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CLEAR FLAME RETARDANT HALOGEN FREE POLYURETHANE TAPE FOR AEROSPACE APPLICATIONS

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Del Lavature is the technical group leader at the Bristol, RI plant for Berry Plastics Engineered Materials Division, Specialty Tape Group. Del has over 39 years experience in the R&D development of new tape products and holds seven patents for new products created in the last eight years.

Del is highly experienced in process engineering, quality control, technical testing of products, tape slitting, and many coating methods, and able to take an idea from a concept to commercialization of a salable product, making novel concepts a reality in a short time period. Del has a wide range of knowledge of materials and material science and able to formulate off the shelf polymers and other materials to create unique products to meet challenging customer requirements.

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Introduction

fFame-retardant additives commonly used in PSA tape industry typically contain halogens and antimony compounds to achieve the required level of flame-retardancy [1, 2]. However, due to growing environmental concerns and regulations, the recent demand in the airspace industry is for new "green" chemistry ointhe tape products, which would use non-halogenated and antimony-free products while still meeting customer-specific requirements.

The new halogen and antimony-free, translucent, light-weight PSA tape with moisture barrier properties was developed to meet the Boeing Material Specification BMS 8-346A for Type I Class II materials and flammability complying with BSS 7230 testing standard Method F2 (twelve seconds vertical burn). The PSA tape construction includes a flame-retardant cross-linked acrylic adhesive. The PU film used as a carrier contains a non-halogenated phosphate-based flame-retardant additive added onto Polyether Polyurethane resin. Customer request for the new tape did not fit into the existing Type I Class II category; therefore, a new Class III was created to incorporate the novel product under current revision BMS8-346.

Some of the technical challenges associated with clear FR PU tape development are: the ability of the adhesive to remain clear after FR additive is incorporated, the ability of PSA to bond to the film carrier without adhesive failure in mass anchorage test, along with limitations in physical properties and commercial availability of raw materials. The objective was to develop a clear 7-mil, 12-mil or 16-mil translucent FR PU tape. The attempts to develop such products in the past were not successful; however, the aerospace industry was in need of such a novel product line.

The technical requirements for the new product are the following:

1. The tape will need to have a clear/translucent PU film backing.
2. The PU film should be fungal and mold resistant.
3. The film thickness needs to be lower than typical commercially-available FR products to provide light-weight tape construction. As a result, the new PU film backing should be around four mils thick. Meeting the light-weight requirement placed the new product outside of the existing BMS 8-346A Type I Class II classification and caused the BMS 8-346A Type I Class III specification to be created.

4. The PU film has to be flame retardant, halogen- and antimony-free and must pass a standalone burn test when bonded to BMS4-20 black floor panels, as shown on Fig.1-3 and as listed in Table 5.

5. The PU film must have a puncture resistance of 10 lb minimum ,as tested per ASTM-D1000 (.166" needle pushing through a .312" hole at 2 in/min), as listed in Table 4.

6. The adhesive must be clear and moisture-resistant, as listed in Table 4.

7. The adhesive have to be halogen- and antimony-free.

8. Other requirements to the finished product are listed in Tables 4 and 5.

Since the adhesive will have to be formulated and coated onto the PU film, the original focus was on developing a clear PU film that would meet customer-specific requirements and was not available commercially. The development of FR PU film was done in close collaboration with the PU film manufacturing company, which extruded PU film and was interested in joining the efforts to develop flame-retardant PU film.

There were several PU formulations that failed before the successful one was created. PU films with higher thickness failed when the bonded burn test was performed, as shown on Fig. 8 and 9. Adding more flame retardant had a negative effect on the physical properties of the film, creating a challenge of finding the balance between the content of added flame retardant required to pass the burn test and the required physical properties.

In spite of limited raw material options and technical challenges associated with the product design, extensive bench-work studies showed that the aromatic ether PU film with 4.0 mil thickness and phosphate-based FR additive met customer-specific requirements, as described in reference (3). Repeated PU film extrusion trials were conducted with successful outcomes to produce the film to be used as a backing of a new tape. As a next step, the FR adhesive formulation had to be developed that could bond to the new PU film and provide required PSA properties of the final tape to meet PSA-related part of the customer specification.

