

Accelerated Aging Of Moulded Foam

Selection of Parameters for a Value-Added Test

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INTRODUCTION

The Flexible Moulded Foam Industry Panel is made up of experienced polyurethane foam scientists, technologists and engineers. They represent major chemical suppliers, foam moulders, seat assemblers and three OEMs, Chrysler, Ford and General Motors.

Initially this panel polled its members and asked them to review the range of physical properties called for in many foam specifications currently in use in North America and in many cases around the world. Members were asked to discuss our request with other informed persons in their respective companies and to bring all of the responses to the panel for collation and ranking. As far as the accelerated aging is concerned, the choice of physical properties was unanimous, namely:

- I. Constant Force Pounding, ASTM D3574, test I3
- II. Wet Compression Set, ASTM D3574, test D,L
- III. Humid Aged Compression Force Deflection Change, ASTM D3574 test, C, J

Constant Force Pounding (CFP) was chosen as the accelerated test has been shown to be correlated to actual in-vehicle cushioning performance.⁽¹⁾ Recently our panel reviewed this pounding test and a number of other accelerated tests used to ensure long term seating performance.⁽²⁾

Wet Compression Set was introduced to the polyurethane industry in the 1970s⁽⁴⁾ and it has been shown to be an excellent compression set test that indicates long term seating behaviour. It has been shown that 50% deflection Wet Set values are correlatable with the 40% deflection IFD loss after CFP⁽³⁾. Wet Sets are performed on small core specimens, 50 x 50 x 25 mm by compressing them to half their original thickness and placing the compressed specimens in an environmental chamber set at 50 ± 2°C, 95 ± 5% RH for 22 hours. After removal from the compression plates, specimens are allowed to recover for 30 minutes (or longer times if so specified by the customer), and the resultant thickness is measured. The Wet Set is the loss in thickness due to compression/aging.

Changes in foam hardness in an accelerated laboratory test have traditionally been measured using the Humid Aged Compression Force (formerly Load) Deflection test, HACFD. These tests call for core specimens, usually 50 x 50 x 25 mm to be humid aged for 3 hours in an autoclave held at 105 ± 3°C or 5 hours at 120 ± 5°C. To achieve and maintain these conditions, the specimens are held under pressure, 21 and 35 kPa respectively. Following autoclaving, specimens are placed in an air circulating oven for 3 hours at 100 ± 5°C for each 25 mm thickness of the specimen. Before CFD testing, specimens are allowed to recondition for at least 2 hours in a controlled environment at 23 ± 2°C, 50 ± 5% RH. CFD may be measured at various deflections, e.g., 25, 40, 50, 65% but the most common deflection is 50%. The percentage CFD change due to humid aging is calculated and reported as HACFD change.

Although our panel had selected HACFD as an acceptable aging treatment, it was considered to be suspect since the concept of autoclave aging had been rejected by us opposite compression sets. We had selected and clearly demonstrated the superiority

of Wet Set and since it correlates with hardness loss in CFP, it has been incorporated into our Industry Panel Moulded Foam specification. We have been unable to find any correlation between any humid aged foam property and in-vehicle cushioning performance.

A review of the polyurethane literature indicates that a number of scientists have considered the humid aging treatment. Saotome et al.,⁽⁴⁾ introduced environmental aging as an alternative to humid aging and this procedure was adopted by all of the Asian OEMs for compression set treatment. At least one European OEM also adopted this aging treatment while others continued to call for humid aging. All North American OEMs specified humid aging for both compression set and CFD change measurements.

Several papers have considered what actually happens to the polyurethane structure during humid aging. Steam aging in an autoclave, especially at 120°C has been shown to actually change the polymer structure^(5,6). Both of these references have shown that the changes that take place during autoclaving will not occur in a vehicle, even at elevated temperatures and humidities such as found in tropical climates. Thus humid aging as an accelerated treatment has long been considered as suspect since it has been clearly shown to modify polymer structure, a phenomenon that does not occur during vehicle usage. Since the original intent of aging under elevated humidity conditions was to determine the hydrolysis resistance of flexible foam products⁽⁷⁾ our panel decided to investigate alternative aging treatments.

Long term hardness behaviour of foam cushioning is very important to ensure that seating remains stable during the lifetime of a vehicle. Accelerated testing that will predict long term hardness behaviour or to ensure that production parts perform consistently is very important.

1. Aging Treatments

As mentioned in the introduction, all Asian OEMs and a few European OEMs age foam at $50 \pm 2^\circ\text{C}$, 95% RH represent tropical conditions. However, even the Asian OEMs have remained with humid aging for treating CFD change specimens. The reason for this is not known at this time but we suspect that they inherited this treatment from North America and Europe and did not consider changing the environmental conditioning.

In this paper, we have considered and performed a number of environmental aging treatments. Obviously the initial condition used was the one favoured for Wet Compression Set, $50^\circ\text{C}/95\%\text{RH}$ with an aging time of 22 hours. Longer aging times under these conditions have also been examined to determine if 22 hours was long enough to obtain reliable change.

We have also measured CFD change after other humidity treatments, $38^\circ\text{C}/95\%\text{RH}$, $100^\circ\text{C}/95\%\text{RH}$ and $80^\circ/75\%\text{RH}$. This last condition was examined since it appears in a recently issued General Motors specification GMW 14124. In this specification the bond strengths and hydrolytic stability of laminated textile materials are measured after these materials have been conditioned for 400 hours (16.7 days). Since this aging duration was deemed to be excessive and would tie up environmental chambers under these conditions for such long periods, we have looked at shorter periods as well to find out if materials equilibrate earlier, i.e., exhibit CFD changes at shorter times similar to those found for longer aging periods.

We have also aged samples at 100°C in an air-circulating oven for up to 168 hours to compare our results with specimens aged under tropical conditions. It has been suggested⁽⁸⁾ that aging foam at 100°C (essentially at zero percent relative humidity) may be a useful treatment for accelerated aging and the results may be correlated with in-vehicle performance.

2. Materials Evaluated

Various grades of moulded foam have been examined in these evaluations. The foams were mostly randomly selected since they were available and had been tested for other physicals. All materials were moulded as 380 x 380 x100 mm blocks using production-ready mixing/metering machines and production-style moulds. All foam blocks were aged for at least 7 days prior to cutting and testing. All testing was performed in temperature and humidity controlled, in certified laboratories.⁽⁹⁾

As the investigation progressed, specific foam types were manufactured. A density range of 32 to 48 kg/m³ blocks were produced. Also since we were aiming our Industry Panel specification as a potential worldwide document, we decided to examine a range of isocyanate chemistries i.e., 100% TDI; 80% TDI/20% MDI; and 20% TDI/80% MDI, over a density range 40 to 48 kg/m³.

3. Accelerated Aging Trials

4.1 Aging at 50°C, 95% RH

Specimens were aged in a chamber for various periods from as short as 4 to as long as 96 hours. Three foam grades, 32, 38 and 44 kg/m³ were evaluated for Wet CFD change and Wet Set simultaneously. Hardness and set measurements were made after 30 minutes reconditioning and the results are shown in Table 1.

Table 1								
Foam Density kg/m ³	Foam Property	Aging Time, hours						
		4	8	16	22	35	48	96
32	Wet CFD change, %	-8	-9	-15	-19	-7	-4	-3
	Wet Set, %	15	18	20	21	24	27	26
38	Wet CFD change, %	-9	-8	-11	-8	-14	-3	-2
	Wet Set, %	13	15	17	19	24	26	24
44	Wet CFD change, %	-9	-14	-8	-11	-5	-5	-2
	Wet Set, %	11	13	15	18	18	24	24

In addition we have measured Wet Set values after 60 minutes recovery and found that the set values only change between zero and 4% but most specimens show 1-2% improvement.

The Wet CFD change data shown in Table 1 leads us to believe that a conditioning time in the humidity chamber of less than 22 hours is too short and that times in excess of 22 hours sometimes resulted in lower change values. These CFD change and Wet Set values are shown in Figure 1. The lower CFD change values found at periods longer than 22 hours may be due to these foams obtaining additional cure during aging treatment, firming-up and hence lower change values result. Hence 22 hours under these conditions may be sufficient and has the added advantage that Wet Sets can be done at the same time. It should be noted that Wet Sets continue to increase with periods longer than 22 hours.

To achieve the best Wet conditioning methodology, we measured the water pick-up of the above 2 foams plus a higher density one. The results appear in Table 2. Some water pick-up inconsistency is apparent, so specimens were exposed in the chamber either uncovered or covered with a sheet of cardboard to prevent any water droplets from the roof of the chamber falling onto the exposed CFD specimens.

Table 2		
Water Pickup, %		
Foam density, kg/m³	Conditioned 22 hours	Conditioned 48 hours
32	+4.61	+0.54
38	+0.05	+4.00
44	+0.01	+0.24
60	+0.07	+0.64

The differences between water pickup is shown in Table 3.

Table 3		
Foam density, kg/m³	Water Pickup, %	
	Uncovered foam	Covered foam
32	+1.3	+1.12
38	+1.1	+0.95
44	+2.4	+0.92
60	+1.1	+0.96

The covered specimens pick-up less moisture and the amount of pick-up from specimen-to-specimen is more consistent. Therefore, covering of specimens in a humidity chamber is recommended for Wet CFD change conditioning.

A second set of foams were manufactured and tested. These foams extended the density range from 26 to 53 kg/m³ and the following results were obtained for specimens aged for 22 hours and reconditioned for 30 minutes.

