Material Change Similarity

Natallia Safronava [1], Richard E Lyon [2]

[1] Technology and Management International LLC

[2] FAA Technical Center

International Aircraft Materials Fire Test Working Group Meeting

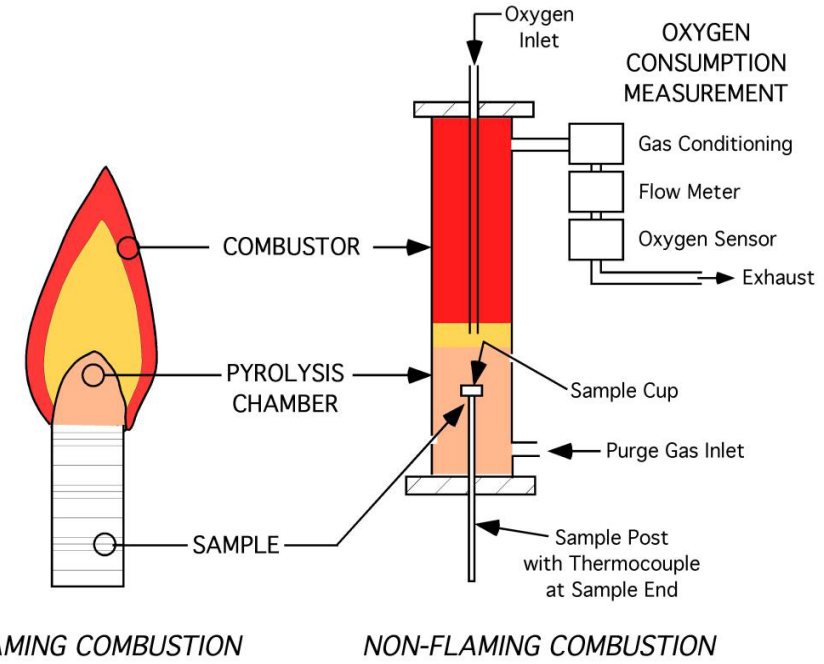
October 19-20, 2015

Atlantic City, NJ

MCC

FAA
Microscale Combustion Calorimeter

- U.S. Patents 6,464,391 & 5,981,290
- ASTM Standard D7309-13



FLAMING COMBUSTION *NON-FLAMING COMBUSTION*

Update on qualification test for adhesives

- ✓ Goal is to use MCC to determine that a small change in composition is a minor change with regard to fire safety in order to avoid recertification.
- ✓ Companies participating in similarity program supply samples with small changes in material composition along with FAR test results for both (2) samples.
- ✓ Microscale Combustion Calorimetry (MCC) testing is performed in FAA lab to determine if 2 materials are “similar” by MCC.
- ✓ MCC Similar = Mean value of fire property of 2 materials differs by less than the Reproducibility Limit (R) from ASTM D 7309.
- ✓ MCC Fire Property used for similarity to be determined.
- ✓ FAR Similar = 95% passing results in FAR tests of 2 materials.

Reproducibility Limit (R)

ASTM standard D7309-13

14.1.2 Reproducibility Limit (R) – Two test results shall be judged ***not equivalent if they differ by more that R value for that material***; R is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

14.4 To judge the equivalency of two test materials, it is recommended to choose the material that is closest in characteristics to the test material

TABLE 3 Heat Release Capacity (J per g-K)

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	s_R	r	R
Acrylic	471.0	9.0	26.7	25.1	74.7
PP	1095.3	32.5	86.4	91.0	242.0
HIPS	715.0	23.0	59.1	64.5	165.5
PC	529.5	25.3	48.2	70.9	134.9
PPSU	208.8	7.4	18.0	20.8	50.5

[A]The average of the laboratories' calculated averages.

