

## RESEARCH ARTICLE

# Fire behavior of pressure-sensitive adhesive tapes and bonded materials

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**Abstract**

Pressure-sensitive adhesive tapes are used in several industrial applications such as construction, railway vehicles and the automotive sector, where the burning behavior is of crucial importance. Flame retarded adhesive tapes are developed and provided, however, often without considering the interaction of adhesive tapes and the bonded materials during burning nor the contribution of the tapes to fire protection goal of the bonded components in distinct fire tests. This publication delivers an empirical comprehensive knowledge how adhesive tapes and their flame retardancy effect the burning behavior of bonded materials. With a special focus on the interaction between the single components, one flame retarded tape and one tape without flame retardant are examined in scenarios of emerging and developing fires, along with their bonds with the common materials wood, zinc-plated steel, mineral wool, polycarbonate, and polymethylmethacrylate. The flame retardant significantly improved the flame retardancy of the tape as a free-standing object and yielded a V-2 rating in UL 94 vertical test and raised the Oxygen Index by 5 vol.%. In bonds, or rather laminates, the investigations prove that the choice of carrier and substrates are the factors with the greatest impact on the fire properties and can change the peak of heat release rate and the maximum average rate of heat emission up to 25%. This research yielded a good empirical overall understanding of the fire behavior of adhesive tapes and bonded materials. Thus, it serves as a guide for tape manufacturers and applicants to develop tapes and bonds more substrate specific.

**KEYWORDS**

adhesives, cone calorimeter, flame retardancy, laminates, phosphorus flame retardants, pressure-sensitive adhesive, tapes

## 1 | INTRODUCTION

Pressure-sensitive adhesives (PSA) are permanent tacky polymers that can adhere to a variety of surfaces by applying light pressure. The permanent tack is achieved using polymers, copolymers, or blends with a low glass transition temperature ( $T_g$ ). PSA tapes became very popular in the construction, transport, and automotive industries in recent decades due to their advantages over mechanical fasteners and liquid

adhesives. To mention just a few of their properties: they can absorb noise and vibrations, have good gap-filling properties, are easy to apply and cause no weak spots at bounded surfaces.<sup>1</sup> Despite all these advantages, there is one major disadvantage, namely poor behavior under high temperature and fire conditions. Most PSA consist of rubber-like polymers and are intrinsically flammable.<sup>2</sup> The improvement achieved by using halogen-free flame retardants such as phosphorus-based ones has been investigated in very few papers,<sup>3</sup> so

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that the general burning behavior of tape-bonded materials is not yet understood. Previous research has shown that combinations of polymeric materials behave differently in fires than the sum of the single components,<sup>4</sup> which indicates that the multi-layer arrangement of bonded substrates has its own specific fire properties. It is well known that thin materials such as films and coatings can change the burning behavior drastically and exhibit special burning characteristics,<sup>5-7</sup> and that the adhesive formulation can have an influence on the burning behavior of bonded construction elements,<sup>8-10</sup> which leads to the assumption that double-sided PSA tapes as thin films will impact the burning behavior of bonded materials. Since the literature shows that non-flammable interlayers in materials can improve the flame retardancy of laminates,<sup>11</sup> it is supposed that varying the carrier (the middle layer in double-sided adhesive tapes) has a strong impact on the burning behavior of bonded materials. All these clues lead to the question as to whether and in which way PSA tapes influence the burning behavior of bonded construction and passenger transport materials. How do PSA tapes impact flammability and flame spread? Are there any direct links between the flame retardancy of the free-standing adhesive tape and the burning behavior of the distinct bonded substrates? Do different tape-substrate configurations and different fire scenarios highlight different phenomena? These are the main issues of this research paper and are investigated to avoid human and economic damage and health hazards. Acrylate based tapes as the most common class of PSA for durable product is intrinsically flammable due to their polyacrylate backbone with hydrocarbon chains. The pyrolysis and the fire properties of a flame-retardant tape and a non-protected tape are investigated, and subsequently these tapes are used to manufacture sandwich-like bonds between common construction and passenger transport materials. The interactions between different material combinations and the effect of flame retardants in the adhesive formulation on different setups are then determined in a variety of materials. To address different carriers, aluminum as a non-flammable material and polyethylene terephthalate (PET) as a flammable polymer were compared in the developing fire scenario. This comprehensive empirical approach delivers a valuable insight in the fire behavior in different fire scenarios of the freestanding tapes, tapes in contact with substrates, and the bonded components. Differing properties are identified as key to understand the distinct fire properties, the conclusions may serve as guideline for future tailored development.

## 2 | MATERIALS AND METHODS

### 2.1 | Materials

Two double-sided tapes with acrylic adhesives coated on a nonwoven PET were provided by Lohmann GmbH & Co. KG (Germany). The tapes differed in their adhesive formulation. The commercially available flame retarded DuploColl® 94 100 FR, referred here as Tape FR, and a tape with the same basis formulation without flame retardant, referred here as Tape RE, were investigated. In addition, transfer

tapes (adhesives without a carrier) were provided for the pyrolysis investigations of the different adhesives. The double-sided adhesive tapes were used in combination with five different substrates, namely beechwood, zinc plated steel, mineral wool, polymethylmethacrylate (PMMA), bisphenol-A polycarbonate (PC) and acrylonitrile butadiene styrene copolymer (ABS). The beechwood specimens were cut from untreated beechwood planks. The wooden samples were all cut in the same fiber orientation since this influences fire testing results.<sup>12</sup> The zinc plated steel was used in a thickness of 1 mm and prepared via guillotine cutting. Rockwool Termarock 50 was purchased and used as an insulating wool with a defined raw density of 50 kg/m.<sup>3</sup> Extruded colorless PMMA (Plexiglas® XT) from Evonik Industries AG (Germany) served as the thermoplastic, non-charring substrate and was cut with a buzz saw to the demanded sizes. PC from Covestro AG (Germany) (Makrolon® GP) was used as a second plastic substrate and cut with a buzz saw. PC and PMMA were purchased from Thyssenkrupp Plastics GmbH (Germany) in the dimensions 1000 × 2000 × 2 mm<sup>3</sup>. ABS plates were purchased from S-Polytec GmbH (Germany) and cut with a buzz saw from a 1000 × 1000 × 1 mm<sup>3</sup> plate. For the investigation of different carriers, an aluminum foil was purchased from VWR International GmbH, Germany in a thickness of 30 μm.

The substrates were chosen due to the large variation in their burning characteristics and their industrial applications. Sandwich-like samples were manufactured by combining them with the acrylic adhesive tapes as intermediate layers (substrate/tape/substrate). The fire behavior of these samples was compared to the homogenous substrates to determine the effect of the tapes. A wide range of sample dimensions was used and varied depending on the material and test scenario. To manufacture coated samples, the adhesive tapes were adhered to the substrates; then air bubbles and inhomogeneities in the surface were eliminated via hand-pressure-roll. The sandwich elements were manufactured by releasing the liner paper of the acrylic adhesive tapes and bonding the second substrate layer on top of the tape surface. Again, the pressure roll was applied to optimize the homogenous contact between tape and substrate. Additionally, sandwich elements with an intermediate aluminum layer were manufactured (PMMA/adhesive tape/aluminum foil/adhesive tape/PMMA). In this case, the aluminum foil was coated with the double-sided tapes on each side and subsequently incorporated into the laminates in the same manner as described above.

### 2.2 | Methods

#### 2.2.1 | Mechanical tests

The mechanical properties of the bonds prepared with the double-sided tapes were analyzed in peel and SAFT tests to investigate the influence of the flame retardant on the adhesive strength and temperature resistance. The peel test was carried out according to DIN EN ISO 29862 (1939) and measured the peel strength at an 180° angle. In order to test them as single-sided tapes, the double-sided tapes were laminated on

either PET or aluminum foil. Specimens  $24 \times 300$  mm in size were laminated onto stainless steel ( $50 \times 200$  mm) according to Afera 5013 and stored for 24 h in a climate chamber at  $23^\circ\text{C}$  and a relative humidity of 50%. The test was performed using an Instron universal testing machine with a peel rate of  $300\text{ mm/min}$ . The SAFT test was carried out according to Afera 5013 and measured the thermal stability of the bond. Single-sided tapes were prepared as described for the peel test. PSA tapes/laminates were bonded to a standard steel ( $50 \times 100\text{ mm}^2$ ) with a contact area of  $24 \times 24\text{ mm}^2$  and placed in the test rack. A heating rate of  $0.5\text{ K min}^{-1}$ , a maximum temperature of  $160^\circ\text{C}$  and a weight of 500 g were applied. For both mechanical tests, five specimens were tested for each tape.

