposed resin, utilising this novel flame retardant, was found to have a vastly improved thermal stability when compared to standard epoxy encapsulants. This unique feature makes it possible for the encapsulation resin to pass through reflow profiles without affecting its performance properties. The novel flame retardant has a much higher efficiency; when attempting to achieve UL94 V-0, it requires only one tenth of the quantity by weight in comparison to systems utilising alumina trihydrate (ATH). As a result, a much lower viscosity encapsulation resin can be formulated, providing a user-friendly resin for applications with complicated geometry, limited space or for specialist application requirements, such as underfilling. The summarised results show that the proposed resin made from the novel flame retardant also has a much lower density than standard materials, providing cost and weight savings for the end user/assembly, without any compromise of the electrical properties. An Electrolube brand epoxy encapsulation resin has been formulated using the novel flame retardant system and is currently available on the market, making it an ideal choice for applications where the resin is to be subjected to very high temperatures, including those seen in typical reflow profiles. Work is now continuing to establish the additional uses and benefits of this novel flame retardant system in a variety of applications.

Table 4: Encapsulation resins of different flame retardant systems

	Formulation D	Formulation B
Base Material	Epoxy	Epoxy
Part A (resin) Density (g/ml)	1.22	1.83
Part B (hardener) Density (g/ml)	0.96	0.92
Part A Viscosity (mPa s @20-23ºC)	800	150,000
Part B Viscosity (mPa s @20-23ºC)	400	200
Mixed System Viscosity (mPa s @20-23ºC)	500	9,000
Shrinkage	<1%	<1%
Thermal Conductivity (W/mK)	0.28	0.45
Cured Density (g/ml)	1.16	1.69
Heat @245ºC for 5 minutes	Not affected	Vapour releasing/Smoking
Dielectric Strength (kV/mm)	10	10
Volume Resistivity (ohm-cm)	10^{14}	10^{14}
Shore Hardness	D50/A90	D85
Colour (Mixed System)	Black	Black
Coefficient of thermal expansion (ppm/ºC)	80-100	40-60
Water Absorption 10 days @20ºC (9.7mm thick disk, 51mm diameter)	<1.5%	<1.5%
Water Absorption 1 hour @100ºC (9.7mm thick disk, 51mm diameter)	<0.5%	<0.5%

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Presentation Overview

- **Introduction** - Why a new Flame Retardant is required
- **Background** – How do Flame Retardants work
- **Results and discussions**
  - Flammability and mixing viscosity
  - Hydrolytic stability
  - Thermal stability
  - Electrical and other properties
- **Conclusions**

Introduction

- *Why a new Flame Retardant is Required*

Requirement From Market

- A gap in the market - An Epoxy Encapsulation Resin:
  - Thermally stable (245^{o}C, at least 2 mins)
  - Very low viscosity, <700m Pa s
  - Flame Retardant to UL94 V-0
- Special requirements
  - Shore hardness D45 – 55
  - Coefficient of Thermal Expansion (CTE) < 100 ppm

Requirement From Market

- **For an encapsulation Resin it also must have:**
- Good electrical properties
    - ➢High Dielectric Strength
    - ➢High Volume resistivity
- Good hydrolytic properties
- Economic aspects

Flame Retardants for Epoxy

- **Halogenated Flame Retardants** (FR)
- Brominated FR – Most effective
  - 15-20% Bromine needed for UL94 V-0
- Chlorinated FR – Less effective
  - Much higher loading than Brominated
- Issues – Toxic gas, possible Dioxin
- Non-halogenated desirable

Flame Retardants for Epoxy

- **Alumina Trihydrate** (ATH)– A metal hydroxide
- Typical loading 50 – 100 parts per 100 parts resin
- Viscosity: ~10,000 mPa s
- Maximum operating temperature: 150 ~ 200ºC
- Limited for certain applications

Flame Retardants for Epoxy

- **Phosphorus Flame Retardants**
- Available mostly in liquid form
- Function also as plasticisers
- Compromised Resin Hardness
- Highest FR achievable UL94 V-2
  - Drip when being FR tested

