Investigation of the Compression Recovery Properties of Polyamide-6 Cellular Solid over the Temperature Range of -5°C to 90°C

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Abstract: It is a fundamental response of any polymeric foam material to undergo non-recoverable deformation following the application of a defined compressive strain, exacerbated by temperature and humidity. This process is commonly referred to as compression set. The ability to predict recovery after the application of a compressive strain is crucial to both the manufacturers and end users of foam materials. Specific compression set test procedures have been established to quantify the extent of non-recoverable deformation in specific foam types but to date no general predictive approach exists. In this work, compression set (fixed strain) tests were undertaken on a cellular polyamide-6 material at various temperatures (-5°C to 90°C) and the foam recovery monitored over time periods in excess of those dictated by standard methods (ISO 1856 [1]). An empirical formula has been proposed to allow the prediction of recovery after compressive strain, covering recovery periods from 10 minutes to 24 hours (up to 168 hours at 23°C).

Introduction

Zotefoams plc is a world leader in the manufacture and distribution of speciality foam materials, manufactured using a unique, proprietary technology. The technology uses nitrogen gas as a physical blowing agent (PBA) which is dissolved into the polymer in a high pressure/temperature process. Once equilibrium is achieved, a thermodynamic instability is introduced to initiate nucleation of the foam structure. This means of production minimises impurities in the product whilst also maximising properties and performance.

A further advantage of this process technology is the wide range of materials to which it can be applied. One such example is a newly developed foam based on polyamide-6 (PA6). For materials such as PA6, foaming has traditionally been an insurmountable technical challenge or otherwise limitations have existed to the reduction in density possible. The ZOTEK N B50 grade foam has a nominal density of 50kg/m^3, equivalent to a 23x volume expansion. The properties of the foam reflects the general properties of this class of polymer with high temperature performance, resistance to hydrocarbon fuels and oils combined with impact performance, buoyancy and low thermal conductivity from the cellular structure. The material is therefore considered ideal for energy absorption and cushioning applications in extreme environmental conditions.

Compression set is one of the simplest standard test procedures to undertake and gives reliable data on one of the foams basic mechanical responses; recovery after fixed strain compressive loading. In the literature there are many instances where compression set tests have been used to monitor the effects on materials following chemical or mechanical ageing or when process/formulation variations are being investigated, for example, the effects of blending two or more polymers [2-4], the effect of the addition of fillers [5], the effect of the level of cross-linking [6] and other aspects of foam ageing [7,8]. Little however has been reported on the data from the compression set test itself. In addition to this there is little reported literature on compression set data outside the scope defined by the most commonly used ISO 1856 and ASTM D345-03 standards. Often compression set data is

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recorded at a few points of recovery time only, and no predictive interpretation of the foam performance outside of the range of these measurements is available.

This study presents results on compression set data for a low density polyamide-6 foam over a range of compressive strain, temperature and recovery time. The paper then describes a predictive formula for the recovery response of the cellular polymer.

Experimental Procedure

All samples were taken from a single foam sheet to minimise the effects of density upon the results. All samples were conditioned for a minimum of 6 days at 23°C (±2°C) and 50% (±5%) relative humidity prior to test. All dimensional measurements were carried out in accordance with ISO 1923 [9] using an MID-F125/150 Mitutoyo Digimatic digital micrometer with an accuracy of ±0.006mm and fitted with an 8mm flat anvil. For mass determination, a Mettler AJ150 balance with an accuracy of ±0.001g was used. Density of the foam sheet was calculated according to ISO 845:1995 [10].

Compression set tests were conducted in accordance with ISO 1856 [1]. Three samples were tested for each test condition and the mean values reported. Nominal test sample size was 50mm×50mm×25mm. The dimensions of the ZOTEK N B50 samples were recorded and the samples were then compressed to a fixed strain (either 25% or 50% of original thickness) and conditioned at the test temperature (either at -5°C, 23°C or 90°C) for the duration of the 22 hrs.

On release of the fixed strain, the samples were maintained at the relevant test temperature. The dimensions of the samples were then re-measured at various time intervals from 10 min through to 24 hrs after release. For the samples tested at 23°C, time intervals were extended to 168 hrs. Recovery at temperature and such extended recovery periods are beyond the scope of the ISO 1856 [1] procedure.

All compression set (*c.s.*) data are reported according to ISO 1856 [1], using Eq. (1)

$$c.s. = \frac{d_o - d_r}{d_o} \times 100 \qquad (1)$$

where d_o is the original thickness of the sample and d_r is the thickness after recovery.

Results and Discussion

Table 1 gives relevant foam properties according to the manufacturers data sheet for the ZOTEK N B50 foam together with the mean results of this study tested under equivalent conditions. There is good agreement between the two data sources.

