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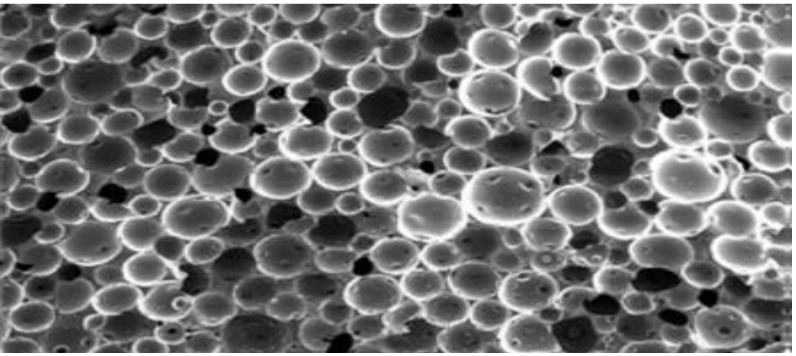

Thermal Decomposition of Mixed Foams used as Encapsulant Materials

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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Contents

Introduction

- Most modeling efforts of abnormal environments assume thermal decomposition of foams that encapsulate electrical assemblies occurs independently of other organic materials in close proximity.
- Where just one type of foam is used for encapsulation, this assumption is likely acceptable.
- Some systems have two, three, or even four different foams within a specific component.
- Each of these foams has a unique decomposition profile and generates different gaseous species from the other foams. The possibility that byproducts from the decomposition of foam A might affect the decomposition of foam B has not yet been explored, to the best of our knowledge.

Description of Foams

- This Study details five foams of interest using thermal gravimetric analysis (TGA).
- Foams investigated were developed at Sandia National Laboratories and have been qualified for use as potting materials.
- The formulations and processing requirements for these foams have been previously documented

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3. Ulibarri, T. A.; Derzon, D. K.; Erickson, K. L.; Casteneda, E. J.; Borek, T. T.; Renlund, A. M.; Miller, J. C.; Clayton, D.; Fletcher, T. *Preliminary Investigation of Thermal Decomposition of Ablefoam and EF-AR20 Foam (Ablefoam Replacement*), SAND2002-0183, Sandia National Laboratories, Albuquerque, NM, January 2002.
4. Russick, E. M. *Electrical Properties of REF308, REF320, EF-AR20, and RSF200 Foam Encapsulants*, SAND2006-0134, Sandia National Laboratories, Albuquerque, NM, January 2006.
5. McElhanon, J. R.; Russick, M. E.; Aubert, J. H. *Removable Foams Based Upon an Epoxy Resin Incorporating Reversible Diels-Alder Adducts*, SAND2001-0954J, Sandia National Laboratories, Albuquerque, NM, March 2001.
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7. Aubert, J. H.; McElhanon, J. R. Saunders, R. S.; Sawyer, P. S.; Wheeler, D. R.; Russick, E. M, Rand, P. B. Loy, D. A. *Progress in Developing Removable Foams, Adhesives, and Conformal Coatings for the Encapsulation of Weapon Components*, SAND2001-0295, Sandia National Laboratories, Albuquerque, NM, February 2001.

Description of Foams

Name	Description	Components	References
GMB Epoxy	GMB-filled epoxy polymer	EPON Resin 828: 75 weight percent (wt%) Diethanolamine: 9 wt% Hollow GMB: 21 wt%	SS1A7955
EF-AR20	EF-Ablefoam replacement (AR) with molded density of 20 lb/ft^3	Resin #1: Epon 830: 60 wt% Resin #2: Epon 8121: 40 wt% Curing agent #1: Ancamine 2049: 25.8 parts per 100 parts resin (phr) Curing agent #2: Epi-cure 3270: 26.2 phr Surfactant: DC-193: 3.0 phr Blowing agent: Fluorinert FC-72: 6.0 phr Nucleating agent: Carbon black: 2.0 phr	SAND98-2538 SAND2002-0183 SAND2006-0134
RSF200	Removable syntactic foam (RSF) with nominal density of 48 lb/ft^3	Removable Epoxy Resin 1: 60 wt% Epon 8121: 20 wt% Epon 8021: 20 wt% Ancamine 2049 curative: 40 phr 3M GMB (D32/4500) 35: phr	SAND2001-0954J SAND2006-0134 SAND2001-0295
REF320	REF with molded density of 20 lb/ft^3	Removable Epoxy Resin 3: 60 wt% Epon 8121 40: wt% Ancamine 2049 curative: 35 phr Ancamine 2205 curative: 14 phr DC-193 surfactant: 1.6 phr Fluorinert FC-72: 18 phr Cab-o-Sil M-5 fumed silica: 0.8 phr	
REF308	REF with molded density of 8 lb/ft^3	Removable Epoxy Resin 3: 60 wt% Epon 8121 40: wt% Ancamine 2049 curative: 35 phr Ancamine 2205 curative: 14 phr DC-193 surfactant: 7 phr Fluorinert FC-72: 41 phr Cab-o-Sil M-5 fumed silica: 0.9 phr	

Sample Preparation/Data Acquisition

Sandia National Laboratories

	GMB Epoxy	EF-AR20	RSF200	REF320	REF308
GMB Epoxy	GMB Epoxy + GMB Epoxy	EF-AR20 + GMB Epoxy	RSF200 + GMB Epoxy	REF320 + GMB Epoxy	REF308 + GMB Epoxy
EF-AR20		EF-AR20 + EF-AR20	RSF200 + EF-AR20	REF320 + EF-AR20	REF308 + EF-AR20
RSF200			RSF200 + RSF200	REF320 + RSF200	REF308 + RSF200
REF320				REF320 + REF320	REF308 + REF320
REF308					REF308 + REF308

- Samples were prepared in an aluminum crucible without (unconfined, UC) and with a lid (partially confined, PC).
- For samples with a lid, the lid was automatically pierced with a 1.0 mm diameter needle attached to the autosampler prior to TGA evaluation.
- No effort was made to mix or crush the combined polymer foam samples

Sample Preparation/Data Acquisition

Sandia National Laboratories

Thermal gravimetric analysis

- Individual material or a combination of two materials
- Ratio of ~ 1:1
- All samples were heated from 35 to 600°C at 10°C/min under an argon flow of 40 mL/min.
- Samples were run in triplicate.