Example:

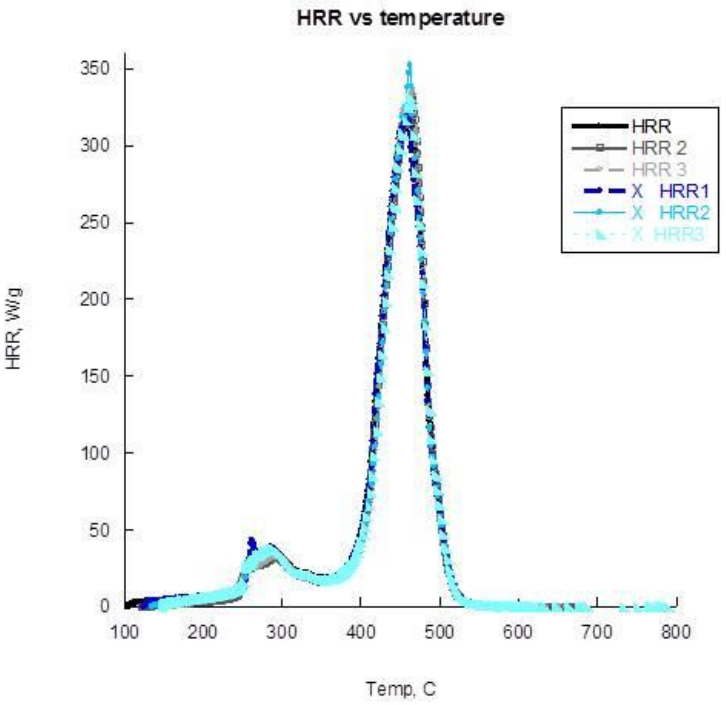
HRC parameter for a 2 samples with HRC around 200 J/g-K should not be different by more than (50.5/209)*100 = 24%, which is approximately the reproducibility standard deviation of OSU tests.

B/E Aerospace samples April 2015

- Two samples of adhesive were submitted for MCC testing
- ***The average values for HRC, HRR, HR, T_{peak} and char yield*** were within reproducibility limit R

MCC Test results

Sample	Initial mass, mg	Char Yield, %	HRC, J/g-K	HRR_{peak} W/g	HR, kJ/g	$Temp_{max}$, C
H3101 1	5.0	9.4	364	323	24.3	464
H3101 2	5.6	9.4	359	331	24.4	463
H3101 3	4.6	8.8	379	333	24.0	464
Average/STDEV		**9.2** ± 0.3	**367** ± 10	**329** ± 5	**24.2** ± 0.2	**464** ± 1
H3101X 1	5.7	10.1	375	320	24.0	460
H3101X 2	4.7	9.7	368	349	24.2	460
H3101X 3	3.9	9.8	369	331	24.0	462
Average/STDEV		**9.9** ± 0.2	**371** ± 4	**333** ± 15	**24.1** ±0.1	**461** ± 1



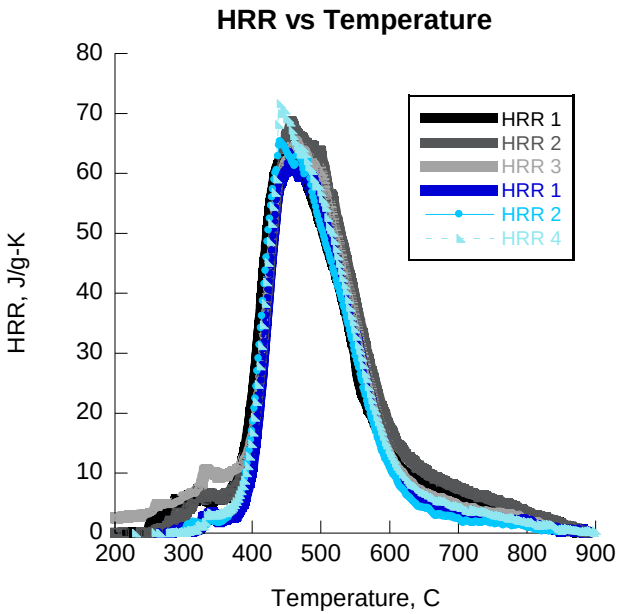
- ***Flammability fingerprints (HRR versus T) were indistinguishable*** with regard to the location and magnitude of HRR
- OSU and NBS testing were completed for the samples. Two sets of testing were completed. The first set showed variation in total heat release. Second set showed no variation with t-test analysis demonstrating that two samples could have come from the same population of test articles.

