AFRL-RX-TY-TR-2009-4577

NOVEL ELASTOMERIC CLOSED CELL FOAM – NONWOVEN FABRIC COMPOSITE MATERIAL (PHASE III)

Davis, Stephen C.

Armacell LLC
7600 Oakwood Street Extension
Mebane, NC 27302

OCTOBER 2008

Final Report for 7 June 2007 – 10 July 2008

DISTRIBUTION STATEMENT A: Approved for public release; distribution unlimited.

AIRBASE TECHNOLOGIES DIVISION
MATERIALS AND MANUFACTURING DIRECTORATE
AIR FORCE RESEARCH LABORATORY
AIR FORCE MATERIEL COMMAND
139 BARNES DRIVE, SUITE 2
TYNDALL AIR FORCE BASE, FL 32403-5323

NOTICE AND SIGNATURE PAGE

AFRL-RX-TY-TR-2009-4577 HAS BEEN REVIEWED AND IS APPROVED FOR PUBLICATION IN ACCORDANCE WITH ASSIGNED DISTRIBUTION STATEMENT.

____//signature//____________________
R. CRAIG MELLERSKI
Work Unit Manager

____//signature//____________________
SANDRA R. MEEKER, DR-IV
Chief, Deployed Base Systems Branch

____//signature//____________________
ALBERT N. RHODES, PhD
Acting Chief, Airbase Technologies Division

REPORT DOCUMENTATION PAGE	*Form Approved* *OMB No. 0704-0188*

1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE**	**3. DATES COVERED** *(From - To)*
28-OCT-2008	Final Technical Report	07-JUN-2007 -- 07-OCT-2009

4. TITLE AND SUBTITLE
Novel Elastomeric Closed Cell Foam - Nonwoven Fabric Composite Material (Phase III)

5a. CONTRACT NUMBER FA4819-07-D-0001

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER 62102F

6. AUTHOR(S)
Davis, Stephen C.

5d. PROJECT NUMBER 4347

5e. TASK NUMBER D2

5f. WORK UNIT NUMBER 4347D23A

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Armacell, LLC
7600 Oakwood Street
Mebane, NC 27302

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Air Force Research Laboratory
Materials and Manufacturing Directorate
Airbase Technologies Division
139 Barnes Drive, Suite 2
Tyndall Air Force Base, FL 32403-5323

10. SPONSOR/MONITOR'S ACRONYM(S) AFRL/RXQD

11. SPONSOR/MONITOR'S REPORT NUMBER(S) AFRL-RX-TY-TR-2009-4577

12. DISTRIBUTION/AVAILABILITY STATEMENT
A: Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES
Ref AFRL/RXQ Public Affairs Case # 09-195. Document contains color images.

14. ABSTRACT

The objective of the effort is to further develop novel closed cell foam-nonwoven fabric composites to commercial scale evaluation. Armacell tasks focused on foam optimization for commercial production, adhesive bonding of foam to fabric and commercial scale-up to produce fabric-like foam 0.10-0.12 inches thick in widths to 60-inches. Self extinguishing, insulating closed cell foam was successfully produced to 60 inch width and furnished in sufficient quantity to produce composites for the target shelter assembly prototypes. This work represents a portion of a broad team effort between AFRL, North Carolina State University, Freudenberg Nonwovens and Haartz Corporation.

15. SUBJECT TERMS
closed cell foam, intumescent polymers, non-volitile halon gas precursor polymers, shelter materials, insulating ability, flame resistance, light weight, intumescent nature

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
U	U	U	UU	25	R. Craig Mellerski
					19b. TELEPHONE NUMBER *(Include area code)*

Reset

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

1. SUMMARY

Fire is a major cause of loss of assets in the US military both during war and peacetime. Tent cities are particularly susceptible to fire due to close proximity of housing and other shelters used for facilities as well as prolonged exposure to extreme environments such as those in the Middle East. The development of lightweight shelters is critical to the Air Force mission of providing prompt logistics support for forward base operations. Structures should have characteristics that enable these shelters to be flexible, and in the case of containing ignitable fuels and other assets such as aircraft, these structures are required to be fire resistant. A key property in providing resistance to fires is the ability of a structure to withstand heat fluxes for a relative long period of time.

The objective of this program was to develop a light weight closed cell foam insulation to serve as a component (layer) of deployable shelters. This foam layer is to provide superior flame resistance to reduce the likelihood of fire propagation via flame spread or burn-through.

The work, which is a Phase III scale-up, focused on optimization of closed cell foam for cost/performance and resolve commercial scale-up issues.

Commercial scale-up issues consisted of:

- Maintaining fine cell structure
- Production of widths to 60 inches
- Skiving bulk foam rolls consistently to 0.10-0.12 inch thickness

Foam optimizations included

- Formula cost reduction to limit quantity of intumescent flame retardant
- Reduction of density to achieve 6 pounds per cubic foot (pcf) or less
- Low temperature flexibility -40°F or lower
- Vertical burn extinguished in less than 2 seconds

2. INTRODUCTION

2.1 Background

Fire is a major cause of loss of assets in the US military both during war and peacetime. Tent cities are particularly susceptible to fire due to close proximity of housing and other shelters used for facilities as well as prolonged exposure to extreme environments such as those in the Middle East. In 2003 at an installation in Kuwait 21 tents were consumed in a fire within 20 minutes before military and Kuwaiti firefighters could respond.[1] The fire was thought to have been caused by faulty wiring in an empty tent. Eight soldiers were treated for smoke inhalation and numerous military personnel were displaced as a result of the total destruction of the tent housing.

Air Force deployments require force projection capability that includes the airlift of a logistics infrastructure to have ready for combat assets within days of orders. The logistics footprint of such an aggressive deployment posture requires the use of materials and supplies that are lightweight, energy efficient and durable under extreme weather conditions. A part of this logistic plan is to deploy crew and aircraft shelters that are manufactured with materials, which are capable of protecting deployed forces and assets. Fire resistant materials are part of the military's strategy to increase readiness while better protecting our war fighters and our material assets. The development of lightweight shelters is critical to the Air Force mission of providing prompt logistics support for forward base operations. Structures should also have characteristics that enable these shelters to be flexible, and in the case of containing ignitable fuels and other assets such as aircraft, these structures are required to be fire resistant. A key property in providing resistance to fires is the ability of a structure to withstand heat fluxes for a relative long period of time.

This report describes the Phase III scale-up and optimization of closed cell elastomeric foam which incorporates intumescent, char forming flame retardants. Commercial scale production of 60 inch wide roll goods is described.

2.2 Objective

The objective of this project was to scale-up and assess commercial feasibility of fire retarded closed cell foam as a component of a lightweight foam-nonwoven fabric composite which displays superior fire barrier properties and thermal insulation.

Closed cell elastomeric foams have inherent thermal insulating properties due to the air trapped inside the small cells. These materials are extensively used in thermal insulation and water vapor barrier applications. The challenge in this work is to produce elastomeric closed cell foam which can act as a fire barrier and yet be light and flexible to enable formation of a flexible composite with a nonwoven fabric. This work, building upon prior development in Phase II[2], provides optimization of the closed cell foam and commercial scale production demonstration.

[1] http://www.arcent.army.mil/news/archive/2003_news/july/fire.asp

[2] Davis, Stephen, C., Kalberer, Jennifer, L. Fire Resistant Composite Closed Cell Foam and Nonwoven Textiles for Tents and Shelters. AFRL-ML-TY-TR-2006-4571

2.3 Approach

Optimization of flame retardant closed cell foam for shelter composites were performed using extensive laboratory scale production in parallel with production scale trials. Armacell laboratories at Mebane and production lines in Conover, North Carolina were utilized in the project.

3. METHODS

3.1 Foam Optimization (Task 1)

Prior development of fire retarded closed cell foam yielded attractive candidates for scale-up. Nitrile-butadiene rubber (NBR) and polyvinyl chloride (PVC) blends provided the most cost effective solutions. Two types of formulas were chosen for optimization. The first, designated here as AF07-A and AF07-B series provided excellent tensile and elongation properties and low temperature flexibility. However, this series displayed poor processing behavior. The second formula type, designated here as AF07-B2B series provided fine cell structure and excellent fire resistance. However, this series displayed poor low temperature flexibility (break at -20°F).