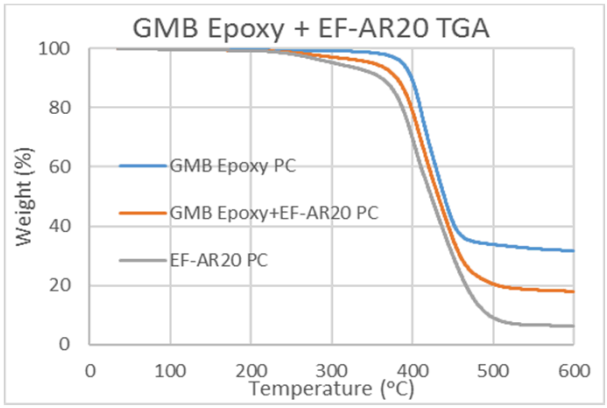
DTG

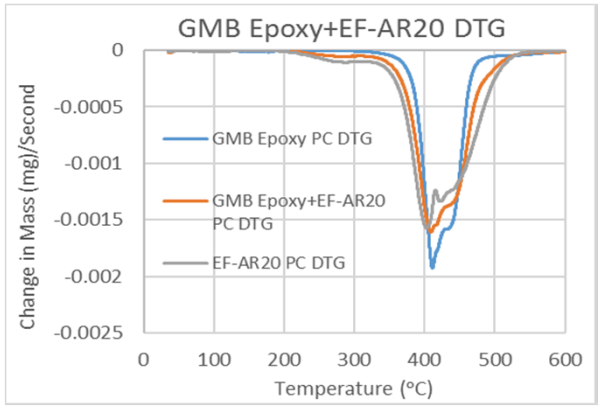
- Overlaid differential thermogravimetric analysis (DTG) traces were used to illustrate subtle changes in rate and weight loss not easily seen in standard TGA plots.

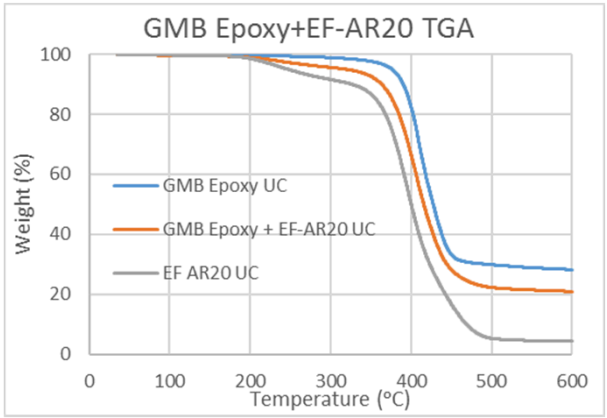
Wt% loss

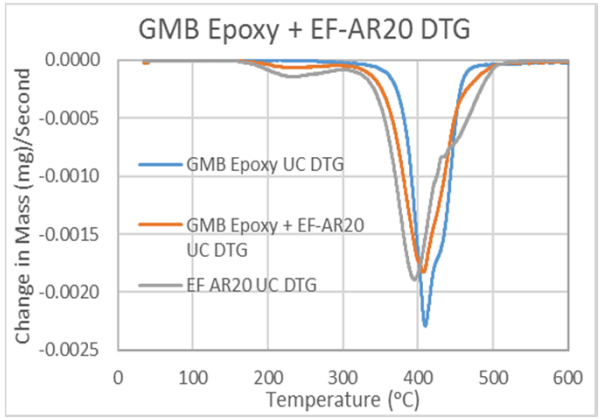
- The weight percent loss of the mixed sample vs the predicted weight loss was calculated and compared to the observed weight percent loss.
- Numbers listed are based on the representative TGA plots shown.

Results: GMB Epoxy + EF-AR20





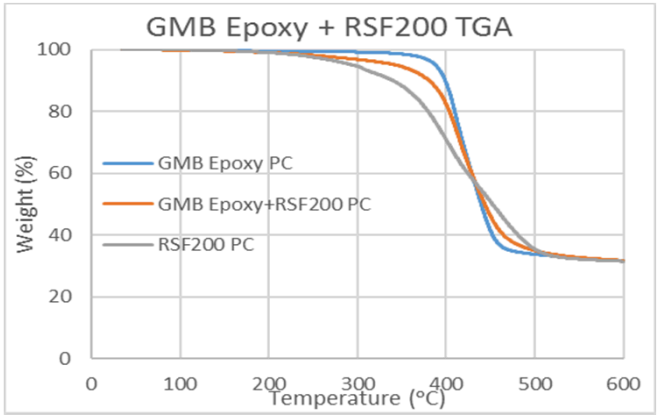


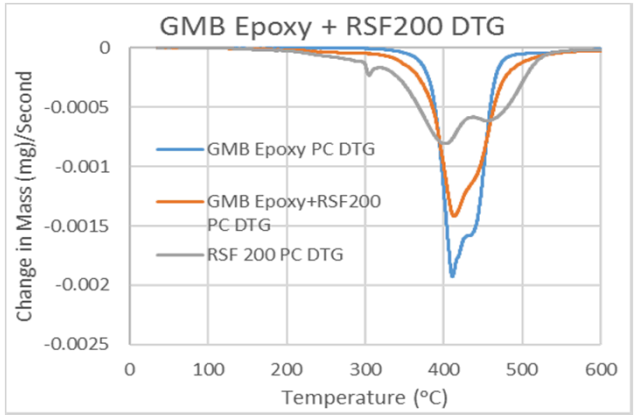


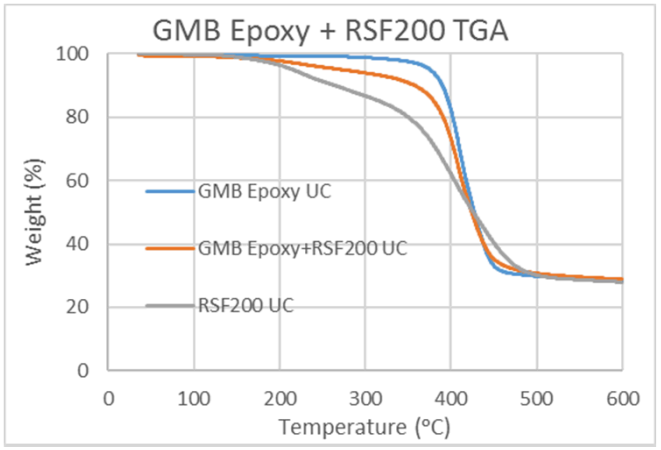
- PC and UC samples showed similar weight loss.
- Onset of decomposition started at a higher temperature for PC samples.
- The DTG curves showed the PC samples underwent a more stepped decomposition than the UC system, particularly at the transition occurring at 415°C.
- There is a difference in the observed vs. expected weight percent loss for partially confined and unconfined samples.
- This is consistent for triplicate samples

