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# Physical and thermal effects on the shape memory behaviour of auxetic open cell foams

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**Abstract** This study examines the processing envelope related to auxetic open cell foams and their shape memory properties, with the analysis of four different phases of multi-component foams (conventional, 1st auxetic, returned and 2nd auxetic). The analysis of the shape memory and its correlation with negative Poisson's ratio behaviour are a novelty in the field of auxetic materials. This study describes the differences between the multi-component foams used as precursors for each phase, exploring their mechanical and thermal characteristics at each stage of the conversion. The results show the important differences related to the mechanical behaviour of the foams, due essentially to the axial compression adopted during the manufacturing process.

## Introduction

Since 1987, when Lakes [1] manufactured a first sample of isotropic auxetic foam, researchers worldwide have drawn more attention on auxetic material (i.e. materials showing a negative Poisson's effect). The unusual kinematic deformation of auxetic materials has led to engineer new solutions for potential applications in structural integrity, sandwich components and passive smart structural devices [2–4]. In fact, auxetic materials show an enhanced mechanical properties compared to their conventional version, as indentation resistance [5], bending stiffness in structural elements and shear resistance [6] and high energy dissipation per unit volume under compressive cyclic fatigue loading [7, 8]. Howell et al. and Scarpa et al. [9, 10] measured the acoustic absorption of negative Poisson's ratio foams. Further measurements were performed by Scarpa et al. [11, 12] on auxetic foams doped with magnetorheological fluid (MRF) particles. Several authors investigated also the dynamic behaviour of auxetic foams [13–15] as well as the possible use of auxetic foams for viscoelastic damping applications [10, 16]. The influence of the manufacturing process over the cell reticulation, pore size and mechanical characteristics of the foams has been investigated in several papers [17, 18]. In [19], a study on a wide batch of different specimens of auxetic foams was carried out; pointing out how the compression ratio imposed during conversion was the most important parameter in the manufacturing process. The foams presented in this article have been manufactured using the technique presented in the latter reference. In [20], some of the authors have analysed for the first time the shape memory properties of auxetic open cell foams under thermal loading. The auxetic foams submitted to appropriate conditions recovered their original dimensions and then, the same specimens, were subsequently converted back again to an auxetic phase. Finally, four different phases of the same native foam were found and referred to 'native or conventional', '1st auxetic', 'returned' and '2nd auxetic'.

In this study, a detailed analysis on the mechanical, thermal and chemical properties of each of the samples described in [20] has been performed. Chemistry and thermal considerations were drawn after differential

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scanning calorimetry (DSC) tests performed on the native foams. In addition, specimens were mechanically tested, in each of the different phase, under both tensile and compressive loadings.

## Specimens preparation, methods and equipment

Native specimens were cut from conventional grey polyurethane (PU)-based open-cell PU (McMaster-Carr Co., Chicago, IL, USA) with 1181–1378 pores/m (30–35 pores/inc), and 27.2 kg/m$^3$ density and from a 27 kg/m$^3$ density conventional light blue open cell foams (SM Upholstery Ltd., Cardiff, UK) with 2047–2244 pores/m (52–57 pores/inc).

Twenty specimens, ten of each foam type, were used to perform this study. The native grey foam was provided in square blocks of 600 mm side length and thickness of 50 mm, whereas the light blue, cube block had 1000 mm side length. The 20 cylindrical specimens of 30 mm initial diameter and 2 different lengths (180 and 120 mm) were cut using a sharp-edged tube. Four batches of five identical specimens each were manufactured.

Since the foams retain their shape memory properties through out the manufacturing processes employed here, the specimens could consequently be converted into auxetic following a modification of the manufacturing process adopted in [21], reverted in the original shape and converted back again into auxetic. Four different families of foams were therefore manufactured and the specimens' mechanical properties were measured uniaxial tensile loading, as explained in [20]. The specimens were cut with two different lengths (batches A and D 180 mm, batches B and C 120 mm), and both subsequently compressed during processing into the auxetic phase until they reached a length of 60 mm. As there were two groups of specimens with different axial compressions during the conversion, four batches of samples were produced, having different characteristics in terms of foam type and processing parameters. All the specimens were subjected to the same volumetric compression ratio when converted into the 2nd auxetic phase. The general manufacturing procedure used to convert the specimens is also described in [19, 21]. All the specimens' manufacturing parameters are shown in Table 1.