Table 4		
Foam density, kg/m³	Wet CFD Change, %	Wet Set, %
26	-0.2	21
26	-0.2	27
33	-1.9	19
34	-1.5	21
43	-2.2	13
43	-2.9	15
53	-2.1	9
52	-1.9	10

At each density, duplicate blocks were evaluated. Essentially the same Wet CFD change and Wet Set values were obtained for these duplicate blocks.

A third set of foams manufactured in Europe were evaluated in two Panel member's laboratories, Table 5.

Table 5				Constant Force Pounding	
Foam Test Location	Foam density, kg/m ³	Wet CFD change, %	Wet Set %	Height change, %	40% IFD change, %
P3T	66	-4	7	4	29
BASF	66	-3	6	1	15
P3T	57	-5	9	5	33
BASF	58	-3	9	-1	22

The agreement between the two laboratories for Wet CFD change and Wet Set is good. In addition, samples were pounded as per ASTM D3574, test I3. The agreement between labs is not so good with the BASF lab obtaining lower height and IFD losses than the P3T lab. This may be partially explained by the fact that CFP (Constant Force Pounding) samples were cut from actual moulded parts, not blocks. When these two laboratories test surrogate moulded blocks, excellent agreement is found. That is the reason the Moulded Foam Industry Panel advocates the use of moulded blocks in this accelerated test.

4.2 Aging at Various Temperatures and Relative Humidities

Two foam grades, 40 and 48 kg/m³ densities have been evaluated using various conditions including autoclaving.

Table 6			Wet CFD Change, %	
Conditioning Time, hours	Temp °C	% RH	Foam density, kg/m ³	
			40	48
24	50	95	-0.2	-1.0
	70	95	-0.5	-1.5
	90	95	+4.0	+0.8
96	80	75	+8	+8
168	80	75	+10	+8
400	80	75	+10	+8
3	105	100	+14	+9
5	120	100	+18	+21

Conditioning at 95% RH for 24 hours gives Wet CFD changes that change from negative to positive as the accompanying temperature is increased from 50 through 70 to 90°C. If the chamber conditions are held at 80°C/75% RH, as called for GMW 15414, cycle Q, for various lengths of time from 96 to 400 hours, the Wet CFD changes resulting are almost the same for these two foam grades. In contrast, autoclaving these foams at 105 or 120°C results in worse CFD changes. Since the CFD results are positive this indicates hardness increase due to further cure in the autoclave even for short times and/or in the drying oven at 100°C. It should be noted that a temperature of 120°C was used formerly as a postcure temperature for 30 minutes to ensure the moulded foam would not take a packing set during transportation.

4.3 Aged at 80°C and 75% RH

These are the GMW 15414, Cycle Q conditions and we have used them as an alternative to either autoclaving or wet conditioning, 50°C/95% RH. We tested the same foams that had been evaluated in section 4.1. The above conditions can be achieved either by using an environmental chamber or placing the specimens in a container with some water but specimens must not sit in the water. For convenience, we used an autoclave heated to 80°C. The pressure relief valve was closed and released after a short time to see if any pressure was generated within but no pressure was found. Specimens were treated for various lengths of time up to 400 hours. Three specimens were extracted and testing after each period, reconditioned and CFD tested. Specimens were retained and retested after 24 or 96 hours to establish any hardness recovery. The data obtained is found in Table 7 and plotted in Figure 2. It can be clearly seen that initially all of these foams lose hardness after aging for 24, 48 and 96 hours. After longer periods 144-400 hours, hardness changes (decreases) are less and for the 32 and 40 kg/m³ grades, hardness increases above the as-produced CFDs are found. The 48 kg/m³ foam only shows a modest CFD change recovery as the aging time is increased beyond 96 hours. Allowing the specimens to recondition in the laboratory for 24 or 96 hours, decreases the Wet CFD changes for the 24 to 144 hours aging periods but the CFD change values at 400 hour aging and reconditioning are even more positive than the non-recovered values, e.g., 32 kg/m³ foam, Wet CFD change = +27%, CFD change after recover = +35%.

Table 7				
Foam Density, kg/m³	Aging Time, hours	Initial Hardness, kPa	Wet CFD change, %	CFD change after Recovery, %
32	24	5.4	-7	-2
40		7.8	-8	-2
48		9.1	-14	-3
32	48	5.8	-9	-3
40		7.7	-9	-5
48		8.9	-11	-7
32	96	5.6	-8	-4
40		8.2	-12	-8
48		8.8	-12	-8
32	144	6.1	-6	-2
40		7.9	-6	-1
48		8.8	-11	-8
32	400	5.6	+27	+35
40		7.4	+11	+22
48		8.8	-9	+1

To try and determine the cause of this Wet CFD change behaviour we have measured some other foam properties. Before cutting the CFD specimens, we measured foam hardness (IFD) and hysteresis loss. Ball rebound (resiliency) samples were then obtained and measurements taken as-made and after various periods as shown in Table 8.

Table 8														
Aging Time, hours														
Foam Density, kg/m ³	0		50		100		200		300		340*		740*	
	BR	HL	BR	HL	BR	HL	BR	HL	BR	HL	BR	HL	BR	HL
40	56	24	53	28	57	29	53	28	54	28	47	35	50	32
48	55	25	54	26	53	27	53	29	51	29	51	31	49	33

BR = Ball Rebound, %
HL = Hysteresis Loss, %

* Samples aged these times by mistake

The ball rebound values (resiliency) decrease slightly with longer aging time, i.e., from 55-56% to 49-50% from initial to 740 hours aging.

The hysteresis loss values exhibit a steady increase with longer aging periods. This may indicate a worsening ability of the foam to recover may partially explain the changes in Wet CFD on aging.

In addition, Constant Force Pounding has been performed on two foam grades, 40 and 48 kg/m³ as-produced and after several aging periods, 48 through 400 hours. The resultant data is plotted in Figure 3. For both foam densities there is a decrease in IFD loss after pounding after aging at 48 and 96 hours, i.e., from 18-20% loss at 0 hours to about 10% loss after 48-96 hours aging. A slight increase in IFD loss occurs after 168 hours aging (11-12%) but the loss values are still less than found for as-produced foam. After 400 hours aging, the IFD losses exceed the initial loss values, i.e., 28-30% compared with 18-20% initially.

Thus we have shown that 80°C, 75% RH aging condition affects several foam properties, i.e., hysteresis loss, resiliency and Wet CFD change values. In order to try and discover what physiochemical changes in the foam structure/chemistry occur on aging under these conditions, a foam swelling study was done. Swelling studies can be used to obtain a measure of the crosslink density of polyurethane. The more crosslinked the polymer, the less it will swell when immersed in a solvent. Specimen dimensions before and after immersion in hexanes were taken and the swollen volumes calculated, as shown in Table 9.

Table 9				
Foam density, kg/m ³	Aging time, hours	Original volume, cm ³	Swollen volume, cm ³	Percentage Swell, %
40	0	70.8	90.2	27.4
	100	74.2	90.6	22.1
	340	74.3	93.6	25.8
	740	71.9	91.9	27.8
48	0	69.8	85.5	22.5
	100	74.1	91.4	23.3
	200	72.9	92.4	26.8
	300	72.9	91.4	25.4

The 40 kg/m³ foam does not show any systematic degree of swell with increasing aging times but the 48 kg/m³ foam appears to exhibit increasing degrees of swell with longer aging times. This may indicate some reduction in crosslink density with increased conditioning time. If this is correct, the lower degrees of crosslink may explain the reduction in foam hardness found with longer aging, times for both CFD and the IFD changes that occur after pounding.

A second technique was used to investigate polymer change. Dynamic Mechanical Analysis (DMA) measures foam modulus (storage and loss) and tan delta (tg) as a function of increasing temperature. This technique can detect polymer modulus changes and chemical/physical changes if they occur on heating. The DMA traces found for these aged foams are very weak and thus uninformative. The storage modulus at the aging temperature of 80°C was found to vary quite widely and no trends could be found versus aging time. Even the tg peaks which normally show the transition due to the SAN particles in the polymer dispersion softening were very weak and scattered between 107 and 138°C. Thus DMA did not help as to elucidate what physiochemical changes may occur during aging.

4.4 Comparison of foams preconditioned/as-received before aging at 80°C, 75% RH

At least one OEM specification calls for foam to be preconditioned in an oven before any testing commences. For instance preconditioning for a minimum of 16 hours at 80°C before humidity treatments may be required. The exact reason for this call-out is unknown at this time but it may be just another of these requirements “handed-down” through time and the original reason has been forgotten. This treatment has been included to ascertain if it has an influence on Wet CFD changes. The same three grades of foam (32, 40 and 48 kg/m³) were evaluated. The data obtained is summarized in Table 10. From this data we find:

1. Pretreatment, 16 hours @ 80°C reduces the percentage Wet CFD change for all densities and all aging times.
2. The maximum percent reduction in Wet CFD changes occur between 48 and 96 hours.
3. The 32 and 40 kg/m³ foams exhibit positive CFD changes after 400 hours aging irrespective of preconditioning or not prior to Wet aging.
4. The 48 kg/m³ foam still has a negative Wet CFD change (lower than its initial value) after 400 hours aging.
5. Data from laboratory #2 (using slightly different aging equipment) show similar results to laboratory #1.
6. The second laboratory also shows that the Wet CFD changes of preconditioned and as-received foams are approximately the same.
7. Wet CFD changes decrease after 24 hours reconditioning in the laboratory and further decreases are found after 14 days recovery.
8. 40 and 48 kg/m³ foams aged for 400 hours at 80°C/75% RH exhibit even more positive Wet CFD changes after 24 hours, 14 days and 5 weeks reconditioning. The 48 kg/m³ foams, which have *negative* Wet CFD changes after aging exhibit *positive* changes after 14 days and 5 weeks recovery.

