Recommended Accelerated Humidification Treatment for Polyurethane Foams

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It is well known that high resiliency polyurethane foam is susceptible to the influence of changes in atmospheric humidity. Increases in humidity affect the polymer in the foam struts, reduces foam hardness and increases compressive set values. The major problem is how to select the best humidity conditions to expose specimens to before performing selected tests that predict the functional performance of foam in automotive cushioning.

Historically all foams were treated to high humidity conditions (95-100% RH) at elevated temperatures (105-125°C) in an autoclave for 3-5 hours. This treatment must be performed at elevated pressures between 21 and 35kPa. It is now recognized that these autoclave conditions do not reflect environmental conditions in vehicles. An alternative humidification treatment is to expose foam in a chamber at atmospheric pressure to tropical conditions, e.g. 40-50°C, 95-100% RH. These conditions are considered to represent actual exposure conditions experienced by cushioning in vehicles in the tropics and during the summer months in other climates.

A wide range of foam densities (27-80 kg/m^3) HR foams were exposed to either tropical or autoclave conditions and changes in properties measured. These properties were monitored for long time periods after treatment to ascertain if the changes caused by humidification treatment remained constant or not. Property recovery of autoclaved specimens was rapid and virtually complete after 14 days of ageing whereas foams exposed to tropical conditions retained a substantial proportion of their exposed properties. This contribution compares these contrasting performances, recommends that foams should be exposed to tropical conditions and that autoclaving should be discontinued as a humidification treatment.

INTRODUCTION

A recent survey indicates that polyurethane foam for automotive cushioning successfully replaced foamed rubber seating from the mid-1950s onwards (1). The succession of polyurethane foam types can be classified as i) fabricated slabstock, ii) hot moulded foam and iii) high resiliency (HR) moulded cushioning. The world automotive industry uses various types of HR moulded foams based on several chemistries and a wide range of foam densities are in use. The density range 25 to 80 kg/m^3 covers most of the moulded products in automotive use today.

During the period (approximately 50 years) when polyurethane has been used, many tests have been developed to determine the best set of properties that can be measured in the laboratory, which will predict the usefulness or functionality of these foams in use. Some of these tests are "carry-overs" from foamed rubber testing but others have been developed specifically for automotive cushioning. Yet others were created for polyurethane foams used in other applications such as furniture and bedding, and then transferred or adapted to automotive foam cushioning. An example of such a test is the Constant Force Pounding (CFP) test which was designed to try and predict the long term performance of slabstock foam grades for various applications. Such a test has proved to be extremely useful in a least eliminating foam grades that will not perform well under repeated compressive forces over long time periods. Thus the CFP test is classified as an accelerated laboratory test (results obtained in one day or less) that has been shown to correlate well with the long term performance of automotive cushioning. Therefore it has been classified as a functional test useful in predicting long term foam performance in vehicles.

A number of other tests have been evaluated extensively to ascertain their functionality and repeatability/reproducibility. These tests have been discussed in several previous papers presented by the Molded Flexible Foam Industry Panel, a group of experts from the OEMs, Tier 1 seating suppliers, foam producers and a number of major raw material suppliers (see www.moldedfoam-IP.org). It has also been the task of this panel to critically review some tests and results for properties that are deemed non-functional (and to recommend their elimination) and to review other tests that are controversial or have been shown to provide data of dubious worth and/or that have poor repeatability or reproducibility between and within laboratories. Specifically, the panel considers tests that use conditions that will not be encountered in actual vehicle useage to be non-value added and has recommended their replacement or elimination from test specifications (2).

One area that has been highly controversial in the automotive seating industry is the influence of atmospheric moisture on foam seating performance. It is well recognized that polyurethane foams are affected by moisture changes in the environment. For instance, full cushions sitting on their actual support pans/structures were evaluated over a wide range of temperature (-30 to +100°C) and relative humidities (0 to 95%). Temperatures and humidities above these recommended for test laboratories (23 ± 2°C, 50 ± 5% RH) resulted in foam softening in IFD testing and hardening when temperatures and RH were lowered (3). However, it was shown that hardness and the other measured properties returned to their original values once the parts had been re-equilibrated within the standard laboratory conditions. Thus, although property changes did occur after the parts were treated/conditioned at non-standard conditions, the properties were reversible and no permanent changes were found.

In laboratory testing, various tests have been used to establish how much a foam will or will not recover from long term compression. From the inception of polyurethane foams until recently, a number of compression tests have been accepted and used by the industry. These tests were "handed-down" from foam rubber testing through slabstock, hot-moulded foam to the current HR foam types. Two of these compressive tests are i) normal or dry and ii) humid aged. The former test simply evaluates the set performance of a foam after compression (e.g. by 50 or 75% of its initial thickness) for 22 hours at 70°C. The specimen is allowed to recover for 30 or 60 minutes after removal from the compressed state and the extent of set calculated and reported.

The humid aged compression set is measured after the foam has firstly been exposed to temperature (105 or 120°C), pressure (21 or 35 kPa) and relative humidity (100%) in an autoclave and then the wet specimens are allowed to dry in an oven at 70 or 100°C usually for 3 hours for every 25mm of specimen thickness.

Finally, after reconditioning under standard laboratory conditions, these specimens are compressed as per normal or dry set conditions and the sets are measured/calculated as before.

An alternative set treatment was proposed in 1977 by Saotome et al (4) as they believed the Wet Set data that they generated was more of a reflection of actual foam exposure to tropical conditions, i.e. the test was more functional than the humid aged compression set (HACS) test. They simply carried out the normal compression set test but in an environmental chamber maintained throughout the test at 50°C/95% RH, i.e. tropical conditions. After removal from the chamber and the restraining clamps, the specimens were allowed to recover for 30 minutes, their instantaneous thickness (height) was measured and the values for Wet Compression Set (WS) was calculated and reported. Usually specimens are compressed by 50% of their original thickness but other deflections may be used.

A number of investigators (2, 5-7) have evaluated the HACS test methodology. They have found that treatment at the elevated temperatures called for in humid ageing cause alterations in the foam polymer structure or some foam mass is lost during autoclaving. These chemical changes are considered to affect foam performance in compression. Such chemical changes do not take place during in-vehicle useage and thus the validity of autoclaving as a humidification treatment is considered to be invalid and indeed these investigators have strongly recommended that autoclaving be eliminated as a pretreatment. The authors are unaware of any correlations between HACS data and the actual performance of cushions in a vehicle.