Aromatic ether polyurethane is a difficult surface to bond an adhesive to. Due to the poor bond of adhesive to film, adhesive anchorage failure was observed at the interface of the film and the adhesive, as shown on Fig. 10. Attempts to achieve a better adhesive anchorage to the film by hot lamination process did not result in an adequate bond.

It was further found that a variety of PSA products, many commercially available, did not bond to the Aromatic ether PU film developed to meet customer-specific requirements of the application. Adding the primer to improve the bond would not be acceptable due to added cost, which would increase cost outside of acceptable range.

The assumption was made that the reason for the poor bond might be related to the flame retardant additive or other components, such as processing aids used in the extrusion process, "blooming" (moving or migrating) to the surface of the film and interfering with the adhesive's ability to stay bonded to the film. Therefore, individual raw materials used to manufacturer PU film were examined to investigate if one of them had the negative effect on the interfacial bond between adhesive and the film. After examination of each of the raw materials used to produce PU film and additional heat aging studies, it was confirmed that the processing aid used in PU resin formulation was migrating to the surface of the film, thus, resulting in low adhesive anchorage to the film backing. After the resin formulation was adjusted to eliminate the processing aid, adhesive bond to film was improved to an acceptable level.

As a result, the development of the components (PU film and adhesive) required to make the final tape product was complete, meeting customer expectations to product performance. As value-added properties, the product also exhibited high cold temperature adhesion to aluminum substrates used in aircraft building (Table 1) and high dielectric strength (Table 3). An accelerated aging test equal to two years of shelf life was completed with positive results, as presented in Table 2.

Through a number of successful production trials and process engineering, the new product was launched and approved per BMS8-346B Type I Class III specification. The novel PU tape described above is patented, US Patent 7, 501,169 B2.

Table 1: Cold Temperature Tests of the Clear FR PU as tested on Aircraft 2024 Aluminum

Temperature(°F)	**Adhesion Observations**
-5	Good Adhesion, Almost Like Room Temperature.
-10	Low Adhesion
-15	Low Adhesion
-40	Adhesive Delaminates from Film with hardly any effort.

Table 2: Test Results for Clear FR 4mil PU, Lot# 700213

Test	**Reference Method**	**PU Initial:** **Lot#700213**	**PU Aged:** **Lot#700213**
Adhesive Thickness (mils)	ASTM-D-3652	1.6	1.6
Film Thickness (mils)	ASTM-D-3652	4.5	4.5
180° Peel off Stainless Steel, <1min Dwell	ASTM-D-3330	31.6	32.7
180° Peel off Stainless Steel, 20min Dwell	ASTM-D-3330	43.9	39.8
180° Peel off Backing	ASTM-D-3330	8.3	18.8
Loop Tack(oz/in)	PSTC-16	50.4	59.5
Tensile(lb/in)	ASTM-D-3759	37.5	36.5
Elongation (%)	ASTM-D-3759	509.5	496.4

Table 3: Dielectric Properties
Dielectric Strength Test (UL510)
Clear FR Tape: CA1579-86

Test #	Breakdown Voltage(kV)	Tape Thickness (mils)	Dielectric Strength(V/mil)	Conditioning
1a	6.76	5.5	1229.1	RT
1	6.67	5.5	1212.7	96hrs @ 83°F, 96% RH
2	6.34	5.6	1132.1	96hrs @ 83°F, 96% RH
3	6.21	5.6	1108.9	96hrs @ 83°F, 96% RH
4	6.28	5.6	1121.4	96hrs @ 83°F, 96% RH
5	6.22	5.6	1110.7	96hrs @ 83°F, 96% RH
Average	**6.34**	**5.6**	**1137.2**	
Std Dev	**0.19**	**0.04**	**43.24**	