A table top 10 kN testing machine (Shimadzu Autograph AGS, Milton Keynes, UK) equipped with a 50-N load cell was used for all mechanical quasi-static cyclical tension and compression tests applying a displacement rate of 20 mm/min at a frequency of 0.03 Hz (triangular waveform). All the specimens were tested in the conventional, 1st auxetic, returned and 2nd auxetic phases between 0 and 10% strain in tension and in compression (separate tests). Longitudinal and radial deformations were measured using an optical system Videoextensometer (Messphysik GmbH, Austria). Two temporary contrasting surface markers (white on the grey and black on the light blue specimens) were adhered to each specimen to allow the Videoextensometer to measure length data in the central part of the specimens away from the ends [20]. The longitudinal ($z$) and radial ($r$) strains, Poisson's ratio, tangent modulus and energy dissipation (hysteresis area) per cycle were calculated. Poisson's ratio $v_{zr}$ was calculated as the negative ratio between the radial and longitudinal strains via a best fit to the strain–strain data. Since the linear stress–strain response of such samples is known to remain up to 25% strain in both conventional and auxetic foams [19], values of tangent modulus, $E$, were calculated as the best fit slope of the linear region of the stress–strain ($\sigma_z$–$\varepsilon_z$) curve between 0 and 10% longitudinal strain. The dissipation of energy in each specimen was calculated from the hysteresis area in the force–deflection data by approximating the sides of each force–deflection curve with 2-s degree polynomial curves [19].

DSC was performed on a TA Instruments Q100 Calorimeter. Three consecutive cyclic scans were acquired between −75 and 200 °C at a heating/cooling rate of 10 °C/min.

**Table 1** Manufacturing features of each specimen; each batch is composed of five identical specimens

| Batch | Conventional foam | | First auxetic stage | | | | Second auxetic stage | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Diameter | Type | Compression ratio | | Temp. (°C) | Cooling method | Compression ratio | | Temp. (°C) | Cooling method |
| | | | Radial | Axial | | | Radial | Axial | | |
| A | 30 | Grey thermoplastic PU foam | 1.58 | 3 | 135 | Water | 1.58 | 3 | 135 | Water |
| B | 30 | | 1.58 | 2 | 135 | Water | 1.58 | 2 | 135 | Water |
| C | 30 | Light blue thermosetting PU foam | 1.58 | 2 | 135 | Water | 1.58 | 2 | 135 | Water |
| D | 30 | | 1.58 | 3 | 135 | Water | 1.58 | 3 | 135 | Water |

## Results

### Thermal analysis

The thermal properties of the two PU-based foams under investigation were examined by DSC in the "native", "1st auxetic", "returned" and "2nd auxetic" states.

No specific information about the chemical composition of the grey foam were supplied by McMaster-Carr Co., whereas the synthetic protocol used to obtain the light blue foam was supplied by the manufacturer and was consistent with the preparation of thermoplastic polyurethane (TPU). This latter kind of foam (SM Upholstery Ltd., with 27 kg/m$^3$ density) was obtained by the step copolymerization