B/E Aerospace samples September 2015

- Samples of parts for aircraft seats were submitted for MCC testing

MCC Test results

Sample	Initial mass, mg	Char Yield, %	HRC, J/g-K	HRR_{peak} W/g	HR, kJ/g	$Temp_{max}$, C
MTM 828 Resin #1	4.39	51.8	90	64	11.2	458
	4.44	50.7	96	66	11.9	465
	4.97	51.3	93	65	11.4	466
Aver ± STDEV		**51.3 ± 0.6**	**93 ± 3**	**64 ± 2**	**11.5 ± 0.4**	**463 ± 4**
MTM 82S Resin # 1	5.27	51.5	95	63	10.0	459
	5.23	50.9	98	66	10.0	442
	5.70	50.7	104	70	10.6	444
Aver ± STDEV		**51.4 ± 0.4**	**99 ± 5**	**66 ± 4**	**10.2 ± 0.3**	**448 ± 9**
Reproducibility Limit (R)		17%	24%	26%	23%	5%

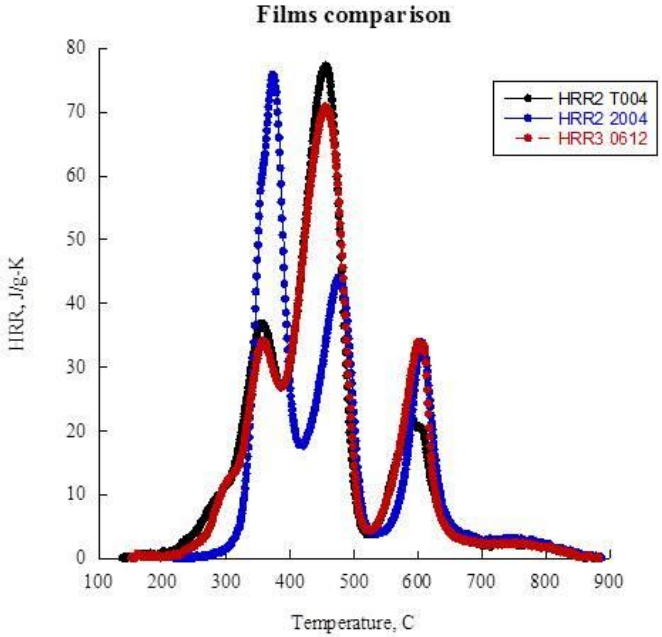


- The flammability fingerprints are very similar
- There are some differences in MCC test parameters (total heat released), but it is within R limit
- FAR testing displayed no significant difference between samples

Hutchinson samples 01/2015

- MCC testing showed one of the films being different from the other two.