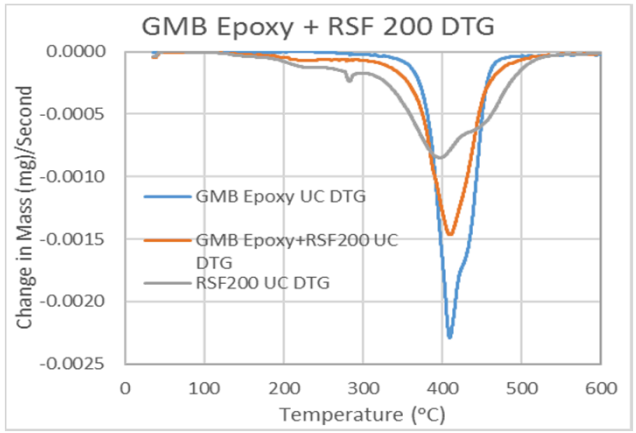
Sample Environment	Foam A	Foam B	w% Foam A	wt% Foam A * individual Foam A wt loss	wt% Foam B	wt% Foam B * individual Foam B wt loss	Expected wt% loss (wt% loss based on weighted wt% loss of individual samples)	Observed wt% loss	Difference: observed wt% loss – expected wt% loss
Partially Confined	GMB Epoxy	EF-AR20	58.56	40.42	41.78	38.58	79.00	81.19	2.19
Unconfined	GMB Epoxy	EF-AR20	59.02	41.01	40.98	35.68	76.69	77.46	0.77

Results: GMB Epoxy + RSF200





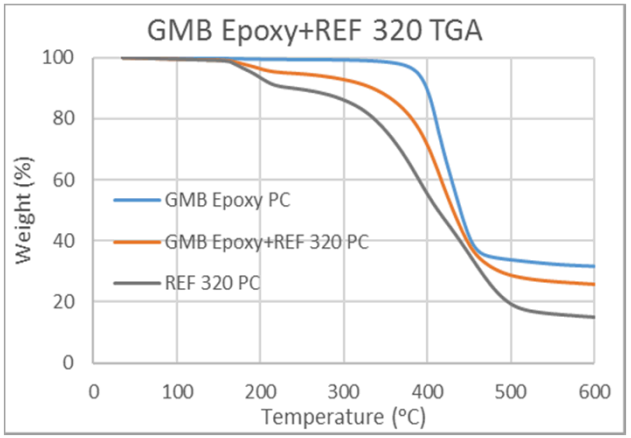


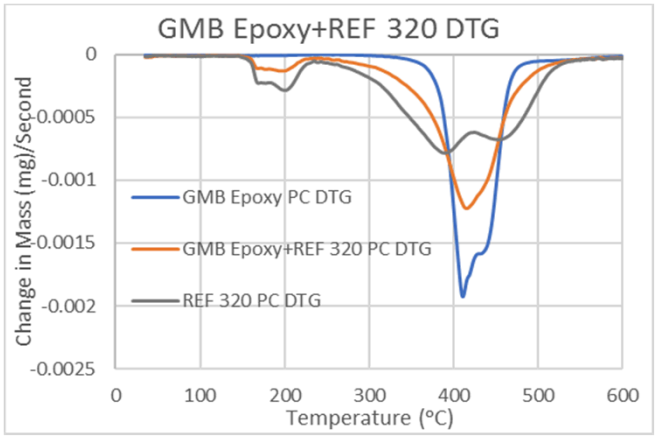


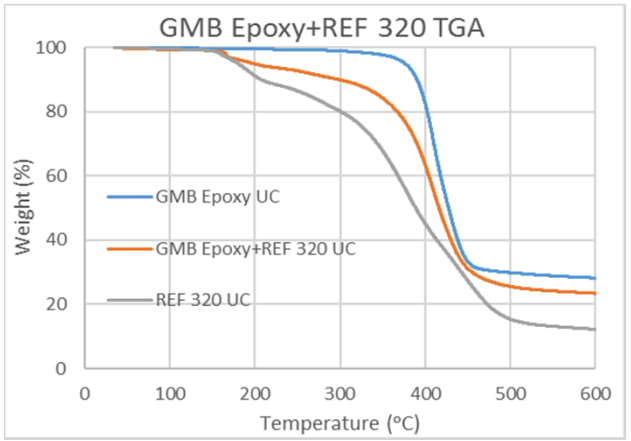
- Both PC and UC samples showed similar weight loss.
- The two systems showed similar decomposition profiles.
- The onset of decomposition started at a higher temperature for partially confined samples.
- The mixed PC and UC systems both show a noticeable decrease in stepped decomposition, particularly at the transition occurring at 415°C. This is more pronounced in the UC sample.

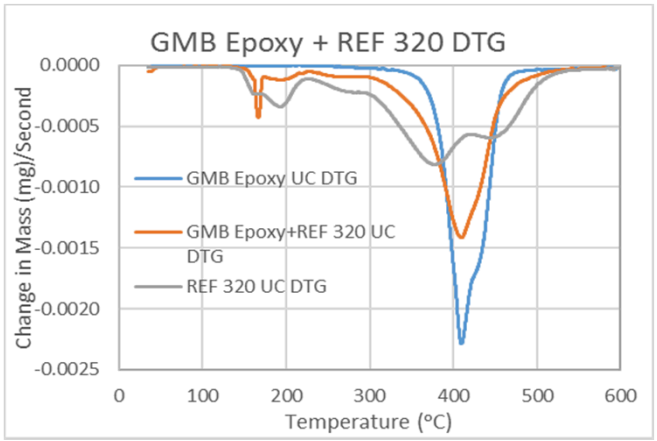
Sample Environment	Foam A	Foam B	wt% Foam A	Weighted wt% A	wt% Foam B	Weighted wt% B	Expected wt% loss	Observed wt% loss	Difference: observed–expected
Partially Confined	GMB Epoxy	RSF200	60.87	42.02	39.13	24.89	66.91	64.82	-2.09
Unconfined	GMB Epoxy	RSF200	64.38	44.74	35.62	24.51	69.24	64.06	-5.18