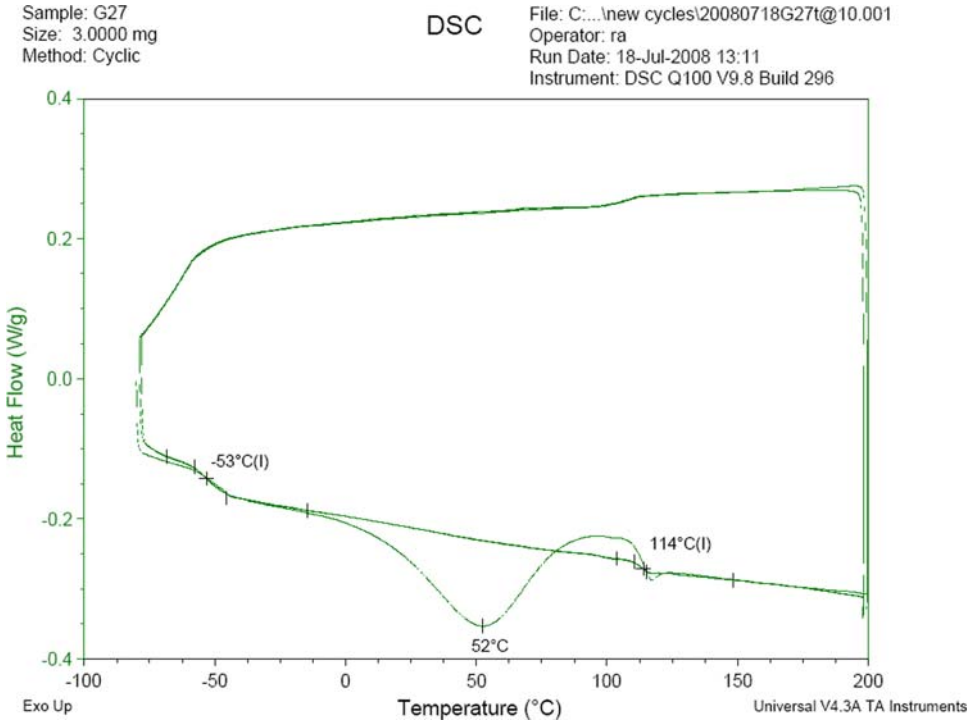


**Fig. 1** DSC experimental results for the grey PU-based foam

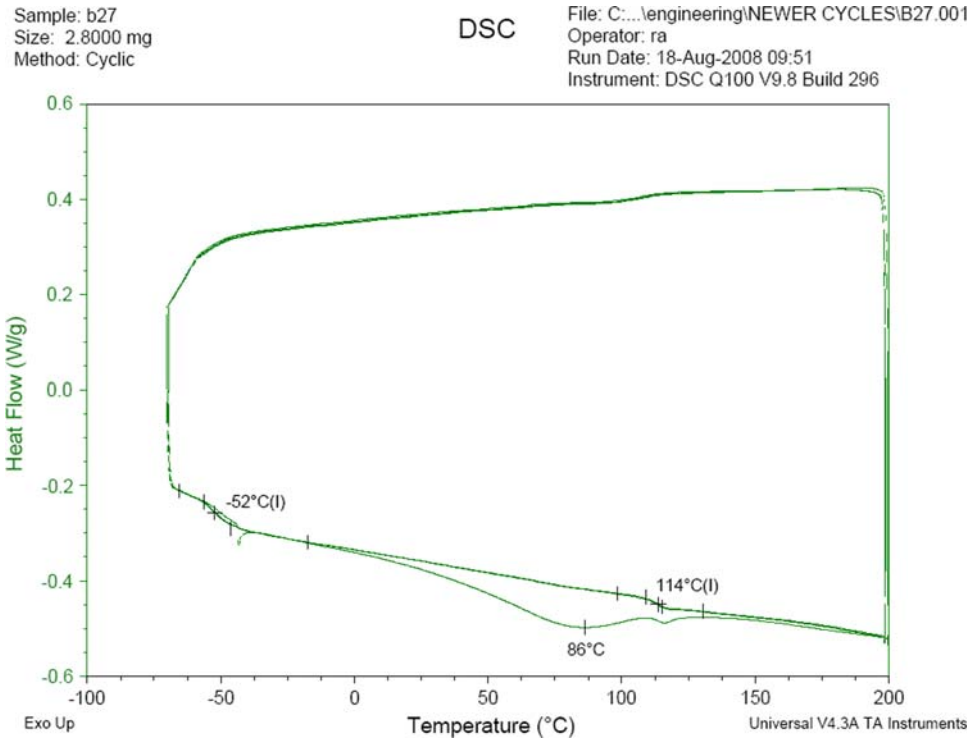


**Fig. 2** DSC experimental results for the light blue PU-based foam

of a modified polyol and a polyether polyol with toluene di-isocyanate in the presence of various catalysts and surfactants. These materials are multiblock copolymers consisting of a long soft block, which in this case is a polyether, and a short hard PU segment. The two blocks are incompatible and microphase separation occurs to afford hard, crystalline domains within a soft matrix. It is these hard phases that act as thermoreversible physical crosslinks that allow for melt processing [22]. It is in this sense that TPUs differ from chemically cross-linked elastomers.

In all the DSC experiments, the first cycle was used to erase the thermal history of the sample and this, consequently, exhibits different features to the subsequent two cycles as shown in Figs. 1 and 2. No significant difference was observed between the latter cycles in any of the experiments and as a result we will discuss the second cycle. On heating the "native" blue foam from −75 °C (Fig. 2), a second-order phase transition was observed at −52 °C (−53 °C for the grey type as shown in Fig. 1). Further heating affords no additional phase change until 114 °C, where another second-order transition is observed.

A comparison of Figs. 1 and 2 indicates no significant difference between the thermal properties of the two foams.