Variation of formula components to resolve the deficiencies were carried out. This included variation of:

1. Plasticizer
2. NBR type
3. Filler level and type
4. Antimony oxide and phosphate intumescent flame retardants

3.2 Scale-up to Commercial Production (Task 4)

Production issues were addressed for formulas based on AF07-B and AF07-B2B type foams. AF07-B type foams displayed marginal processing with material lacking green strength on milling resulting in stock crumbling. Also, excessive volatilization of plasticizer resulted in smoke production in the ovens. These issues were addressed via laboratory trials to optimize plasticizer type and rubber type.

AF07-B2B type foams were scaled to production with optimization of the final formula to enable production of low density foam with fine cell structure. Scale up optimization consisted of variation of production conditions during full scale trials.

3.3 Sample Synthesis

Closed cell elastomeric foam samples were synthesized in the R&D laboratories at Armacell LLC in Mebane. Production scale samples were produced at the Armacell facility in Conover, NC. Laboratory scale formulations were mixed in a 0.9 pound scale using a banbury mixer and a 6 inch heated rubber mill and extruded into flat strips 4 inches wide and ¼ inch thick with a rubber extruder. Sections of these strips were cured and expanded in an oven. Closed cell foam samples produced were cut to size for laboratory screening fire tests. Factory scale formulations were mixed in a 600 pound scale using a banbury mixer. Strips were continuously extruded using a 6 inch diameter extruder screw and cured/ expanded in a 300 foot, six zone oven.

3.4 Vertical Fire Test – Laboratory Screening

Fire testing was carried out in the laboratory using a vertical mounting bracket and a gas burner as shown in Figure 1. This test allowed each sample to be quickly evaluated to determine if the formulation met the flammability requirements. The sample was suspended 19 mm above the top of the burner. A flame 38 mm high was applied to the test sample for 12 seconds while the afterflame, afterglow and any melting or dripping were timed and noted. No afterflame or an afterflame lasting no greater than two seconds was considered a pass.

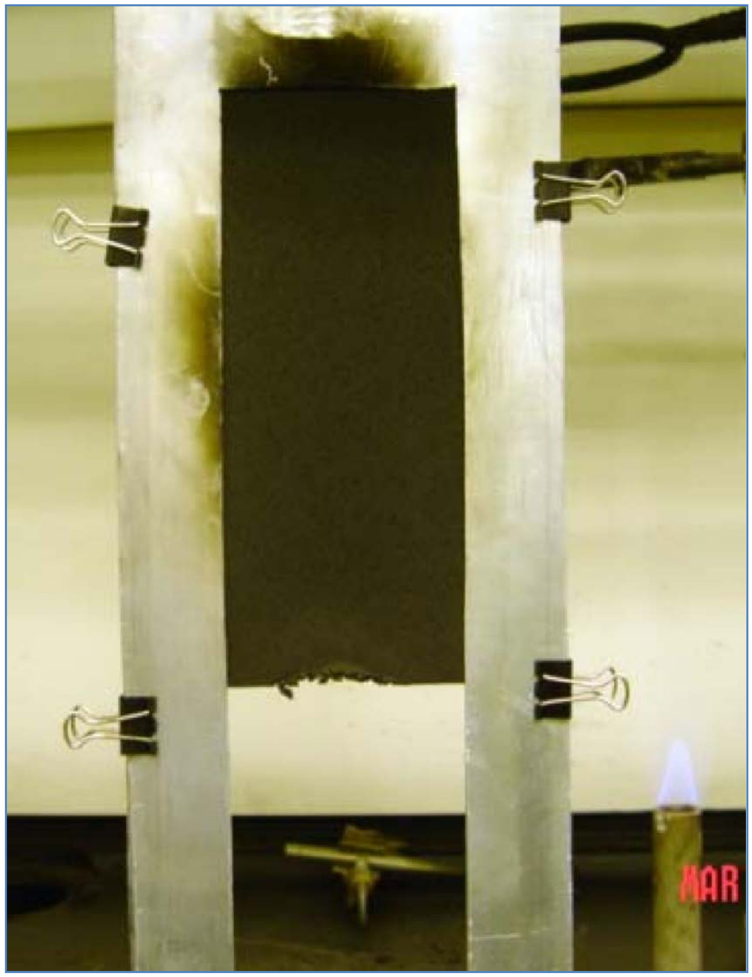

Figure 1 Laboratory Screening Vertical Fabric Test

3.5 Physical Property Testing

Tensile and Elongation were measured according to ASTM D412 (die A). Compression Deflection (CD at 25% compression), Compression Set (CS at 50% compression) and Density were measured according to ASTM D1056.

Limited Oxygen Index (LOI) was measured using a Stanton Redcroft FTA Flammability Unit.

Low temperature flexibility was measured according to ASTM D1056, using ¼ inch thick x 1 inch bent around a 1 inch diameter mandrel at temperature.

3.6 Thermal Insulation Property

Thermal conductivity of foam was measured using a Netzsch Heat Flow Meter (Model: Lambda 2000). Thermal conductivity was measured on 1 inch thick foam samples to ensure reliable data.

4. RESULTS AND DISCUSSION

4.1 Foam Optimization

Two formulas were optimized for commercial scale-up:

Type I: AF07-A and AF07-B

PVC/NBR with low temperature plasticizer and antimony oxide.

Type II: AF07-B2B

PVC/NBR with phosphate intumescent and aluminum endothermic flame retardants. The B2B foams contain half the PVC compared to B type foams with only 4 wt% chlorine and no antimony and less than 2 wt% phosphorous.

4.2 AF07-A and AF07-B Foams

Initial work focused on optimization of PVC/NBR foams which used antimony trioxide as a flame retardant component. These foams, represented by series AF07-A and AF07-B provided excellent low temperature flexibility, tensile and elongation. However, the foam had problems in processing such as poor milling behavior and volatilization of plasticizer. Significant efforts were expended to address these problems. Variables studied included:

1. Plasticizer type
2. Filler level and type
3. NBR type
4. Changes in the inorganic components (metal oxides)
5. Oven cure/expansion conditions

Tensile and elongation of AF07-A foams are shown graphically in Figure 2.

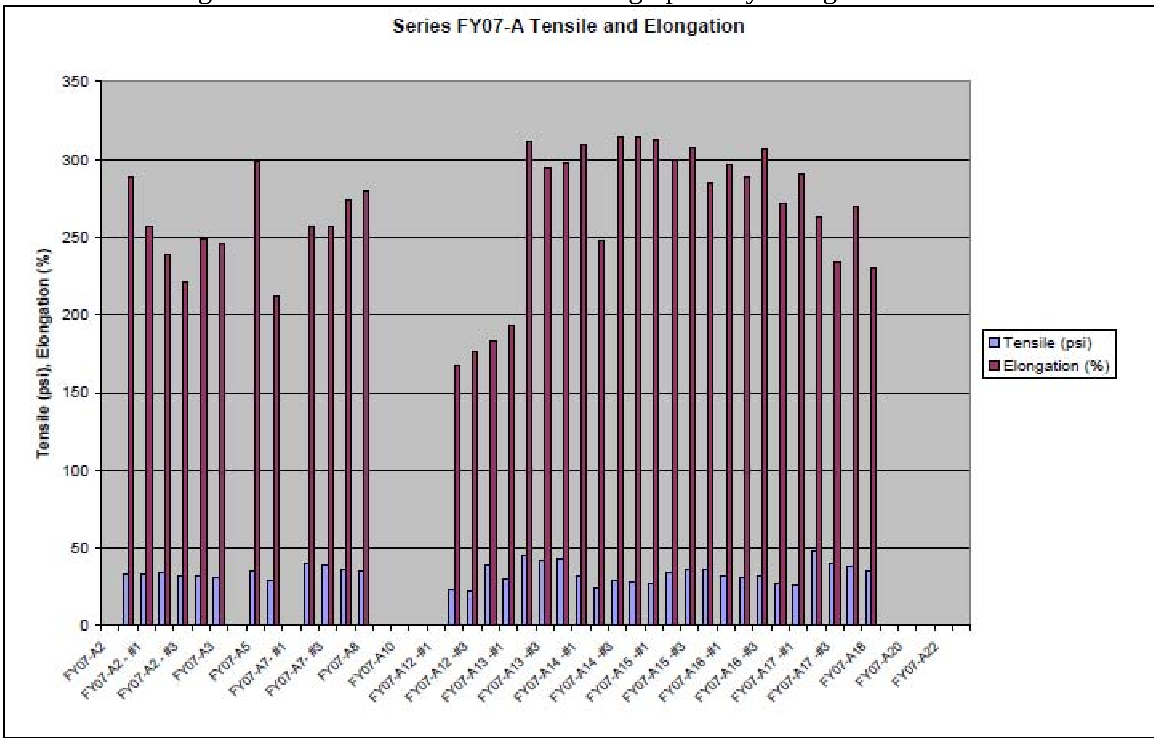

Figure 2 T&E of AF07-A foams (summary of optimization trials)

Tensile and elongation of AF07-B foams are shown graphically in Figure 3.