## 2.2.2 | Pyrolysis analysis

To analyze the thermal decomposition of the adhesive, a transfer film was investigated by TGA in a Netzsch TG 209 F1 Iris (Germany) under nitrogen atmosphere (flow:  $30\text{ mL min}^{-1}$ ). 10 mg of adhesives were cut out of a representative transfer film sheet and subsequently adhered to the bottom of the crucible. The sample was heated from  $30^\circ\text{C}$  to  $900^\circ\text{C}$  at a heating rate of  $10\text{ K min}^{-1}$ . The emerging gases were transferred to a Bruker Optics Tensor27 infrared spectrometer, where the IR analysis took place. The transfer line was heated up to  $270^\circ\text{C}$ .

The transfer films were investigated in Py-GC/MS to investigate the emerging gases during pyrolysis.  $30\text{ }\mu\text{g}$  adhesive samples were pyrolyzed in a micro-furnace double-shot pyrolyzer (PY3030iD, Frontier Laboratories, Japan) at  $500^\circ\text{C}$  and subsequently led via split-/splitless inlet port to a gas chromatograph (7890B, Agilent Technologies, USA). The following column parameters were used: Ultra Alloy  $+-5$  capillary column ( $l = 30\text{ m}$ ,  $iD = 0.25\text{ mm}$ , film thickness =  $0.25\text{ }\mu\text{m}$ ), helium flow:  $1\text{ mL min}^{-1}$ . Column temperature:  $40^\circ\text{C}$  for 2 min. Then a heating ramp of  $10^\circ\text{C min}^{-1}$  to  $300^\circ\text{C}$  followed. This temperature was kept constant for 10 min. The mass spectrometer used was a mass selective detector (5977B, Agilent Technologies, USA) using  $70\text{ eV}$  ionization energy and a scan range of  $15\text{--}550\text{ amu}$ . The split was adjusted to 1:30 and the GC injector was used at  $300^\circ\text{C}$ . The peaks were referenced with the NIST14 MS library.

Hot stage FTIR can give useful insights into the chemical processes taking place in the condensed phase during pyrolysis.<sup>13,14</sup> Halogen-free flame retardants, based on phosphorus, can act in different modes in the condensed and gas phases. The adhesives of Tape RE and Tape FR were measured horizontally as transfer films in a THMS600 cell from Linkam, UK. The IR transmission spectra were recorded by a Lumos 2 IR microscope from Bruker, USA. Representative pieces ( $3 \times 3 \times 0.1\text{ mm}^3$ ) of the films were cut out of DIN A4 sheets and placed on a plain KBr window. The samples were heated from room temperature to  $600^\circ\text{C}$  at a heating rate of  $25^\circ\text{C min}^{-1}$ . The first spectra were recorded at  $100^\circ\text{C}$  followed by measurement intervals of  $50^\circ\text{C}$ . After  $350^\circ\text{C}$  was reached, the interval was decreased to  $10^\circ\text{C}$  until a temperature of  $600^\circ\text{C}$  was reached.

PCFC measures the heat release rate of pyrolysis gases of materials on a small scale and can give information about the combustion

of the gases released from a material during pyrolysis.<sup>15,16</sup> Representative 5 mg adhesive samples were cut out of transfer films of adhesives and subsequently stuck to the bottom of the crucible. The measurements were performed in a FAA Micro Calorimeter (Fire Testing Technology Ltd., UK) according to ASTM D7309-21b. At a heating rate of  $1\text{ K s}^{-1}$ , the samples were heated from  $100$  to  $750^\circ\text{C}$  in a nitrogen atmosphere with a flow of  $80\text{ mL min}^{-1}$ . Subsequently the gases were led to the combustor, where the oxidation process took place at  $900^\circ\text{C}$  under an additional oxygen flow of  $20\text{ mL min}^{-1}$ . The heat capacity was determined by analyzing the oxygen depletion and subsequently applying the Huggett's relation.<sup>17</sup>

## 2.2.3 | Flammability tests

The vertical UL 94 test is one of the most widely applied flammability assessment tests for polymeric materials.<sup>18</sup> It was performed in an UL 94 chamber from Fire Testing Technology (UK). The thickness of the tapes was increased, and an eight-layer specimen was manufactured to avoid the evasion of the  $20\text{ mm}$  flame. The samples were  $13 \pm 0.5\text{ mm}$  wide,  $125 \pm 1\text{ mm}$  long and  $0.9 \pm 0.1\text{ mm}$  thick.

The OI was determined according to ISO 4589 in a standardized apparatus from Fire Testing Technology (UK) and is a common small burner test to investigate the flammability of materials in the scenario of an emerging fire. A representative piece of tape was cut and subsequently folded to ensure that the bending stiffness of the tape was sufficient. The emerging sample was then able to stand free without support in the clamp of the test apparatus. The dimensions of the resulting specimen were  $70 \times 65 \times 3\text{ mm}^3$ . The specimen size was chosen to minimize the amount of sample needed and to achieve sufficient bending stiffness for the free-standing material in the clamp.

The fire behavior of the tapes that were glued on one side of a substrate plate was determined in the single flame source test according to ISO 11925-2 to systematically investigate the interaction between tape and substrate. Edge application of the flame was chosen and a burning chamber from Fire Testing Technology was used. The  $20\text{ mm}$  flame was applied for 30 s.

## 2.2.4 | Fire behavior in the cone calorimeter

The cone calorimeter (Fire Testing Technology, UK) was used to investigate the fire behavior in developing fires under forced flaming conditions according to ISO 5660. A distance of  $35\text{ mm}$  between the sample and cone heater and an irradiation of  $50\text{ kW m}^{-2}$  were chosen to avoid contact of the samples with the cone heater and provide for homogenous heat and irradiance distribution over the specimen surface.<sup>6</sup> A distance longer than the standard ( $25\text{ mm}$ ) was used due to the charring and expansion of PC in the cone calorimeter test. The high irradiation level is the same for a standard cone calorimeter test used to assess railway vehicle components according to EN 45545. Specimens of  $100 \times 100\text{ mm}^2$  were prepared with a thickness of  $4\text{ mm}$  for PMMA and PC and  $8\text{ mm}$  for wood. Each material was

**TABLE 1** Peel and SAFT test of Tape RE and Tape FR with the use of different carriers.

Tape	Carrier	Peel test		SAFT test	
		$F_{\text{mean}}$ (N/mm)		$T$ (°C)	
Tape RE	Al	1.22	±0.17	119.3	±1.0
	PET	0.82	±0.03	71.6	±5.7
Tape FR	Al	0.65	±0.12	116.6	±2.7
	PET	0.66	±0.05	104.6	±11.4

measured as a monolithic substrate plate and the burning behavior was compared to that of two tape-bonded substrates. The thickness of the substrates was 2/4 mm. The samples were measured in an aluminum tray without a retainer frame, resulting in an irradiated surface area of 100 cm<sup>2</sup>. For wooden samples, four metal wires were mounted as a grid to avoid distortion or bending of the flat sample. Both acrylic double-sided tapes and the silicon-based tape were measured as a coating on beechwood, zinc plated steel and mineral wool. Sandwich elements of beechwood, PC and PMMA were measured under the same conditions. Time to ignition ( $t_{\text{ig}}$ ), heat release rate (HRR), peak of heat release rate (PHRR), maximum average rate of heat emission (MARHE), fire growth rate (FIGRA) and the total heat evolved (THE) were emphasized as the most important result values.