Table 4: BMS8-346A Test Results

Property	**Tape Lot # 1**	**Tape Lot # 2**
Weight (oz/ft^2)	0.5	0.6
Thickness(Mil)	5.5	6.1
Elongation %	506.9	500.6
Tensile Strength (psi)	10556.1	10200.1
180° Peel(lb/in of Width)	Environmental Condition	
Test Substrate	24hrs, Room Temp.	
Aluminum	2.6	2.8
BMS4-20	2.7	2.7
	7 Days 160F Circ. Air	
Aluminum	3.5	3.9
BMS4-20	4.1	4.2
	7 Days 120F/95%RH	
Aluminum	2.3	2.5
BMS4-20	1.1	1.6
Water Vapor Transmission Rate (grams/100in^2)	5.37	5.39
Environmental Resistance	Pass	Pass
Flammability (12 Seconds Vertical): Tape Only		
Extinguishing Time(s)	0.0	0.0
Burn Length(in)	3.4	3.7
Drip Extinguishing Time(s)	0.0	0.0
Moisture Absorption (% Weight Gain)	0.49	0.54
Puncture resistance (lbs)	11.5	12.0

Table 5: BMS8-346B Test Results

Test	Customer Requirements	Lot #1	
Weight (oz/ft^2)	0.70 Max	0.50	8.1
Total Thickness(Mil)	5.0 Min	5.50	8.1
180° Peel(lb/in of Width)	Environmental Condition		8.3
Test Substrate	24hrs, Room Temp.		
Aluminum	0.9 Min	2.6	
Flammability 12 Seconds Vertical) [Unsupported]			8.6
Extinguishing Time(s)	5.0 Max	0.0	
Burn Length(in)	5.0 Max	3.40	
Drip Extinguishing Time(s)	2.0 Max	0.0	
Flammability (12 Seconds Vertical) [Bonded to BMS4-20 Floor Panel]			8.6
Extinguishing Time(s)	10.0 Max	0.0	
Burn Length(in)	8.0 Max	3.77	
Drip Extinguishing Time(s)	2.0 Max	1.5	