In contrast, the tropical conditions called for in WS may be encountered within a vehicle. Compressing a specimen by 50% of its original thickness is quite arbitrary. Other conditions have been investigated as have other temperatures. No additional useful information was gleaned from these other conditions (8) and thus we have used the standard deflection (50%) in this study. Almost all of the Asian OEMs, some European and recently some North American OEMs have standardized on the original WS conditions of 50% deflection held for 22 hours in a controlled chamber at 50°C/95% RH and the set is calculated after reconditioning for 30 minutes at 23 ± 2°C/50 ± 5% RH. We have used the latter conditions throughout our extended testing period.

FOAM AUTOCLAVING – HISTORICAL PERSPECTIVE

We will consider the ASTM standards that have been used since 1965. Initially there were two foam standards – one for slabstock foam (ASTM D1564T) and one for moulded foam (ASTM D2406). ASTM D1564T called for autoclaving at 104.4 or 121.1°C for 3 and 5 hours respectively whereas ASTM D2406 only called for the higher temperature and longer time. After these standards were combined to form ASTM D3574 in 1977, both autoclaving treatments were included and they were called

J1 (3 hours @ 105 ± 3°C) and J2 (5 hours @ 125 + 0, -5°C). Before autoclaving, all foams were to be dried for 3 hours at 100 ± 5°C and then re-dried after autoclaving for the same time at the same temperature ***unless*** 100°C adversely affected the final properties, then the drying temperature was lowered to 70°C. ASTM D3574 has regularly undergone several reviews by the ASTM D20 Committee and in the 2001 edition, the pre-drying procedure before autoclaving was eliminated (presumably to reflect on the fact that foam in use is not pre-dried). Therefore autoclaving treatment and post-treatment have been changed over the years but usually the OEM specifications that have to be met have remained unchanged. An exception is the General Motors Moulded foam specification FBMS 7-7. Due to the data and arguments presented by Union Carbide Corporation (now Bayer Material Science), the GM specification for humid ageing requirement was changed from J2 to J1 conditions since GM understood that the J2 conditions did not reflect functionality in a vehicle.

The cushioning supplied to the GM assembly plants continued to remain fully functional over the foam density range called for in their vehicles and long term performance continued to be maintained for the duration of vehicle life. Thus it appears that there does not appear to be any correlation between humid aged (autoclaved) compression sets and cushioning performance in the field. Since HACS data is a non-functional property the Molded Flexible Foam Industry Panel has rejected this test but recognizes that it has to be replaced with another humidification test procedure that will produce data that correlates well with long term cushioning performance in vehicles.

WET COMPRESSION SET DISCUSSION

As mentioned earlier, performing the compression set test under tropical conditions may be a viable alternative treatment. At least the treatment conditions are more realistic than either J1 or J2 conditions in ASTM D3574. However, the question remains – "Does compression set performed under tropical conditions correlate with actual in-vehicle performance of seat cushioning?" Alternatively it may be possible to correlate Wet Set with another foam property that can be shown to be related to the long term, in-vehicle performance of seat cushioning. For example, an earlier study by one of the authors showed good correlation between Constant Force Pounding data (an accelerated laboratory durability test performed within 24 hours) and the long term behaviour of seat cushioning over a period of up to five years in a police vehicle fleet (14). Also it has been shown that there is a fairly good correlation between Wet Compression Set values for a range of HR foams and the extent of hardness loss (IFD at 40% deflection) of these same foams after the 80,000 cycle Constant Force Pounding test (8).

Thus: *Wet Set correlates with CFD IFD loss which in turn correlates with Seat Cushion performance in actual vehicles.*

Therefore, Wet Set can be considered to be a measure of foam behaviour that correlates with foam cushioning performance in vehicles. Based on this evidence, Chrysler LLC (9) and Ford Motor Company (10) have recently issued new foam performance specifications in which the only compression set requirement is Wet Set, i.e. normal or dry set and HACS requirements have been eliminated. General Motors has considered replacing their set requirements with only Wet Set but as yet some of their overseas divisions still want to remain with normal and humid aged set requirements and these properties still appear in the recently issued GMW 15471 foam specification (11).

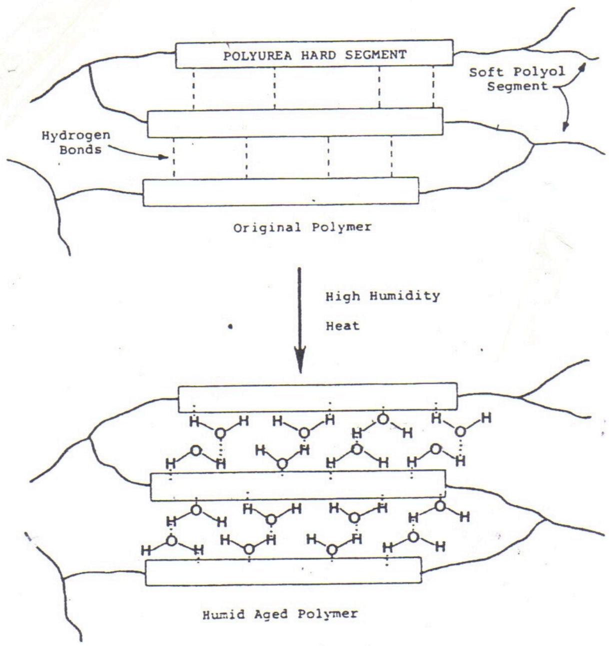
Most of the Asian OEM specifications call for Wet Set but they still retain normal set as well. The only Asian OEM exception is Mazda who still call for J1-type of HACS perhaps due to their affiliation with Ford.

In Europe, the OEMs call for a variety of humidification treatments. These range from Daimler-Benz requiring foam to be subjected to the J2 conditions (3 cycles). It may be noted that repeated exposure to J2 conditions does not worsen HACS values. Experiments on other HR foams produced values of 19, 21, 21% and 16, 14, 15% for successive HACS values, indicating that one cycle is enough to obtain HACS values (8). Fiat requires treatment for 200 hours at 90°C/100% RH; GM-Opel requires J2 conditions (although the GM parent company changed to J1 requirements as mentioned earlier); PSA require 96 hours at 70°C, 90% RH and Volvo requires foam to be extensively conditioned for 22/168/1400 hours at 50°C/20% RH and for the same time periods at 38°C/95% RH. Only Renault requires a similar environmental treatment to that specified by most of the Asian OEMs, i.e. 95 hours at 40°C/95% RH or 22 hours at 40°C/95% RH. The latter conditions in the Renault specification are used for compression set but test specimens must have skin on one of the faces perpendicular to the compression direction and instead of using a 50% deflection, they call for a 70% deflection.