Results: GMB Epoxy + REF320





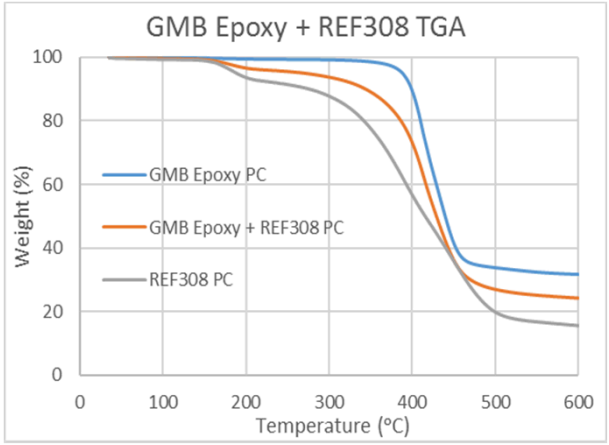


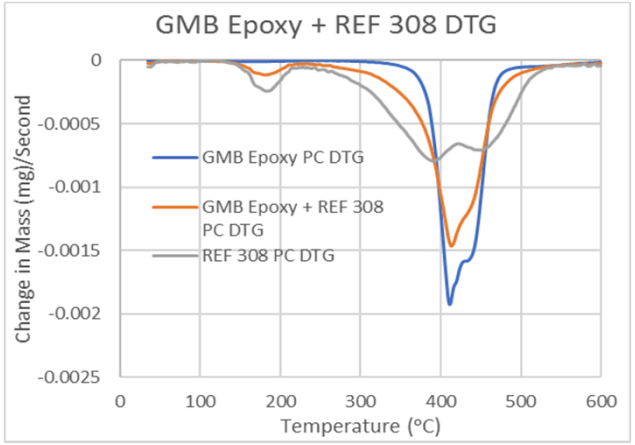


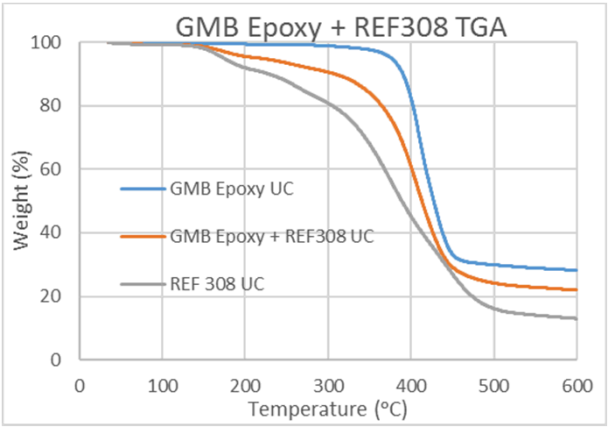
- PC and UC samples showed similar weight loss.
- The decomposition profiles of the PC system showed a more rapid weight loss for the initial event than did the UC sample, indicating a change in the mechanism in the PC system.
- For the UC system, variations between the individual samples and the mixed sample are more pronounced, most notably at temperatures below 300°C.

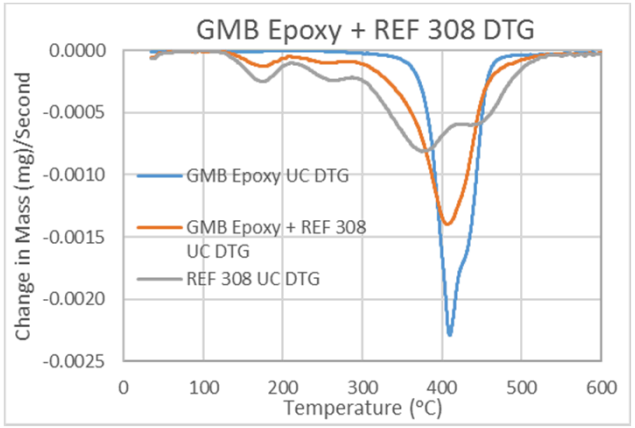
Sample Environment	Foam A	Foam B	wt% Foam A	Weighted wt% A	wt% Foam B	Weighted wt% B	Expected wt% loss	Observed wt% loss	Difference: observed–expected
Partially Confined	GMB Epoxy	REF320	50.47	34.84	49.53	41.29	76.14	72.70	-3.44
Unconfined	GMB Epoxy	REF320	53.16	36.94	46.84	40.07	77.02	74.75	-2.27

Results: GMB Epoxy + REF308





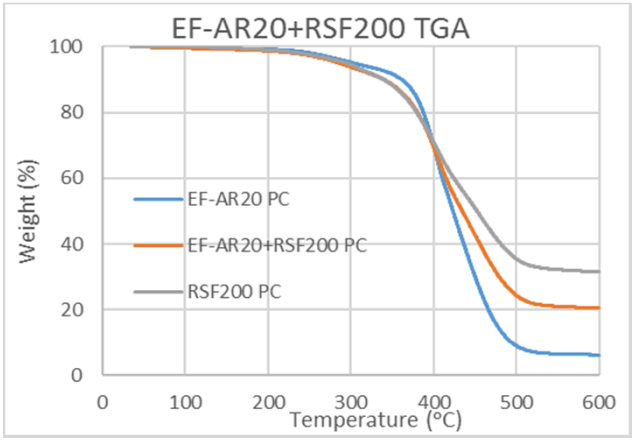


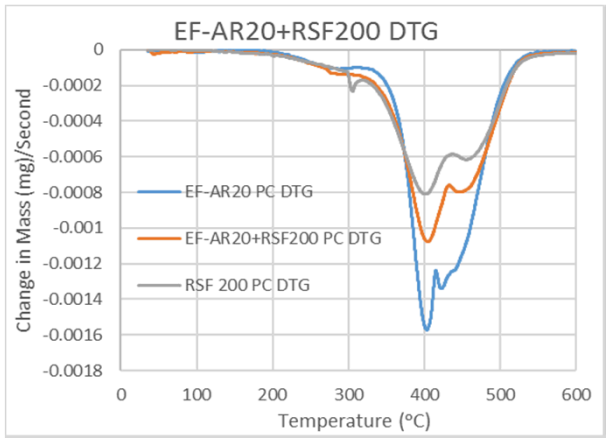


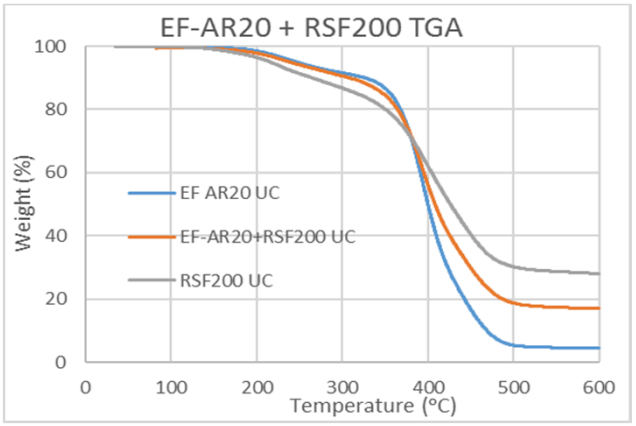
- PC and UC samples showed similar weight loss.
- Decomposition profiles of the PC system show less of a stepped weight loss initially followed by more of a stepped weight loss compared to the samples in the UC system, indicating a difference in the decomposition mechanism between the two samples.
- For the UC system, the onset of decomposition is slightly shifted to start at a lower temperature