Some of the data in Table 10 is plotted in Figure 4 and the trends are essentially similar to the data in Figure 1. The effects of pre-curing can be seen and the same trends found by the two laboratories that evaluated these foams can be observed.

Table 10

			Wet CFD Change %	Wet CFD Change %	Wet CFD Change %	Wet CFD Change %
Foam density, kg/m ³	Pre-treated at 80°C?	Aging time, hours	After aging (lab #1)	After 24 hours recovery	After 14 days recovery	After 5 weeks recovery
32	No	48	-13 (-17)*	-7	-5	-2
	Yes		-10 (-15)*	-4	-2	+1
	No	96	-9 (-21)*	-7	-	-
	Yes		-8 (-15)*	-5	-	-
	No	168	-10 (-20)*	-9	-2	-1
	Yes		-4 (-18)*	-3	-2	+5
	No	400	+15	+26	+29	+23
	Yes		+25	+34	+36	+37
50	No	48	-14 (-16)*	-8	-6	-5
	Yes		-11 (-16)*	-5	-3	-1
	No	96	-12 (-21)*	-8	-	-
	Yes		-12 (-17)*	-8	-	-
	No	168	-11 (-21)*	-10	-4	-3
	Yes		-9 (-21)*	-7	-1	-1
	No	400	+10	+18	+21	+22
	Yes		+9	+19	+21	+23
48	No	48	-14 (-21)*	-9	-7	-5
	Yes		-12 (18)*	-6	-4	-2
	No	96	-16 (-23)*	-12	-6	-
	Yes		-7 (-22)*	-11	-	-
	No	168	-13 (-27)*	-13	-7	-8
	Yes		-13 (-24)*	-11	-6	-6
	No	400	-8	-3	0	+1
	Yes		-7	0	+2	+3

Table 11		
Foam density, kg/m ³	Pretreated at 80°C?	Humid Aged CFD change, %
32	No	+28
	Yes	+16
40	No	+6
	Yes	+2
48	No	-19
	Yes	-18

4.5 Autoclave Aging

Since one of our objectives was to demonstrate the superiority of the wet conditioning over humid aging in an autoclave, we tested the same three foams using ASTM D3475 test C, J2 conditions, i.e., 5 hours at 120°C followed by 3 hours at 100°C drying before testing. The data in Table 11 resulted:

Humid aging of the 32 and 40 kg/m³ foams increases foam hardnesses i.e., positive CFD changes. Pre-treatment reduces the extent of CFD pick-up. Humid aging reduces the CFD of the 48 kg/m³ and pretreatment has essentially no effect.

Recall that when these foams were wet aged at 80°C/75% RH, all three foams exhibited a negative CFD changes. Similarly, aging at 50°C/95% RH results in negative CFD changes. Thus the two wet aging treatments cause hardness reductions whereas the autoclave treatment produces positive and negative changes. The reason for these inconsistent responses caused by autoclaving is not known at the present.

4.6 Influence of Foam Compressing/Crushing on Wet CFD change

It is well established in the HR moulded foam industry that some foam mechanical properties can be modified by compressing the foam specimens prior to testing. For example, foam permeability and resiliency can be positively affected by pre-compressing the test specimens several times. Some OEM specifications require specimens to be pre-compressed 2 to 10 times before measuring resiliency.

One of the foams tested previously has been subjected to multiple compressions by about 75 to 80% of the specimen thickness before CFD testing and Wet aging with the following results:

One of our objectives in this compression investigation was to establish if the Wet CFD change increases found after aging for 400 hours could be reduced or eliminated. Our results indicate:

1. Specimens not pre-compressed before testing all show positive Wet CFD change values, especially the 400 hours aged specimens (+50% increase).
2. Specimens pre-compressed 5 times are softer than initially after aging.
3. Increasing pre-compression to 10 times further decreases the Wet CFD values resulting in greater negative changes after aging (24 through 144 hours).
4. Even after the 400 aging, pre-compression reduces the magnitude of Wet CFD change, i.e., 0, 5 and 10 pre-compressions results in Wet CFD change increases of 50, 30 and 17% respectively. Thus severe pre-compression will affect the degree of Wet CFD change and may even change the CFD value from positive to negative, see Figure 5.

Table 12			
Foam density, kg/m ³	Number of Compressions	Aging time at 80°C/75% RH, hours	Wet CFD change, %
32	0	24	+2
	5		-1
	10		-10
	0	48	+4
	5		-6
	10		-8
	0	96	+8
	5		-3
	10		-7
	0	144	+11
	5		-6
	10		-6
	0	400	+50
	5		+30
	10		+17

5. Phase II

In this phase, foams made using three different isocyanates were formulated at nominally 40 and 48 kg/m³ densities. Thus six foam grades were moulded:

We will refer to these foams as M/T, T and T/M types and will reference their measured densities. Each type was subjected to three aging tests;

- I. Wet CFD change after aging 22 hours at 50°C/95% RH
- II. Constant Force Pounding
- III. Wet Set at 50°C/95% RH

Isocyanate type	Target foam density, kg/m ³	Measured foam density, kg/m ³
80% MDI/20% TDI	40	40
	48	50
100% TDI	40	40
	48	48
80% TDI/20% MDI	40	46
	48	58

5.1 Wet CFD change and Wet Set

Wet CFD change procedure was modified for convenience of the test technicians. The reconditioning time after removal from the chamber was increased to 2 hours. However, Wet Set reconditioning remained at 30 minutes (tests have indicated that the change in Wet set values does not change significantly even after 24 hours recovery before measurement).

The Wet CFD change and Wet set results for these foams are shown in **Table 13**:

The individual results for Wet CFD change are for duplicate sets of specimens (3 specimens tested in each set) to ascertain the reproducibility of this test. The wet CFD changes for these three isocyanate chemistries are all negative and the change values are quite small. There does not appear to be any systematic trend with foam density and data sets are quite consistent. In contrast, the wet set values are much greater and it is well known that wet set values are dependent on foam density, i.e., the lower the density, the higher the wet set. 100% TDI foam may have lower wet sets than the mixed isocyanate foams.

After 5 days recovery, all of these foams recover their CFD hardnesses, some exactly, some a small percentage lower or higher but the resultant differences are small. Essentially, we can say that these foams recover hardness and that the softening caused by the elevated temperature and relative humidity is reversible. This has also been shown to be true for complete cushions tested for IFD before, after treatment and then after recovery in the laboratory for a few days⁽¹⁰⁾. Wet sets also show some recovery but even after 2 weeks, some set is still recorded, see Figure 6.

Foam Type	Measured density, kg/m ³	Wet CFD change (50°C/95% RH, 22 hours), %	Wet Set, %
M/T	40	-2, -3	14 (9)*
	50	-2, -4	6 (3)
T	40	-3, -4	10 (7)
	48	-2, -3	6 (5)
T/M	46	-1, -3	16 (10)
	58	0, -3	11 (8)

()* Sets after
2 weeks recovery

5.2 Constant Force Pounding after Aging at 50°/95% RH

These foams have been pounded as-produced and after aging. The data is found in Table 14.

Table 14					
Isocyanate, Type	Measured foam density, kg/m ³	Aging at 50°C/95% RH, hours	Constant Force Pounding		Initial Hysteresis Loss, %
			Thickness change, %	40% IFD change, %	
M/T	40	0	2	17	23
		48	3	14	
		96	3	18	
		168	3	15	
		400	3	14	
	50	0	2	17	23
		48	3	18	
		96	3	13	
		168	3	12	
		400	4	19	
T	40	0	2	15	22
		168	2	17	
		400	2	16	
	48	0	1	11	19
		168	1	12	
		400	1	17	
T/M	46	0	2	24	27
		168	2	26	
		400	2	21	
	58	0	2	25	27
		168	2	20	
		400	2	25	

The CFP thickness changes are all low and within acceptable limits. The IFD losses are also acceptable except perhaps for the values for T/M foams since they exceed 20% loss. There does not appear to be any trend in IFD changes with aging time for any of these foam types. We recorded the initial hysteresis losses of these foams from the IFD/deflection traces specifically to determine if the correlation that we had established previously held true for these different types of foams, i.e., does the 40% IFD loss after pounding correlate linearly with the hysteresis loss numbers⁽²⁾. In Figure 7, the initial (un-aged) IFD loss values are plotted for all foam types and densities against these hysteresis loss numbers and a linear relationship has once again been found. With a correlation coefficient of $R^2 = 0.9893$, it is obvious that initial IFD loss is very strongly dependent on foam hysteresis loss irrespective of foam density over the range 40 to 58 kg/m³ or type of isocyanate used to manufacture these foam (M/T, T or T/M).

There is also a reasonably good correlation between the 40% deflection IFD losses and hysteresis losses for foam samples aged for 168 or 400 hours at 80°C/75% RH. The lower correlation coefficients found for aged samples may indicate variations in foam response to heat/humidity cure during aging.

6. Phase III – Comparison between selected aging treatments

Four treatments were selected:

- i) Aging at 100°C, ~95% RH in a desiccator/oven for 72 and 168 hours
- ii) Aging at 38°C, ~95% RH in an environmental chamber for 72 and 168 hours
- iii) Aging at 50°C, ~95% RH in an environmental chamber for 72 and 168 hours
- iv) Aging at 100°C in an air circulating oven
- v) Aging at 95°C, 95% RH for 100 hours in a jar/oven or environmental chamber

In the case of the last treatment, specimens were aged in either an environmental chamber or placed in a jar with water added and aged in an oven at 95°C. These comparative tests were done to find out if aged CFD changes resulting from the two different storage conditions would produce the same results or otherwise.

