THE MELT/SHRINK EFFECT OF LOW DENSITY THERMOPLASTICS INSULATES Cone Calorimeter Tests

by

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The melt/shrink effects on the fire behavior of low density thermoplastic foam have been studied in a cone calorimeter. The experiments have been performed with four samples of expanded polystyrene foams having different thicknesses and two extruded polystyrene foams. Decrease in surface area and increase in density, characterizing the melt/shrink effect have been measured at different incident heat fluxes. Three of these foams tested have been also examined by burning tests at an incident heat flux of 50 kW/m². It was assessed that the fire behavior predictions based the current literature models provided incorrect results if the cone test results were applied directly. However, the correct models provided adequate results when the initial burning area and the density of the molten foam were used to correct the initial cone calorimeter data. This communication refers to the fact that both the effective burning area and the density of the molten foam affect the cone calorimeter data, which requires consequent corrections to attain adequate predictions of models about the materials fire behavior.

Key words: melt/shrink behavior, cone calorimeter, flammability, thermoplastic foam, fire behavior

Introduction

Low density thermoplastic foams are widely encountered in soft furnishings and construction industries. The poor flammability performance of thermoplastic based products causes fire to be a significant contributor to annual fire losses. An illegal fireworks show, for example, has ignited a thermoplastic insulation and the resultant fire gutted a 30-story building in Beijing's Central Business District, China, on February 9, 2009. One fireman died, while six firemen and two construction workers were injured. The cost of the fire was estimated at more than 23.44 million US\$. In this context, the fire at the Kiss nightclub in the south Brazil's college town of Santa Maria killed 235 people on January 27, 2013, and it appears to have started when

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the band lit a flare that ignited the flammable sound-proofing foam boards. The fire safety performance of thermoplastic foam raises great concerns.

In NIST Flammability of Thermoplastic Materials project [1], characterizing melt/drip effects is one of the key goals of designing and assembling the necessary apparatus for developing bench scale flammability measurement methods. The melting effect is especially important for characterizing flammability of thermoplastic foams. It causes the foam to shrink and drip during a test. Therefore, the density and surface area exposed to the heat flux undergoes a significant change in bench scale tests.

Numerical and experimental tests have been performed in melt burning research [2, 3] Moreover, experimental research on the melt/drip effects have been carried out at different scales by standard and self-designed facilities [4-7]. In these experiments, specimens from eight commercial products (all densities are more than 550 kg/m³), have been tested with UL94 in vertical burning test conditions [4]: mass loss rate, first drop mass and diameter, first dripping time were used to describe the melt/drip effect in the experiment. Thermal stabilities of another eight thermoplastic polymers have been studied by bench scale UL94 test and micro scale thermogravimetric (TG) test regarding the ignition mechanism of melting polymers [5, 6].

In prediction of fire behavior of materials both modeling and experiments need to be performed together providing mutual information for improving the fire tests. There is a variety of experiments in this direction, still sporadic to some extent, but providing step-by-step enough amount of data about the fire behavior of thermoplastics in fire, especially the foams. In this context, simulation of polymer burning under UL94 vertical test conditions based on mass loss rate to investigate the change in density to the predicted have been carried out [7]. As a supporting technique, TG experiments have been used to estimate the onset of melting and then to provide adequate results for modeling both the melting and pyrolysis stages [8]. Increasing in the scale of the experiments resulted (with ISO 9705) to study polymer melt flow behavior and subsequent pool fire [9] in room scale tests, also the interaction between a vertical polymer sheet fire and the induced pool fire were studied. Other factors, regression, char layer and fire spread, which influence the burning behavior of polymer, have also been studied by [10-12].

A T-shape trough to study the flowing behavior of melting thermoplastics [13], and large scale burning tests were carried out for thermoplastics of different thickness [14]. A meth odology to record the real time melt and burning/dripping behavior of thermoplastics quantita tively in an electric furnace heated by connective heat flow have been developed [15]. Multi-scale test was also adopted to study the fire behavior of expanded polystyrene (EPS) foam [16].

In fact, it is difficult to characterize the fire behavior of foams [17] due to melt/drip effect of samples when tested. Melt/drip effects not only change the ignition mechanism of thermoplastics resulting in ignition time scatter, but also raise the uncertainties in heat release rate (HRR) measurements in cone calorimetric tests. The HRR is an essential parameter in fire testing and flammability assessment, especially for performance based fire safety design in which HRR of materials is used for fires design in enclosures. Many software packages use HRR as input data for full scale fire predictions and material or product evaluation in light of fire behavior. Thus, reliable HRR data are essential pool of information for correct evaluation materials and prediction their fire behaviors.