### 2.2.5 | Flammability of bonded substrates

The flammability of bonded materials was determined via OI and UL 94. For UL 94 tests, sandwich samples in the dimensions of 125 × 13 × 4 mm<sup>3</sup> were manufactured. Samples of ABS and zinc plated steel (2 mm) were prepared to investigate the behavior of thinner materials bonded by tapes. For OI measurements, samples 100 × 10 × 4 mm<sup>3</sup> in size were manufactured corresponding to the standard.

## 3 | RESULTS AND DISCUSSION

### 3.1 | Tape analysis

The peel test in Table 1 shows that the Tape FR has less peel strength compared to Tape RE. This behavior is often to be found in adhesives where additives are added, as part of the adhesive formulation that is responsible for the cohesive and adhesive strength is exchanged for the flame retardant. The peel strength is still sufficient for a non-structural bond. The SAFT test in contrast, shows that the heat resistance of Tape FR is similar and with the PET carrier even higher than the heat resistance of Tape RE.

The pyrolysis and fire behavior of both tape adhesives were analyzed to obtain information about the decomposition of the basic formulation of the tape and the efficiency of the flame retardants. The TGA curve of the tapes (Figure 1a) shows a shift in the temperature at

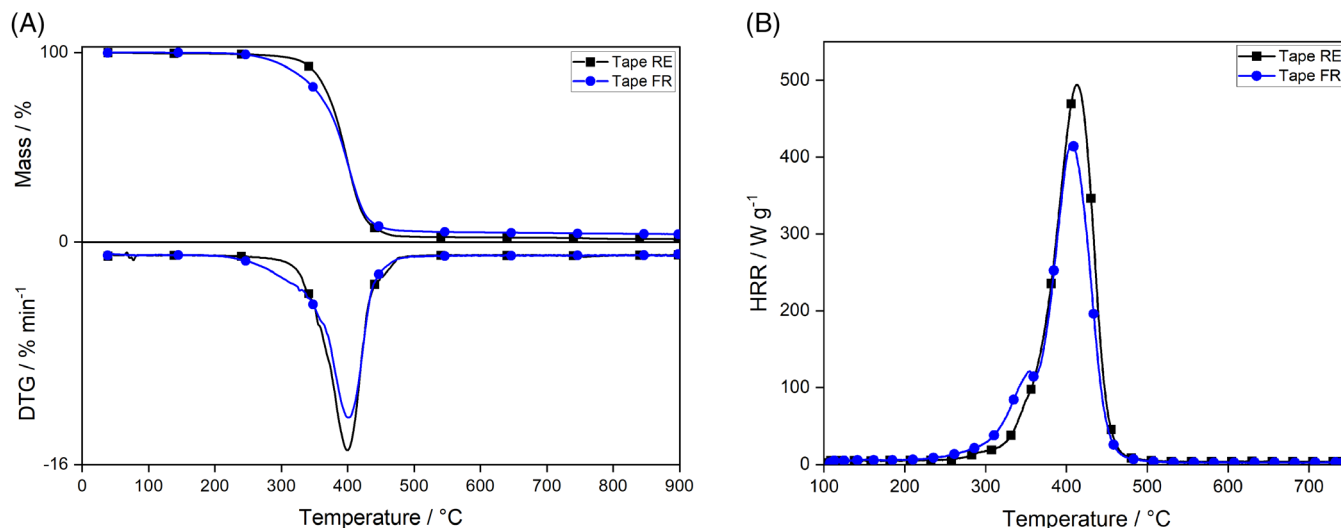
the beginning of decomposition. Tape RE starts to decompose later and loses 5% of its mass at 333°C, whereas Tape FR loses 5% mass at 292°C (Table 2). The main decomposition temperatures of both tapes are around 400°C, which leads to the assumption that the flame retardant decomposes and is mainly released earlier. The residue of Tape FR is higher by 2.3 wt.%, which proves that there is a slight condensed phase action of the flame retardant.

The PCFC measurements of the adhesives in Table 3 show distinct behaviors for Tape RE and Tape FR (Figure 1b). Tape FR has a shoulder at 350°C, which can be attributed to the flame retardant that volatilizes earlier. Thus, the HRC, which is defined as the PHRR/heating rate, decreases by 15.7%. The fire growth capacity, which is an indicator for the fire hazard of materials,<sup>19</sup> decreases by 7.1% compared to Tape RE, because Tape FR has improved charring behavior and some fire load was replaced by flame retardant. The residue of Tape FR (2.2 wt.%) is higher than the residue of Tape RE (1.4 wt.%). All these results agree with those of the TGA and confirm the slight condensed phase action of the flame retardant.

To analyze the condensed phase during pyrolysis, the sample was heated under nitrogen atmosphere while recording the IR transmission spectrum. Figure 2 shows the hot stage FTIR transmission spectra of Tape RE and Tape FR at different temperatures. The spectra were normalized based on the maximum of the CO peak at 1735 cm<sup>-1</sup>. Figure 2a shows the spectra of Tape RE and Tape FR at 100°C for comparison. Both adhesives show the typical peaks for acrylates, with maximum absorption at 1740 cm<sup>-1</sup> due to the strong CO vibration. The C—O—C stretching of the ester group can be found between 1300 and 1100 cm<sup>-1</sup>.<sup>20</sup> In this area, the absorbance of the films is too high for the differentiation of the peaks and yields in a cut-off peak. The C—H bending vibrations of the alkyl rest takes place at 1454 and 1376 cm<sup>-1</sup>.

The peaks at 832, 894, 1072, and 1315 cm<sup>-1</sup> exist only for the Tape FR, which leads to the conclusion that these peaks are specific to the flame retardant, as this was the only difference between the two adhesive formulations. Some of their bands are also found in many phosphorus flame retardants that have been investigated in the literature.<sup>21,22</sup> For instance, the 1315 cm<sup>-1</sup> peak is typical for PO vibrations and 1070 can be referred to a P—O—C vibration. Figure 2b shows a decrease in the flame retardant peaks already at a low temperature (250°C), which shows that the flame retardant or its decomposition products are volatilized and can act in the gas phase. Figure 3 shows both tapes after heating them to 600°C. Tape RE decomposes and has a smooth surface with bubble-like charred spots on its surface, while Tape FR builds a char network at around that temperature. The char network is the effect of the flame retardant acting with the nonwoven PET used as carrier for the flame retarded adhesive, increasing the residue and potentially acting as a barrier.

The Py-GC/MS was used to identify the volatile pyrolysis products of the tape adhesives. Figure 4 depicts the chromatogram of the Tape FR adhesive and chemical information of the pyrolysis products. The decomposition of the acrylate is proven to be a typical depolymerization as shown in the literature.<sup>23,24</sup> The volatile products can be identified as shown in Table 4.



**FIGURE 1** (A) TGA mass loss and DTG comparison between Tape RE and Tape FR adhesives. (B) Pyrolysis combustion flow calorimeter measurements of Tape RE and Tape FR.

**TABLE 2** 5% mass loss,  $T_{\max}$ , and residue comparison of Tape RE and Tape FR adhesives in TGA.

Sample	$T(m = 95\%)$ (°C)	$T_{\max}$	Residue (wt.%)
Tape RE	$333 \pm 2$	$398 \pm 1$	$1.4 \pm 0.2$
Tape FR	$292 \pm 2$	$399 \pm 1$	$3.7 \pm 0.5$

**TABLE 3** PCFC results for Tape RE and Tape FR.

Sample	Residue (wt.%)	HRC ( $\text{W g}^{-1}$ )	FGC ( $\text{W g}^{-1} \text{s}^{-1}$ )
Tape RE	$1.4 \pm 0.3$	$497 \pm 6$	$402 \pm 2$
Tape FR	$2.2 \pm 0.1$	$419 \pm 5$	$326 \pm 7$

The decomposition products are either educt-acrylates that emerged during the depolymerization, copolymer educts, flame retardant or inhibitor. The good volatilization of the flame retardant corresponds to the behavior of flame retardants with a gas phase mode of action and is coherent with the information from TGA and PCFC. It is very common and effective to protect films like adhesive tapes by gas phase active flame retardants but not yet understood, how these tapes and flame retardants work in bonds.