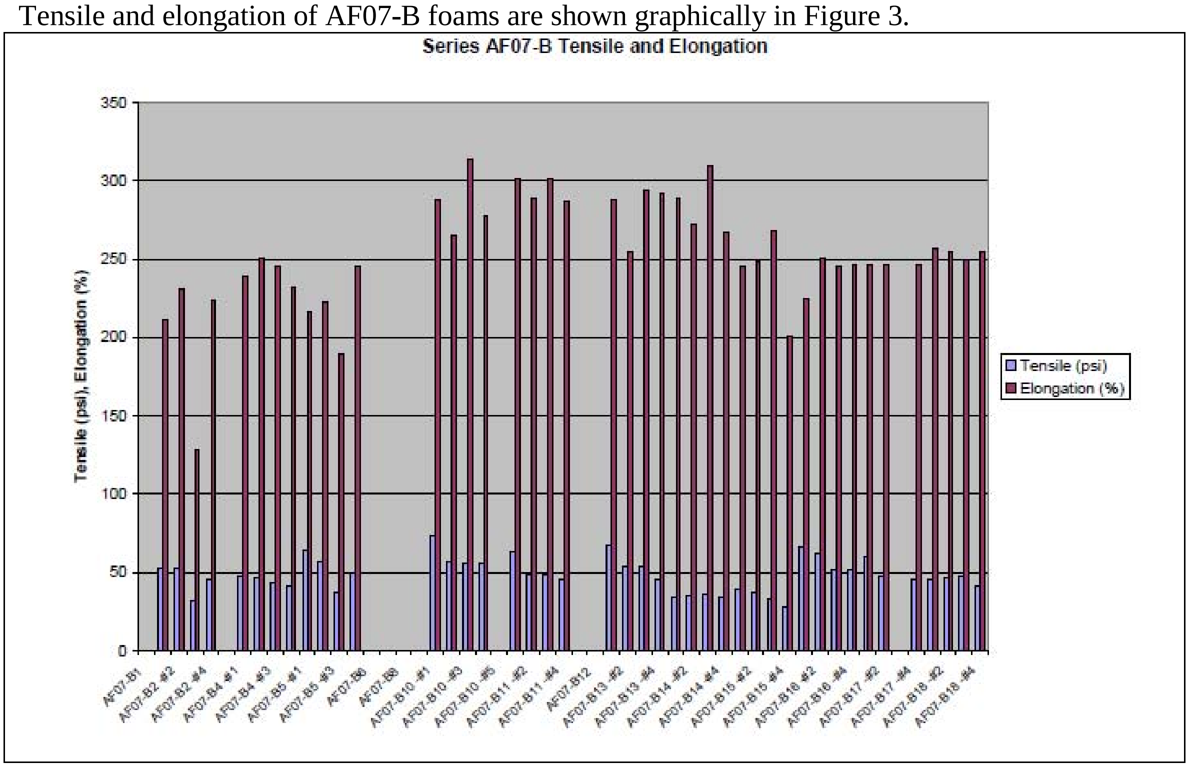

Figure 3 T&E of AF07-B foams (summary of optimization trials)

From the figures, it can be seen that the AF07-B type foams displayed higher tensile strength. This was accomplished by increasing the polymer content of the foam. From laboratory studies, processing was found to improve by using different types of NBR rubber.

The AF07 B type foam was scaled for commercial production trials resulting in the following properties:

Type A: Hot polymerized NBR

Type B: Cold polymerized NBR

Blend: 50/50 Type A and Type B

Table 1 Results of Production Trial for AF07-B foam

Type	Process	Density	Tensile	Elongation	LOI
"A"	poor	7.0	50	248	27.5
"B"	Best	6.7	51	258	27.0
"Blend"	better	7.0	48	251	27.3

These initial production trials resulted in marginal processing quality and excessive smoke generation.

Laboratory studies were performed to improve processing of the AF07-B type foam. Changes of PVC type improved processing further with a reduction in tensile strength and elongation when a lower molecular weight (K value) PVC was substituted. A summary of the effects on tensile and elongation properties is shown in Figure 4.

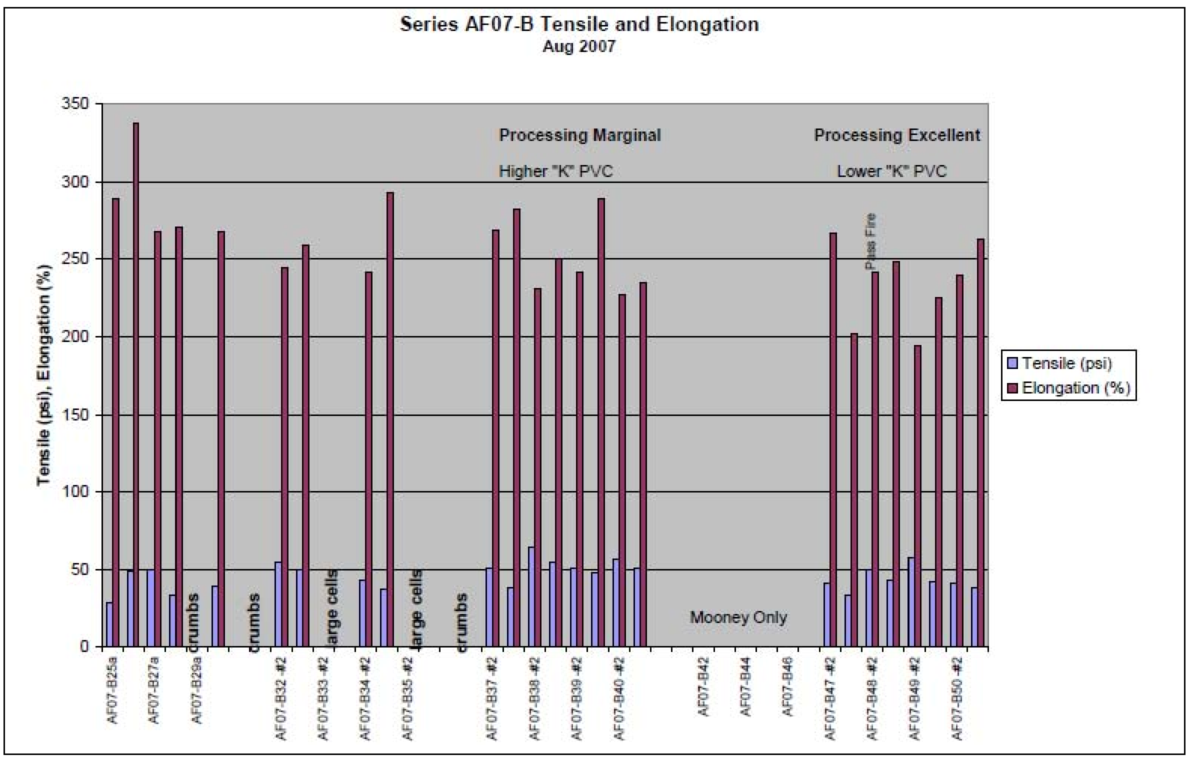

Figure 4 T&E Properties of AF07- foams using high and low K PVC

In addition the type of plasticizer was varied. Dioctyl adipate (DOA) was initially utilized in AF07-B foam to enhance low temperature flexibility. It was found that DOA was the contributor to smoke due to its high volatility. Therefore, diisononyl adipate (DINA) was used as a lower volatility replacement. Effects of DINA levels and PVC types and levels were studied during foam optimization. A 40/60 blend of low K and high K PVC with 60 parts DINA was optimal. Figure 5 shows the effects of PVC and DINA on tensile and elongation. Maxima are clearly seen. Figure 6 shows the effect on the limited oxygen index (LOI) for the AF07-B series optimization with DINA and PVC types. Increases in DINA result in decrease in LOI. All of the LOI were below 29.