Flame Retardants for Epoxy

- **A new Flame Retardant System is therefore required that meets the requirement:**
- Thermally stable – up to 245° C for 2 mins.
- High efficiency – low loading to achieve low viscosity
- No adverse effect on resin's electrical and other performance

Background

- *How do Flame Retardants Work*

Three required components for a fire

FUEL

HEAT

OXYGEN

Brominated Flame Retardant

- **Targeted at FUEL** in ***vapour phase***
- Active fragments (AF) from combustion
- AF + O_2 = Free Radical (exothermic)
  - Lead to more AF generation
- Hydrogen Bromide – A very effective Free Radical deactivator
- Stop flame in the vapour phase

Phosphorus Containing Flame Retardant

- **Targeted at FUEL** in ***condensed phase***
- Generate Phosphoric Acid ($H_3PO_{4)}$ on Burning
- H_3PO_4 Catalyse Char formation
- Char: acting as barriers at interface
- Stop **FUEL** provision to vapour phase

Metal Hydroxide Flame Retardant

- **Targeted at HEAT & OXYGEN**
- Metal Hydroxide decompose to give out water
- **HEAT** absorbed from endothermic reaction and water evaporation
- **OXYGEN** diluted or even eliminated by the non-combustible water vapour

The New Flame Retardant System

- **Phosphorus (P) and Nitrogen (N) containing system**
- **N** synergises with **P** to enhance flame retardancy
- It is proprietary at the present

Results & Discussions

- *What has been done and what has been achieved*

What Has Been Done

- The novel flame retardants evaluated against two conventional controls:
  - Alumina Trihydrate
  - Phosphorus containing
- Flammability, hydrolytic stability, thermal stability, electrical and other performance properties of the 3 resin systems have been compared.