### 3.2 | Flammability tests

The vertical UL 94 test of both tapes measured as a single layer film did not achieve a vertical rating, because it either escaped the burner flame or ignited and burned to the clamp within a few seconds. The multilayer samples of Tape RE no longer shrank away from the flame, instead igniting and burning up to the clamp within 30 s, which results in no UL 94 V rating. Tape FR, by contrast, ignited and self-extinguished by dripping. The drops ignited the cotton wool, leading

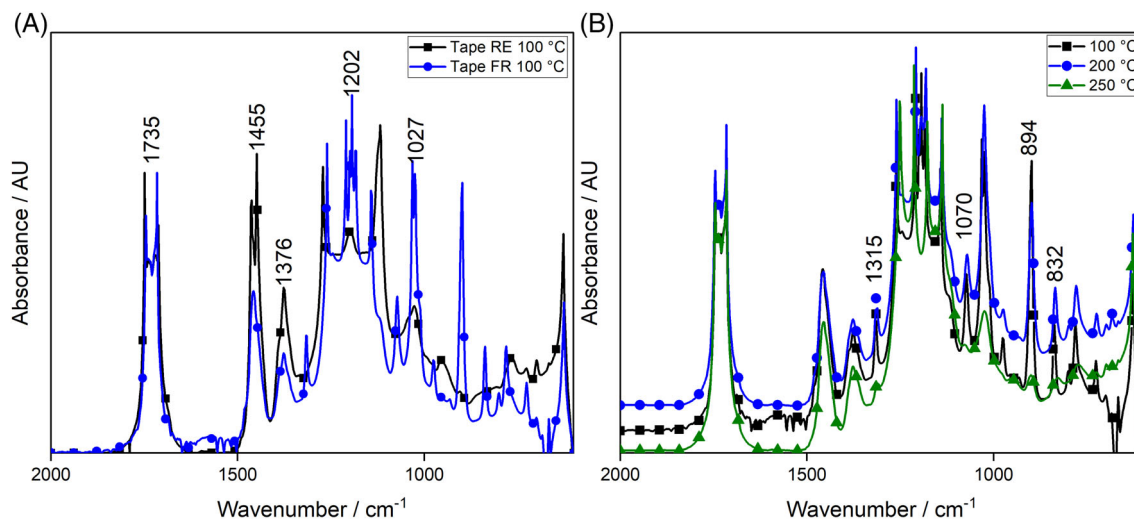
to a V-2 rating for the tape. Folded tapes were used to avoid shrinking of the tapes and in order to obtain consistent results. Tape RE burned self-sufficiently at an oxygen concentration of 17.5 vol.%. Tape FR needed an oxygen concentration of 22.8 vol.% to burn continuously. The large difference of 5.3 vol.% confirms the good protective effect of the flame retardant. UL 94 Test and OI Values are to be found in Table 5.

### 3.3 | Tapes as coatings

To investigate the interaction between tape and substrate, the above-mentioned tapes were investigated as free-standing films and coatings in the single flame source test (according to ISO 11925-2) as illustrated in Figure 5. All free-standing films dodged the burner flame by shrinking away from the burner flame so that the films were self-extinguishing. As coatings, the burning characteristics varied strongly on different substrates. On the zinc plated steel substrate (Figure 5a), neither tape ignited within 30 s and almost no harm to the tape was recognized. After ignition on wood (Figure 5b), both tapes burned differently. Tape FR extinguished after the burner flame was removed, whereas Tape RE kept burning self-sufficiently until the whole tape and large parts of the substrate were consumed by the flame. On mineral wool (Figure 5c), both tapes ignited after approximately 2 s. All of Tape RE was consumed after 23 s, whereas Tape FR was self-extinguishing and only consumed in the area surrounding the burner flame. In contrast to the free-standing tape, the substrates served as mechanical support, so that shrinking away from the burner flame and dripping were prevented.

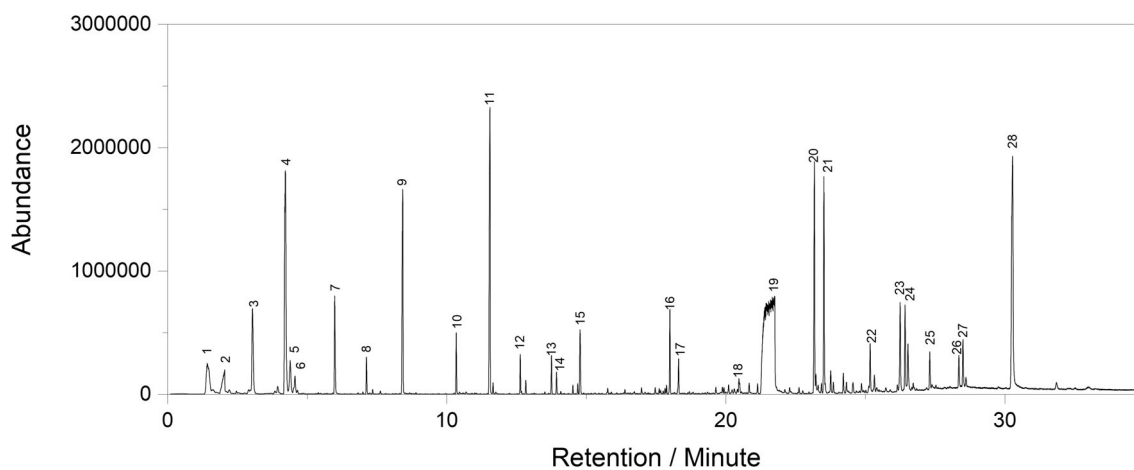
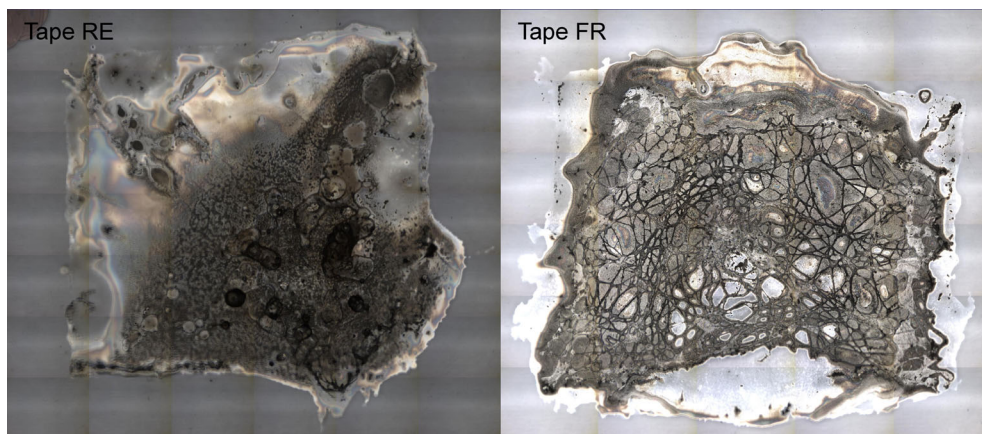
The different burning characteristics of the adhesive tape on the varying substrates can be easily explained by the different thermal conductivity, specific heat capacity, and the density of the substrates, or better, by the different square root of the product of these





**FIGURE 2** Hot stage measurements of Tape RE and Tape FR adhesives: (A) comparison of IR spectrum at 100°C; (B) Tape FR at 100, 200, 250°C.

**FIGURE 3** Hot stage images of Tape RE and Tape FR after pyrolysis at 600°C.



**FIGURE 4** Pyrolysis gas chromatogram of Tape FR.

properties, their heat effusivity (Table 6). The values were taken from the literature and shall only depict the magnitude<sup>25–27</sup> of the materials' ability to discharge thermal energy from the tapes.