Thus it is obvious that there are a plethora of conditions that have to be used to satisfy OEM requirements around the world. The mandate of the MFIP is to determine which test requirements truly reflect on the functionality of foam cushioning in use over the lifetime of a vehicle and to ascertain if these tests can be performed repeatedly and reproducibly in various laboratories. With these objectives in mind a series of experiments were devised aimed at determining the best humidification treatment to which foam should be subjected to obtain functional results that relate to the performance of cushioning in vehicles.

HUMIDFICATION PHENONMENON

It is not the intent of this work to try and elucidate what happens when HR foam is subjected to a humidification treatment. As stated earlier, we want to ensure that any test involving the presence of high relative humidity is meaningful and can be performed easily in foam test laboratories. However, we consider it useful to quote from a paper by Dounis, Wilkes and Turner (12) that appears to explain what occurs when foam is exposed to moisture especially at elevated temperatures: "The mechano-sorptive phenomenon observed is caused by the polar water molecules interacting with the hydrogen bonds within the cellular structure of the foams, particularly the hard segment regions". This phenomenon is best described in a figure that first appeared in an excellent publication on HACS by Herrington and Klarfeld (13):



Proposed representation of humid aged foam polymer.

Here we can see how the water molecules bond to the hard urea segments in the foam structure. The humidified structure thus obtained is considered to be "plasticized" by the water molecules and thus will be susceptible to slippage between the hard segments if a stress is applied to the foam while "plasticized". This may result in deformation that might be permanent after the stress is removed.

When a foam specimen is subjected to the Wet Compression Set test, it is stressed (compressed) first between metal plates by a specified deflection, e.g. 50 or 70%, and then exposed to temperature and high humidity for a relatively long time period, 22 hours. The foam is removed from the environmental chamber, the stress is released and the specimen is reconditioned for 30 or 60 minutes before the "final" height or thickness is measured. From the initial and "final" thicknesses the Wet Compression Set value is calculated and expressed as a percentage of the original specimen thickness except in the Renault specification where it is required as a function of the deflection. Further thickness determination is not required by any OEM or in any standard. Thus Wet Set is determined on a specimen that has returned to ambient temperature (23 ± 2°C) but in all probability still retains a higher level of humidity i.e. above 50 ± 5% RH.

In contrast, a specimen treated to humidification in an autoclave is always dried in a forced-air convection oven say at 70 or 100°C, before being compressed and treated at 70°C for 22 hours in an oven. It is believed that when desorption of water molecules occurs during oven drying, it leaves a "free volume" within the polymer structure. If this "free volume" still exists wholly or partially after specimen reconditioning in the laboratory (usually 2 hours or longer), it may contribute to the amount of set found for that foam. Obviously the greater the extent of the "free volume" after desorption, the greater its influence on compression set. This may partially explain why foams made with higher formulation water contents and thus higher percentages of hard urea have higher HACS values due to their higher "free volume" contents. Also, most foams have higher HACS values after exposure to the J2 treatment (5 hours @ 120°C) than after J1 treatment (3 hours @ 105°C). The higher temperature and autoclave pressure will allow more moisture to be hydrogen-bonded to the hard domains. After drying for 3 hours at 100°C, foams exposed to J2 treatment may have greater percentages of "free volume" and thus higher compression sets result.

As mentioned throughout this brief summary, all set values are measured on specimens reconditioned in the unconstrained state for only 30 or 60 minutes after removal from the oven and release from the compression plates. It is known that many foams regain thickness at longer recovery times (13). An extensive reconditioning trial by one of the authors (3) indicated that some foams will recover all but a few percent of their original thicknesses if allowed to age for several weeks. Some foams that have very high HACS values when measured in the specified recovery time, e.g. 30% set or greater, will partially recover but the residual sets even after long ageing periods may still be significant,

For example:

Block #	HACS*, % 30 minutes	(J2 Conditions) 11 days
27	26	6
40H	37	8
50H	34	6
70L	10	2
80L	8	2

*HACS measured using GMW14357E procedure where specimens are dried for 3 hours @70°C instead of at 100°C as per ASTMD3574E.

Thus it is obvious that compression set of foams treated in an autoclave/dried/reconditioned before clamping is a non-permanent phenomenon. Specimen thickness recovery progresses rapidly in the first few hours after stress release and then more slowly with increasing time until equilibrium thickness is reached. The value of this extended recovery time thickness is governed by the initial set value measured after the standard time interval. The asymptotic set values measured after weeks of recovery under standard conditions are considered to be due to irrecoverable damage that occurs in the foam structure during compression. For example, polymer struts might break during compression or covalent bonds might rupture or the foam structure may take up a new equilibrium state via plastic deformation while under stress/elevated temperature.

Although it has been reported that little set recovery occurs after 24 hours or even a week for wet set specimens (8), it is not known if long term ambient ageing allows specimens to recover thickness and to what extent. One of the purposes of this investigation was to determine the long term recovery behaviour of typical HR seating foams after they had been subjected to Wet Set treatment in an environmental chamber or pressure-treated in an autoclave specifically under J2 conditions.

TEST SPECIMENS

In order to eliminate the contribution of any residual "ageing-cure" at ambient temperature, foams that had been stored for over three years, either under laboratory conditions or in a clean storage room (reasonable degree of temperature control) were selected for testing. These HR moulded foam blocks (380 x 380 x 100 mm) had been poured on a commercial production moulding line, cured for about 8 minutes, crushed via progressive nip rollers and carefully stored as indicated. After several weeks of storage, the blocks were tested for hardness and selected physical properties. The data obtained at that time is shown in Table 1. At each core density except for the foam at 27 kg/m^3, two polymer polyol solids levels are reported as L=low and H= high levels. The exact solids levels vary slightly from density to density but are approximately, L = 5 %, H = 21% based on the resultant foam composition.

Table 1

Block #	Core Density, kg/m3	Hysteresis Loss, %	Wet Set, %	Resiliency, %	Repeat Wet Set, %
27	24	30	--	--	32
30L	31	29	27	56	23
30H	29	33	30	55	28
40L	39	25	16	60	17
40H	39	41	31	49	27
50L	53	22	12	63	10
50H	52	39	24	50	26
60L	63	22	8	64	9
60H	62	29	10	57	13
70L	73	20	4	63	5
70H	73	32	7	54	9
80L	83	19	3	63	4
80H	84	25	4	59	5

Table 1 shows that at each density, the hysteresis loss (HL) values increase with increased solids content. This is in agreement with other findings in the literature. For the lower solids foam (marked with an "L"), the HL values found decrease with increasing densities. Wet Set values decrease quite significantly with increasing foam density but it should be observed that at all densities, the H-foams have higher wet sets than the L-foams. Resiliency values range between 49 and 64%. At each density, the higher solids foams have lower resiliencies.