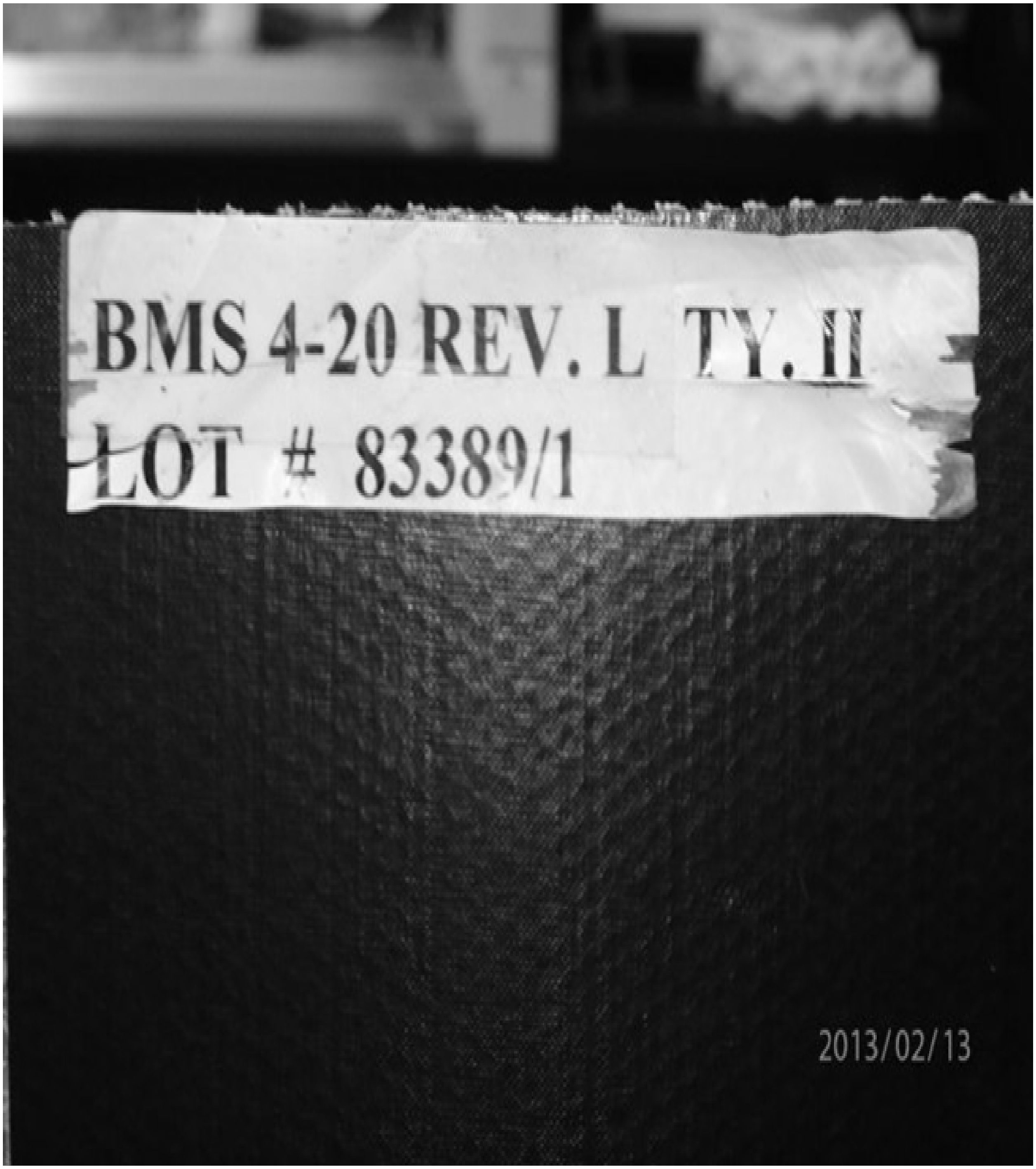

Fig. 1

Original black aircraft floor panel

Fig. 2

Aircraft black floor panel with the flame retardant clear PU tape bonded to it.

Fig. 3

Aircraft black floor panel with the honeycomb construction.