Cone calorimeter has been widely used in evaluating fire behavior of polymers in fire safety engineering. When the flammability of low density thermoplastic a horizontal placement of the sample is measured by the cone calorimeter and the material can melt and shrink in the sample holder. The sample can not drip but it may shrink during the melting stage and thus melt/drip effects is changed to melt/shrink effect. This is quite different from the test material

which is liquid [18] or solid [19, 20] can hold its shape or burning area. If the sample is shrinking, then the exposed area to incident heat flux should be significantly smaller than the nominal exposure area used to calculate the HRR per square meter. It was found that the sample size can influence the cone test results [21], the ignition of smaller PU adhesive samples was attributed to an enhanced mixing of volatiles and oxygen as a result of an increased distance between the conical heater and the sample surface. This interpretation refers an effective area of sample which has to be determined in order to perform an adequate ignition procedure as well as for the consequent calculations of HRR per area. As a support of these comments, an incorrect HRR obtained from a cone calorimeter test with a nominal sample surface (for example 0.008848 m²) as burning area would cause the fire behavior predictions invalid if the current models would be directly applied.

As examples of direct application of the current model predictions to cone calorimeter data obtained with melting materials we have to mention some research carried out. The fire behavior of sandwich panels [22] using expanded polystyrene core (EPS). EPS cores was classified as flashover-category (FO-category) 4 by Ostman and Hovde model [23] and Hansen and Tsandaridis model [24] from cone calorimeter tests using 50 kW/m² incident heat flux. Thus an ISO 9705 room fire test on a room lined with EPS panels (without skins) is predicted to reach flashover before 2 min after the beginning of the test. Six out of eight tests reached flashover, all the flashover happened after 5 min, only two of them reached flashover before 10 min. These models are not able to be applied to data about composite materials and can not predict the time to flashover for sandwich panels [22].

Heating and ignition tests were conducted in our research to study the melt/shrink effect in cone calorimetric tests. Density and surface area changes are measured for test samples at different incident heat fluxes in heating tests. Ignition tests were carried out at 50 kW/m² incident heat flux. Density and surface area of melt/shrink foams is used to correct the 50 kW/m² results and input to fire behavior prediction models. The low density foams are classified by Ostman's and Hansen's model using 50 kW/m² test data directly and the corrected data. The difference of prediction results is analyzed. The main goal of this research is to deduce the correlation between melt/shrink effect and the HRR measurement and explore a method to use the test results in fire behavior prediction.

Test descriptions

Test facilities

The tests were performed with the cone calorimeter in the State Key Laboratory of Fire Science, Hefei, China, and this cone is in accordance with ISO 5660. All test specimens were wrapped in aluminum foil with the upper surface uncovered, put into a specimen-holder and exposed in the horizontal orientation. Specimens were packed to the appropriate test level height using Kaowool ceramic fiber. The specimen-holder's edge frame retains the specimen as allowed in the standard. The edge frame reduces the test surface area to 0.008848 m², and this is the area used in calculations. The nominal exhaust system flow rate for all tests was 0.024 m³/s. Prior to testing, a polymethyl methacrylate (PMMA) reference material was tested to ensure that all systems were working correctly.

Materials and tests arrangement

Six kinds of thermoplastic foams were tested in this research. Four of them were EPS foam panel, and two were extruded polystyrene (XPS) foam panel. These materials, parameters and labels are listed in tab. 1. Photos of tested samples are shown in fig. 1.

	EPS	EPS	EPS	EPS	XPS	XPS
$ ho_{ m n} [m kgm^{-3}]$	6.5	5.3	7.8	8.2	52.6	37.8
Thickness [mm]	8	12	18	48	20	18
Color	White	White	White	White	Red	Grey
Material label	EPS_8	EPS_12	EPS_18	EPS_48	XPS_red	XPS_grey
Test label	EPS_8_*	EPS_12_*	EPS_18_*	EPS_48_*	XPS_red_*	XPS_grey_*

Table 1. Materials used in the tests

^{*} Incident heat flux in cone test, for example, EPS_8_25 is the EPS specimen with the thickness of 8 mm at the incident heat flux of 25 kW/m²



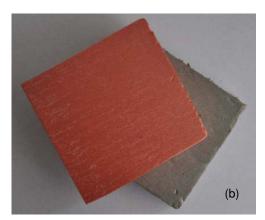


Figure 1. Prepared specimens for cone tests; (a) EPS, (b) XPS

The tests were conducted in two stages. In the first stage, burning tests were conducted for EPS_48, XPS_red, and XPS_grey at incident heat flux of 25, 35, and 50 kW/m². Triplicate specimens of each material were tested at each incident heat flux, except for XPS_red and XPS_grey at 50 kW/m², only one test was conducted for each of them.