The PVC level was increased to increase LOI (lower flammability) further. Figure 7 illustrated the change in properties as the PVC level increased. LOI increased to 32 and the elongation decreased, tensile increased and compression deflection (firmness) increased. The conclusion was high PVC levels were undesirable because the foam flexibility was lost.

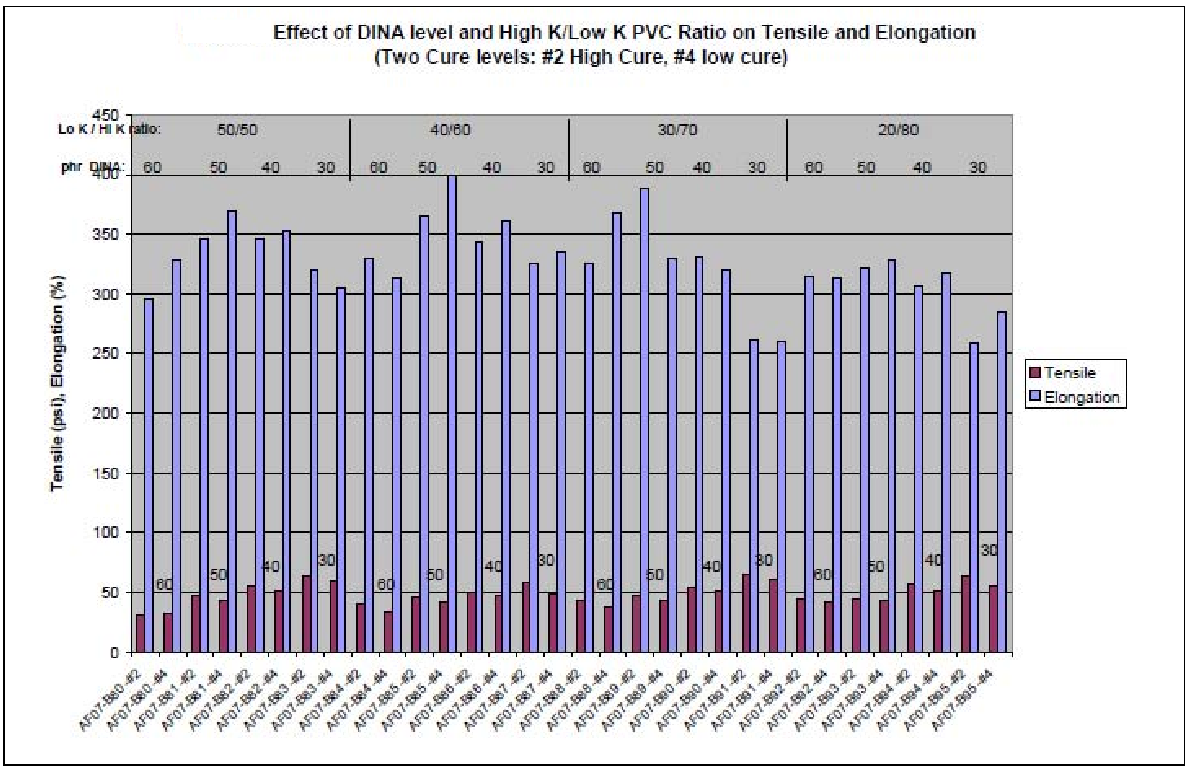

Figure 5 DINA Plasticizer and PVC effects on T&E for AF07-B foams

DINA at high levels also resulted in flashing during flame exposure. Efforts were made to minimize the DINA level. Results, shown in Table 2 show undesired stiffness results as the LOI was increased by decreasing the DINA.

Therefore, DINA plasticizer, used to generate low temperature flexibility, resulted in flashing during fire tests. The flashing was immediately self extinguishing upon flame removal. However there was concern that the flashing could be an issue in large scale testing. Efforts to minimize the DINA resulted in an undesirable increase in stiffness at low temperature. A compromise using phosphate ester plasticizers resulted in improved fire properties and less stiffening at -20°F. This “B” series formulation optimization was a partial success with flexibility still suffering.

Due to processing issues and the plasticizer flashing, further optimization of the AF07-B type foams was abandoned.