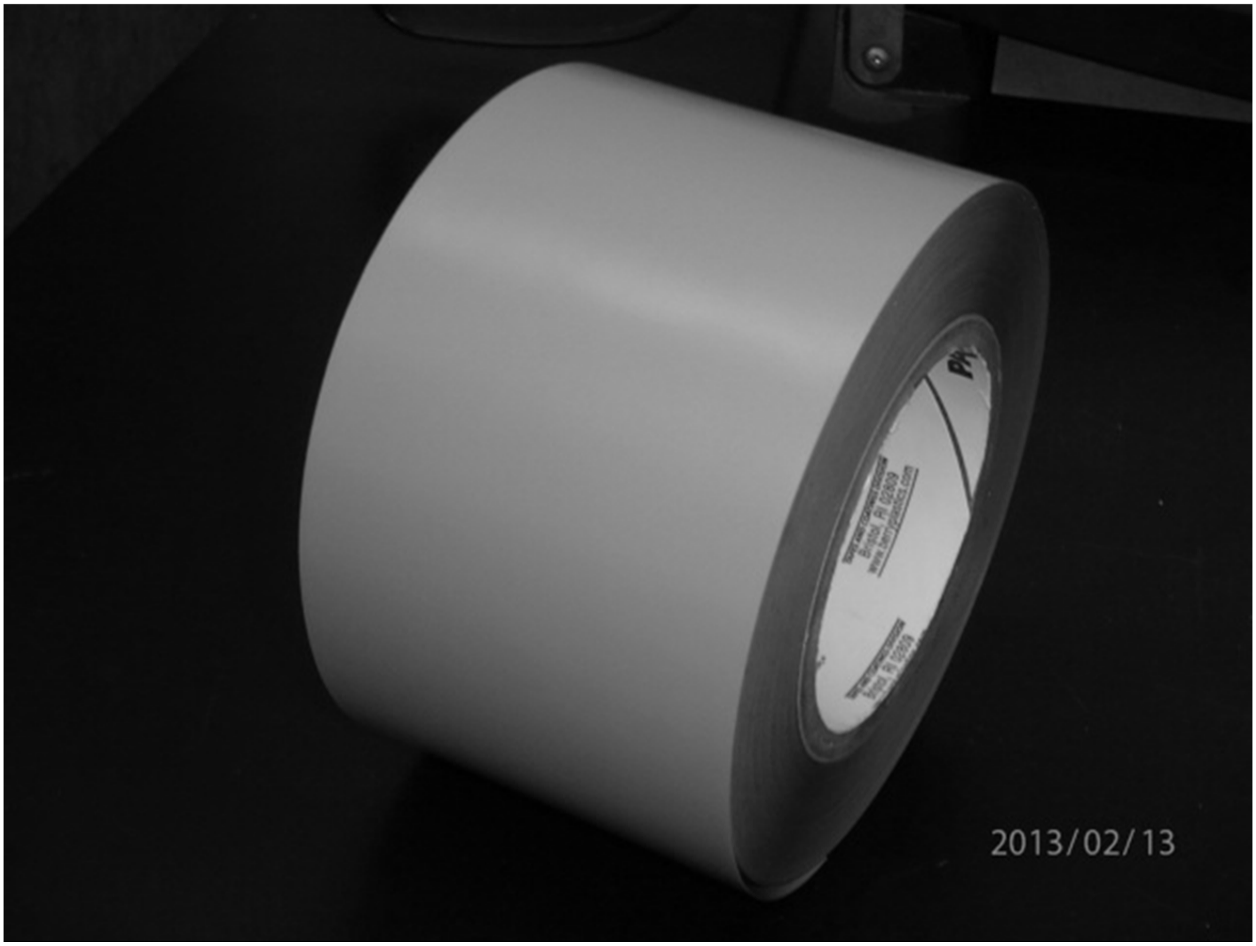

Fig. 4

A roll of the clear flame retardant PU tape laminated to a blue release liner.

Fig. 5

The photo shows the burn length of the clear flame retardant PU after the 12-second vertical burn test in bonded condition.

Fig. 6

Unsupported 12-second vertical burn test setup.

Fig. 7

The photo shows the end of the 12 seconds burn test and the burn length.