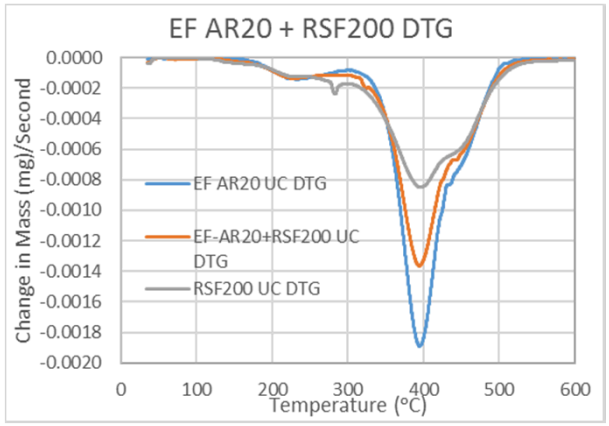
Sample Environment	Foam A	Foam B	wt% Foam A	Weighted wt% A	wt% Foam B	Weighted wt% B	Expected wt% loss	Observed wt% loss	Difference: observed–expected
Partially Confined	GMB Epoxy	REF308	55.81	38.52	44.19	36.58	75.11	74.55	-0.56
Unconfined	GMB Epoxy	REF308	54.62	37.96	45.38	37.87	75.83	75.86	0.04

Results: EF-AR20 + RSF200





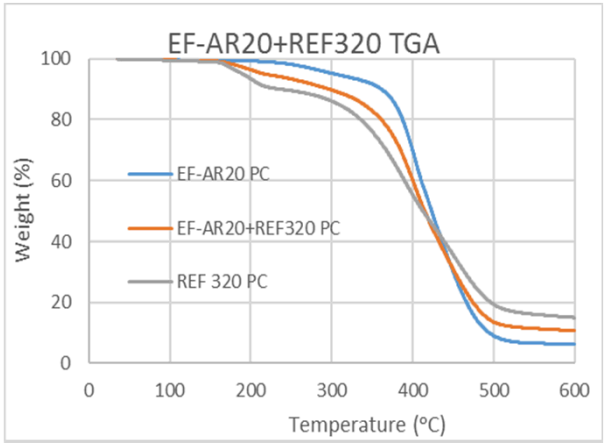


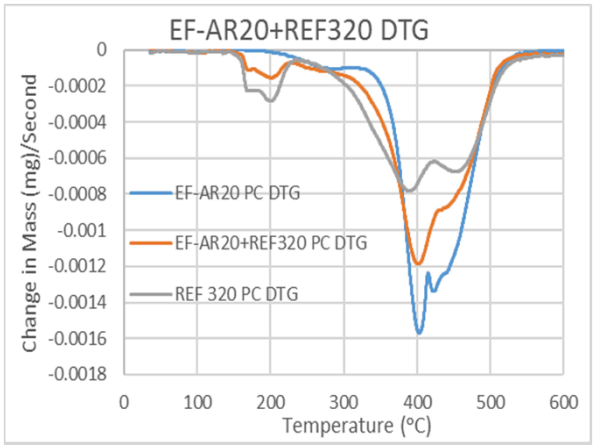


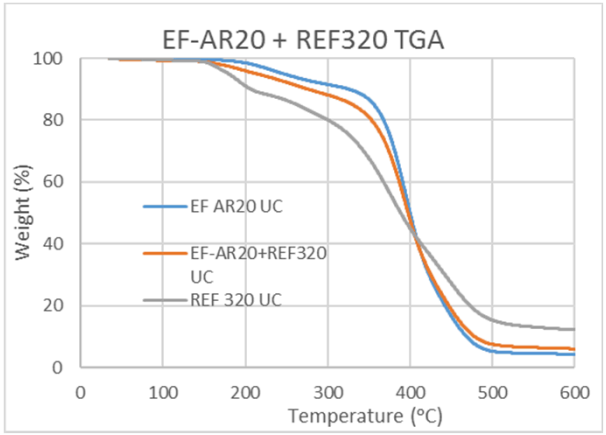
- PC and UC samples showed similar weight loss.
- The PC system exhibited a much more stepped weight loss indicating a difference in the decomposition mechanism for the two samples.

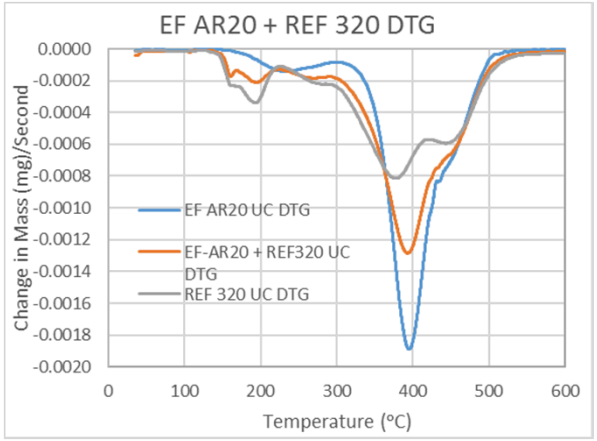
Sample Environment	Foam A	Foam B	wt% Foam A	Weighted wt% A	wt% Foam B	Weighted wt% B	Expected wt% loss	Observed wt% loss	Difference: observed–expected
Partially Confined	EF-AR20	RSF200	39.08	36.09	62.36	39.68	75.76	78.04	2.28
Unconfined	EF-AR20	RSF200	48.82	42.51	51.18	35.21	77.72	81.47	-3.75