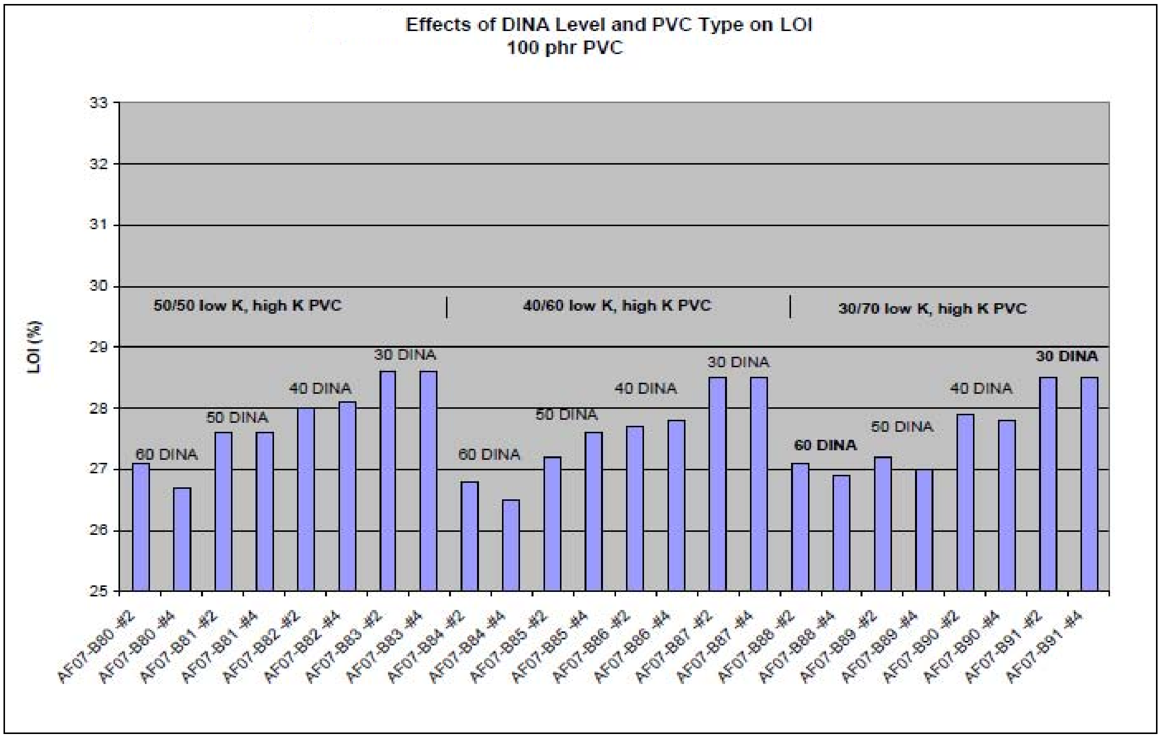

Figure 6 DINA Plasticizer and PVC effects on LOI for AF07-B foams

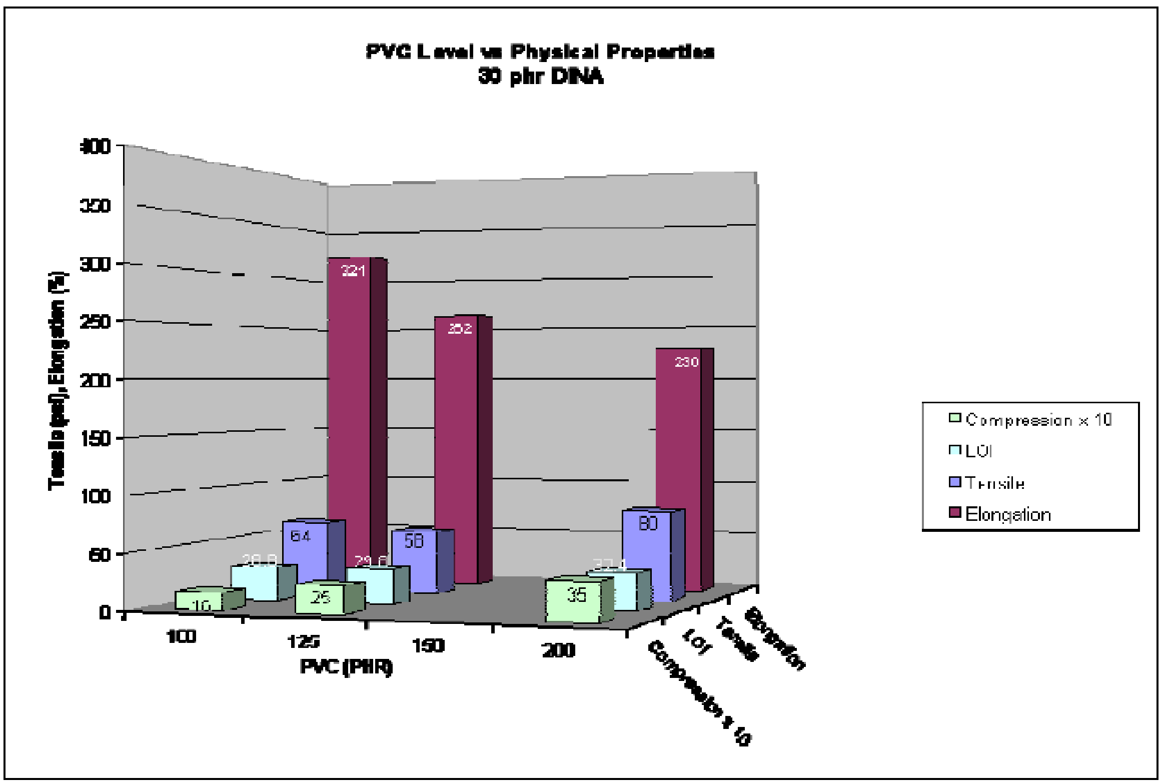

Figure 7 Effect of PVC level on properties of AF07-B foams

Table 2 Effect of DINA Plasticizer Level on LOI and flexibility (at -20°F)

Compound	Mooney (ML1+4)@270F	LOI (%)	Plasticizer PHR	Plasticizer	PVC	Stiffness @-20°F
B96	34	27.1	60	DINA	High K	↓ increases
B97	36	27.9	50	DINA	High K	
B98	43	28.4	40	DINA	High K	
B99	51	29.1	30	DINA	High K	

4.3 AF07-B2B Foams

The second foam type is based on the formula U9B2B reported previously. This foam produces very fine cells but becomes quite stiff at low temperature. This series of compounds was labeled AF07-B2B-. Parameters studied were:

1. Plasticizer type
2. NBR type
3. Filler level
4. Addition of antimony oxide as a synergist to the intumescent flame retardant
5. Hybrids of the FY07-B2B and FY07-B formulas

TGA analysis of AF07-A, -B and –B2B foams are shown in Figure 8. It can be seen that the B2B foams do not display the rapid weight loss in the 250-300°C range as does the type A or B foams. This sharp weight loss in the B type foams correlates to the observed surface flashing upon flame exposure. This is due to the volatile plasticizer and filler system. The intumescing B2B type foams display a more gradual weight loss upon heating and do not display the initial flashing of the flame.

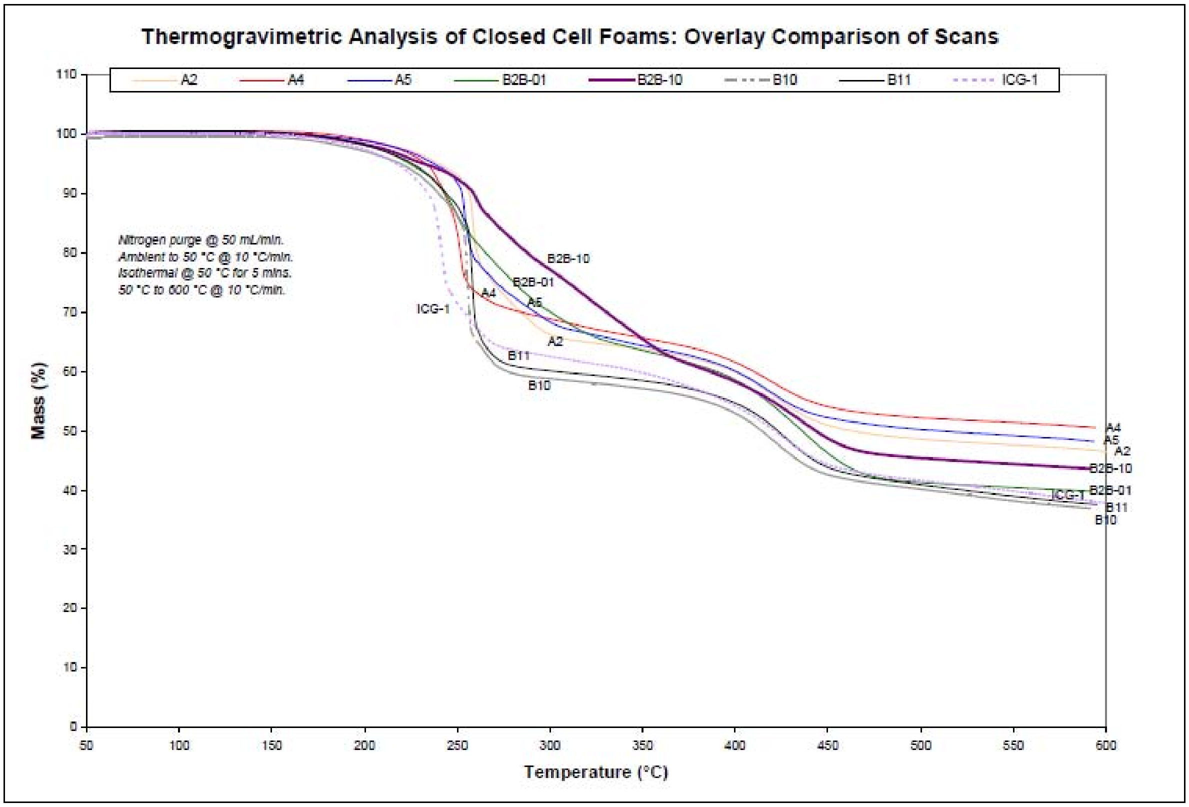

Figure 8 TGA Analysis of AF07-A, -B and –B2B type foams

Figure 9 shows tensile and elongation results for AF07-B2B type foams during optimization studies. It can be seen that the tensile strength and elongation are lower than the AF07-B type foams. Efforts were made to increase the tensile strength. In addition, the AF07-B2B type foams had an unpleasant odor when fresh or freshly skived. This odor dissipated with time (much like that of a vinyl shower curtain). Efforts were made to identify the primary contributor to the odor. Table 3 shows results which clearly point to the ammonium polyphosphate component of the intumescent flame retardant.