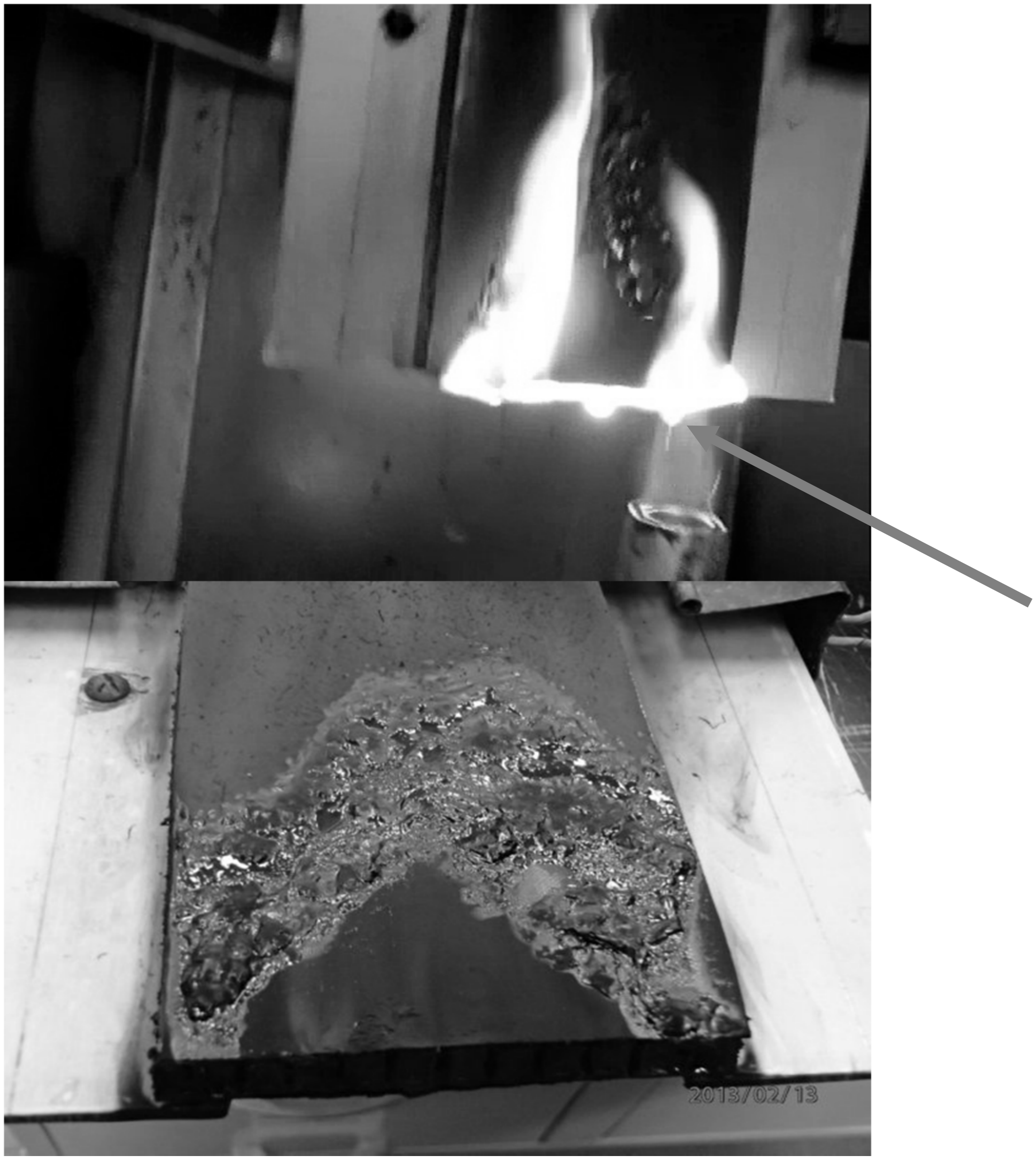

Fig. 8

The photo shows the sample that failed FR test. In the top photo the honeycomb structure at the base of the floor panel is filled with melted burning PU. The arrow is pointing to the flame below the edge of the test panel.