Results: EF-AR20 + REF320





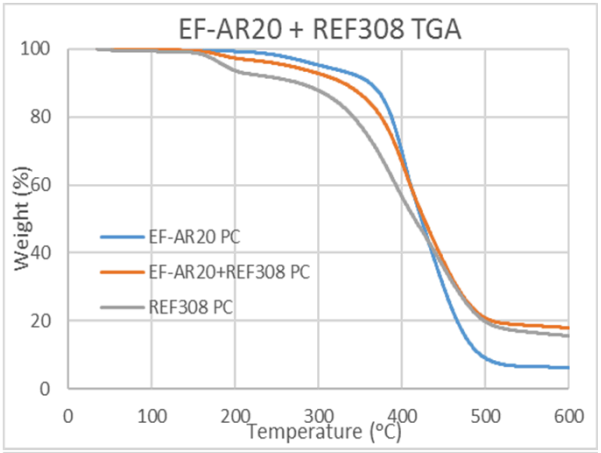


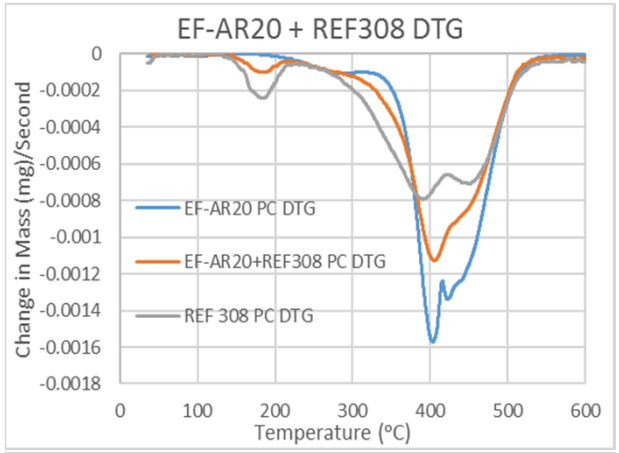


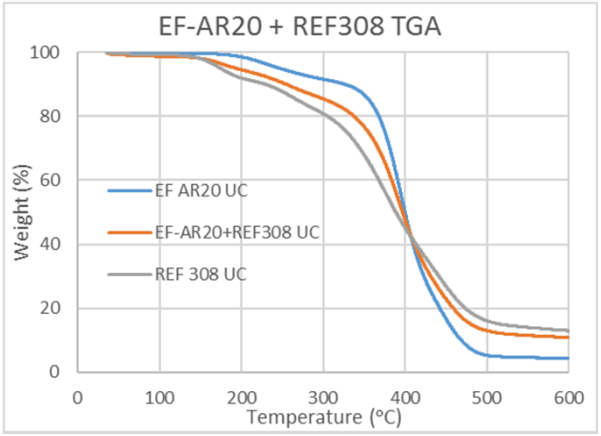
- The sample in the PC system showed a slightly lower wt% loss than in the UC system.
- The TGA curve for the PC sample shows decomposition as expected.
- The UC sample exhibits an unusual behavior.
  - The curve initially lies closer to REF320, while at higher temperatures, the mixed sample converges to EF-AR20.
  - May indicate that when REF320 decomposes simultaneously with EF-AR20, less char is formed than if REF320 decomposes alone.
  - The DTG curves for the UC sample mimic this behavior.
- The wt% loss of the mixed vs individual samples was calculated and compared to observed. The PC showed only a 0.23 wt% difference. The UC showed a discrepancy in the expected vs. observed of 7.58 wt%.

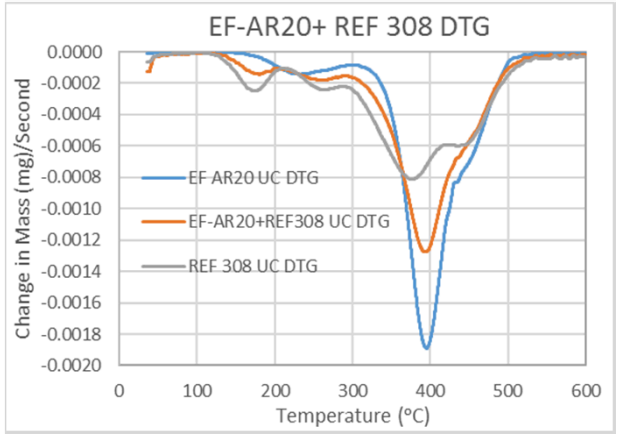
Sample Environment	Foam A	Foam B	wt% Foam A	Weighted wt% A	wt% Foam B	Weighted wt% B	Expected wt% loss	Observed wt% loss	Difference: observed– expected
Partially Confined	EF-AR20	REF320	50.74	46.86	49.26	41.07	87.93	87.70	-0.23
Unconfined	EF-AR20	REF320	42.24	36.77	57.76	49.42	86.19	93.78	7.58

Results: EF-AR20 + REF308









- The weight loss between PC and UC EF-AR20 + REF308 samples varied.
- The TGA profile for the PC mixed sample shows a decomposition curve that converges on the REF308 curve at high temperatures.
  - This behavior may indicate that when REF308 decomposes simultaneously with EF-AR20, more char is formed than if REF308 decomposes alone.
  - This is the opposite effect from what is seen with EF-AR20 + REF320. This trend is less noticeable in the DTG curves

Sample Environment	Foam A	Foam B	wt% Foam A	Weighted wt% A	wt% Foam B	Weighted wt% B	Expected wt% loss	Observed wt% loss	Difference: observed–expected
Partially Confined	EF-AR20	REF308	52.31	48.30	52.66	43.59	91.89	81.31	-10.58
Unconfined	EF-AR20	REF308	47.84	41.65	52.16	43.53	85.18	86.37	1.19

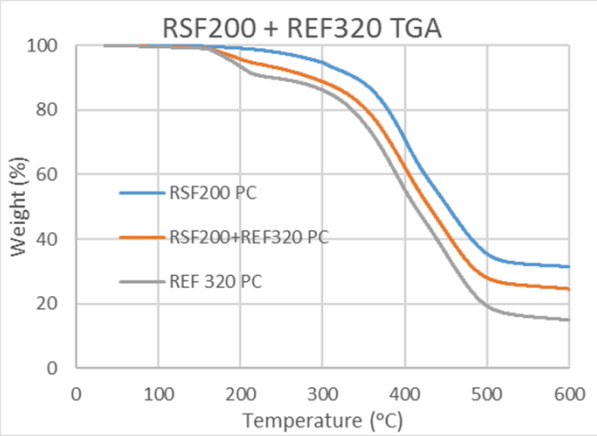
- The behavior of EF-AR20 when combined with REF320 and REF308 is unusual compared to when EF-AR20 is combined with other foams.

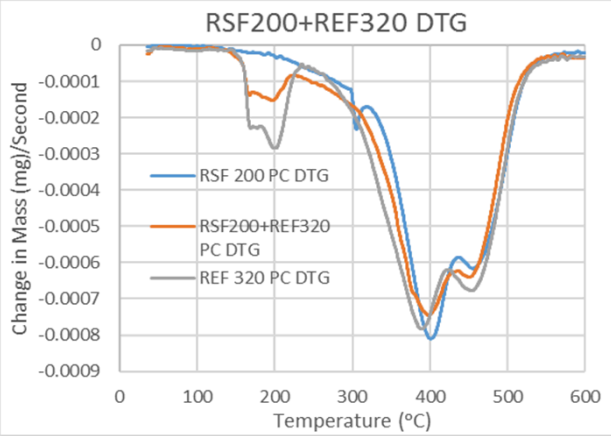
- More analysis is needed in order to determine the mechanism of decomposition for these foam combinations, however it makes sense that if EF-AR20 + REF320 exhibited an unusual interaction then EF-AR20 + REF308 would as well since the two foams are similar.

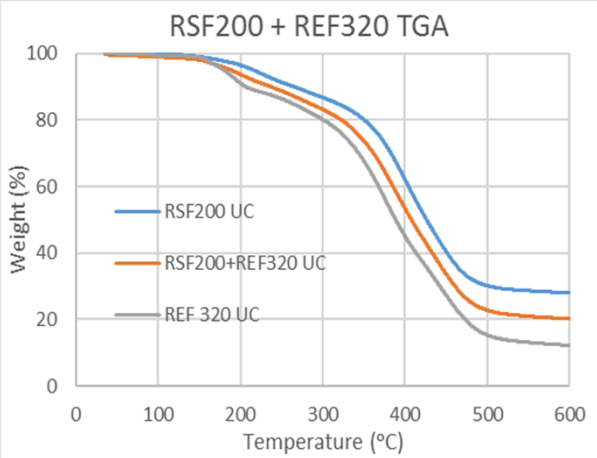
  - REF320 and REF308 are formulated from the same components in different quantities to achieve a different final density.