In order to determine if the three year storage period had any influence on these foams, the wet sets were re-measured and the results appear in the last column of Table 1. When these repeat Wet Set values are plotted against the original set values, see Figure 1, the correlation coefficient, R^2, is 0.9641, indicating excellent agreement between the original and repeat sets. These wet sets plotted versus foam core density exhibit reasonably good correlation coefficients for low (L) and high (H) solids foams, Figure 2. Therefore it was concluded that these foams had retained their original set properties during long term storage. This is in agreement with some earlier data that compared a range of physical properties (but not wet set) for moulded cushions that were used in a police fleet study (14). The foams listed in Table 1 have been used in our humidification study.

Fig. 1 Comparison of Original and New Wet set values

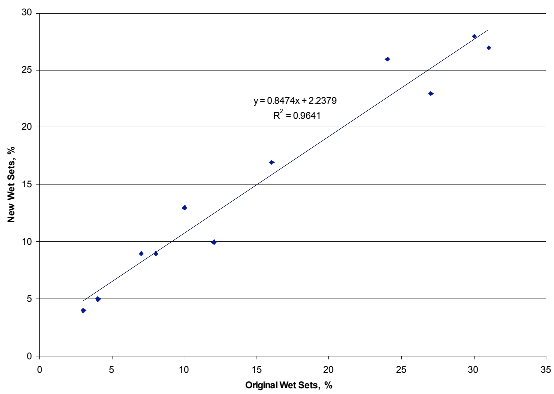
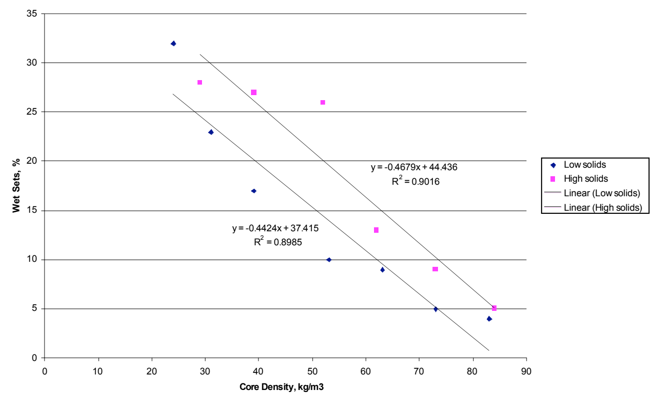


Fig. 2 Wet Sets versus Core Density



AUTOCLAVE HUMDIFICATION

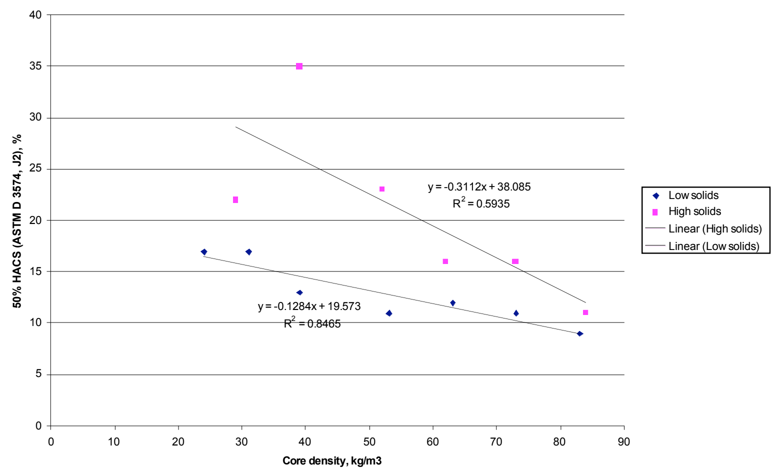
Humid aged compression set (HACS) should properly be termed compression set after humid ageing in an autoclave. To fully describe the pretreatment, the correct terminology should be autoclave-humid ageing/oven drying prior to compression set determination. In deference to previous work, we shall call this type of set, HACS. We used the procedure specified in ASTM D3574-08, test D, J2, i.e. 50% deflection compression set after autoclaving for 5 hours at 120°C under a pressure of 35 kPa. Specimen drying and reconditioning was exactly as outlined in ASTM D3574. The HACS values found are shown in Table 2. These HACS values are expressed as a function of specimen thickness.

Table 2

Block #	Core Density, kg/m^3	ASTM D3574, J2 HACS, %
27	24	17
30l	31	17
30H	29	22
40L	39	13
40H	39	35
50L	53	11
50H	52	23
60L	63	12
60H	62	16
70L	73	11
70H	73	16
80L	83	9
80H	84	11

These data show the same trends as the Wet Set values, i.e. HACS values decrease with increasing foam density and higher solids foams have worse sets than lower solids foams. If these HACS values for low (L) and high (H) solids foams are plotted versus core densities, Figure 3, the correlations are not as good as found for Wet Set data. This is due to the wider scatter in data found for HACS although the specimens were all processed at the same time in a certified autoclave and convection oven (ISO 17025- accredited equipment, operators and laboratory).

Fig.3 50% HACS (ASTM D3574, J2) versus Foam core density



COMPRESSION SET RECOVERY

Both Wet and humid aged specimens are measured 30-40 minutes after they have been allowed to recondition in the laboratory (23 ± 2°C, 50 ± 5% RH). That is the requirement for these set tests. Since it was known that HACS specimens normally increase in thickness (height) after longer conditioning periods, we decided to check both sets of specimens at known intervals to determine thickness recovery if any. Specimens were stored in a mesh tray-basket in the conditioned laboratory and thicknesses remeasured at convenient intervals. Thus the new set values could be calculated and compared with the standard (i.e. 30-40 minutes recovery) values. The data sets are found in Table 3.

Table 3

	WET SET VALUES, %						ASTM D3574 HACS, %					
Block #	30 min	10 days	14 days	33 days	50 days	81 days	30 min	7 days	14 days	33 days	50 days	80 days
27	32	25	24	22	21	20	17	7	4	3	4	3
30L	23	18	18	17	17	17	17	5	4	5	6	5
30H	28	22	22	20	20	20	22	6	5	5	6	6
40L	17	14	13	12	13	12	13	3	2	3	3	3
40H	27	24	24	23	23	22	34	6	5	6	6	5
50L	10	9	9	8	8	8	11	2	2	2	3	2
50H	26	22	21	20	20	19	22	4	3	4	4	4
60L	9	8	8	8	9	9	12	2	2	3	3	3
60H	13	12	12	11	12	12	15	3	2	3	3	3
70L	5	5	5	5	6	6	11	3	2	3	3	3
70H	9	8	8	8	9	9	15	3	3	3	3	4
80L	4	4	4	3	4	4	9	2	1	3	3	3
80H	5	5	5	5	5	6	10	2	2	2	3	2