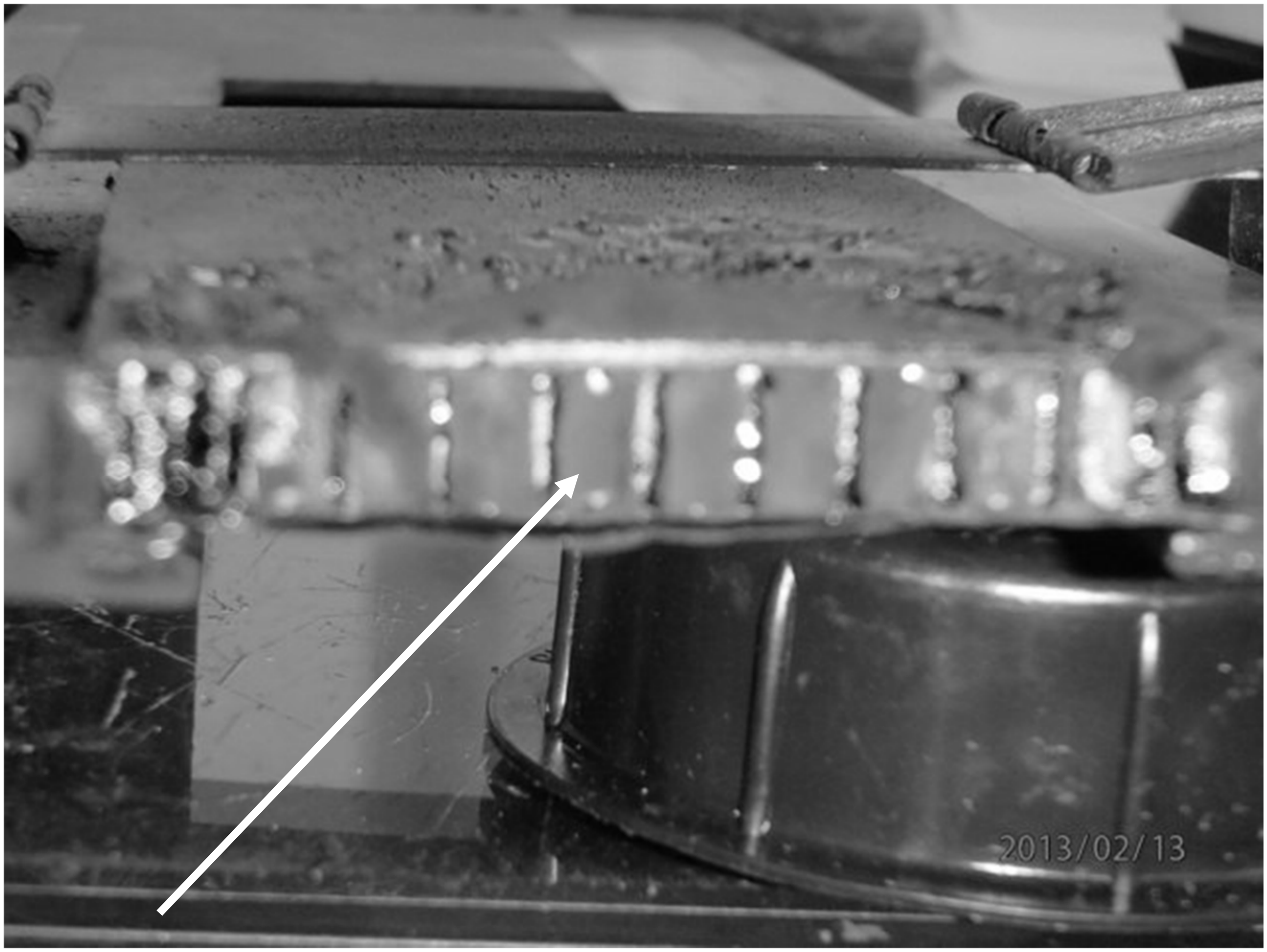

Fig. 9

The arrow is pointing to the honeycomb structure at the bottom of the floor panel that is filled with PU after the burn test.