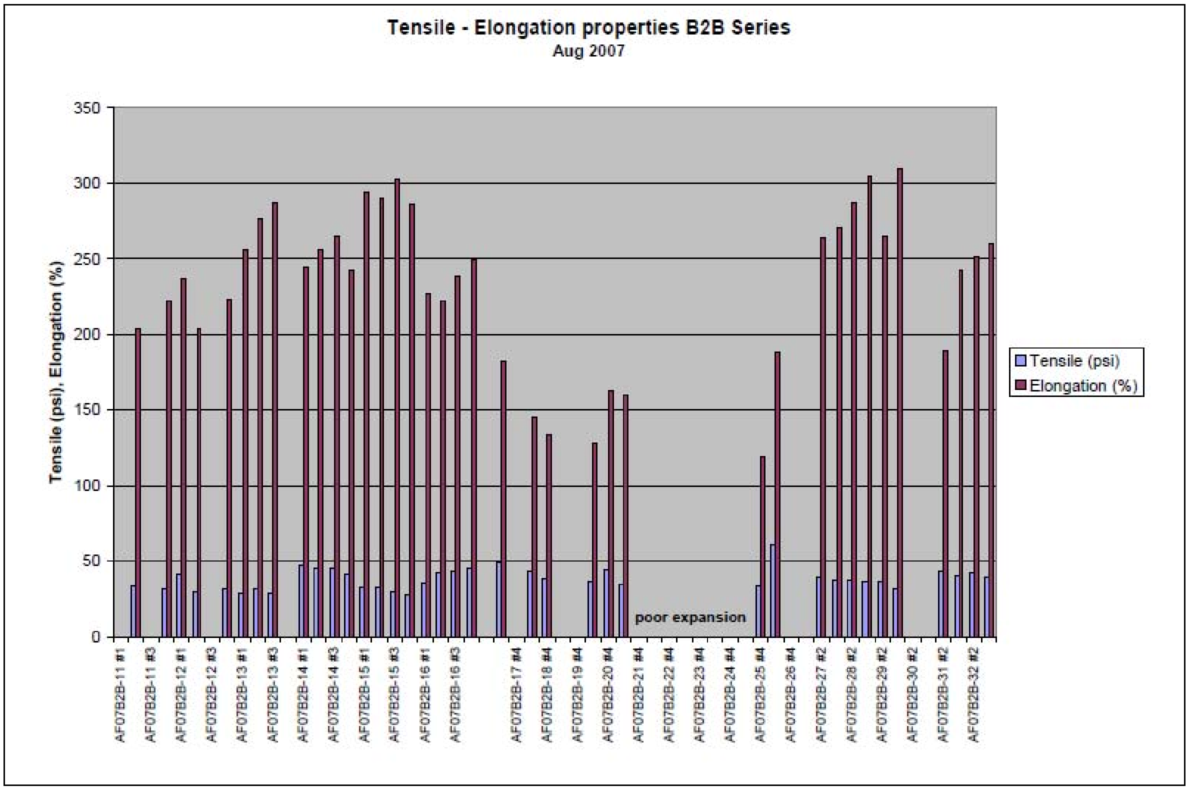

Figure 9 Physical properties of AF07-B2B type foams before optimization

Table 3 Effects of formulation on properties and odor in AF07-B2B foams

Trial	MB Mooney	FB MDR	Density pcf	Tensile psi	Elongation %	25% Comp Defl lbs	Formula focus	ODOR Change
AF07B2B-82 #2	**37**	3.88	**12.7**	56	160	2.5	**"B2B" cure**	0
AF07B2B-83 #2	**37**	2.85	**9.2**	43	152	1.9	**"B" Cure cure**	+
AF07B2B-84 #2	**37**	3.95	**11.6**	54	153	2	**"B" Cure cure, "B2B" levels**	+
AF07B2B-85 #2	**39**	3.90	**11.8**	49	148	2.1	**"B2B" cure, change accelerator type**	0
AF07B2B-86 #2	**42**	3.74	**12.8**	45	157	2.5	**"B2B" cure, change accelerator levels**	0
AF07B2B-87 #2	**32**	3.26	**9.3**	–	–	--	**no PNP**	++
AF07B2B-88 #2	**25**	3.10	**5.5**	16	110	--	**no PNP**	++

0 = same odor
+ = lower odor

The data of Table 3 also shows higher tensile values. However these resulted from higher than target density (11 pounds per cubic foot [pcf] vs 6 pcf target).

Efforts to optimize tensile/elongation properties with lower density resulted in slight improvements as shown in Table 4.

Table 4 Density and tensile/elongation results from formula optimization of AF07-B2B foams

Trial	MB Mooney	FB MDR	Density pcf	Tensile psi	Elongation %	25% Comp Defl lbs	LOI	Formula focus
AF07B2B-69 #2	42	2.68	6.8	24	123	1.8	31.6	Control for tensile/density optimizations
AF07B2B-89 #2	39	3.88	12.0	42	191	2.2	31.7	control high cure B2B
AF07B2B-90 #2	40	3.92	12.5	44	190	2.3	31.6	control high cure B
AF07B2B-91 #2	42	4.68	22.9	63	123	5.1	poor foam	Higher Cure B2B + higher Foaming agent
AF07B2B-92 #2	41	4.03	15.4	48	174	2.8	poor foam	High Cure B + higher Foaming agent
AF07B2B-93 #2	42	4.84	16.2	38	131	3.3	32.5	Lower iron oxide
AF07B2B-94 #2	35	3.51	9.8	34	173	1.8	30.3	No Iron Oxide
AF07B2B-95 #2	30	3.8	12.6	37	140	2.3	31.6	high cure B2B
AF07B2B-96 #2	38	2.98	8.6	27	129	1.9	30.8	Lower cure
AF07B2B-97 #2	38	3.23	7.4	22	127	1.4	31.7	Lower cure
AF07B2B-98 #2	32	3.16	10.7	39	152	1.7	30.1	higher DINA(60) std cure
AF07B2B-99 #2	28	2.26	7.4	27	134	1.5	30.2	higher DINA (60) lower DETU

These changes did not improve the tensile elongation at 6 pcf density. Foam density was the dominant influence.

Further optimization compared lower flammability phosphate ester plasticizer (S- 148) with DINA at different filler levels using 50/50 blends of aluminum trihydroxide and intumescent ammonium polyphosphate containing flame retardants. Results, which show improved tensile-elongation-density properties, are shown in Table 5.

Table 5 Properties of AF07-B2B foams using S-148 and DINA at various filler levels

Trial	Cells	MB Mooney	FB MDR	Density pcf	Tensile psi	Elongation %	LOI	Formula focus
AF07B2B-151 #2	fine	29	3.21	6.1	42	198	29.6	200 FR, 60 S-148
AF07B2B-153 #2	fine	37	4.38	6.1	33	159	27.6	200 FR, 50 S-148
AF07B2B-155 #2	fine	43	4.04	9	45	165	31.8	300 FR, 60 S-148
AF07B2B-157 #2	fine	53	5.97	9.7	–	–	–	300 FR, 50 S-148
AF07B2B-159 #2	fine	51	4.37	9.9	–	–	–	350 FR, 60 S-148
AF07B2B-161 #2	fine	59	5.44	11.9	–	–	–	350 FR, 50 S-148
AF07B2B-152 #2	fine	24	2.58	6.3	28	177	25.3	200 FR, 60 DINP
AF07B2B-154 #2	fine	30	3.68	8.1	26	189	25.8	200 FR, 50 DINP
AF07B2B-156 #2	fine	36	3.41	8.8	39	156	29.1	300 FR, 60 DINP
AF07B2B-158 #2	fine	45	4.21	9.5	–	–	–	300 FR, 50 DINP
AF07B2B-160 #2	fine	43	3.72	10.8	–	–	–	350 FR, 60 DINP
AF07B2B-162 #2	fine	51	4.83	14.2	–	–	–	350 FR, 50 DINP

To further lower the density, changes in polymer were made. Introduction a flexibilizing polymer, LCBR, resulted in a combination of fine cell foam with adequate tensile and elongation. This combined with intumescent flame retardant and ATH produced high LOI results (>30%). The data is summarized in Table 6.

Table 6 Properties of AF07-B2B foams using LCBR

AF07B2B-205 #2	med-fine	**5.8**	28	151	2.2	29.8	**PASED**	cf 211: 250FR, 56 S141, no LCBR
AF07B2B-206 #2	med-fine	**6.5**	25	135	2.1	31.4	**PASED**	cf 212: 300FR, 60 S141, no LCBR
AF07B2B-207 #2	med-fine	**5.5**	26	165	1.8	29.1	**PASED**	cf 213: 250FR, 50/10 S141/DINA, no LCBR
AF07B2B-208 #2	med-fine	**6.0**	19	127	1.7	31.5	**PASED**	cf 214: 300FR, 50/10 S141/DINA, no LCBR
AF07B2B-209 #2	med-fine	**6.4**	25	143	2.1	31.4	**PASED**	300FR, 40/20 S141/DINA
AF07B2B-210 #2	med-fine	**5.7**	22	136	1.7	29.3	**PASED**	250FR, 40/20 S141/DINA
AF07B2B-211 #2	fine	**5.7**	23	120	1.9	29.7	**PASED**	cf 205: 250FR, 56 S141, +10 LCBR, -10 NBR
AF07B2B-212 #2	fine	**6.7**	18	98	2	31.5	**PASED**	cf 206: 300FR, 60 S141, +10 LCBR, -10 NBR
AF07B2B-213 #2	fine	**5.9**	21	119	1.7	30.9	**PASED**	cf 207: 250FR, 50/10 S141/DINA, +10 LCBR, -10 NBR
AF07B2B-214 #2	fine	**6.8**	25	111	1.8	31.4	**PASED**	cf 208: 300FR, 50/10 S141/DINA, +10 LCBR, -10 NBR

Fine cell AF07-B2B foams below the target density (<6 pcf) were produced. These improvements were scaled to production trials.