For both sets of data, foam recovery (height increase) was found. However, there is a clear difference in the data sets. Wet sets show some recovery after 10 days reconditioning and very little change in set values after that. HACS values show significant change after seven days reconditioning and even more recovery (low set values) after fourteen days, especially for the lower density foams (27-30 kg/m^3). This may be seen in Figure 4 where some of the sets for these foams are plotted versus recovery time. Two each of the low (L) and high (H) solids foams can be compared in this figure. Wet set recovery is greater for lower density foams (30 and 40 kg/m^3) but quite small for high density foams, (60 and 70 kg m^3) i.e. after 10 days reconditioning the four foams shown in Figure 4 exhibit the following changes in set:

Fig. 4 Wet set and HACS Recovery versus Time

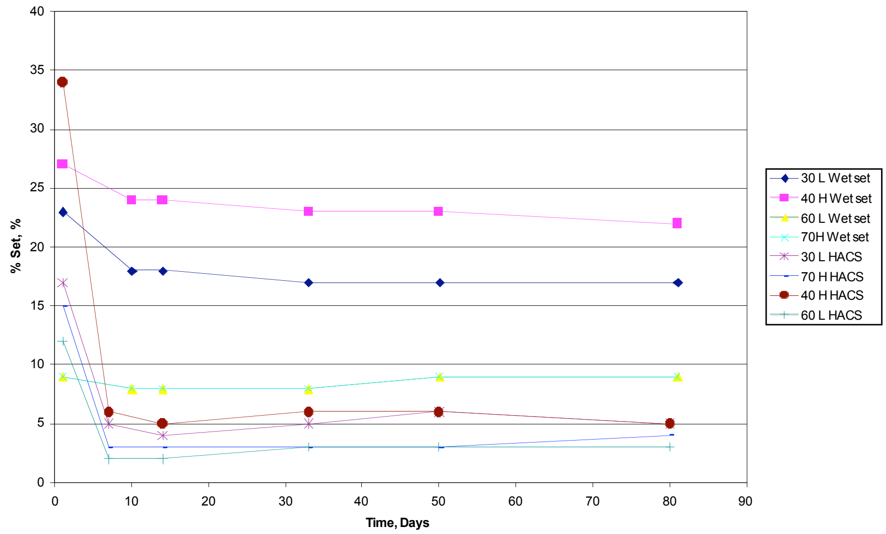


Table 4

Foam Type	30L	40H	60L	70H
Wet Set change	5	3	1	0.3
HACS change	12	28	10	8.5

Table 4 includes the much larger changes shown for HACS values. High density foams exhibit smaller changes in HACS.

It is apparent that the HACS phenomenon and the rapid and significant recovery that takes place in a few days, e.g. 7 days, indicates that whatever causes HACS to occur must be quite reversible even under ambient conditions. On the other hand, wet set while showing some recovery over the same time frame is relatively static in value after longer periods.

Since wet sets occur due to the foam being compressed under humidification, the “bounce-back” shown after a short recovery period may be due to elastic forces in the struts causing these struts to return partially to their original state. The portion of the wet set that does not recover even after long reconditioning periods is most likely due to plastic deformation of the struts under the compressive force and promoted by the elevated temperature (50°C) and high humidity (95% RH).

MOISTURE PICK-UP DURING HUMIDIFICATION

Specimens were either autoclaved (5 hours at 120°C, 35 kPa) and dried, 3 hours at 100°C or compressed by 50% and treated in an environmental chamber for 22 hours at 50°C, 95 %RH (as per Wet Set procedure). Before treatment specimens were weighed using a four decimal place electronic balance (normally used for weighing density specimens). The autoclaved/dried specimens were reconditioned overnight and reweighed whereas the specimens from the chamber were weighed 30 minutes after removal and release from the compressed state. All foam grades have been tested but we will report the same four grades that were potted in Figure 4. After reweighing specimens were stored in the laboratory under standard conditions and then reweighed. The mass changes found were:

Table 5

Block Type	Mass changes, %			
	Wet Set Conditions		ASTM D3574, J2 conditions	
	30 mins	33 days	Overnight	48 days
30L	+0.476	-0.29	-1.878	-1.06
40H	+0.545	-0.28	-0.804	-0.88
60L	+0.661	-0.35	-1.030	-1.17
70H	+0.652	-0.32	-0.877	-1.00

As expected the wet set conditioned specimens exhibited a mass increase averaging about +0.6%. The autoclave treated specimens all lost mass averaging about -0.9%. This magnitude of mass loss had been reported previously and was attributed to some material in the foam being leached out of structure in the autoclave and then removed by evaporation during drying (7).

Both sets of specimens were reconditioned for long periods in the laboratory and reweighed. Enviro-aged specimens now show a mass loss of about -0.3% and the autoclaved specimens an average mass loss of -1.0%. Thus the enviro-aged specimens on reconditioning lost the moisture picked-up in the chamber plus some material loss from the foams resulting in masses lower than the originals. The autoclaved specimens after reconditioning for 48 days had an average mass loss of 1%, a slight increase of about 0.1% over the average mass measured earlier (-0.9%).

These mass determinations appear to indicate that in both humidification treatments some mass is removed from these foams. The amount removed on enviro-ageing in a chamber is less than one third of the mass removed during autoclaving/drying. Since both humidification processes are essentially at the same relative humidity (95-100%), the mass loss differences must be attributed to temperature, time duration of humidification and treatment pressure of 34 kPa. Thus, foams treated for 22 hours at 50°C in the compressed state (50% deflection) do not lose as much material as foams pressurized for 5 hours at 120°C. Presumably the combination of higher processing temperature and elevated pressure forces more moisture into the foam struts and this promotes the solubilising of non-crosslinked material and this material evaporates with the moisture during convection-oven drying.

Specimens compressed by 50% of their original thickness will “increase” in apparent density by a factor of two. So specimens of 30 and 80 kg/m^3, which have about 2.6 and 7% polymer volume respectively (assuming solid polyurethane has a density of 1150 kg/m^3) will increase to 5.2 and 14% by volume respectively. Thus there is still a large amount of air volume in the compressed specimens to allow moisture to penetrate and hydrogen-bond with the foam hard segments during the relatively long humidification period of 22 hours.

At this time, we cannot explain why foams with such a wide range of density (30 to 70 kg/m^3) should pick-up the same amount of moisture on enviro-ageing or lose the same amount on long term ageing. Similarly on autoclaving these same foams, they lose the same mass approximately and have similar mass losses after ageing.