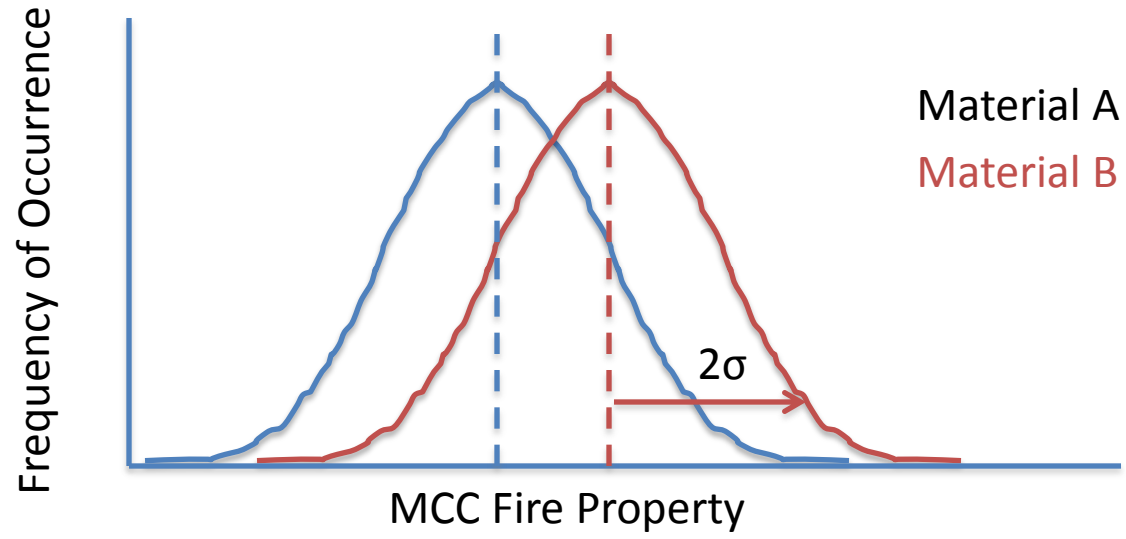
Sample name	Sample mass, mg	Char Yield, %	HRC sum, J/g-K	HRR peak, W/g	HR, kJ/g	T peak, C	T onset, C
Film T004	6.1	34	159	80	10.4	466	270
	4.6	33	143	77	10.7	455	252
	5.8	34	134	70	10.4	446	232
Average		***34***	***145***	***76***	***10.5***	***456***	***251***
Film 0612	6.2	39	135	55	9.7	457	270
	5.9	37	155	79	10.4	472	280
	5.3	36	141	71	10.6	456	271
Average		***37***	***144***	***68***	***10.2***	***462***	***274***
Film 2004	6.1	38	148	76	9.2	372	317
	6.4	38	139	74	9.1	377	317
	7.2	36	152	81	9.5	377	316
Average		***38***	***146***	***77***	***9.3***	***375***	***317***