Specimens were also humid aged in autoclaves for 3 hours at 105°C and 5 hours at 120°C, oven dried and tested.

Hysteresis loss for each specimen was measured before and after treatment to determine how much change, if any occurs in this property after aging.

We used a rapid CFD test cycle with essentially no hold time at 50% deflection. Specimens were then allowed to recover at ambient temperature before hysteresis loss (HL) was measured. Thus for each specimen the CFD and HL was recorded before any aging treatment, then after 72 hours aging, followed by further aging for a total of 168 hours before retesting again. However, the autoclaved specimens were only humid aged for 5 hours at 120°C or 3 hours at 105°C, dried for 3 hours at 100°C, reconditioned in the laboratory under standard conditions and retested for CFD and HL changes.

Seven different foam grades were selected for testing. These foams, in the form of 380 x 380 x 100 mm test blocks had been poured on production equipment into a block mould on a commercial production line. The foam grades were:

Block Number	Core Foam Density, kg/m ³
530	37
545	50
551	33
566	38
571	46
740	38
742	38

The Wet CFD changes and HL changes (initial – final HL) results are found in Table 15. It is immediately obvious that the Wet CFD changes vary between negative and positive values, e.g., CFD change values after treatment at 100°C/95% RH vary between -35 and +11% after 72 hours of aging. The changes after wet aging treatments (38°C/95% RH and 50°C/95% RH) are

much smaller and the two data sets are quite similar. The Wet CFD changes after 100°C oven treatment are all negative, varying between -5 and -17%. This is the only treatment to produce consistent, i.e., all negative results.

HL changes after each of the three treatments at high humidity (95%) all show positive increases with the changes after wet aging conditions (38°C/95% RH and 50°C/95% RH) all less than 4%. Greater changes were found after treatment at 100°C/95% RH, i.e., 80- 20% increases. Similar increases were found after 168 hours. After aging at 100°C, HL changes are very small but show some changes when the treatment time is increased to 168 hours.

Specimens autoclaved and dried showed considerable variation in CFD change but all exhibit positive increases in HL, Table 16. After aging for 5 hours at 120°C, CFD changes vary between -36 and +18% but less change was found after autoclaving for 3 hours at 105°C. It should be noted that each of the CFD and HL change results recorded are an average of three replicate determinations. The data spread for all foams was much wider for autoclaved-aged specimens than for environmental chamber-aged specimens. This has been determined to be due to the uneven distribution of moisture in an autoclave with specimens lower in the chamber picking up more moisture than those near the top of the autoclave. Thus, thorough drying of specimens before CFD/HL measurements is necessary. This can be checked by weighing the specimens before further testing. In Table 16, foam 551 has two results. Two of the specimens had an average CFD change of +8% and a third at +36% giving an overall average of +17%. Other foams, even those autoclaved at 105°C, also show inconsistent results within sets.

The HL changes for both humid aging treatments are much more consistent. After five hours at 120°C, all HL changes are positive, varying between 9 and 17%. Smaller changes were found after aging for 3 hours at 105°C, i.e., 2-6%. Thus foam HL changes after autoclave aging are more consistent than CFD changes.

To complete the environmental aging measurements, we measured the wet sets of these seven foams, Table 17. Sets between 9 and 28% were found. A wet set of 9% is exceptional but it should be noted that this was the highest density foam tested and it has been established that wet set is highly dependent on foam density.

We attempted to correlate the % CFD changes with foam density. No correlation exists for specimens treated at 100°C/95% RH and since the % changes after wet aging at 38 or 50°C/95% RH are quite small, correlation is difficult. After aging at 100°C in an oven, there may be some correlation with density but quite weak.

There is better correlation between % HL change and density after treatments at 38 or 50°C/95% RH and 100°C but no correlation for specimens aged at 100°C/95% RH or those that were autoclaved.

Table 15					
Aged 72 hours				Aged 168 hours	
Treatment	Foam, ID	% CFD change	% HL change	% CFD change	% HL change
100°C/95% RH	530	-35	+8	-48	+9
	545	-31	+10	-42	+11
	551	+11	+18	+29	+23
	566	+2	+14	0	+18
	571	+9	+18	+14	+21
	740	+8	+19	+18	+26
	742	+3	+20	+36	+26
38°C/95% RH	530	+4	+3	+1	+4
	545	+2	+3	-1	+3
	551	0	+3	-1	+4
	566	+2	+3	+1	+3
	571	+2	+3	0	+4
	740	-1	+3	-1	+4
	742	0	+3	-1	+5
50°C/95% RH	530	+4	+3	+5	+3
	545	0	+2	0	+2
	551	-1	+3	-1	+4
	566	+2	+3	+3	+3
	571	+1	+3	0	+3
	740	-1	+3	-2	+3
	742	0	+2	-1	+3
100°C	530	-5	-1	-2	-1
	545	-5	0	-12	0
	551	-17	0	-18	+1
	566	-10	0	-13	0
	571	-5	0	-13	+1
	740	-12	0	-16	+1
	742	-10	0	-15	+1

Humid Aged Treatment	Foam ID	% CFD change	% HL change
5 hours at 120°C +3 hours drying at 100°C	530	-36	+9
	545	-34	+10
	551	+8 or +17	+17
	566	-8	+13
	571	-8	+13
	740	-1	+16
	742	+18	+17
3 hours at 105°C +3 hours drying at 100°C	530	-6	+2
	545	-14	+3
	551	-6	+5
	566	-1 or +4	+4
	571	-12	+4
	740	-13	+4
	742	-3 or 0	+6

Treatment	Foam ID	Wet Compression Set, %	Core Density, kg/m³
22 hours at 50°C/95% RH	530	13	37
	545	9	50
	551	28	33
	566	18	38
	571	21	46
	540	21	40
	742	28	38

If the HL change values are plotted versus time, Figure 8 results. The data can be divided into three “zones”. After treatment at 100°C/95% RH, large HL changes are found (9-26%). Both wet aging treatments result in lower changes (2-5%) zone 2 and oven aging at 100°C results in only a few percent change in HL zone 3.

In Table 18, the Wet CFD changes resulting from specimen treatment at 95°C/95% RH in an environmental chamber or in a jar/oven are compared to determine if these two methods would produce the same or at least similar results. Five out of the six foam types had similar % CFD changes, the exception being foam 530. We believe that further investigations should be done to establish if the jar/oven treatment can be used instead of an expensive chamber.

Constant Force Pounding was performed on the same six foams and the results are found in Table 19. These foams all exhibit low thickness/height losses after pounding. IFD losses vary between 16 and 25% and these values may be considered to be fairly acceptable for the foam density range investigated.

Table 18		
BASF CFD Change Results after Aging at 95°C/95% RH for 24 hours		
Foam ID	% CFD Change	
	Chamber	Jar/Oven
530	14.3	5.2
551	17.2	18.5
566	12.6	9.7
571	15.7	14.3
740	15.6	13.7
742	14.3	13.5

Table 19					
Constant Force Pounding Results					
Block ID	Thickness loss, %	40% IFD loss, %	Initial HL %	Final HL %	HL Improvement %
530	3.8	15.9	25.6	23.5	2.1
551	2.5	21.5	28.4	26.2	2.2
566	2.8	23.7	22.5	19.3	3.2
571	3.6	20.2	27.3	23.8	3.5
740	3.3	25.4	28.6	25.1	3.5
742	3.1	23.9	35.6	32.0	3.6

The HL numbers obtained are interesting. All foams show an improvement in HL after pounding with an average improvement of about 3%. These HL changes may be attributed to foam cell walls being ruptured during the 80K cycles of pounding. If HL numbers decrease, the foams should become more “comfortable”. The hardness losses (16-25%) will decrease (i.e., IFDs will firm-up) with recovery time and most but not all of the initial foam hardness will be regained. Once again, the 40% IFD losses in pounding correlate reasonably well with the wet set values found for these foams.

In summary this third phase has shown:

1. % CFD changes after different aging treatments vary widely and some conditions result in inconsistent results.
2. % CFD change versus foam core density may be useful after oven treatment at 100°C.
3. HL changes versus density may be useful after Wet aging treatments.
4. % CFD changes versus aging time may be useful after 100°C aging but the other treatments produce inconsistent or only small changes.
5. % HL changes versus time after 100°C/95% RH aging are all positive (+8 to +20%) but are virtually zero after oven aging (100°C) and wet aging gives small changes of 2 to 3%.
6. There is a reasonable correlation between wet CFD changes (95°C/95% RH) for environmental chamber and jar/oven aged specimens.
7. Pounding improves HL numbers
8. CFP IFD losses can be correlated with wet sets and this study also indicates that HL changes are also correlated with wet set changes.

Compression Force Deflection (CFD) change values after aging treatments do not appear to be useful indicators of accelerated foam performance. This is especially true for any materials that have been humid aged in an autoclave.

Hysteresis loss changes are a better indication of long term foam performance as determined by an accelerated lab test.

Constant Force Pounding hardness and hysteresis losses are reasonably well-correlated with the static fatigue property measured under tropical conditions (50°C/95% RH) as determined by wet compression set.

DISCUSSION

Recently Sonnenschein et al, published data that included changes in CFD at 40% deflection using a VW testing methodology. In this test, samples 70x70x30 mm, are compressed 4 times by 70% of their original height. On the 4th cycle the 40% deflection force is measured. Foam was then aged in an environmental chamber for 200 hours at 90°C and 95% RH. After removal from the chamber and re-equilibration under standard conditions for 24 hours, the CFDs were re-measured. Their 40% CFD measurements on original foam did not correlate well with the humid aged CFD changes measured by these investigators.