The condition for ignition in this test is that the fuel gases emerging from thermal decomposition of the tapes or samples produce an ignitable mixture with the surrounding air.<sup>12</sup> For zinc plated steel, this

**TABLE 4** Decomposition products of Tape FR in Py-GC/MS.

Peak number	Time (min)	Decomposition product
1	1.409	CO <sub>2</sub>
2	2.04	Acetic acid
3–6	3.036–4.638	Aliphatic copolymer
7	5.99	Aromatic copolymer
8 + 9	7.12–8.42	Alkanol rest from acrylates
10–15	10.3–15.0	Educt-acrylates and decomposition products
16 + 17	17.9–18.4	Dimers of acrylate decomposition products
18 + 19	20–22	Phosphorus based flame retardant
20–21	23–25	Dimers and trimers of acrylates
22–28	26–30	Inhibitor and decomposition products

**TABLE 5** UL 94 test and Oxygen Index of both adhesive tapes as layered specimens.

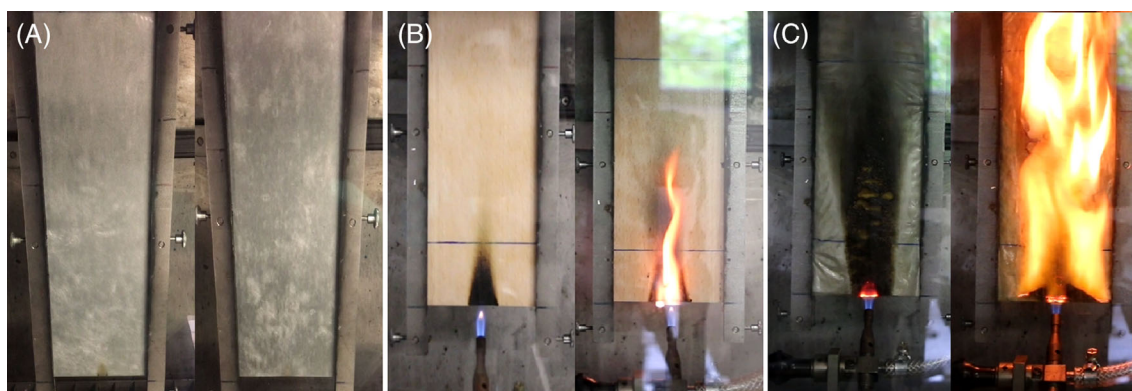
Adhesive tape	UL 94 V rating	Oxygen index (vol.%)
Tape RE	N.R.	17.5 ± 0.2
Tape FR	V-2	22.8 ± 0.2

condition is not fulfilled due to the low temperatures at the application point of the flame. Metals are very good heat conductors, and thus the heat of the applied flame was distributed too quickly to reach the temperatures required for decomposition. The maximum temperature is 210°C at 60 s (end of test) (Figure 6a). The maximum surface temperature of mineral wool in contrast, exceeds 500°C (510°C), which is quite sufficient for decomposition and ignition (Figure 6b).

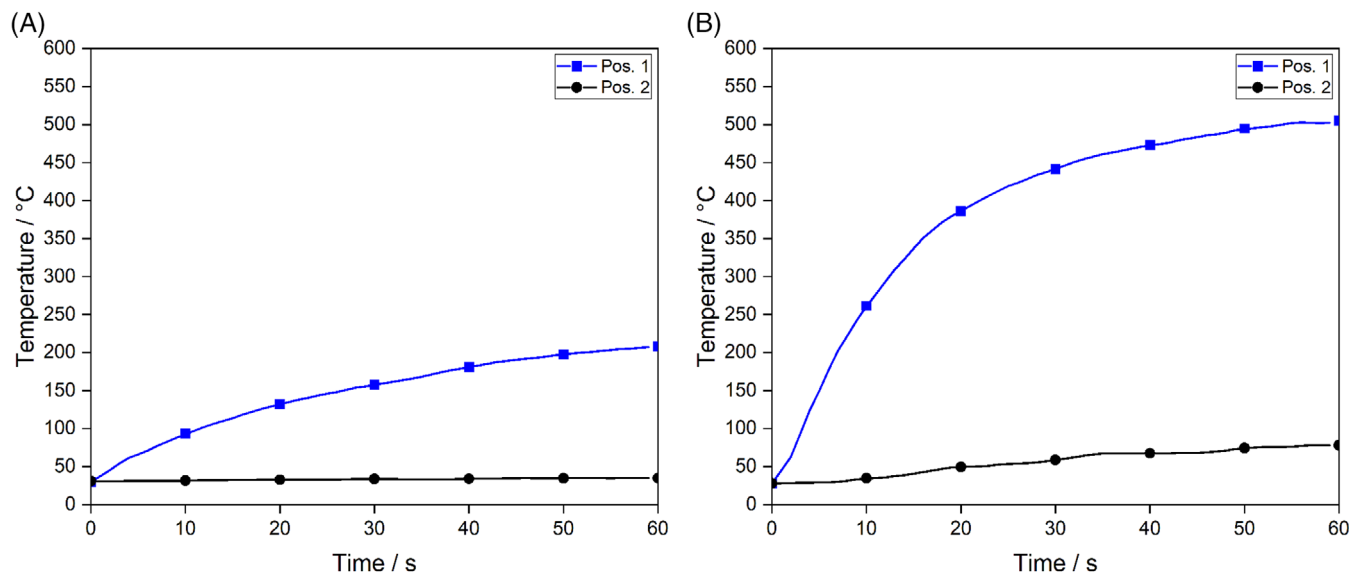
The flammability ranking of the tapes as coatings on substrates corresponds with the flammability ratings in UL 94 and OI. Comparing the substrates yields that heat dissipation is the main factor for the flammability of the coated sample. For materials with a high heat conductivity such as steel, the heat loss  $q''_{\text{loss}}$  via conduction is so high that ignition cannot take place. For insulating wool and wood,  $q''_{\text{loss}}$  is much smaller so that the tape heats up until there are enough fuel gases for ignition and the exothermic chain reaction of the burning process is started. The results are expected to be transferrable to one-sided tapes.

### 3.4 | Glued laminates

Laminates (substrate/tape/substrate) were investigated in different fire and pyrolysis tests to investigate the influence of an adhesive gap and PSA tapes for different bonded materials. The flammability of the materials was assessed by UL 94 test (Table 7).

**FIGURE 5** Burning behavior of Tape FR (left) and Tape RE (right) on (A) zinc plated steel, (B) beechwood, and (C) mineral wool in the single burning item test according to ISO 11925-2.**TABLE 6** Thermophysical properties of the substrates used in EN ISO 11925-2 test.

	Thermal conductivity (W m <sup>-1</sup> K <sup>-1</sup> )	Specific heat capacity (J kg <sup>-1</sup> K <sup>-1</sup> )	Density (kg m <sup>-3</sup> )	Heat effusivity (J m <sup>-2</sup> K <sup>-1</sup> s <sup>-½</sup> )
Beechwood	0.2	1400	700	443
Zinc	115	380	7000	17 490
Insulating wool	0.004	840	50	13
Steel	45	480	7840	13 013



**FIGURE 6** Surface temperature development during EN ISO 11925-2. (A) Zinc plated steel and (B) insulating wool by comparison. Pos.1 = surface temperature in the burner flame application area. Pos. 2 = surface temperature 200 mm above the burner flame application zone.

**TABLE 7** Vertical UL 94 test of the sandwich-like specimen. Substrate thickness: Wood: 4 mm, steel: 1 mm, PMMA: 2 mm, polycarbonate: 2 mm.

Tape	Wood	Steel	PMMA	Polycarbonate
Tape RE	N.R.	V-0	N.R.	V-2
Tape FR	N.R.	V-0	N.R.	V-2
Monolithic	N.R.	V-0	N.R.	V-2

**TABLE 8** Burning velocity of monolithic PMMA compared with Tape RE and Tape FR bonded to PMMA in the same thickness.

Adhesive joint	Burning velocity (mm/min)
PMMA monolithic	32.5 ± 0.2
PMMA + Tape RE	36.7 ± 0.4
PMMA + Tape FR	32.4 ± 0.6

The wood samples ignited and burned up to the clamp within 30 s and did not pass the vertical test. The adhesive gap widened when the flame was applied, but no delamination and dripping took place. The bonded steel element did not ignite within 10 s and achieved a V-0 rating with no dripping. There was no visible change in the adhesive gap. The PMMA sample burned up to the clamp, with burning drops falling off the sample and igniting the cotton wool. The sample did not achieve a rating in the vertical test. Polycarbonate samples were ignited, and the sample burned up to 40 mm. At the bottom of the sample where the flame was applied, a char layer built up on the surface. After the burner flame was removed, the sample extinguished by dripping. Part of the sample fell off and ignited the underlying cotton wool. A V-2 rating was achieved. The substrates dominated the flammability of the glued materials which had the same



**FIGURE 7** UL 94 vertical test of (A) polycarbonate 4.1 mm, (B) wood 8.1 mm, (C) ABS 2.1 mm.

UL 94 ratings. In the horizontal test, the PMMA samples differed in their burning velocity (Table 8). The PMMA sandwich element with Tape RE and the monolithic PMMA burned faster than the Tape FR PMMA sample.