Results of AF07-B2B foam produced at production scale (trials B2B-4) are shown in Table 7.

Table 7 Properties of B2B-4F (factory) foam

Two 75 ft rolls 54" x .68-.87 gauge

w/o skin	**Roll 3**	**Roll 4**
Density (pcf)	7.7	8.0
CD 25%	3.1	2.7
Tensile	46	46
Elongation	225	199
LOI	30.7	30.4
-20°F flex	Pass	Pass
Burn: NFPA 701 lab	Pass	Pass
wt (oz/yd2) at 0.10"	9.2	9.6
wt (gsm) at 0.10"	314	326
Compression Set with skin	30.6	30.9
Compression Set without skin	26.3	33.3

It can be seen that at a factory scale, density was above target but properties were excellent. During the factory trials, the foam had large (unacceptable) cell size. Changes in cure levels were made and cell size became acceptable as shown in Figure 10.

Figure 10 Comparison of cell size for AF07-B2B-4F before (left) and after cure adjustments

Additional laboratory optimization for tensile-elongation-density resulted in further improved properties as shown in Table 8.

Table 8 Properties of AF07-B2B foam further optimized for density and physical properties

Trial	Cells	MB Mooney	FB MDR	Density pcf	Tensile psi	Elongation %	25% Comp Defl lbs	LOI	Fire pass/fail	FR phr
AF07B2B-215 #2	fine	27	2.45	6.2	28	145	2.3	31.8	PASSED	300
AF07B2B-216 #2	med-fine	27	3.37	6.6	30	135	2.6	31.6		300
AF07B2B-217 #2	med-fine	27	1.98	6.2	28	128	2.1	31.0	PASSED	300
AF07B2B-218 #2	fine w/ blisters	31	2.04	7.7	28	130	2.0	31.4		300
AF07B2B-219 #2	fine w/ blisters	28	1.76	8.1	27	142	2.2	30.9		300
AF07B2B-220 #2	medium-fine	35	1.79	7.4	31	119	2.0	34.1	PASSED	350
AF07B2B-221 #2	medium-fine	35	1.79	7.3	30	118	2.3	33.9	PASSED	350
AF07B2B-222 #2	medium-fine	34	1.55	7.4	27	122	2.6	33.1		350
AF07B2B-223 #2	fine	20	2.25	9.4	32	125	3.0	33.4		350
AF07B2B-224 #2	fine	20	2.3	9.9	29	113	3.0	33.7		350
AF07B2B-223R #2	mix blisters	36	1.64	7.4	29	113	2.1	33.8		350
AF07B2B-224R #2	mix blisters	36	2.02	7.7	27	98	2.1	34.0		350
AF07B2B-225 #2	fine - good	37	1.98	8.1	26	114	2.8	34.1		350
AF07B2B-226 #2	fine - good	35	1.89	8.0	19	101	2.4	33.7		350
AF07B2B-227 #2	medium-fine	29	1.47	7.4	24	128	2.2	34.0		350
AF07B2B-228 #2	fine - good	28	1.38	7.2	23	121	2.1	33.6	PASSED	350
AF07B2B-229 #2	fine - good	31	1.60	7.5	22	124	2.3	33.8		350
AF07B2B-230 #2	fine - good	31	1.58	7.7	25	128	2.3	33.7		350
AF07B2B-231 #2	v fine -good	25	2.14	6.4	40	200	2.6	28.4	PASSED	200
AF07B2B-232 #2	fine - good	24	1.71	5.8	31	180	2.3	28.2		200
AF07B2B-233 #2	v fine -good	25	2.55	6.3	32	170	2.4	28.2		200
AF07B2B-234 #2	med-fine	25	2.12	5.7	28	184	2.3	28.0		200
AF07B2B-235 #2	v fine - dense	26	2.35	7.6	39	136	2.9	30.0		250
AF07B2B-236 #2	fine - good	26	1.78	6.9	36	150	3.0	30.1	PASSED	250
AF07B2B-237 #2	fine - good	26	2.39	7.5	41	165	3.0	29.4		250
AF07B2B-238 #2	fine - good	27	2.02	7.2	36	175	3.0	29.7		250
AF07B2B-239 #2	coarse	28	1.31	7.5	24	160	2.3	31.9		300
AF07B2B-240 #2	fine - dense	27	1.79	8.3	34	168	3.2	31.4		300
AF07B2B-241 #2	coarse	27	1.31	7.5	25	154	2.3	31.6		300

Very fine celled foams close to 6 pcf target density were produced. At 7 pcf density, foams were produced with very high LOI of 34%. A trade-off of LOI and density was seen. Evan at 28.4 LOI using this intumescent flame retardant, the foam passed the vertical flame test.

Low temperature flexibility was measured for a series of foams produced using a low temperature polymer (LTP) added as a minor component to AF07-B2B foams. Flexibility, as measured by bending a strip of foam around a 1 inch mandrel and observing cracking or no cracking, is indicated in Table 9.

Table 9 Use of LTP additive for property improvement of AF07-B2B foams

Trial	Cells	MB Mooney	Density pcf	Tensile psi	Elongation %	25% Comp Defl lbs	LOI	Fire pass/fail	Cold Flex -20F	Cold Flex -40F	Cold Flex -65F
AF07B2B-260 #2	medium	27	5.3	21	178	2.1	32.6	pass	pass	pass	fail
AF07B2B-261 #2	fine	33	7.2	32	154	2.5	34.3	pass	pass	pass	fail
AF07B2B-262 #2	fine	21	5.9	22	146	2.0	32.2	pass	pass	pass	fail
AF07B2B-263 #2	fine	25	6.2	19	149	2.2	33.5	pass	pass	pass	fail
AF07B2B-264 #2	v fine-clv	22	6.5	22	126	2.4	32.6	pass +1	pass	pass	fail
AF07B2B-265 #2	fine	27	6.7	26	116	2.5	33.9	pass	pass	pass	fail
AF07B2B-266 #2	fine - clv	19	5.8	23	156	2.2	31.8	pass +2	pass	pass	pass
AF07B2B-267 #2	fine	23	6.3	19	129	2.2	33.4	pass +1	pass	pass	fail
AF07B2B-268 #2	m-fine - clv	19	5.7	21	145	2.0	32.0	pass +1	pass	pass	pass
AF07B2B-269 #2	m-fine - ok	22	6.3	19	142	2.0	33.1	pass	pass	pass	fail
AF07B2B-270 #2	m-fine - ok	29	6.0	24	135	2.5	32.6	pass +1	pass	pass	fail
AF07B2B-271 #2	fine	34	6.1	21	127	1.8	34.1	pass	pass	pass	fail
AF07B2B-272 #2	coarse	21	5.2	23	158	1.9	32.1	pass +2	pass	pass	fail
AF07B2B-273 #2	coarse	25	6.0	23	148	1.9	32.8	pass	pass	pass	fail
AF07B2B-274 #2	medium	23	5.4	20	143	2.1	32.8	pass	pass	pass	fail
AF07B2B-275 #2	m-fine - ok	26	6.8	26	120	3.1	34.1	pass	pass	pass	fail
AF07B2B-276 #2	medium	16	5.2	22	172	2.0	31.8	pass	pass	pass	fail
AF07B2B-277 #2	medium	18	5.9	22	166	2.0	32.9	pass +1	pass	pass	fail
AF07B2B-278 #2	medium	17	5.3	23	170	2.2	30.8	pass	pass	pass	fail
AF07B2B-279 #2	medium	20	5.8	25	171	2.3	31.7	pass +2	pass	pass	pass
AF07B2B-280 #2	medium	17	5.4	20	163	1.9	30.6	pass	pass	pass	fail

It can be seen that this series of optimizations results in fine to medium size cells (all acceptable) with low density, high LOI and all passing cold crack at -40°F. Cracking at - 65°F is a limitation for these AF07-B2B foams.