Using small angle x-ray scattering techniques, Sonnenschein et al, have inferred that foam with a low humid aged CFD loss has a much better defined phase separation than foam exhibiting high HACFD change. They suggest that foam with superior HACFD loss has much better defined phase separation and that by changing foam formulation to improve this phase separation should result in improved HA foam properties. Foams with good aging properties show good separation of the hard segment (due to the water-isocyanate reaction) from the rest of the foaming mass caused by the slower gelation reactions (due to formation of a polymer network). By changing the amounts of blow and gelation catalysts, some improvements in humid aged CFD losses were obtained. However, increases in both gel and blow catalyst levels reduced these CFD losses and the correlations obtained were very low. It is not clear, at the present time if the data presented by Sonnenschein et al, can be compared with the results presented here.

Fortunately, they did choose to environmentally age their foam and not autoclave them for their CFD change work although they do refer to humid aging (normally a term associated with autoclave conditioning).

It may be possible to re-investigate our results or to repeat some of the environmental aging to determine if hard segment separation can explain some of the data we have obtained. For example, does the reduction in CFDs after short term aging (22-35 hours) followed by steady increases in hardness after longer aging times (200-400 hours) correlate with changes in hard segment separation? Our DMA investigations or swelling studies did not indicate the kinds of morphological changes that would explain the considerable changes found in hardness with prolonged aging. Maybe only sophisticated small-angle x-ray scattering will elucidate the structural changes that may be occurring with increased aging times.

Autoclaving results in variable amounts of water pick up depending where the specimens are located in the vessel. All specimens show moisture pick-up, lowest pick-up amounts for specimens, farthest away from the water reservoir and greatest nearest the water. Specimens autoclaved for 5 hours at 120°C pick-up more moisture than those treated for 3 hours at 105°C. Drying specimens in an air-circulating oven (3 hours at 100°C), removes the moisture but the specimens now weigh **less** than originally, so the autoclaving treatment must leach out something from these foams. Even after reconditioning for 18 days under controlled laboratory conditions (23 ± 2°C, 50 ± 5% RH), the specimens still weigh less than originally, with those treated at 120°C showing greater losses than after 105°C autoclaving.

Specimens aged in an environmental chamber at 50°C/95% RH all gain weight but much less than those autoclaved. For foams with densities of 32-60 kg/m³ the gains were about 1%. After 18 days reconditioning, these specimens all retained some moisture but only 0.1% or less and masses were all greater than originally. Chamber-aged specimens show further weight increases if the aging time is increased, say from 22 to 48 or longer hours.

Thus, since foams all exhibit decreases in CFD hardness after environmental chamber aging, we believe the absorbed/adsorbed moisture must soften the foam struts via plasticization. Longer exposure times up to about 48 hours result in further softening but even longer treatment times produce foams with smaller CFD losses. This may be due to some of the absorbed/adsorbed moisture being released from the struts during these long treatment periods. Alternatively, the polymer in the struts may be “postcured” during the conditioning periods. Both water removal and “postcuring” would stiffen the polymer and result in firmer foams. Since extremely long aging periods, e.g., 400 hours, results in foams that are harder than the original specimens, it is postulated that initially CFD hardness recovery may be due to moisture desorption followed by polymer “postcure” resulting in harder foams. However, we have not been able to measure any changes in polymer structure using either solvent swelling or DM techniques. This area warrants further study.

RECOMMENDATIONS

Our panel does not believe the treatment of foams in an autoclave to be a value-added aging method. Since earlier work has indicated that autoclaving changes polymer structure, a phenomenon that will not take place in a vehicle, and our results indicate that autoclaving may remove mass from the foam, we advocate foam conditioning by autoclaving be removed from foam testing. In contrast, we advocate the use of an environmental chamber or a surrogate test, e.g., closed jar containing water placed in an oven to age foam. The actual wet aging conditions selected may vary from OEM to OEM depending on vehicle requirements. We advocate the same wet conditions that are used extensively by all the Asian OEMs for Wet Compression Set aging, i.e., 22 hours at 50°C/95% RH. Longer aging times may produce greater hardness changes but for quality control purposes, a duration of 22 hours will clearly indicate hardness changes and ensure excellent foam-part functionality in vehicles over the duration of the vehicle’s lifespan.

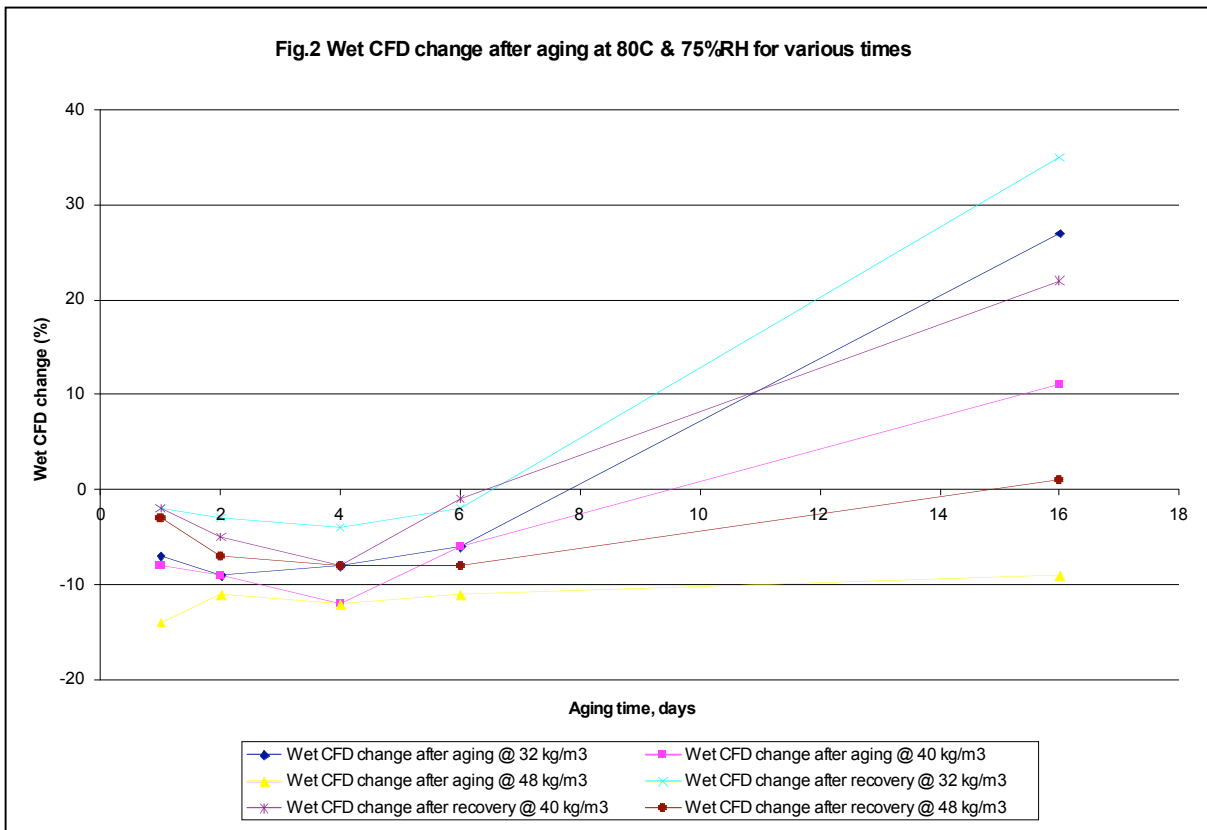
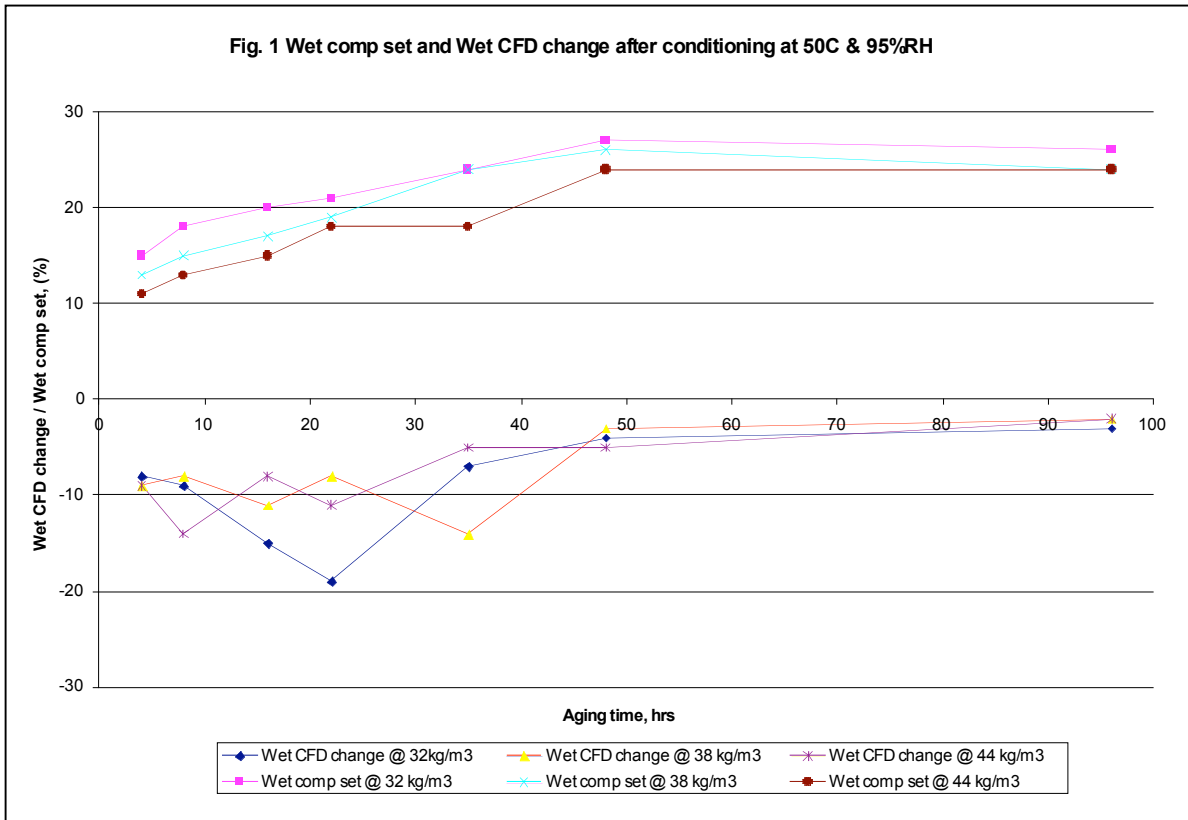