Table 1: Comparison of Experimental Results with Manufacturers Data

Parameter	Datasheet	Experimental
Density	52 kg/m^3	48.5 kg/m^3
c.s. (25%, 30 min, 23°C)	14 %	14.8 %
c.s. (25%, 1440 min, 23°C)	12 %	11.1 %
c.s. (50%, 30 min, 23°C)	29 %	28.6 %
c.s. (50%, 1440 min, 23°C)	24 %	23.5 %

Eq. (2) is the base predictive equation for compression set data as a function of time, for 25% and 50% initial fixed compressive strain.

$$c.s.(\%) = a\ln(t) + b \quad (10 \le t \le 1440) \tag{2}$$

where a and b are the temperature dependant coefficients, given in Table 2, and t is recovery time in minutes.

Table 2: Coefficients of Logarithmic Empirical Fitting Function

Test Temperature [°C]	Compressive Strain = 25%		R^2	Compressive Strain = 50%		R^2
	a	b		a	b	
90	-1.531	22.454	0.977	-1.422	33.842	0.982
23	-0.952	18.059	0.991	-1.321	33.083	0.988
-5	-1.084	17.408	0.963	-1.319	32.750	0.972

The data, when plotted, shows a high logarithmic dependency as a function of recovery time. This is confirmed by the coefficient of determinations (R^2) given in Table 2.

With reference to the 23°C data shown in Fig.1, this logarithmic time dependency appears to be maintained to a time-frame of at least 7 days irrespective of the initial fixed compressive strain.

There is generally a better correlation in the results from 50% initial fixed compressive strain tests than the 25% tests. This is possibly a result of permanent damage caused to the foam structure by the initial compression stage, thereby affecting the base polymer elasticity. The recovery in this case is thought to be dominated by lowering of the gas pressure within the cells, thus when the energy is dissipated during the gas expansion, the recovery slows. This is illustrated by the data in Fig.1 for all temperatures investigated.

This observation cannot be applied to the 25% initial fixed compressive strain data. As shown in Fig.1 this data is more widely scattered than that obtained from the 50% initial fixed compressive strain tests. It is postulated that this is due to the recovery being a combination of the expansion of the gas within the cells and the viscoelastic properties of the cell wall. Nonetheless the ordering of the series on the graph for all temperatures is consistent irrespective of initial fixed compressive strain level.
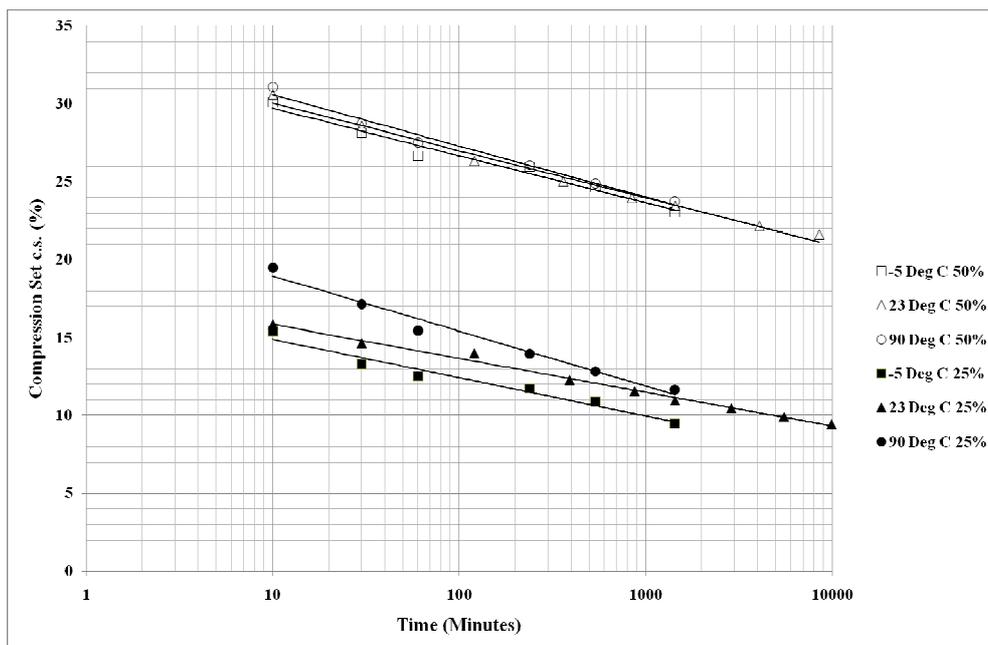

Fig.1: Experimental Data from Compression Set Tests Including Fitting Functions

Conclusions

The compression set of a low density polyamide-6 foam (ZOTEK N B50) material has been assessed over a range of fixed strain, test temperatures and recovery times.

As a function of time and at temperatures in the range -5°C to 90°C, the recovery behaviour appears to show a close correlation to a logarithmic function. The logarithmic correlation to the experimental data is statistically strong with coefficient of correlation, R^2, often higher than 0.97.

It is worth noting that:
a) The coefficients determined in this study are only directly applicable to the ZOTEK N B50 foam but it would be expected that similar recovery mechanisms/relationships exist in other foam types.
b) There is evidence that this logarithmic rule may be applicable over extended time periods.
c) There is evidence that the logarithmic function coefficients *a* and *b* are temperature dependent.

In all of these cases further experimental work would be required to confirm these observations.

Acknowledgements

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