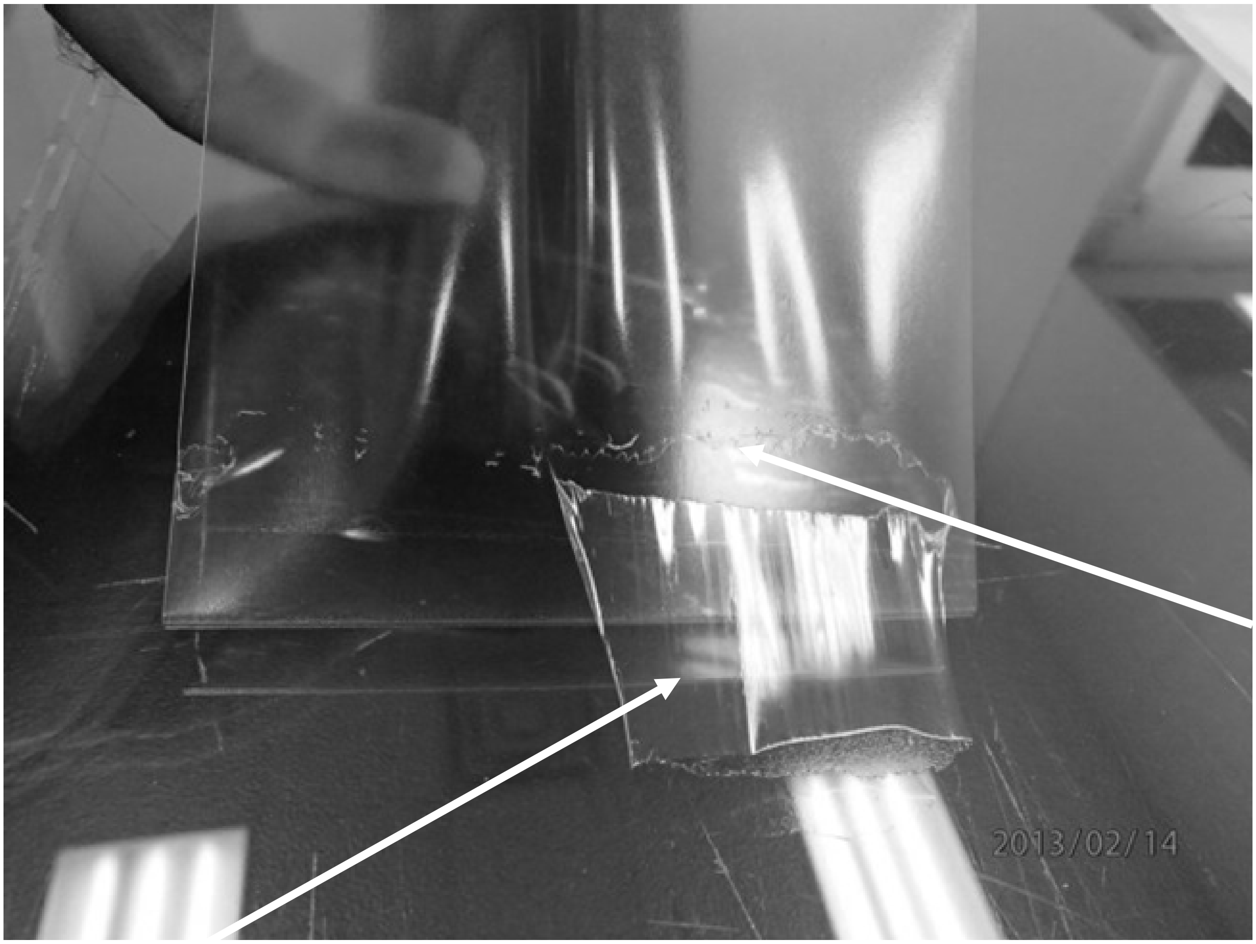

Fig. 10

Adhesive is failing to anchor to the film. The bottom arrow is pointing to the delaminating adhesive and the top arrow is pointing to the area of the film where the adhesive is coming from.

Conclusion

Due to stringent environmental regulations, industry around the world will require "green chemistry" of raw materials and products. Future PSA flame-retardant products need to be non-halogenated and antimony-free. The chemical industry is facing a challenge to meet the growing needs of customers to replace traditional halogenated and antimony-based FR in existing products and develop new "green" products.

The flame retardant additives generally have a negative effect on the physical properties of the base polymer and final product. The challenge is to identify the alternate environmentally-friendly flame-retardant raw materials, allowing formulators, scientists and engineers to produce high-performance products equivalent in properties to the products made with traditional flame-retardant chemistry. Furthermore, the new product made with more environmentally-friendly chemistry has to be recognized by the customer as equivalent to "traditional" products, and tested and approved for use. Creating a new product may require a change in existing customer specification or writing a new specification or standard.

References:

1. "Pressure Sensitive Adhesive Tapes. A guide to their function, Design, Manufacture, and Use" John Johnston, PSTC, 2003

2. "Handbook of Pressure-Sensitive Adhesive Technology", Don Satas, 1989, P.654-656.

3. "Translucent Flame Retardant Tape", Del Lavature, US Patent No. 7,501,169 B2.