**TABLE 9** Oxygen index of adhesive joints in vol.%

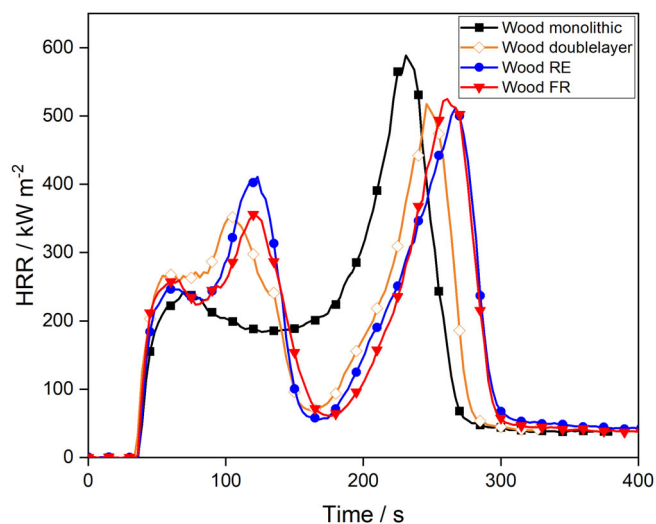
Adhesive joint	PMMA $\pm 0.2$	PC $\pm 0.3$	Wood $\pm 0.2$	Steel
Monolithic	17.7	27.1	27.7	NR
Tape RE	17.6	26.1	27.9	NR
Tape FR	18.0	27.0	27.9	NR

If samples with a reduced thickness (2.1 mm) are tested in UL 94 vertical test, the single layers of the substrates wrap themselves up and expose more material, and especially tape, to the flame. This can lead to a faster burning process and was observed for 1 mm thick ABS plates that were taped with Tape RE and Tape FR resulting in a 2.1 mm specimen (Figure 7c).

A popular test for assessing burning behavior in an emerging fire scenario is the oxygen index (OI). The results are concluded in Table 9. Monolithic PMMA and taped PMMA with Tape RE burned self-sufficiently at an oxygen concentration of 17.6 vol.%. This value corresponds with the manufacturer information (17.5 vol.%). The taped sample with Tape FR tended toward a higher OI. As a charring polymer, PC had a higher OI. Monolithic and Tape FR samples had a similar OI of 27 vol.%, which corresponds with the manufacturer statement of 28 vol.%. Tape RE lowered the OI slightly, to 26.1%. All wood samples had a very similar OI, which corresponds with the literature values for different kinds of wood.<sup>28–30</sup> The char layer builds up on taped samples as well and there is no delamination. For PC, the taped samples started to delaminate during the burning process. The area of the tape exposed to the propagating flame increased accordingly, thus increasing the impact of the tape on the OI value, which explains the lower OI value for the Tape RE sample of bonded PC.

The sandwich elements were investigated in the single flame source test according to ISO 11925-2. Wood, steel and insulating wool were all self-extinguishing as monolithic material and as taped material. In zinc plated steel and insulating wool, no ignition of the sample was possible. The temperature in the adhesive gap was measured via thermocouple and did not exceed 130°C, which is not sufficient to form sufficient fuel gases for ignition. The adhesive tape did not drip or act as a wick in steel or insulating wool as suspected. Monolithic and bonded wood were both self-extinguishing, whereas the adhesive gap in the latter widened. Since there are no relevant wick effects or delamination in this thickness, the ratio between the exposed surface areas of tape and substrate (1:40) can explain that the burning behavior is dominated by the substrate material.

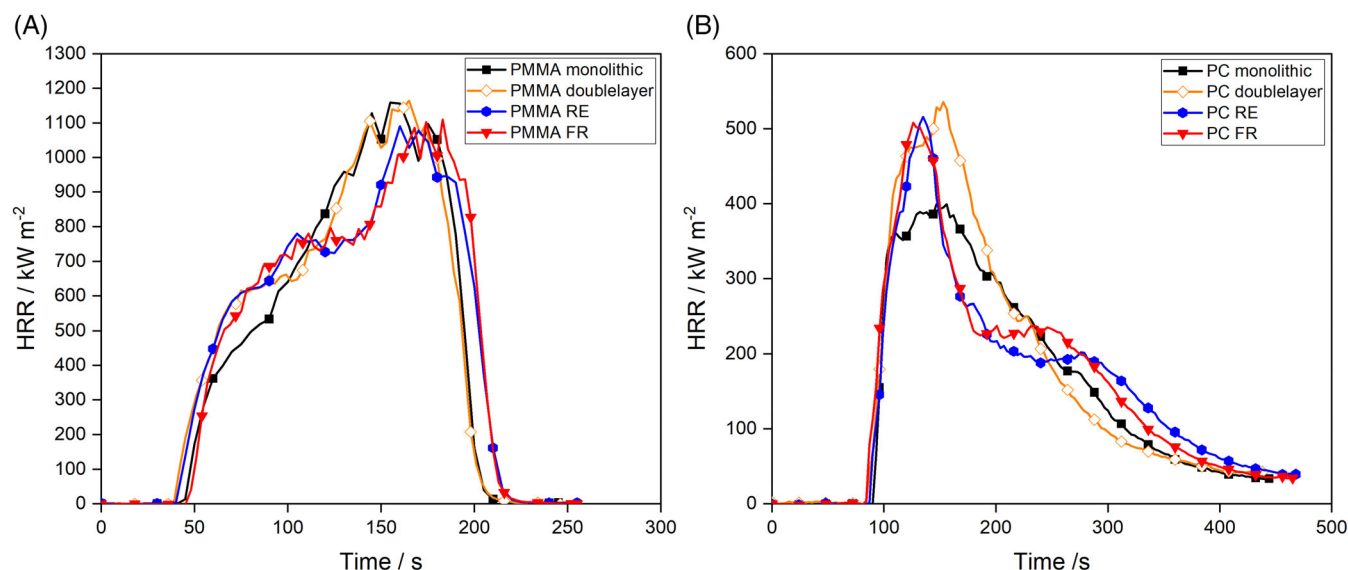
The cone calorimeter was used to determine the behavior in a forced flaming condition depicting a developing fire. Figure 8 shows the HRR curve of a monolithic beechwood sample of 8 mm thickness compared with layered wood (two layers of wood, each 4 mm) and two taped (via Tape RE and Tape FR) samples of the same thickness. The monolithic sample had two PHRRs, which is typical for wood.<sup>31–33</sup> The first peak occurred immediately after ignition of the volatile products emitted from the surface. Then a char layer built up and the HRR decreased due to the heat and fuel barrier effect. After around 180 s the protective layer cracked, fuel gases were emitted

**FIGURE 8** Cone calorimeter HRR curves of monolithic, layered, and taped wood samples. Sample thickness: 8 mm. Irradiation: 50 kW m<sup>-2</sup>. Distance: 35 mm.