Resin Formulations

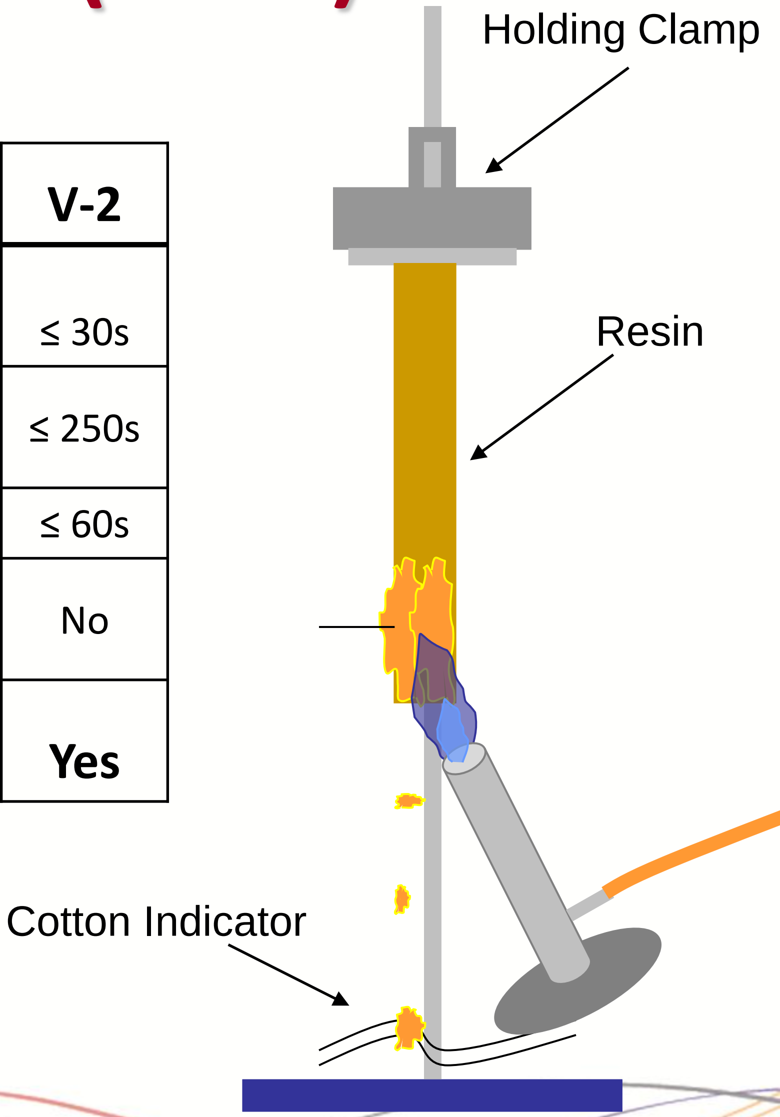
- **Part A (resin)**

Formulation	A	B	C
FR001 (proprietary FR)	23		
ATH01 (alumina trihydrate)		300	50
PFR01 (phosphorus FR)			22
Base Epoxy Resin	85	85	85
Additives	60	60	60

- **Part B (Hardener)** – same for all

Flammability test (UL94)

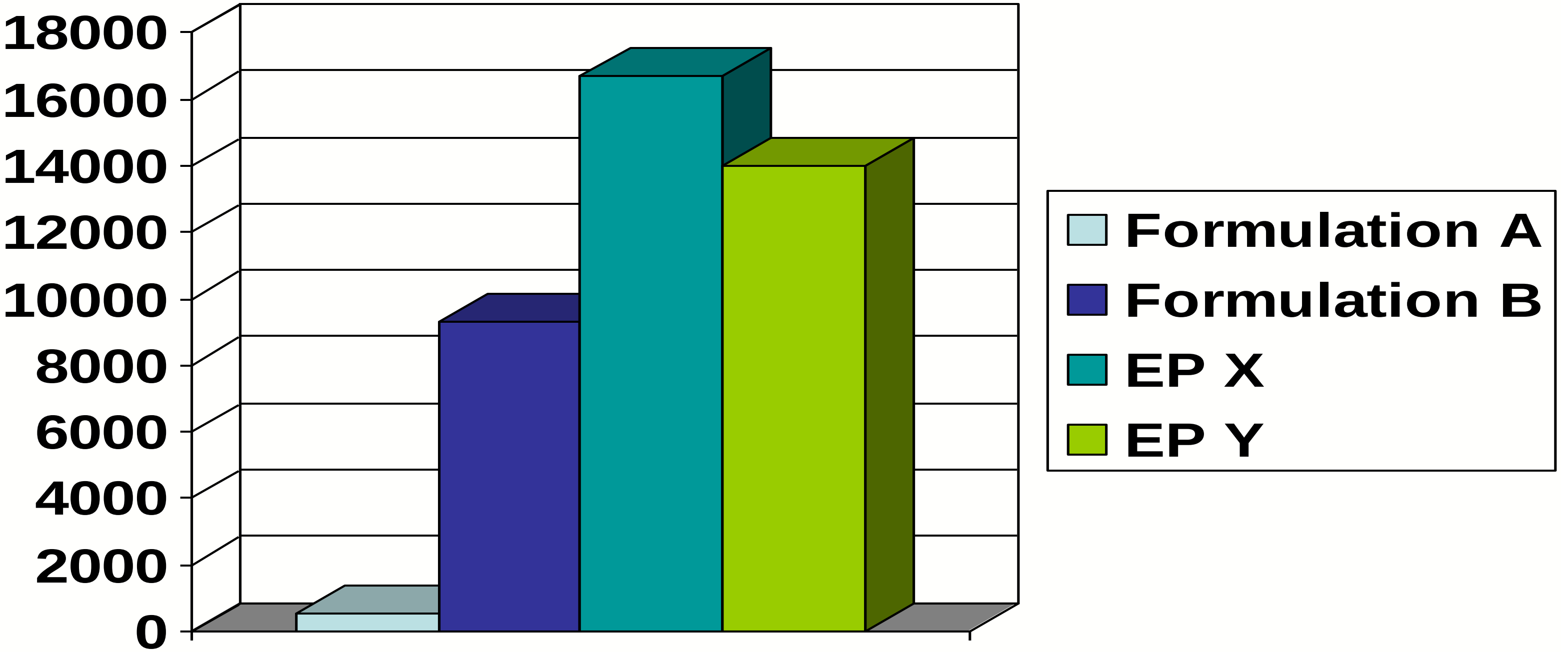
Criteria Conditions	V-0	V-1	V-2
After flame time for each individual specimen t_1 or t_2	≤10s	≤ 30s	≤ 30s
Total after flame time for any condition set $t_1 + t_2$ for the five specimens	≤ 50s	≤ 250s	≤ 250s
$t_2 + t_3$	≤ 30s	≤ 60s	≤ 60s
After flame or afterglow of any specimen up to the holding clamp.	No	No	No
Cotton indicator ignited by flaming particle or drops	**No**	**No**	**Yes**