In the second stage, only heating tests were conducted. Three EPS panels with thickness of 8, 12, and 18 mm were heated with the cone under incident heat flux of 25, 35, and 50 kW/m² for 10 to 15 seconds. Triplicate specimens of each material were tested under each incident heat flux. Heating tests were also conducted for EPS_48, XPS_red, and XPS_grey under incident heat flux of 25, 35, and 50 kW/m², the heating procedure stopped 10 to 20 seconds before the mean ignition time for each materials, as obtained from first stage tests. Triplicate specimens of each material were also tested under each incident heat flux. The heating times chosen for EPS_48 are 90, 60, and 40 seconds, for XPS_red are 80, 50, and 30 seconds, for XPS_grey are 110, 40, and 10 seconds.

Test results

Melt/shrink effect of foams

The original remains of second stage tests are shown in fig. 2. The melt/shrink effect is clear for all the tested thermoplastic foams.

To quantify the effect, all remains are taken from the aluminum foil with their original shape and put on a 94×94 mm blue (for EPS specimens) and white (for XPS specimens) paper to take photos. Processed photos are illustrated in figs. 3 and 4. The reduced surface areas of the molten specimens are calculated and listed in tabs. 2 and 3. The volume of each residue was

also measured, thus its density is calculated, also these are listed in tabs. 2(a), 2(b) and 3. Incident heat flux has more influence on density than surface area.

With low nominal density and the lowest mass, EPS_8 specimens shrank to a very low effective surface area decrease to a very low level, see tabs. 2(a) and 2(b). For EPS specimens, the more nominal density and mass, the more effective surface area would be reached, see also tabs. 2(a) and 2(b).

Influence on flammability prediction

In the second stage, burning tests were conducted for EPS_48, XPS_red, and XPS_grey at incident heat flux of 50 kW/m². The results are illustrated in fig. 5.

The cone calorimeter results of these thermoplastics from tests at 50 kW/m² are used as input data to [23] empirical linear regression model and [24] multiple discriminant function analysis model to predict the flashover category (FO) in the ISO9705 room corner test.





Figure 2. Heating test remains

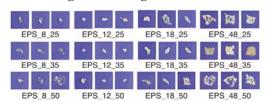


Figure 3. Processed images of EPS remains

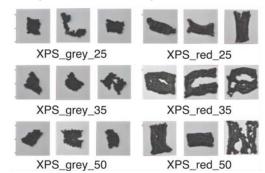


Figure 4. Processed images of XPS remains

			EPS	-8			EPS – 12					
Incident heat flux, [kW/m ⁻²]	Speci- men mass, [g]	Mass remain, [g]	Mass loss, [g]	$A_{\rm e}$, [mm ²]	R _a , [%]	$ ho_{ m m}, \ [m kgm^{-3}]$	Specimen mass, [g]	Mass remain, [g]	Mass loss, [g]	$A_{\rm e}$, [mm ²]	R _a , [%]	ρ _{m,} [kgm ⁻³]
25	0.52	0.51	0.01	250	2.83	217	0.63	0.61	0.02	381	4.30	231
25	0.51	0.49	0.02	293	3.31	203	0.64	0.61	0.03	311	3.51	252
25	0.52	0.50	0.02	246	2.78	208	0.61	0.60	0.01	231	2.61	249
Mean	0.52	0.50	0.02	263	2.97	209	0.63	0.61	0.02	307	3.47	244
SD	0.01	0.01	0.01	26	0.29	7	0.02	0.01	0.01	75	0.85	11
35	0.51	0.50	0.01	388	4.39	238	0.61	0.59	0.02	181	2.05	298
35	0.53	0.50	0.03	326	3.68	192	0.61	0.58	0.03	267	3.02	239
35	0.51	0.49	0.02	260	2.94	222	0.60	0.56	0.04	252	2.85	264
Mean	0.52	0.50	0.02	325	3.67	217	0.61	0.58	0.03	234	2.64	267
SD	0.01	0.01	0.01	64	0.73	23	0.01	0.02	0.01	46	0.52	30
50	0.52	0.50	0.02	312	3.53	248	0.62	0.59	0.03	388	4.39	268
50	0.52	0.49	0.03	265	2.99	245	0.63	0.58	0.05	221	2.50	297
50	0.55	0.50	0.05	910	10.28	166	0.65	0.60	0.05	181	2.05	302
Mean	0.53	0.50	0.03	496	5.60	220	0.63	0.59	0.04	264	2.98	289
SD	0.02	0.01	0.02	359	4.06	47	0.02	0.01	0.01	110	1.24	18