Recall that these same foams had very different wet sets and HACS values after 30 minutes removal from compression, Figure 4. The wet set recovery values, Table 3, indicate greater recovery for the 30L foam than for the higher densities and the 70H foam had almost no recovery set at all. Yet these foams picked up the same amount of moisture and lost about the same amount of ageing. Similarly, the autoclaved specimens showed wide differences in HACS recovery but almost the same mass losses after autoclaving/drying, Table 3. Thus the loss of mass for each sub-set does not seem to explain the differences in either wet set or HACS recoveries. However, the difference in mass loss between enviro-aged specimens and autoclaved/dried specimens after reconditioning, i.e. average losses of approximately 0.3 and 1.0% respectively may account for the significant differences in wet set and HACS recovery on reconditioning.

In Figure 4, it is obvious that HACS (compression set after autoclaving/drying) is a much more unstable condition than wet set. The latter sets do show some recovery (improvement) after 10 days of reconditioning but little or zero recovery after that period. As foam density is increased the extent of recovery decreases and lower polymer solid foams recover less than the higher polymer solids foams of the same density.

All specimens that were autoclaved/dried recovered substantial thickness in less than 10 days (7 days was the shortest recovery period examined in this study) as shown in Table 6.

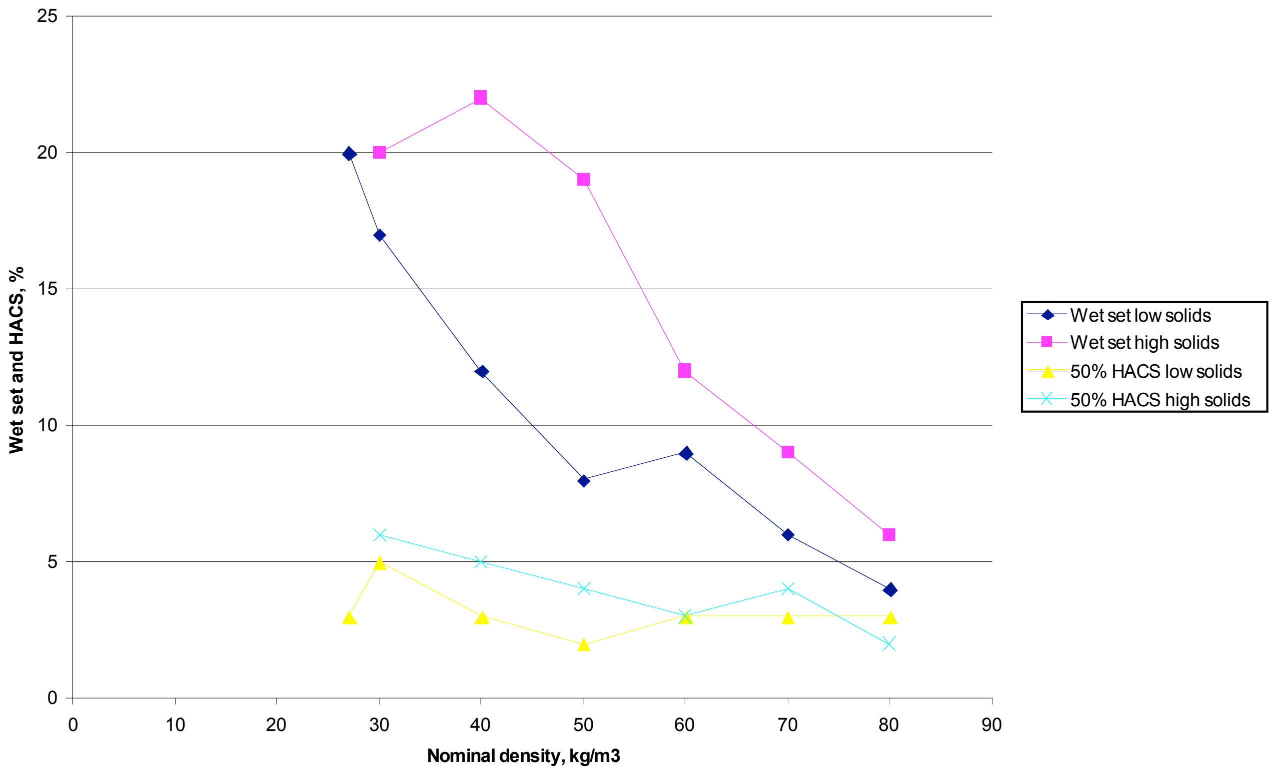
Table 6

Block type	27	30L	30H	40L	40H	50L	50H	60L	60H	70L	70H	80L	80H
HACS recovery in 7 days, %	10	12	16	10	28	8	18	9	12	8	12	7	8

Once again these set recoveries decrease with increasing density and lower solids foams have lower recovery values than higher polymer solids foams. The very large difference between the recovery values for the 40 kg/m^3 foams was considered abnormal. When the higher solids foam was visually examined, it had a more irregular structure than all of the other foams in the series in that it had zones of coarse and fine cell structures thus indicating some mixing problem during manufacture. Thus the data from this foam is suspect and must be treated with reserve.

After about 80 days reconditioning, the set values may be considered to have stabilized, see Table 3. The data is plotted in Figure 5 where it can be seen that all HACS values are 6% or lower. If we eliminate the problematic 40H foam, all HACS lie between 2 and 5%. At 50 kg/m^3 and greater, the range is 2 to 4% with an average value of about 3%. These low values may be considered to be the “residual” HACS and further ageing will not result in improved (i.e. lower) values.

Fig. 5 Wet set and HACS after 80 days recovery



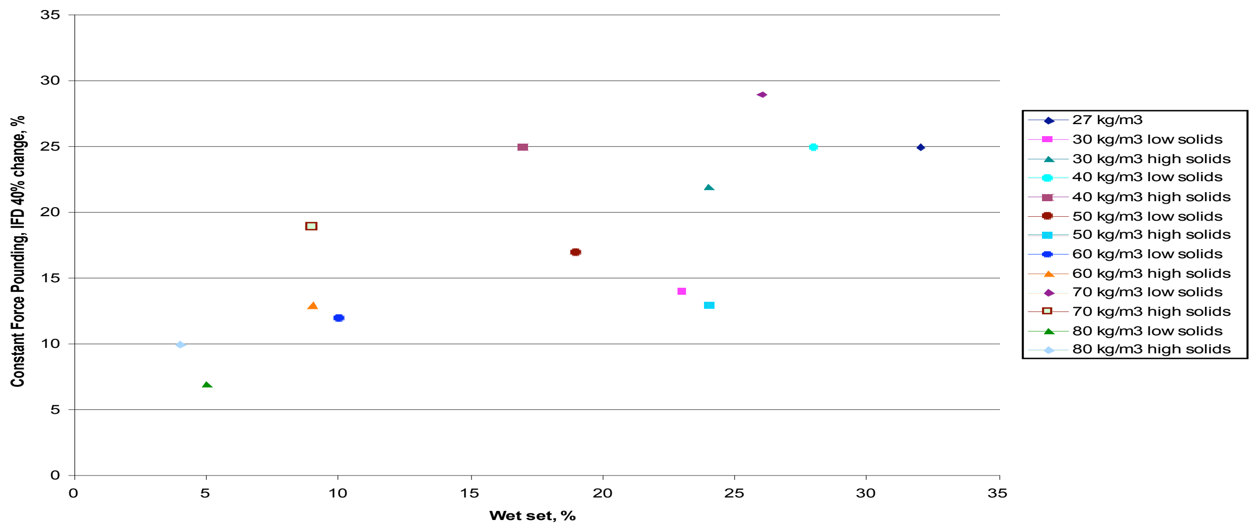
Wet set values remain substantially higher than the HACS values except at 70-80 kg/m^3 density. There is a wide separation between the set values for the low and high solids content foams especially at lower densities. Since the values plotted in Figure 5 are almost the same as the sets obtained after shorter recovery times 14, 33 or 50 days. We believe that these 80 day values can be considered as the "residual" wet set values for these foams.