more freely into the flame zone and the sample started to burn intensely, causing the second peak (main peak) in HRR. After 240 s the sample reached the PHRR and extinguished immediately afterwards. The layered and taped samples (double layer, RE, FR) behaved differently. After the slightly earlier ignition at 34 s, a first PHRR emerged which tended to be more pronounced for the layered samples. Then a char layer built up and the first local minimum of HRR was observed. Subsequently the char layer cracked, volatiles passed the char layer, the first layer of the sample delaminated and the tape in the bonded samples was consumed, all of which resulted in the second PHRR. The second PHRR was the main difference between the monolithic and the layered samples and the “additional” peak. This peak led to a higher ARHE at this time and a higher MARHE<sub>1</sub> (200  $\pm 14$  kW m<sup>-2</sup>) for the first two peaks (PHRR<sub>1+2</sub>) compared with the MARHE<sub>1</sub> (153  $\pm 10$  kW m<sup>-2</sup>) for the first PHRR of the monolithic wood. After the first wood layer was consumed by the flame, a char residue was left on top of the second wood layer on all laminates. The thick char residue of the first wood layer and the thin char layer of the second wood layer worked as a strong heat and volatile barrier, resulting in the all-time minimum of HRR, which is around 60% lower than in the monolithic sample. This minimum is characteristic for layered wood materials which delaminate in fires, such as plywood.<sup>34</sup> After the layer cracked, volatiles passed through the char layers again and PHRR<sub>3</sub> was observed. PHRR<sub>3</sub> was lower and shifted to a later time for all laminates compared to the PHRR<sub>2</sub> of monolithic wood. After the consumption of the second layer, the sample extinguished quickly and exhibited a wood-typical afterglow. Table 10 shows the most important parameters to evaluate and assess the burning properties of the wood specimen. The layer-wise burning of the laminates shows some characteristics that lead to improved fire properties, such as a stronger insulating layer and a very low HRR at around 180 s, shifting the last PHRR by around 30 s and lowering it by around 10%. But the separate burning of the layers leads to an additional PHRR

**TABLE 10** Cone calorimeter data for monolithic, layered, and taped wood samples. Sample thickness: 8 mm. Irradiation: 50 kW m<sup>-2</sup>. Distance: 35 mm.

Sample	$t_{ig}$ (s) ± 2	PHRR <sub>1</sub> (kW m <sup>-2</sup> ) ± 10	PHRR <sub>2</sub> (kW m <sup>-2</sup> ) ± 30	PHRR <sub>3</sub> (kW m <sup>-2</sup> ) ± 50	FIGRA (kW m <sup>-2</sup> s <sup>-1</sup> ) ± 0.5	MARHE <sub>1</sub> (kW m <sup>-2</sup> ) ± 10	MARHE <sub>2</sub> (kW m <sup>-2</sup> ) ± 10	THE (MJ m <sup>-2</sup> ) ± 3	Residue (%) ± 1
Wood monolithic	37	245	589	-	4.0	153	229	66	15.8
Wood double layer	34	271	363	547	5.3	204	220	64	15.8
Wood RE	33	249	392	499	4.6	202	218	69	14.6
Wood FR	34	258	366	521	4.7	197	219	67	14.7



**FIGURE 9** (A) Cone calorimeter comparison between monolithic PMMA, layered PMMA without adhesive tape, sandwich-like PMMA bonded with Tape RE, and sandwich-like PMMA bonded with Tape FR. (B) Cone calorimeter data of monolithic, layered, and taped polycarbonate samples. Sample thickness: 4 mm.

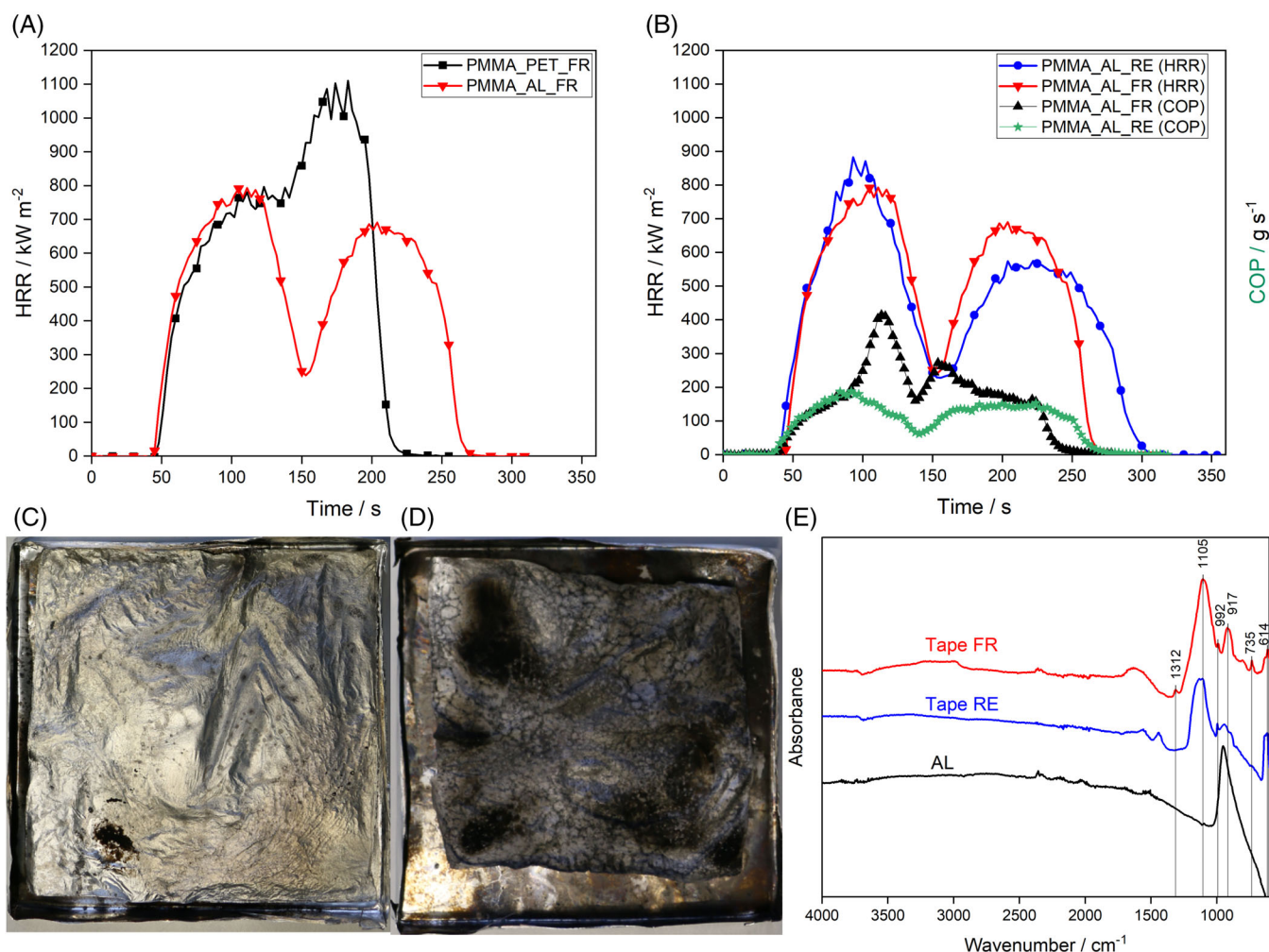
which emerges at around 110 s, leading to a MARHE<sub>2</sub> that is 33% higher than the MARHE<sub>1</sub> of monolithic wood. This additional PHRR presents a fire risk that needs to be addressed and can determine the potential for certifications for the material based on MARHE, such as the EN 45545. Also, the FIGRA, which is defined as the max (HRR(t)/t) and is an indicator for the fire hazard of a material, is 15% higher for bonded wood due to that first PHRR<sub>1</sub>.