Table 2(b). Area and density change of EPS specimens

	EPS – 18							EPS – 48				
Incident heat flux, [kW/m ⁻²]	Specimen, mass,	Mass remain, [g]	Mass loss, [g]	A _e , [mm ²]	R _a ,[%]	ρ _m , [kg/m ⁻³]	Specimen mass,	Mass remain, [g]	Mass loss, [g]	$A_{\rm e}$, $[{ m mm}^2]$	R _a , [%]	ρ _m , [kg/m ⁻³]
25	1.41	1.38	0.03	611	6.91	403	3.92	3.87	0.05	1787	20.20	719
25	1.44	1.43	0.01	1186	13.40	450	3.83	3.80	0.03	2073	23.43	639
25	1.45	1.61	0.04	534	6.04	468	3.95	3.91	0.04	1820	20.58	680
Mean	1.43	1.47	0.03	777	8.78	440	3.90	3.86	0.04	1894	21.40	679
SD	0.02	0.12	0.02	356	4.02	34	0.06	0.06	0.01	156	1.77	40
35	1.44	1.39	0.05	635	7.18	411	3.93	3.89	0.04	1831	20.69	850
35	1.44	1.41	0.03	580	6.56	463	4.03	3.98	0.05	2087	23.59	781
35	1.46	1.40	0.06	488	5.51	487	4.06	3.99	0.07	2271	25.67	776
Mean	1.45	1.40	0.05	568	6.42	454	4.01	3.96	0.05	2063	23.32	802
SD	0.01	0.01	0.02	75	0.84	39	0.07	0.06	0.02	221	2.50	41
50	1.49	1.43	0.06	489	5.53	514	3.64	3.58	0.06	2126	24.03	661
50	1.46	1.41	0.05	477	5.39	487	3.74	3.67	0.07	1932	21.83	779
50	1.45	1.41	0.04	1103	12.47	509	4.05	3.99	0.06	2147	24.26	789
Mean	1.47	1.42	0.05	670	7.80	503	3.81	3.75	0.06	2068	23.37	743
SD	0.02	0.01	0.01	358	4.05	14	0.21	0.22	0.01	119	1.34	71

Table 3. Area and density change of XPS specimens

			VDC	and .			VDC curvi						
	XPS_red							XPS_grey					
Incident heat flux, [kW/m ⁻²]	Specimen mass, [g]	Mass remain, [g]	Mass loss, [g]	$A_{ m e}$, $[{ m mm}]^2$	R _a , [%]	$ ho_{ m m}$ [kg/m $^{-3}$]	Specimen mass, [g]	Mass remain, [g]	Mass loss, [g]	$A_{\rm e}$, $[{ m mm}^2]$	R _a , [%]	$ ho_{ m m}$, $ ho_{ m m}$, $ ho_{ m m}$	
25	10.32	9.83	0.49	3066	34.66	520	6.34	6.28	0.06	1497	16.93	569	
25	10.11	9.84	0.27	2131	24.08	509	5.92	5.90	0.02	1701	19.23	651	
25	10.31	9.89	0.42	2074	23.44	588	5.76	5.73	0.03	1555	17.57	705	
Mean	10.25	9.85	0.39	2424	27.39	539	6.01	5.97	0.04	1585	17.91	642	
SD	0.12	0.03	0.11	558	6.30	43	0.30	0.28	0.02	105	1.19	69	
35	10.73	10.16	0.57	4342	49.07	534	6.29	6.26	0.03	1725	19.50	706	
35	11.31	11.01	0.30	4702	53.14	782	6.32	6.27	0.05	1594	18.01	789	
35	11.11	10.67	0.45	4322	48.85	734	6.26	6.19	0.07	2101	23.74	600	
Mean	11.05	10.67	0.44	4455	50.35	683	6.29	6.24	0.05	1807	20.42	698	
SD	0.30	0.45	0.14	214	2.42	131	0.03	0.044	0.02	263	2.97	95	
50	10.55	10.13	0.42	3730	42.16	733	6.01	5.95	0.06	1602	18.10	788	
50	10.09	9.63	0.46	2642	29.86	911	5.95	5.93	0.02	2175	24.58	801	
50	10.13	9.51	0.62	5400	61.03	839	5.90	5.79	0.11	1444	16.32	826	
Mean	10.26	9.76	0.50	3924	44.35	828	5.95	5.89	0.06	1740	19.67	805	
SD	0.26	0.33	0.11	1389	15.70	90	0.06	0.09	0.05	385	4.35	19	