Fig. 3 Constant Force Pounding 40% defl. IFD losses after aging at 80C & 75%RH

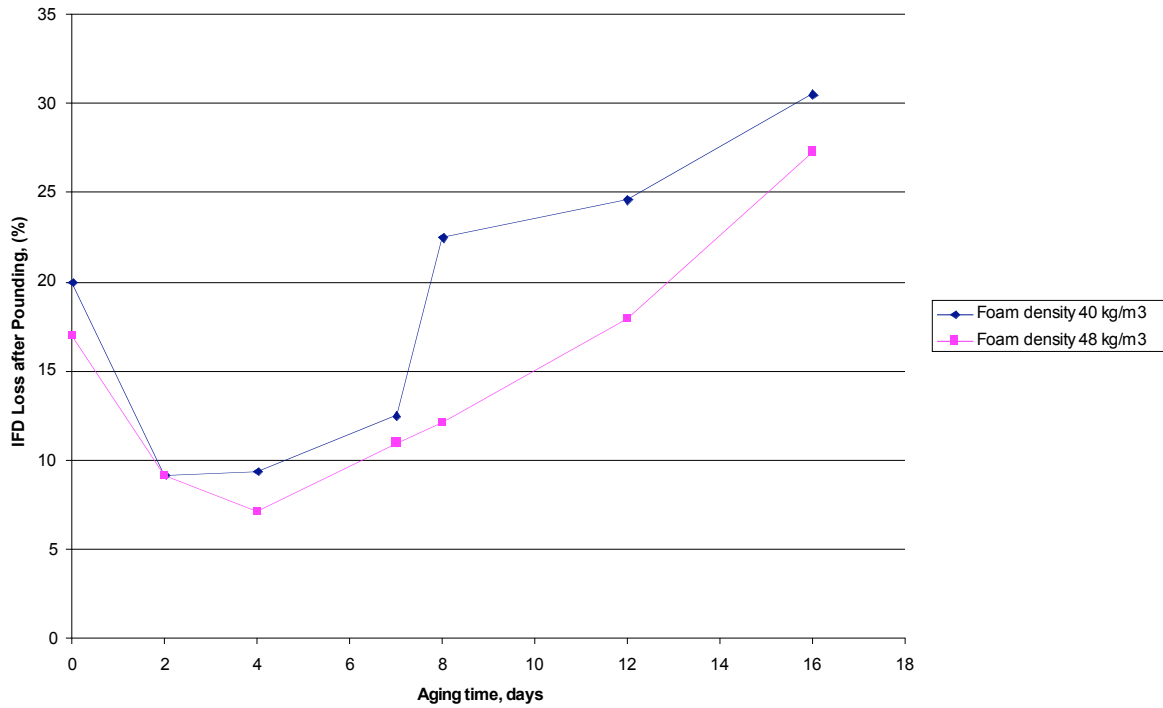
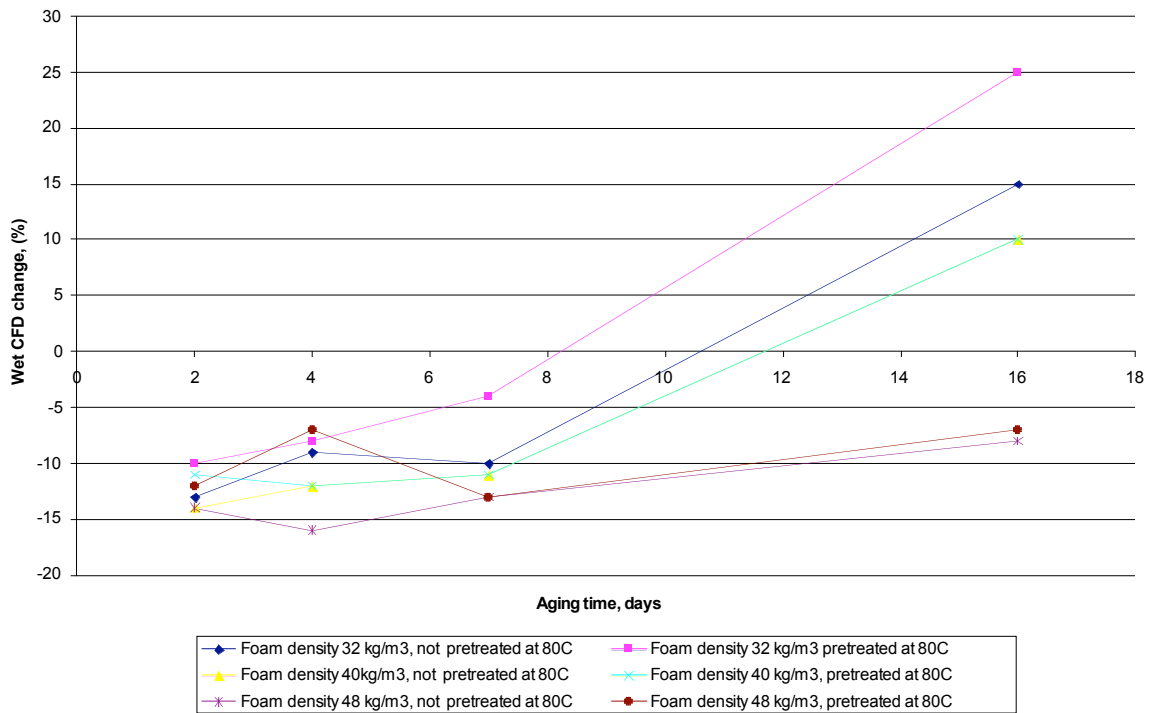
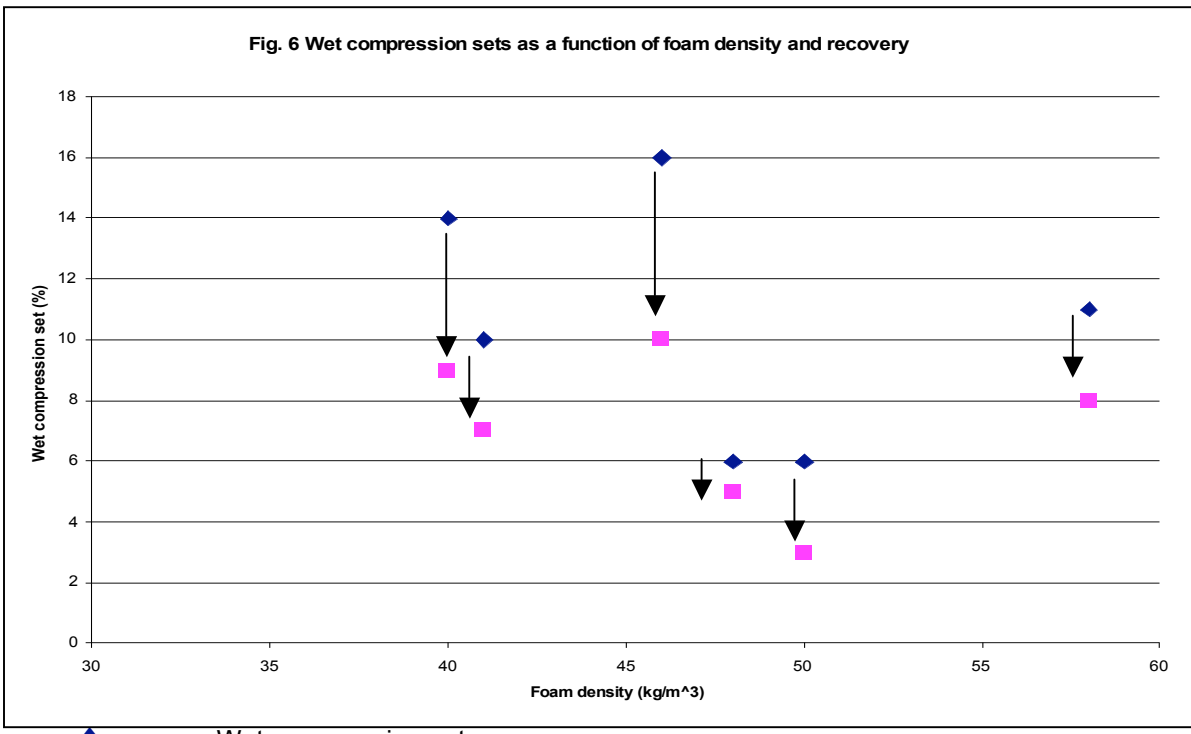
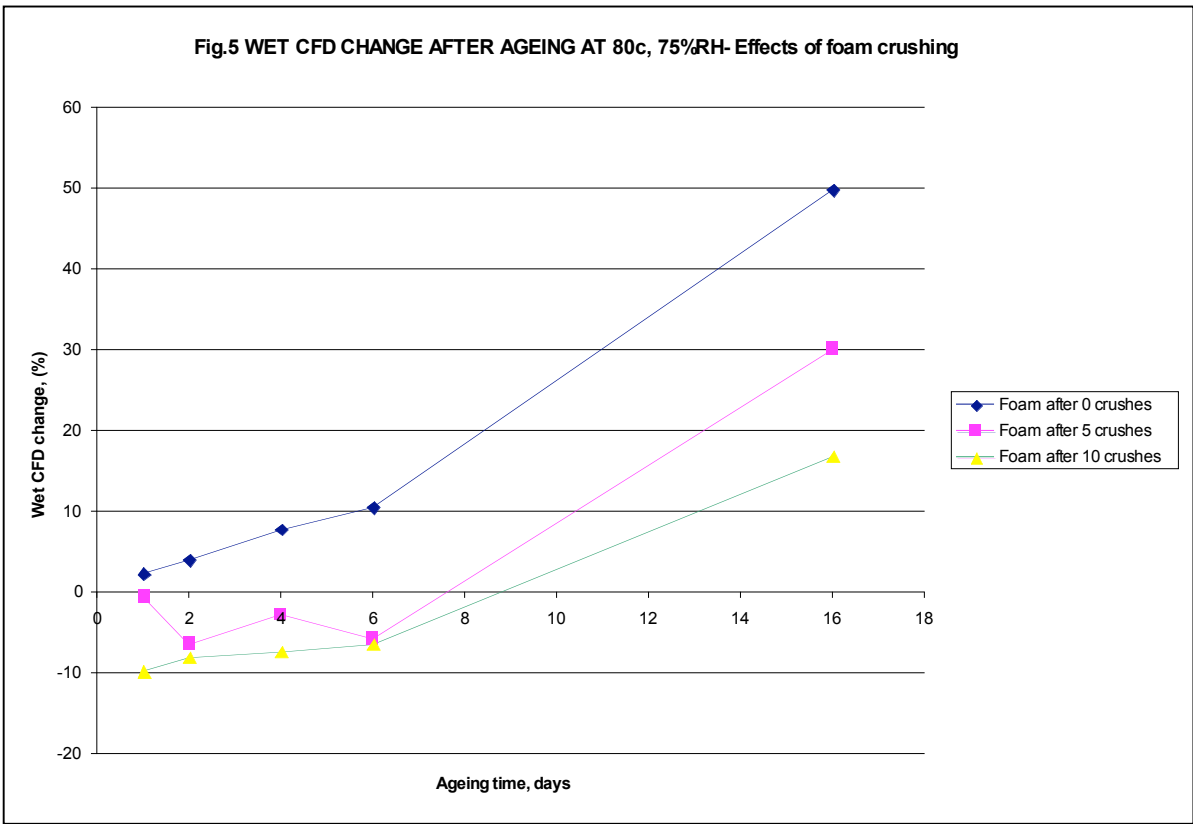