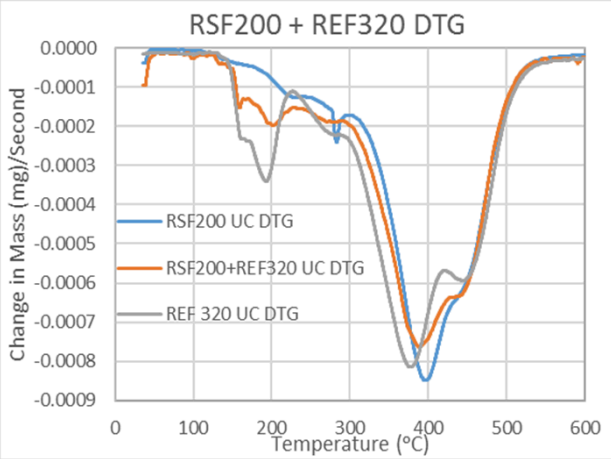
  - The differences in formulation between REF320 and REF308 may be attributed to the increased or decreased char formation seen when these foams are combined with EF-AR20 as different amounts of off-gasses from the decomposing foam can contribute to different side reactions between the decomposing foam and off-gases in the system.

Results: RSF200 + REF320





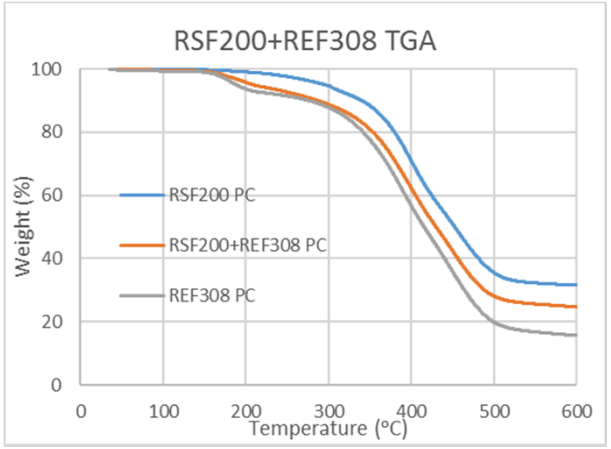


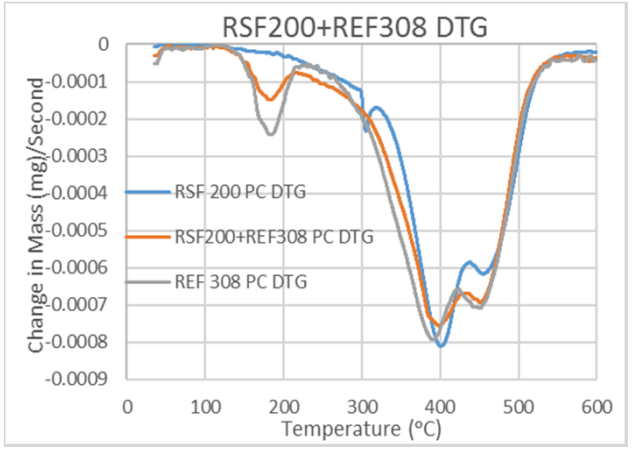


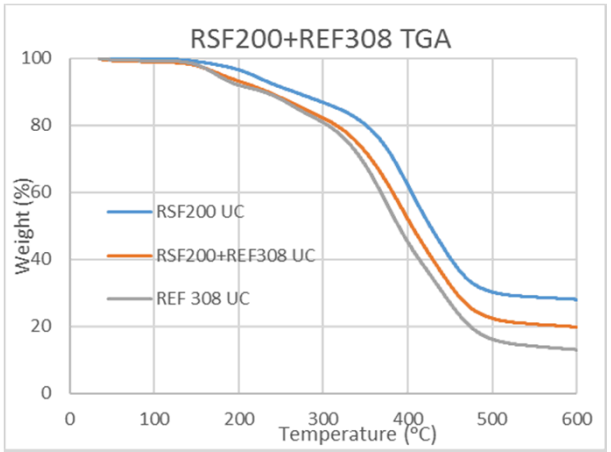
- The PC system showed a lower percent weight loss than the UC system.
- The DTG curves for the partially confined and unconfined sample show that the mixed sample lies between the two individual samples.

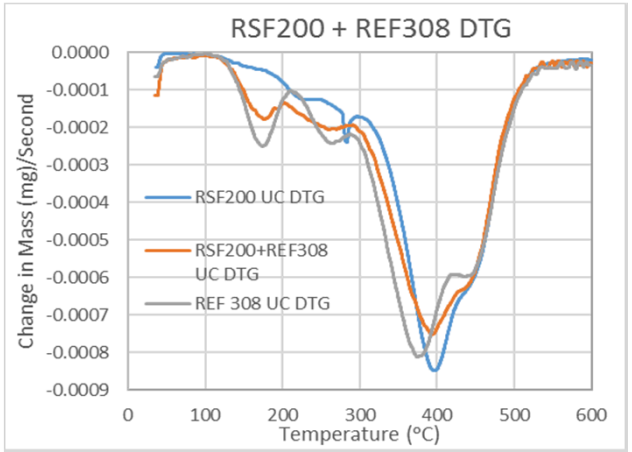
Sample Environment	Foam A	Foam B	wt% Foam A	Weighted wt% A	wt% Foam B	Weighted wt% B	Expected wt% loss	Observed wt% loss	Difference: observed– expected
Partially Confined	RSF200	REF320	55.24	35.14	44.76	37.32	72.46	73.95	1.49
Unconfined	RSF200	REF320	55.36	38.09	44.64	38.19	76.28	77.28	1.00