The FO-category is determined by application of the following set of rules:

- The FO-category 1: products not reaching flashover during 1200 seconds of testing time,
- The FO-category 2: 600 seconds ≤ tfo < 1200 seconds,
- The FO-category 3: 120 seconds ≤ tfo < 600 seconds, and
- The FO-category 4: tfo < 120 seconds.

The regression model [23] is expressed in the following equation:

$$t_{FO} = 0.07 \frac{t_{\rm ig}^{0.25} \rho^{1.7}}{THR_{300}^{1.3}} + 60$$
 (1)

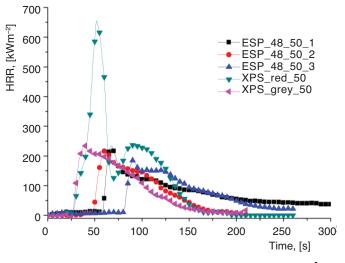


Figure 5. The HRR of at incident heat flux of 50 $kW/m^2\,$

where $t_{\rm fo}$ is the time to flashover in the room corner test, $t_{\rm ig}$ – the time to ignition in the cone calorimeter at 50 kW/m², THR₃₀₀ – the total heat release during 300 seconds after ignition at 50 kW/m² and ρ – the mean density. All of these three foams are classified to FO-category 4, which would reach flashover in ISO room test within 120 seconds, see tab. 4.

Table 4. Classification of results based on original 50 kW/m² test data

	EPS_48_50-1	EPS_48_50_2	EPS_48_50_3	XPS_red_50	XPS_grey_50
$t_{\mathrm{ig}},[s]$	45	34	68	45	23
z_1 - $\rho_{\rm f}$, [kgm ⁻³]	8.2	8.2	8.2	52.6	37.8
z_2 -THR ₃₀₀ , [MJm ⁻²]	21.17	12.96	14.72	27.29	16.14
z_3 -ln(FIGR A_{cc})	1.23	1.31	0.715	2.54	1.77
$t_{\mathrm{fo}}, [\mathrm{s}]$	60.12	60.21	60.21	62.08	61.98
F _{FO1}	-7.8523	-7.2774	-7.9617	-6.1556	-6.4927
F _{FO2}	-4.2778	-4.4030	-5.4608	-0.9503	-2.9904
F _{FO3}	-1.3307	-4.4709	-5.3701	5.1187	-1.6639
F _{FO4}	2.5850	0.0258	-1.5179	9.4533	2.7957
FO-category	4	4	4	4	4

In [24] model, the four classification functions that are expressed:

$$F_{\text{FO1}} = 0.01789z_1 - 0.06057z_2 + 0.971z_3 - 7.910 \tag{2}$$

$$F_{FO2} = 0.01492z_1 + 0.03354z_2 + 1.877z_3 - 7.418$$
 (3)

$$F_{FO3} = 0.008589z_1 + 0.409z_2 + 2.721z_3 - 13.406$$
(4)

$$F_{FO4} = 0.0000256z_1 + 0.347z_2 + 3.621z_3 - 9.215$$
 (5)

The selected parameters were:

- $z1 = \rho$, [kgm-3] = density of samples,
- z2 = THR300, [MJm-2] = total heat release during 300 seconds after apparent time to ignition, and
- z3 = ln(FIGRAcc) where FIGRAcc is the maximum value of the ratio between HRR and time when HRR was measured.

The parameter values and calculated results are also listed in tab. 4. All $F_{\rm FO4}$ give the largest value of the four functions, the foams can be determined as a member of FO-category 4, which would reach flashover in ISO9705 test within 120 seconds and they are the same as the results from [23] model. The values of all $t_{\rm fo}$ are all close to 60 seconds.

The effective area at 50 kW/m^2 heat flux is used to correct THR₃₀₀ (z_2), and density of molten foam is used in eq. (1) and as z_1 in Hansen's model [24], the calculated results are listed in tab. 5. Three out of five results from Ostman's model [23] and all results from Hansen's model classify the foams as a member of FO-category 3. Obviously, in both Ostman's and Hansen's models, the effective surface and density of molten play important role in the flammability prediction. The fig. 6 shows the influence of R_a and R_d on t_{fo} with Ostman's model.