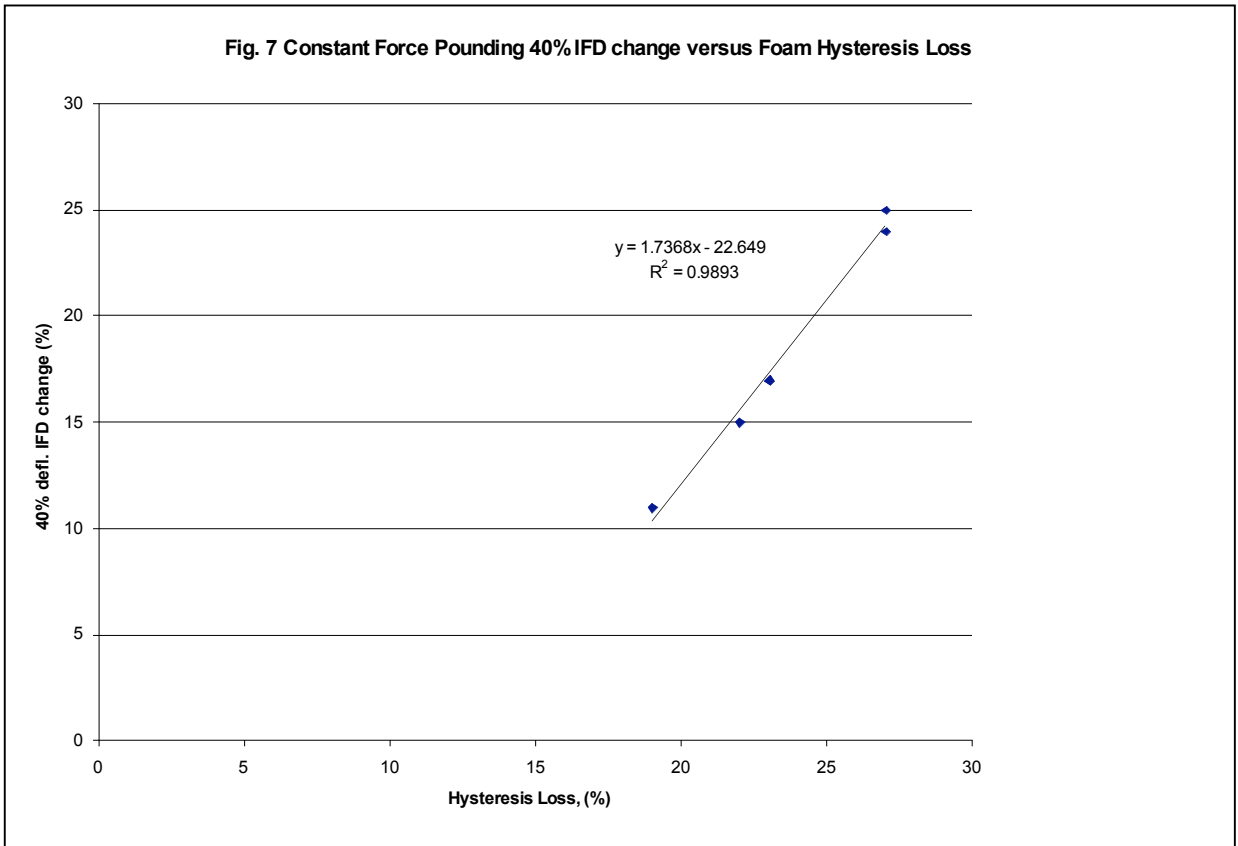
Fig.4 Wet CFD changes for Non-Pretreated and Pretreated Foams





- ◆ - Wet compression set
- - Wet compression set after 2 weeks recovery

Fig. 7 Constant Force Pounding 40% IFD change versus Foam Hysteresis Loss



Acknowledgements

The Authors would like to thank their respective employers for supporting the work presented in this paper. We would also like to thank all the members of the Molded Foam Industry Panel for their encouragement and critiques during the progress of this investigation. Special thanks to Kathy Lane of Woodbridge Foam Corporation for preparing the text of the paper. Technical support from Julian Milakowski, Senior Manager and his staff at the P3T Lab (www.P3Tlab.com) and BASF's Urethanes R&D Polymer Physics lab is greatly appreciated. We would like to especially thank our colleagues on the Panel from General Motors, Diane McQueen, Wayne Reeder and Joe Pellerito for suggesting that we consider the Cycle Q conditions found in GMW14124.

Appendix

Effect of Test Conditions (Heat and Humidity) on Chemical Changes that affect the Measured Properties of PUF

An estimate of the effect of test conditions can be made from first principles (*ab initio*) if a number of assumptions are allowed. The major assumption is that chemical changes in the polymer structure caused by heat and humidity are responsible for the changes in the measured properties of the foam. We recognize that this is likely a significant over-simplification since it ignores physical changes such as phase reorganization (hard phase versus soft phase segregation). However it is still useful to gain an appreciation of the time scales involved in chemical reactions.

A further assumption is that the reactions are primarily hydrolytic; water is directly involved in the reactions with the polyurethane. Tests done at higher humidity generally show greater changes in foam properties than similar tests done at lower humidity. There are at least two hypotheses to explain this observation: water may interact with the polymer linkages (primarily urethane bonds) resulting in cleavage and loss of CO₂ or the initial reaction is thermal cleavage of some bonds resulting in reactive species which then react with water rather than reversibly reverting to the original bond. In either case water is a required reactant.

All tests for polyurethane foam specify temperature and relative humidity (RH) requirements with specific tolerances. When considering the effects of water on the chemical bonds as polyurethane it is important to know the absolute humidity. The relationship between relative humidity and absolute humidity (grams of water per kg of air) is shown in Figure A1.