In Figure 9a, the HRR curves of monolithic, layered, and taped PMMA are shown. The first step in the burning process is the ignition of the volatiles, which resulted in an immediate increase in the HRR. This built up the shoulder in the diagrams of all four samples. The HRR within this shoulder was higher for the taped and layered materials due to the air gap or tape that is located between the plates. This leads to a reduced thermal thickness and thus a higher HRR and fire risk at the beginning of pyrolysis.<sup>35</sup> After the first shoulder, the thermal thickness and heat dissipation within all samples decreased, which results in faster heating of the sample, a larger pyrolysis zone, more volatiles and a higher HRR until the PHRR was reached. Then the

sample extinguished rapidly, and no afterglow occurred. This HRR curve is typical for PMMA under these conditions and is comprehensively discussed in the literature.<sup>36–38</sup> For both bonded samples, the HRR stagnated after 120 s and built a small plateau which is caused by the tape that has PET as a carrier. The carrier disturbs the melt flow in the pyrolysis zone and has a higher melting range (270°C) than PMMA (160°C). After finishing the plateau, the PHRR was reached at a temperature around 10°C lower than the PHRR of monolithic PMMA, and subsequently the samples extinguished quickly without any afterglow and complete consumption of the material. Due to the lower PHRR and MARHE, and the shift of the PHRR to a later time, the MARHE indicates a slightly lower fire risk for the bonded materials compared to the layered and monolithic PMMA. In the layered PMMA plates, the air gap is eliminated quickly due to the melt zone, where there happens to be much convection, and which even increases with a reduced sample thickness.<sup>39</sup> Thus, the sample behaves similar to monolithic PMMA. The FIGRA for the bonded samples, determined by the first shoulder in the HRR curve, is the same

**TABLE 11** Cone calorimeter data of monolithic, layered, and taped PMMA samples.

Sample	$t_{ig}$ (s) $\pm$ 4	FIGRA ( $\text{kW m}^{-2} \text{s}^{-1}$ ) $\pm$ 0.4	PHRR ( $\text{kW m}^{-2}$ ) $\pm$ 16	MARHE ( $\text{kW m}^{-2}$ ) $\pm$ 5	THE ( $\text{MJ m}^{-2}$ ) $\pm$ 2
PMMA monolithic	38	8.1	1181	587	118
PMMA double layer	35	8.5	1155	601	118
PMMA RE	44	8.2	1029	565	118
PMMA FR	37	7.9	1059	575	121
PC monolithic	85	3.4	411	199	66
PC double layer	79	4.0	529	232	71
PC RE	79	4.0	519	198	72
PC FR	81	4.1	512	197	71



**FIGURE 10** (A) Comparison between bonded PMMA (with Tape FR) with and without an aluminum middle layer. (B) Cone calorimeter HRR and CO formation (COP) of taped PMMA samples with Tape RE and Tape FR with an aluminum middle layer (AL). Sample thickness: 4 mm. (C) Cone calorimeter residues of Tape RE and (D) Tape FR with an aluminum middle layer used to connect two PMMA plates. (E) ATR-FTIR spectrum of cone calorimeter residues of Tape RE and Tape FR with an aluminum middle layer used to connect two PMMA plates. Comparison to the untreated aluminum foil that was used as a middle layer.

for monolithic samples where it is determined by the PHRR. Table 11 contains the most important parameters of the PMMA cone calorimeter measurements.

Figure 9b shows the HRR curves of the different PC samples. The monolithic PC showed typical behavior for a charring material.<sup>40</sup> After ignition and the rise of the HRR, a char layer built up and the HRR

decreased until fuel depletion and transition to a strong afterglow. The same effects occurred for the layered and taped samples, but the HRR had a higher peak after ignition. Due to the separation of layers by tape or air, the heat transfer within the sample is disturbed, so that the first layer heats up faster and shows a higher PHRR.<sup>6</sup> After the first pronounced PHRR, the HRR dropped fast to a plateau, where the char, mainly formed by the first layer, protected the underlying material. This manner of protection by a first layer is known for laminates,<sup>41</sup> and the plateau is characteristic for a strong protective char layer.<sup>42</sup> The double layer PC showed a slightly higher and wider PHRR and a MARHE 15% higher, and developed no char plateau. The missing connection between the first and second layers leads to faster growth of the sample into the cone heater, which exposes the surface to higher irradiation and thus results in a higher HRR (Table 11). After the second PC layer started to form less fuel gases, since more and more char was building up and less fire load was available, the HRR decreased to the same amount of afterglow as the monolithic PC.

To investigate the influence of different carriers within the PSA tapes, an aluminum foil layer was placed between two layers of double-sided PSA tape. Figure 10a shows the HRR curve of bonded PMMA samples. The black curve shows the laminates of PMMA bonded with the PET carrier tape, and the red curve shows the laminate with an aluminum layer as a middle layer (AL), simulating a different carrier. The aluminum layer acts as a non-flammable interlayer and protects the second layer of PMMA. The positive effect of metal foils or flame retardant interlayers is known from the literature<sup>11</sup> and is in this case responsible for the minimum of HRR at 150 s and the lower, shifted PHRR of the PMMA laminate.

Figure 10b shows the HRR and the COP curve of the Tape RE and Tape FR-bonded samples with the aluminum middle layer. Because phosphorus flame retardants can increase CO production in cone calorimeter measurements,<sup>9,43</sup> the COP was used to determine the time when the pyrolysis front reached the tapes. The HRR curves show that there is a minor impact of the flame retardant on the PHRR<sub>1</sub> and the time of the first peak. The action of Tape FR, which was active in the gas phase, was shown by the increased COP at around 100 s. After the aluminum layer was reached and the all-time minimum of HRR took place at about 150 s, the second layer of PMMA started to burn and led to a second peak of HRR. Again, the flame retardant on the backside of Tape FR showed its effect on the COP. The shape of the PHRR<sub>2</sub> depended on whether Tape RE or Tape FR was used. Tape RE exhibited a lower second peak of HRR and a longer burning time. In contrast, the second peak of HRR was higher for Tape FR and the second PMMA layer was consumed faster. This can be explained by Figure 10c–e where the residues of both samples are depicted. For Tape RE (c), the area of the remaining aluminum foil is much larger than for Tape FR (d). This explains the better barrier effect of the aluminum carrier with Tape RE and the associated later, lower PHRR<sub>2</sub>. The reduced area of the aluminum Tape FR sample can be explained by the ATR-FTIR results in Figure 10e, which shows the ATR-FTIR spectra of the residue surfaces from the cone calorimeter tests compared with the untreated aluminum foil used to

manufacture the cone calorimeter samples. The aluminum foil shows an uncluttered spectrum that shows mainly the 950 cm<sup>-1</sup> Al–O vibration. Tape RE shows a dominant peak around 1100 cm<sup>-1</sup> which is attributed to C–H vibrations, which are also present in the spectrum of Tape FR. The Tape FR spectrum also shows peaks at 1312 and 735 cm<sup>-1</sup>, which are attributed to P–O vibrations and suggest that the phosphorus flame retardant reacted with the aluminum surface.

## 4 | CONCLUSIONS

Phosphorus-based flame retardants have a major impact on the flame retardancy of PSA tapes as free-standing films and drastically improve flammability ratings in UL 94 vertical test and OI.

Bonded to substrates on one side, the flame retardancy of the tapes is no longer the only factor that determines the fire characteristics of this connection. The thermal effusivity of the substrate plays a significant role and determines the flame spread over the material. The fire behavior of PSA-bonded laminates (substrate/tape/laminate) depends on how the tape works as an insulation layer between the bonded layers, the flammability of the tape itself becomes a minor factor. The burning behavior then depends on the substrates and the tape used in the gap, and on how these materials interact with each other. The interfaces determine the burning behavior in different ways depending on the substrate and the carrier properties. In wood and PMMA there are positive as well as negative aspects influencing the fire behavior of monolithic and bonded materials. In wood, there is an additional PHRR at the beginning, but a strong insulating effect of the tapes. In PMMA the separation of the layers by tape leads to a faster heating up of the first layer in cone calorimeter testing, but a barrier effect that shifts and lowers the PHRR. In PC, there is a significant increase in the fire risk when bonded materials are used. The PHRR at the start is much higher than in monolithic PC, which leads to a higher MARHE and FIGRA. Also, the carrier has a major impact on the fire behavior: for example, when PET and ALU carriers are compared in PMMA, the aluminum improves the fire properties crucially, lowering PHRR and working as a barrier for the second layer. All these complex interactions yielded fundamental knowledge about how tape-bonded materials behave in fires and how the modification of these tapes can improve the fire behavior of bonded substrates. This paper may feed the communication between tape developers and applicants and serve as guide to develop flame retarded tapes tailored to achieve the distinct protection goals of the bonded components.

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## CONFLICT OF INTEREST STATEMENT

The authors declare no conflict of interest.

## DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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