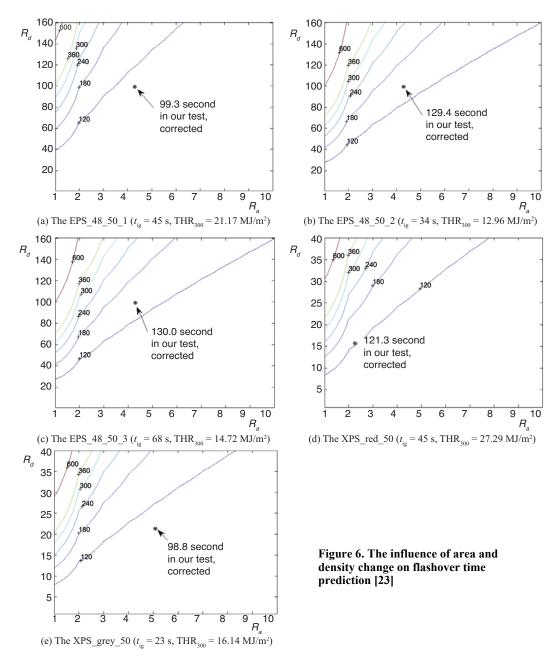
	EPS_48_50_1	EPS_48_50_2	EPS_48_50_3	XPS_red_50	XPS_grey_50
$t_{ m ig}, [m s]$	45	34	68	45	23
z_1 - $\rho_{\rm m}$, [kgm ⁻³]	743	743	743	828	805
z ₂ -THR ₃₀₀ [MJm ⁻²]	90.6	55.5	63.0	61.5	82.1
z_3 -ln(FIGR A_{cc})	2.69	2.76	2.17	3.35	3.40
$t_{ m fo}, [m s]$	99.3	129.4	130.0	138.3	103.3
FO-category	4	3	3	3	4
F _{FO1}	2.5066	4.7006	3.6734	6.4307	4.8201
F _{FO2}	11.7554	10.7096	9.8537	13.2864	13.7280
F _{FO3}	37.3505	23.1851	24.6472	27.9745	36.3384
F _{FO4}	31.9827	20.0565	20.5226	24.2770	31.6057
FO-category	3	3	3	3	3

Table 5. Classification of results based on corrected 50 kW/m² test data

Conclusions

The HRR per area of the foam depends on the effective burning area and the heat release of the molten pool. This would cause the estimation of the foam flammability more complicated than it is suggested by the existing in the literature models. Because of that, when the foam melts and shrinks, the effective burning area should be smaller than the standard burning area used in calculations of the HRR per area and this would cause a lower data of HRR. Shrunk foam would get less total heat flux from the cone heater and this would influence the ignition and subsequent burning process.

In cone calorimeter tests, both the effective burning area and the density of molten foam have significant effect on the results. The initial burning area could be used to correct the HRR per area of sample, but this is still a subject to a certain amount of uncertainty due to the burning area changing during the combustion process.



The current models used for prediction of fire behavior as well as the flammability classification methods are incorrect in case of low density thermoplastics foams. In this context, even results based on corrected cone calorimeter test data need to be verified by full scale tests. In a real fire scenario, two key factors affect the fire behavior of the thermoplastic foams: the effective burning area and the flowing (or dripping) ability of the melts.

The fire behavior prediction model for low density thermoplastics foams should include the effective burning area caused by melt/shrink effect, and the HRR of molten foam. For

real fire scenarios the prediction of the flowing or dripping performance should be considered due to increase in the complexity in predicting the fire behavior. Moreover, it is necessary to study the melt/shrink performance of low density thermoplastic foams when evaluating the flammability hazard.

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Nomenclature

A_{e}	– effective area, [mm ²]	t_{ig} – ignition time, [s]
$R_{\rm a}$	- ratio of area, $(=A_nA_e^{-1})$	Construction In
$R_{ m d}$	– ratio of density, $(=\rho_{\rm m}\rho_{\rm n}^{-1})$	Greek symbols
THR	 total heat release in specific time 	$\rho_{\rm m}$ – density of molten foam, [kgm ⁻³]
	after ignition, [MJm ⁻²]	$\rho_{\rm n}$ – nominal density of foam, [kgm ⁻³]
$t_{ m fo}$	predicted flashover time, [s]	

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