CONSTANT FORCE POUNDING (CFP)

In a previous report (15) it was indicated that there was a fairly good relationship between the extent of hardness loss (IFD change at 40% deflection) after pounding for the standard 80K cycles as called for in ASTM D3574-08, I3 and Wet compression Set values, i.e. the greater the change in IFD 40% def. after pounding, the greater the percentage of Wet Set found. The correlation between hardness loss and wet set was approximately linear, although as might be expected when comparing such widely differing test methodologies, there was some scatter in the results. However, as was indicated earlier, if there is indeed a useable correlation between these properties, it would prove to be advantageous. Also, earlier work (14) has shown that the hardness loss after pounding (80K cycles) correlated with actual cushion performance in police vehicles. The advantage of the Wet Set test is that it is rapid and easy to perform. An ILS (InterLaboratory Study) or round robin for Wet Set, between ISO 17025-certified and non-certified laboratories has shown good reproducibility/repeatability using the ASTM E 691 evaluation procedure. Thus our confidence in the Wet Set test is strong. Although Wet Set, in common with all compression set tests is an overnight test, it has the advantage over a pounding test in that many specimens of various foam formulations and/or densities can be performed simultaneously whereas only one pounding test can be done within the same time frame. Also the Wet Set test does not require any specially-designed equipment but an environmental chamber, commonly available in a well equipped laboratory, should be used. Alternatively it is possible to place compressed specimens in a closed container such as a desiccator containing water and to heat it to 50°C in an oven but the chamber method is recommended.

Constant Force Pounding results for the foams listed in Table 1 plus a few others of intermediate polymer solids levels at nominally 30, 40 and 50 kg/m^3 densities are found in Table 7. Also included in this table are the Wet Set values for these foams. Plotting IFD 40% deflection change versus Wet Set produces Figure 6. There is a fair amount of scatter in the relationship but it can be seen that greater Wet Sets predict higher hardness changes after pounding. This relationship is useful in that, if foam has a very high wet set; it predicts that constant force pounding will cause severe hardness loss and usually high losses in thickness as well.

Fig. 6 Constant Force Pounding; IFD 40% change (loss) versus Wet set



CFP hardness changes are dependent on foam density as shown in Table 7. Some of the scatter in data is due to these foams having different percent solids contents which were varied between 5 and 21% in the finished foam.

Table 7

Constant Force Pounding and Wet Set Data

Block #	Constant Force Pounding % Thickness Change	Constant Force Pounding % IFD Change	Wet Set, %
27	3	25	32
30L	3	14	23
30I	3	22	24
30H	3	25	28
40L	3	25	17
40I	1	17	19
40H	5	13	24
50L	2	12	10,6
50I	0	13	9
50H	9	29	26
60L	3	19	9
70L	2	7	5
80L	1	10	4

N.B. Blocks with an "I" designation following the nominal density value have intermediate percent solids levels.

FOAM SWELLING IN FABRIC CLEANER FLUID

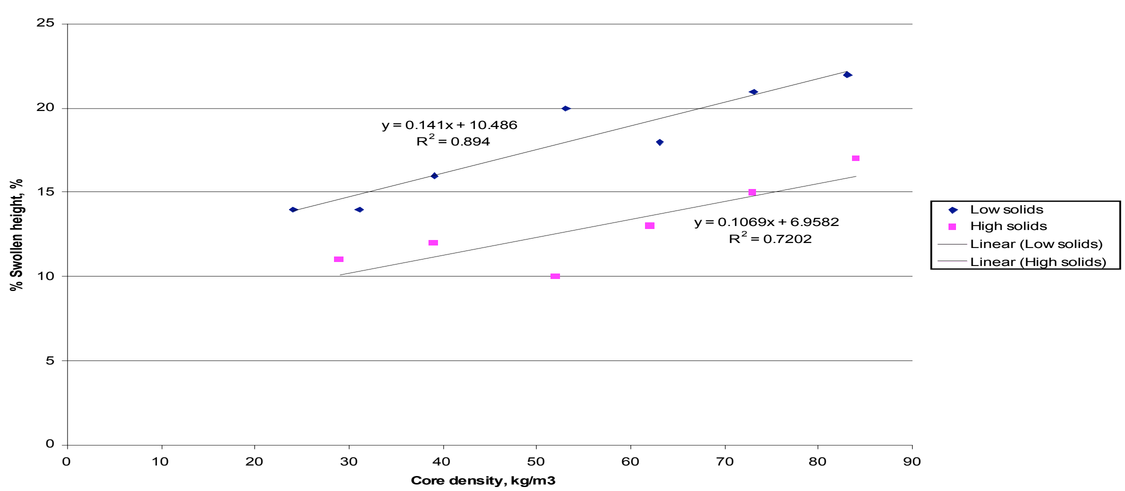
As a means of understanding how these foams behave in the CFP and Wet Set test, we have subjected them to a swelling test in a solvent-type fluid. This immersion normally allows the foam to swell and will give an indication of the degree of cross-linking of the polymer in the struts. The greater the amount of cross-linking, the less the foam should swell on immersion.

Specimens 50 x 50 x 25 mm were measured for thickness using a Mitutoyo height gauge. They were then completely immersed overnight in a bath containing a fabric cleaner, MS-3185 which is a mixture of petroleum distillates (mostly consisting of light and medium naphthas and 1,2,4-trimethylebenzene). Specimens were then removed from the cleaner and their swollen thicknesses measured rapidly using callipers reading to the second decimal place. The percentage increase in thickness was calculated and is reported in Table 8. After squeezing most of the fluid out of the specimens they were dried in an air-circulating oven for 96 hours at 105°C. The dried thicknesses were then measured using the Mitutoyo height gauge and the dried percentage thicknesses calculated. The swollen thickness data is plotted versus foam core densities in Figure 7.