Figure A1: Water content in air as a function of temperature and relative humidity

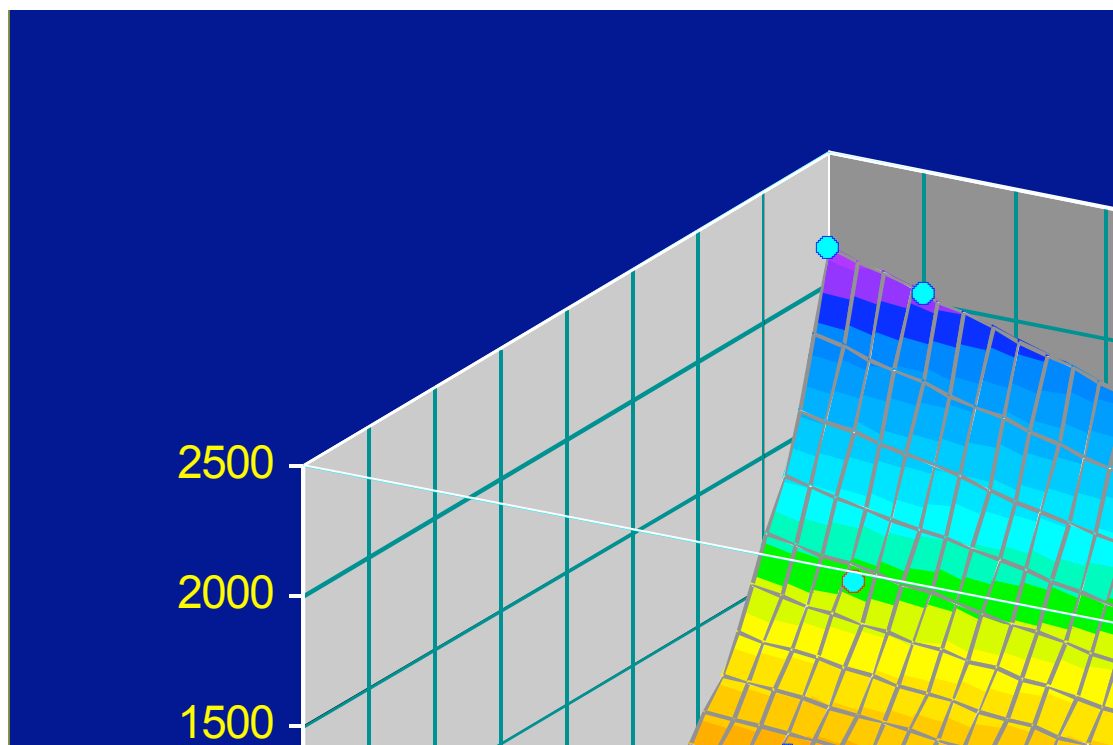


Figure A1 was generated using Tablecurve 3D from Jandel Scientific and data calculated using software from Lenntech, a company associated with the Technical University of Delft. The conversion program is available at <http://www.lenntech.com/calculators/relative-humidity.htm>. The graph clearly shows that the amount of water present increases tremendously as temperature increases. The concentration of water in the air increases by approximately a factor of 100 going from 20° to 100°C.

One approximation often used in chemical kinetics (based on the Arrhenius equation) is that reaction rates double with every 10°C increase in temperature. This means that a reaction may proceed approximately 250 times faster at 100°C than at 20°C.

Combining the effects of water concentration and reaction rate as a function of the reaction temperature and presenting the results as the reaction half-life, the graph in Figure A2 can be generated.

Figure A2: Half-life of chemical bonds as a function of relative humidity and temperature.

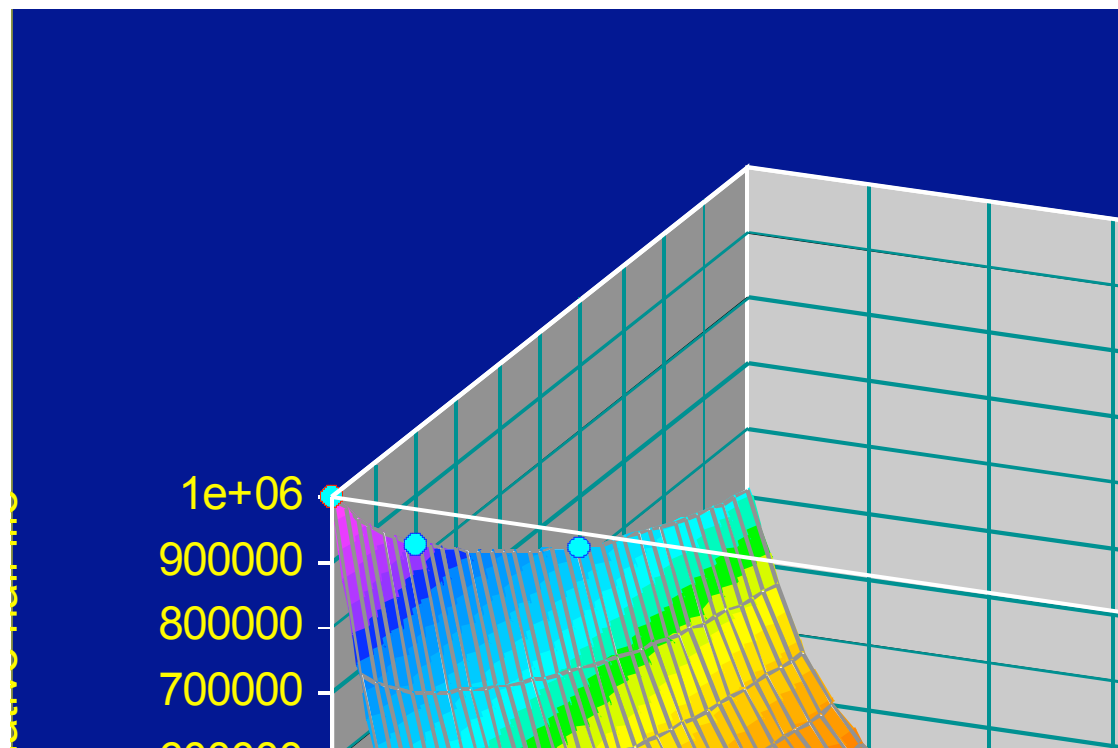


Figure A2 shows the relative lifetimes of whatever chemical species may be responsible for reactions during humid aged conditioning. The data used to generate Figure A2 is given in Table A.

Table A: Half-life of chemical bonds as a function of relative humidity and temperature.

Temp (C)	RH (%)	Half life (relative)
20	50	1000000
20	60	829545
20	80	623932
30	50	268382
30	80	167431
50	50	19251
50	75	12852
50	80	12007
50	95	10139
60	50	5155
60	75	3456
60	95	2716
70	50	1383
70	75	920
80	50	370
80	75	247
80	95	195
90	75	66
90	95	52
100	95	14
100	100	13

If for example, some species has a half-life of 1,000,000 hours at 20°C and 50 % RH (half of that species will decompose in a million hours under those conditions), then at 100°C and 95% RH half the material will decompose in 14 hours.

There are some major assumptions used in generating these data, so they should not be taken in any way as predictive. We fully recognize the limitations and potential errors in doing these calculations. It was the sole purpose of this exercise to illustrate how significantly extremes of temperature and RH could affect the chemical species in a polyurethane foam.

More extensive hydrolytic conditioning studies of various flexible polyurethane foams under various temperature and humidity treatments may elucidate the mechanisms involved that result in foam mechanical changes.

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BIOGRAPHIES

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Ron Blair has received his degrees in Glasgow Scotland. He continued his studies at the University of British Columbia before joining Royal Dutch Shell Plastics Laboratory in Holland. During his six years with Shell, he worked in various functions including fundamental research, plastics testing and latterly, polyurethanes. In 1976 he joined Monsanto Canada, which became Woodbridge Foam Corporation in 1978. Ron has held various processing and chemistry positions and recently retired from managing the Woodbridge P3T Laboratory, Woodbridge, Ontario, Canada. He is now a consultant to the polyurethane industry and can be reached at ronblair@hotmail.com.

R. Dawe

Bob Dawe joined Dow in 1986 and worked on enhanced crude oil recovery, pulp and paper applications and nuclear magnetic resonance spectroscopy. Bob joined Polyurethanes as a TS&D specialist in 2000 for molded foam applications and is based in Sarnia, Ontario, Canada. Bob has a Ph.D. (1982) in synthetic organic chemistry from the University of Waterloo and held postdoctoral positions at the Australian National University and the National Research Council of Canada.

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Roy Pask has been with BASF Corporation since 1968 where he is currently Supervisor of Polymer Physics in the Urethanes R&D Department. With over 30 years of foam testing experience, Roy also represents BASF on a number of industry associations including the Center for the Polyurethanes Industries, the Polyurethane Foam Association, the Carpet Cushion Council, the Society of Automotive Engineers, the Molded foam Industry Panel and the American Society of Testing and Materials, where he serves as subcommittee chairman for cellular material and urethane raw material standards. Roy did his undergraduate and graduate studies at Wayne State University in Detroit Michigan.

James T. McEvoy

Jim McEvoy has worked for Johnson Controls for 17 years in various roles, most recently focusing on polyurethane foam development. He received his Bachelor of Science in chemistry from the University of Wisconsin and MBA in Managing Technology from the University of Phoenix. His entire career has been spent in either polyurethane foam manufacture or seating development.

Mark Weierstall

Mark Weierstall has received an Associates Degree in General Studies from Macomb County Community College and is currently pursuing a Mechanical Engineering Degree. He has 22 years of experience the automotive foam industry where his activities have mainly been focused on product development, physical testing and specification development for seating and energy management foams. He currently manages Woodbridge's Corporate Comfort Laboratory in Troy, Michigan.

Marcela Rusan de Priamus

Marcela-Elena Rusan de Priamus joined Woodbridge Foam Corporation in 2001 and worked on testing and characterization of the Polyurethanes materials. Marcela is involved in different R&D projects on experimental laboratory testing for mechanical and physical properties of polyurethanes applications. She has her Bc. Eng. in Material Science and Engineering (1995) with previous European experience in testing and analysis of different materials for their microstructure, mechanical, physical and thermal properties. At the present time, Marcela is a Supervisor in the Woodbridge P3T Laboratory, Woodbridge, Ontario, Canada.