Further work yielded finer cells and consistently low density foams in the laboratory as shown in Table 10. Adjustments of plasticizer revealed what level will be required to make the viscosity 30 Mooney in preparation for factory trials. Runs B2B-303-305 used high PNP (intumescent flame retardant) levels and B2B-306 – 308 used low PNP levels. Each required different levels of plasticizers.

Lowering of iron oxide (RIO) levels, to make the foams more rubber rich, had no effect on the viscosity as seen in B2B-310 – 312 and B2B-314 – 316. The RIO levels did not affect passing the flame test. The formulations are still border-line on cold crack at -65°F.

Table 10 Optimization of AF07-B2B foam for production trials

Trial	Cells	MB Mooney	FB MDR	Density pcf	Tensile psi	Elongation %	25% CD lbs	LOI	Fire pass/fail	Cold Flex -20F	Cold Flex -40F	Cold Flex -65F
AF07B2B-303 #2	med	22	2.2	**6.1**	27	132	2.9	29.6	**PASS**	**PASS**	**PASS**	FAIL
AF07B2B-304 #2	fine/med	25	2.67	**6.2**	35	127	3.6	30.0	**PASS**	**PASS**	**PASS**	FAIL
AF07B2B-305 #2	fine/med	30	3.21	**6.2**	36	121	4.1	29.8	**PASS**	**PASS**	**PASS**	FAIL
AF07B2B-306 #2	med	16	1.6	**5.8**	27	117	3.4	29.8	**PASS (+2)**	**PASS**	**PASS**	**PASS**
AF07B2B-307 #2	fine/med	22	2.45	**5.8**	33	96	4.1	31.0	**PASS**	**PASS**	**PASS**	**PASS**
AF07B2B-308 #2	fine	28	3.15	**5.7**	29	83	4.1	31.2	**PASS (+2)**	**PASS**	**PASS**	FAIL
AF07B2B-309 #2	fine/med	26	2.68	**5.8**	24	174	2.4	31.6	**PASS**	**PASS**	**PASS**	FAIL
AF07B2B-310 #2	fine	33	3.56	**6.3**	30	145	2.6	31.7	**PASS**	**PASS**	**PASS**	FAIL
AF07B2B-311 #2	fine	32	3.54	**5.7**	26	134	2.6	31.3	**PASS**	**PASS**	**PASS**	FAIL
AF07B2B-312 #2	fine/med	33	3.3	**5.6**	26	144	2.4	32.3	**PASS**	**PASS**	**PASS**	FAIL
AF07B2B-313 #2	med	23	2.11	**5.4**	27	115	3.2	32.8	**PASS**	**PASS**	**PASS**	FAIL
AF07B2B-314 #2	fine/med	29	2.89	**5.6**	32	103	3.2	32.4	**PASS (+1)**	**PASS**	**PASS**	FAIL
AF07B2B-315 #2	fine/med	27	2.88	**5.0**	27	109	3.1	31.8	**PASS**	**PASS**	**PASS**	FAIL
AF07B2B-316 #2	fine/med	29	2.78	**5.2**	29	105	3.3	32.8	**PASS**	**PASS**	**PASS**	FAIL

These formulas were scaled to production with the goal of producing fine cell foam in a width of 60 inches and a minimum of 1 inch thick.

Results of the factory trial (AF07-B2B-5 and -6 (factory)) are shown in Table 11.

Table 11 Properties of AF07-B2B foams produced at factory scale

	Rolls 60" Wide x 75 ft length x 1.1"-1.4" thickness					
	B2B-5		B2B-6			
	Roll 1	Roll 2	Roll 1	Roll 2	Roll 3	Target
Density (pcf)	5.6	6.0	**5.5**	5.4	6.3	**6.0**
CD 25%	2.1	3.0	**3.3**	3.2	4.1	
Tensile	21	31	**38**	34	44	
Elongation	247	199	**225**	202	250	
Compression Set%	44	36	**26**	24	17	
LOI	29.7	29.6	**31.1**	31.6	31.2	
-40°F flex	PASS	PASS	**PASS**	PASS	PASS	
Burn: NFPA 701 lab	PASS	PASS	**PASS**	PASS	PASS	
wt (oz/yd2) at 0.10"	6.8	7.2	**6.6**	6.5	7.6	**7.2**
wt (gsm) at 0.10"	229	243	**224**	220	257	**244**

Target densities were better than target and the foams had fine cells and excellent physical properties. The foams were produced to 60 inch width but later shrank to 56 inches wide. Target densities and weight of 0.10 inch thick foam are also shown in Table 11. All targets were met at production scale except the final width of 60 inches.

Further production trials using optimized AF07-B2B formulas were carried out which resulted in very fine cell foam with low density and excellent physical properties. Rolls were produced with an equilibrated width of 62 inches as shown in Table 12.

Table 12 Properties of final production optimization of AF07-B2B foams

	Rolls 62" Wide x 75 ft length x 1.1"-1.4" thickness			
	B2B-7			
	Roll 1	Roll 2	Roll 3	Target
Density (pcf)	4.3	4.5	5.0	**6.0**
CD 25%	3.6	3.5	3.4	
Tensile	27	36	42	
Elongation	139	193	167	
Compression Set%		18		
LOI	31.9	32.0	31.8	
-40°F flex	PASS	PASS	PASS	
Burn: NFPA 701 lab	PASS	PASS	PASS	
wt (oz/yd2) at 0.10"	5.2	5.4	6.0	**7.2**
wt (gsm) at 0.10"	175	183	204	**244**

It can be seen that the optimized production trial yielded very lightweight foam with fine cells and passed the vertical flame test.

4.4 Skiving Foams to Produce Foam "Fabric"

Rolls of foam were produced 75 feet long and 1-1.25 inches thick in the factory. These foams were then "skived" with a continuous vacuum bed skiver at our customer, CGR Products in Greensboro, North Carolina. The foams were initially leveled by cutting off thin layers including the skin. Then the foams were skived, slice by slice the length of the foam and rolled. Precision of the skiving operation was acceptable to maintain 0.10-0.12 inch gauge control. The rolls of foam "fabric" were sent to Haartz Corporation for laminating to coated non-woven fabric.

4.5 Adhesion of Foam to Nonwoven Fabric

Several adhesive systems were evaluated for bonding with the nonwoven fabric to form a composite structure. The best system found was a flame retarded pressure sensitive adhesive (PSA) manufactured by 3M Company. The product, 9372W, was a 2 mil acrylic PSA. Rolls were sent to Haartz Corporation for testing. Haartz found the lamination with this adhesive to not be satisfactory for their process. However, a suitable adhesive was found by Haartz and used in commercial scale composite formation.

5. CONCLUSIONS

Optimization and scale-up of lightweight, flexible, flame retarded closed cell foam was successful after extensive optimization studies. The production of the foam in full production scale at a width of 60-inches was demonstrated. Optimization of production conditions yielded foams with low density (4.5 pcf or 5.2 ounces at 0.10-inch thickness), which passed the vertical flame test. Several hundred yards of 5.4 pcf density foam were delivered to Haartz Corporation to produce a foam-nonwoven fabric composite in sufficient quantities to fabricate a full scale shelter assembly prototype. The closed cell foams produced are water proof, have thermal insulating properties, self extinguish upon flame exposure and form a char barrier to reduce spread of fire. Upon flame exposure the foams do not melt and provide a superior barrier to flame spread than conventional materials.

6. RECOMMENDATIONS

The use of lightweight, flexible, flame retarded closed cell foams as components of shelter systems should be fully assessed to reduce flame spread in a fire event and reduce heat loss due to the thermal insulation properties of closed cell foam.

7. REFERENCES

1. ASTM D412, 2006. Standard Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension.

2. ASTM D1056, 2007. Standard Specification for Flexible Cellular Materials—Sponge or Expanded Rubber.