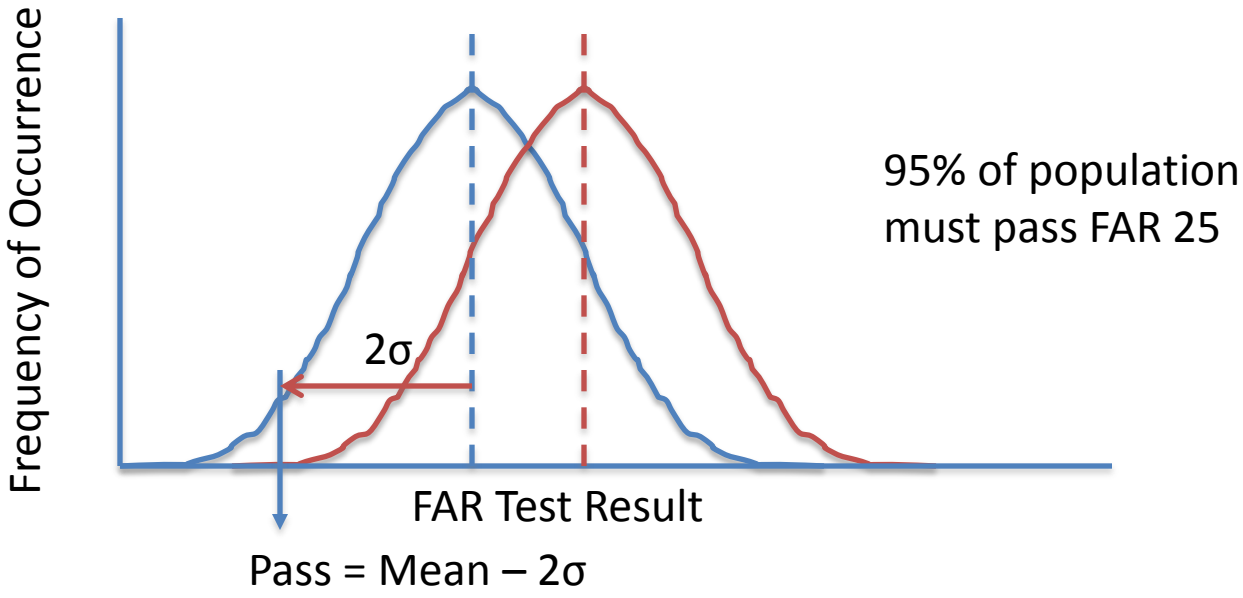


- Higher onset temperature, lower total heat release and presence of CO gas in combustion products make film# 2004 more flame resistant then the other two

- FAR testing showed some failures of film #2004 , probably due configuration of blankets

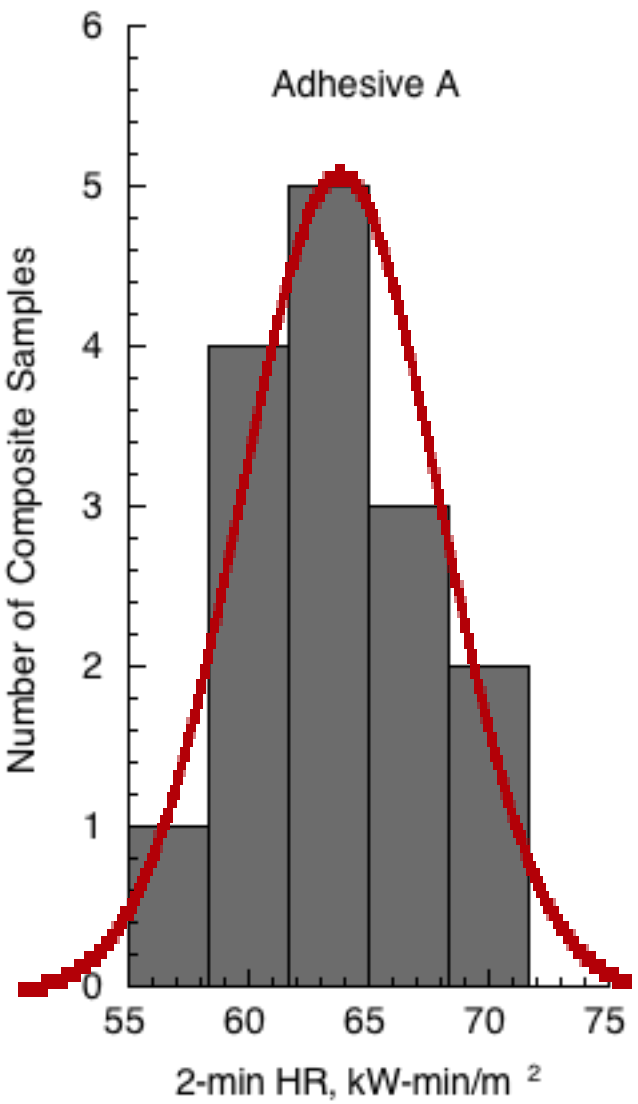
How to Compare 2 populations in 2 Different Tests?