Flammability and Viscosity

Formulation	A (FR001)	B (ATH01)	C (PFR01)
Flammability (UL94)	V-0	V-0	V-2
Mixed System Viscosity (mPa S)	520	9,300	500

Mixing Viscosity of Typical Epoxy Resins



Hydrolytic Stability

Water Absorption % = [(WS2 / WS1) – 1] x 100

WS1 - Weight before water treatment (g)

WS2 - Weight After water treatment (g)

Specimens were treated at 100ºC for 1 hour.

Hydrolytic Stability

Formulation	A (FR001)	B (ATH01)
Water Absorption (%)	0.13	0.12
Conductivity of Water Bath Before Test (µS/cm)	10.9	10.9
Conductivity of Water Bath After Test (µS/cm)	20.5	20.3

Thermal Stability

- **Three parameters assessed**:

- Co-efficient of Thermal Expansion (CTE)
- Hardness Change after heat treatment
- Physical Change after heat treatment

Thermal Stability

$$\text{CTE} = (1/L)\ \text{X}\ \frac{\text{Length Change}}{\text{Temperature Change}}$$

Where: L = Length at 20°C

Thermal Stability

- Formulation A failed CTE (requirement was <100ppm)
- Normally a harder resin has a lower CTE
- Modification leads to Formulation D, a slightly harder resin

Thermal Stability

Formulation	A	D
Flame Retardant	FR001	FR001
CTE (ppm)	117	92
Hardness before heat treatment	D45	D50
Hardness after heat treatment	D42	D50

Thermal Stability

	Heated @ 245ºC for 5 minutes
Formulation D (FR001)	Not Affected
Formulation B (ATH)	Smoking (Vapour Releasing)

Thermal Stability – Epoxy using ATH flame retardant (245ºC for 10 mins)

Thermal Stability – Epoxy using The novel flame retardant(245ºC for 10 mins)

Electrical and Other Properties

Formulation	D (FR001)	B (ATH01)
Dielectric Strength (KV/mm)	10	10
Volume Resistivity (Ohm-cm)	10^{14}	10^{14}
Cured Density (g/ml)	**1.16**	**1.69**

	Formulation D ER2218	**Formulation B**
Base Material	Epoxy	Epoxy
Part A (resin) Density (g/ml)	1.22	1.83
Part B (hardener) Density (g/ml)	0.96	0.92
Part A Viscosity (mPa s @20-23ºC)	800	150,000
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Conclusions

- An exceptionally low viscosity, high temperature resistant and flame retardant epoxy encapsulation resin (**Electrolube ER2218**) has been formulated using a novel flame retardant system.
- The thus formed resin can pass through reflow profiles without affecting its performance properties.

Conclusions

- The cured density of the encapsulation resin is much lower than that of flame retarded resins using metal hydroxide flame retardant, leading to significant potting cost saving for end users.
- Work is continuing to establish the additional uses and benefits of this novel flame retardant system in a variety of applications.

Thank You!