Table 8

Block #	Percentage Swollen Thickness	Percentage Dried Thickness
27	14.3	2.4
30L	13.6	0.8
30H	11.4	1.8
40L	16.2	0.2
40H	12.0	1.1
50L	20.0	-0.1
50H	9.5	0.3
60L	17.8	-0.1
60H	13.2	0.5
70L	21.2	-0.1
70H	15.0	0.4
80L	22.0	-0.5
80H	16.7	0

Fig. 7 Foam percentage thickness swell in organic solvent versus foam core density



The percentage of thickness swelling increases with increasing foam density and the lower solids containing foams (L=low solids) swell more than those foams with higher (H) solids. The increase in swollen thickness with increasing density can be attributed to the lower crosslink density of the denser foams (since lower amounts of water and isocyanate were used to make these foams, they have a lower urea content). It is uncertain at this time why the foams with higher solids contents have lower percentage swelling than those at the same densities with lower solids contents.

After drying the thickness trend is reversed in that the lowest density foams exhibit the greatest residual swelling but the extent of residual swell is only about 2%. For foam densities of 50 kg/m^3 and above, the extent of residual swell/shrinkage is about zero, so the specimens have essentially returned to their original thicknesses. It is surprising that the lower density foams which do not swell as much as their higher density ones do not return to their original thicknesses. This may indicate that swelling in this fluid causes some physiochemical change in the struts which does not completely reverse on drying.

The data shown in Table 9 indicates that percentage thickness swell decreases both with the percent polymer solids content increase and the calculated theoretical percentage of ureas in these foams. The latter relationship is understandable as the urea contents represent the crosslinked portion of the resultant foams. Our intuition is that the higher solids containing foams would swell to a greater extent than those with lower solids but the reverse was found. Future work may elucidate the reason for this unexpected phenomenon.

Table 9

% Foam Swollen Thickness and Polymer Solids/Urea Contents

Block #	% Swollen Thickness	% Polymer Solids	% Urea Content
27	14.3	4.2	21.5
30L	13.6	5.3	19.0
30H	11.4	12.0	19.2
40L	16.2	5.4	17.1
40H	12.0	22.0	17.3
50L	20.0	5.7	13.7
50H	9.5	26.7	13.7
60L	17.8	15.2	11.4
60H	13.2	26.8	11.4
70L	21.2	11.0	10.0
70H	15.0	24.2	10.0
80L	22.0	10.2	9.5
80H	16.7	18.7	9.3

Foams that exhibit more swelling in solvent show lower hardness losses after pounding. Higher amounts of swelling indicate lower crosslinked structures which are more flexible and are able to withstand the repeated compression and recovery experienced in pounding for 80K cycles. In other words, the foams that swell more have higher resiliency ("bounciness") and thus will flex more easily during pounding, less strut damage will occur and less IFD loss will result. Therefore, foam swelling indicates the 'inherent resiliency' of foams and gives a good indication how they will behave in a pounding test.

A swelling test is yet another simple-to-perform procedure that can be used to rapidly predict the pounding behaviour of foams. All swelling tests using a flammable solvent such as MS-3185 should be performed in a laboratory fume hood using recommended safety equipment, etc.

SUMMARY AND CONCLUSIONS

The Wet Compression Set test provides data that indicates how foams will perform in a vehicle under tropical conditions. Since foam specimens are held in compression while being exposed to tropical conditions for a relatively long period of time, i.e. 22 hours and not subjected to elevated pressure conditions such as occurs in an autoclave, we believe Wet Set is a more realistic test than autoclaving. Thus we recommend this humidification treatment and encourage its adoption to replace all HACS tests, i.e. compression sets after autoclaving and oven drying.

Wet Sets can be used to rapidly evaluate a large number of foam types simultaneously. Since Wet Set values have been shown to be correlated with the hardness loss that takes place after Constant Force Pounding, it can be used to predict these losses. If the Wet Set values obtained are high, e.g. 30% or greater, it is reasonably certain that high pounding losses will occur.

Foam swelling in a solvent is also a useful predictor of pounding behaviour. Foams that swell considerably in a solvent can be considered to have a degree of 'inherent resiliency' that will allow them to flex in the pounding test with acceptable losses in foam thickness and hardness.

The Wet Set test has been shown to give set data that only partially recovers on long term ageing under ambient conditions. Specimens subjected to the HACS cycle rapidly recovery thickness and within about two weeks have almost regained their original dimensions. Thus the HACS values approach zero values. Since the Wet Set test produces more stable sets, we recommend it as the preferred humidification treatment especially for automotive foams.

REFERENCES

1. G.R. Blair, J. Reynolds and M. Weierstall, "Automotive Cushioning through the Ages", available on the www.moldedfoam-IP.org web site, 2008.

2. R. Pask, G.R. Blair and M. Weierstall, CPI conference 2008, September, San Antonio, Texas.

3. G.R. Blair, A. Russ, D.E. Bradt and R.J. Bailey, Polyurethanes Expo 2001, Columbus, Ohio, September 30-October 3, pp 285-308.

4. K. Saotome, K. Matsurbara and T. Yatomi, J. Cellular Plastics, 1977 May/June, vol. 13, pp 203-208.

5. R. Brasington and H. DeRoeck, Proceedings of the PU Conference 2000, October 8-11, Boston, Mass. pp 267-278.

6. R. Volland and H.M. Rothermel, SAE Technical Paper 830486, 1983 February 28-March 4, Detroit, Michigan.

7. G.R. Blair, R. Pask, M. Weierstall, R. Dawe, J. McEvoy and M. Rusan de Priamus, CPI Conference, 2008 September.

8. www.moldedfoam-IP.org, internal document, available on request.

9. Chrysler LLP Specification MS-DC649.

10. Ford Motor Company Specification, WSB-M1520-B1/B2.

11. General Motors Specification, GMW15471.

12. D.V. Douris, G. Wilkes and R. Turner, J. Applied Polymer Science, volume 50, 1993, pp 293-301.

13. R.M. Herrington and D.L. Klarfeld, J. Cellular Plastics, 1983, pp 177-182.

14. G.R. Blair and R.J. Horn, Utech Conference 1996 March 26-28, The Hague, The Netherlands, Paper 5.

15. G.R. Blair, M. Weierstall, J. McEvoy and A. Ali, CPI Conference 2006, pp 526-542.

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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.