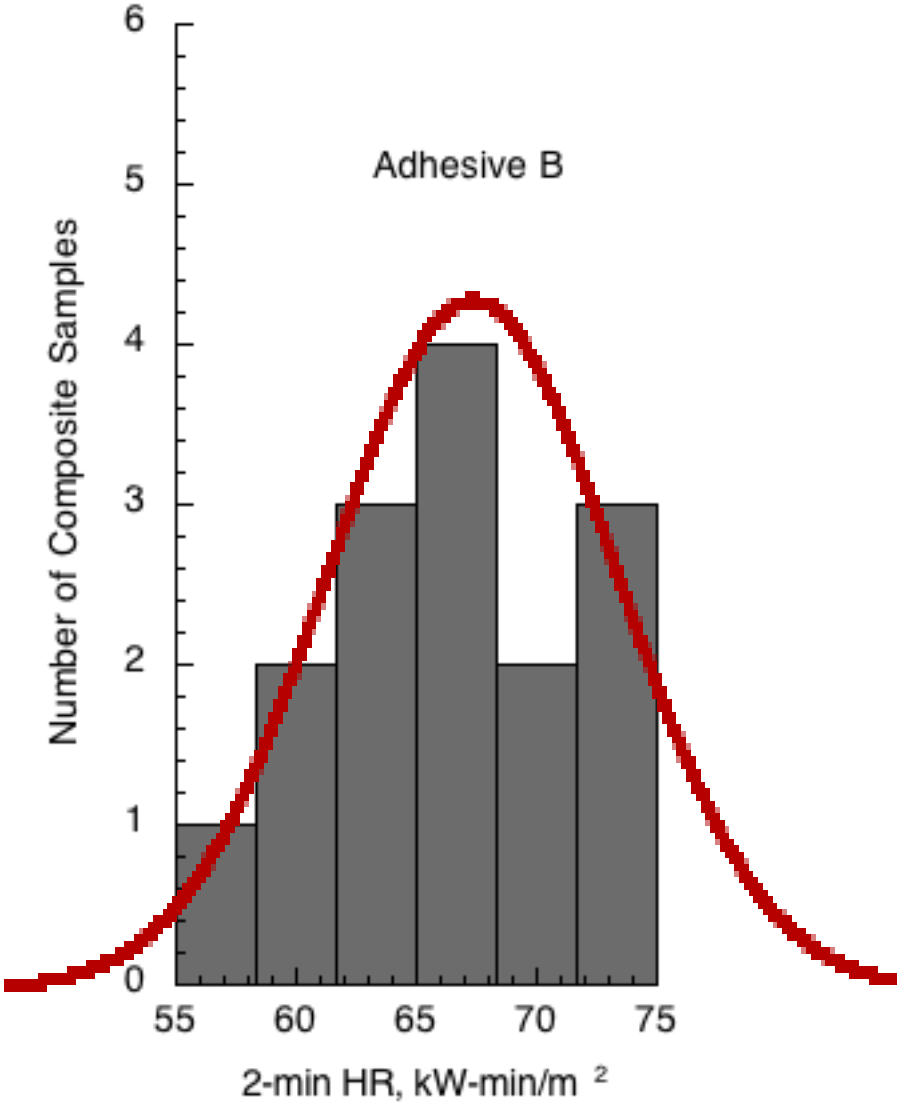




95% of population must pass FAR 25

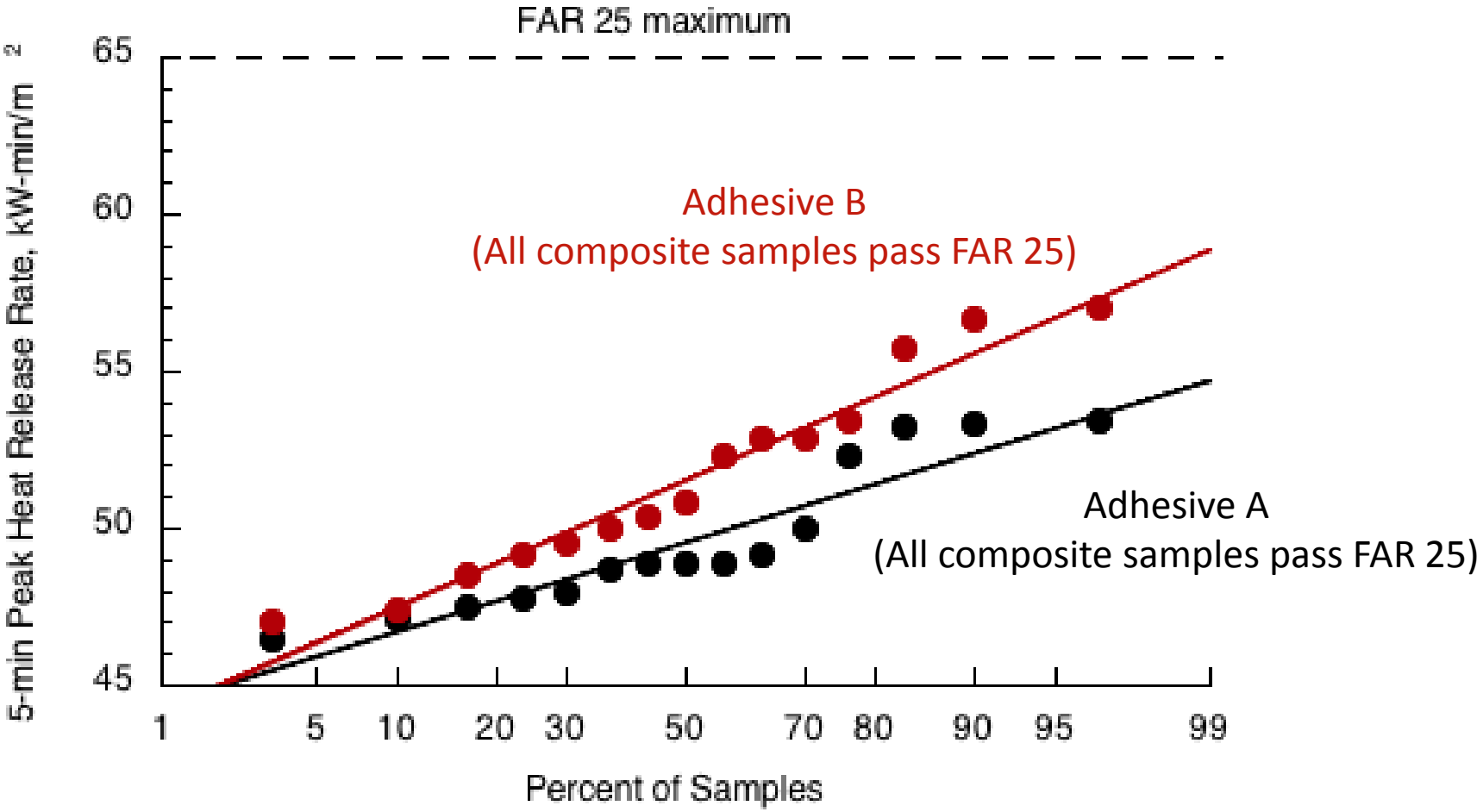
- Plot frequency distribution of FAR results
- Fit normal probability function to data
- Calculate percentage of FAR results below particular value