Results: RSF200 + REF308





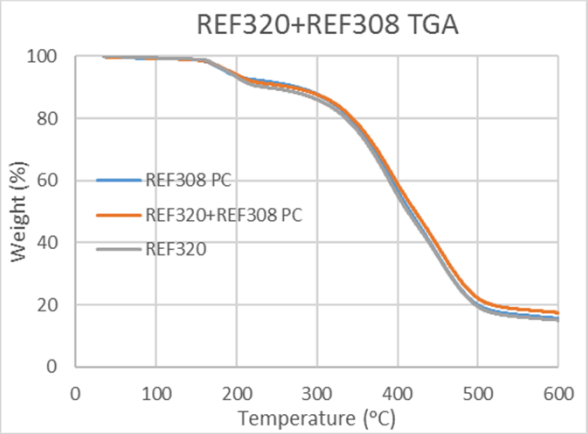


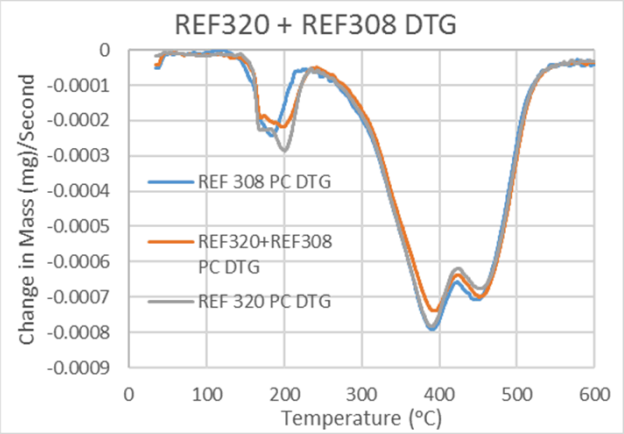


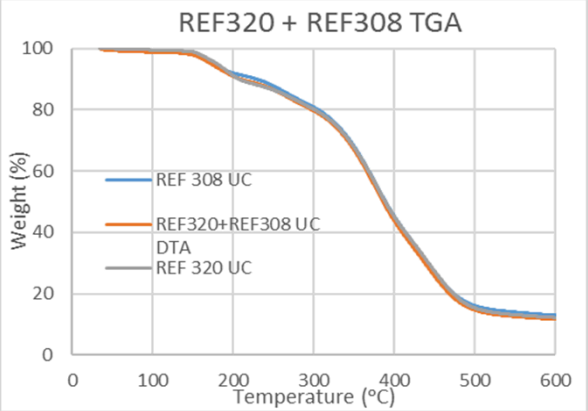
- PC and UC samples showed similar weight loss.
- The initial weight loss for the unconfined sample is much greater
- In the second weight loss event, the unconfined sample underwent a smaller weight loss
- The weight percent loss of the mixed sample versus the weight loss for the two individual materials was calculated and compared to the observed weight loss

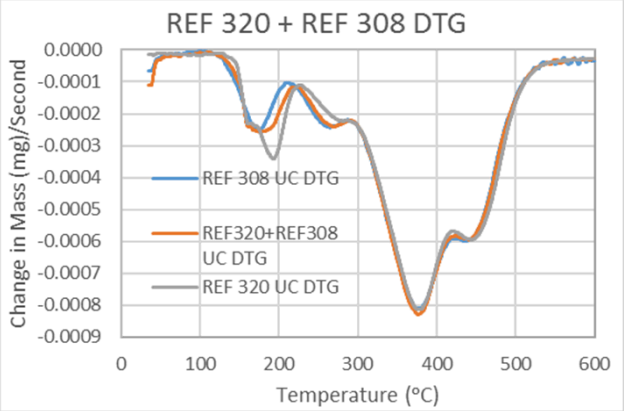
Sample Environment	Foam A	Foam B	wt% Foam A	Weighted wt% A	wt% Foam B	Weighted wt% B	Expected wt% loss	Observed wt% loss	Difference: observed– expected
Partially Confined	RSF200	REF308	48.00	30.54	52.00	43.04	73.58	77.09	3.51
Unconfined	RSF200	REF308	52.04	35.80	47.96	40.02	75.83	77.83	2.00

Results: REF320 + REF308









- PC and UC samples showed similar weight loss.
- The TGA decomposition profiles of both the UC and PC samples were similar in shape.
- The DTG curves show a more stepped decomposition profile in the UC system sample.
- The DTG curves also show that the mixed sample lies between the two individual samples
- These curves are similar since REF320 and REF308 are similar foams with different densities.

Sample Environment	Foam A	Foam B	wt% Foam A	Weighted wt% A	wt% Foam B	Weighted wt% B	Expected wt% loss	Observed wt% loss	Difference: observed–expected
Partially Confined	REF320	REF308	58.73	48.61	41.27	34.17	82.78	81.15	-1.63
Unconfined	REF320	REF308	35.76	30.59	64.24	53.61	84.20	86.37	2.17

Conclusions

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Partially Confined vs. Unconfined Foams

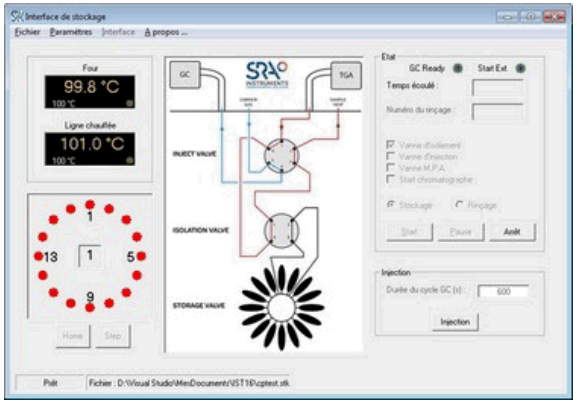
- Foams showed different decomposition profiles in PC and UC environments. The degree of variation depended on the foam.
- All foam samples showed a higher onset decomposition temperature in a PC environment
- In most measurements, PC samples exhibited a smaller overall wt% loss than UC samples due to char buildup inside the partially confined system.
- Foam environment affects the decomposition mechanisms. Foams decomposing in a PC environment undergo reactions with volatile species in the aluminum pan. In UC systems, the reactive species are swept away by the purge gas.

Mixed vs. Individual Foams

- Side reactions between the decomposing foam and off-gases in a PC environment are more evident for mixed foams.
- The mixed foam systems deviate from the weighted-average of the individual constituents.
  - REF320 and REF308 combined with EF-AR20 showed the most deviation
  - For EF-AR20 + REF320 less char is formed than if REF320 decomposes alone. This behavior is reversed for EF-AR20 + REF308 foam mixtures.
  - Possibly due to the slight difference in formulation between REF320 and REF308.
- More information is needed in order to determine the mechanism of decomposition for these systems
- Changes in the amounts of off-gasses from the decomposing foam can contribute to different side reactions between the foam and off-gases. Identifying gaseous species emitted during decomposition could elucidate mechanistic differences.
- Not all foams and/or combinations of foams will be present in every system however, this demonstrates that the mechanism of decomposition of combined foams varies from that of the single foam.

Future Work

- Analyze the off-gasses from individual and mixed foam samples using TGA-GC/MS
- Compare degradation products for individual and mixed foams observed with TGA-GC/MS to the modeled systems.



Software interface
http://www.sra-instruments.com/en

The IST-16 collects samples from TGA during the thermal transition according to a user-defined sequence. The samples are stored in up to 16 loops. GC analysis starts automatically after the storage completion.

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Thank You