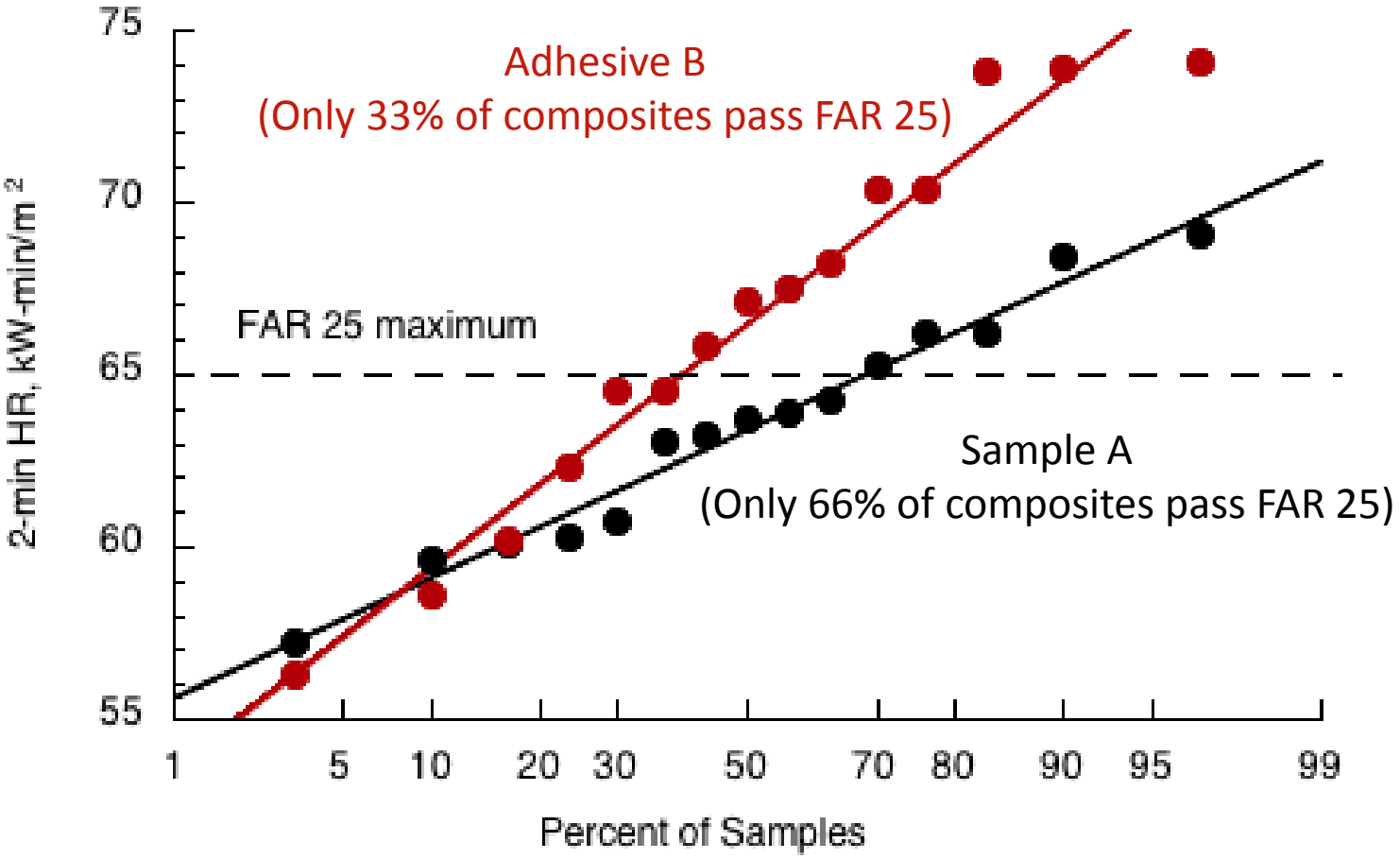
FAR Peak Heat Release Rate of Composites in OSU is Similar for 2 Adhesives (Both Pass)

KYDEX/Adhesive/Aluminum Composite



FAR 2-min Heat Release of Composite in OSU is Similar for 2 Adhesives (Both Fail)

KYDEX/Adhesive/Aluminum Composite



NO FALSE POSITIVES OBSERVED IN TESTING TO DATE

Company	MCC Similar?	FAR Similar?	FAR Test Configuration	MCC DETERMINATION
B/E Aerospace (April 2015)	YES	YES	OSU Kydex/Adhesive/Aluminum	POSITIVE
B/E Aerospace (September 2015)	YES	YES	60 s VBB OSU Smoke density	POSITIVE
Hutchinson	NO	NO	Radiant Panel	POSITIVE
3M	NO MAYBE	NO MAYBE	VBB 12 s	POSITIVE POSITIVE

MCC Testing

MCC Can Determine Similarity of Individual Components By Comparing:

- Heat Release Capacity
- Total Heat Release
- HRR Signature
- CO/CO_2 Ratio

For Dissimilar Components, MCC may be able to determine Better or Worse using Flammability Index:

$$F_{index} = \frac{\text{Heat Released by Combustion}}{\text{Heat Required for Ignition}} = \frac{HR\ (750^{\circ}\ C)}{c_p(T_{onset}-T_0)}$$

Similarity Determination ?