## Mechanical analysis

Figures 3 and 4 show the behaviour of the Poisson's ratio versus the tangent modulus for all the specimens produced in this study. Figure 3a illustrates the comparison between batches A and B (axial compression ratios of 3:1 and 2:1, respectively), highlighting the differences due to a different axial compression during the manufacturing. Conversely, Fig. 3b compares the results gathered from batches A and D, which present similar manufacturing characteristics, but are made up from two different native foams. Similar considerations can be made in Fig. 4a, b, where the comparisons between batches C, B and D (blue versus grey foams with both 2:1 compression ratio and grey foams with

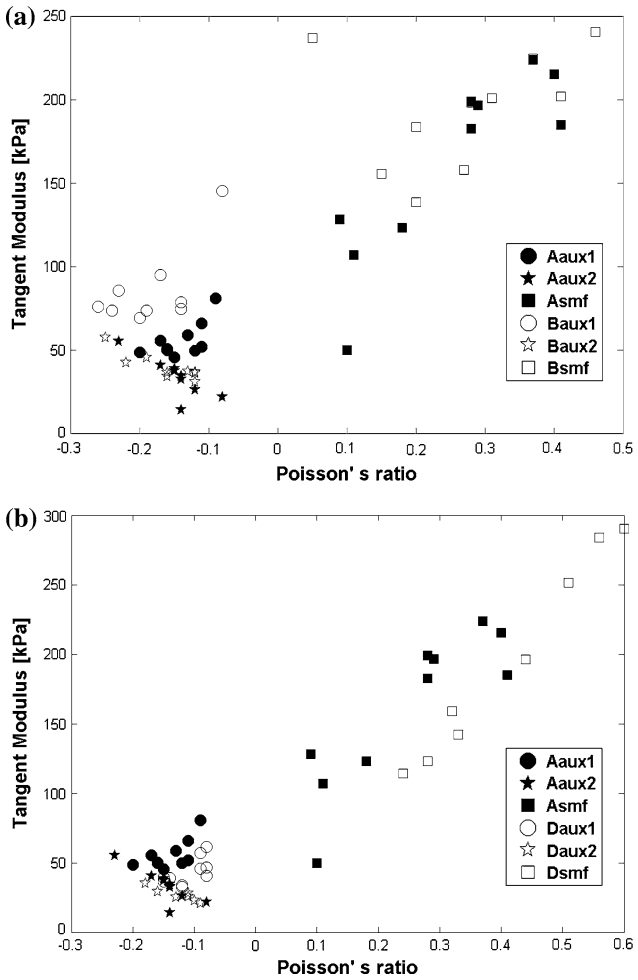


**Fig. 3** Tangent modulus versus Poisson's ratio resulted from compressive and tensile loadings in the 1st and 2nd auxetic (aux1 and aux2) and returned (smf) phases of batches A–B (**a**) and A–D (**b**)

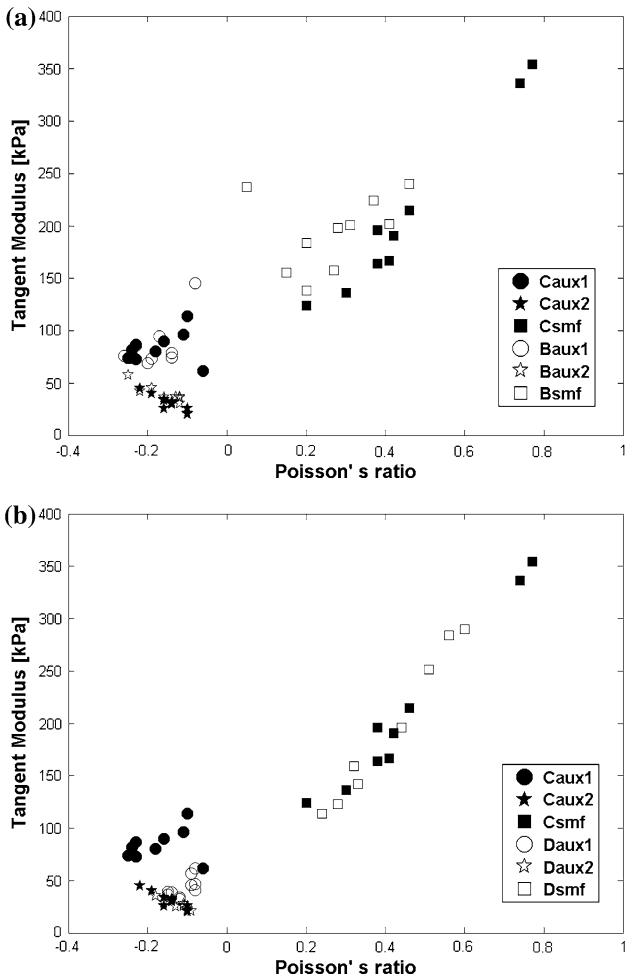


**Fig. 4** Tangent modulus versus Poisson's ratio resulted from compressive and tensile loadings in the 1st and 2nd auxetic (aux1 and aux2) and returned (smf) phases of batches B–C (**a**) and C–D (**b**)

**Table 2** Results gathered from the quasi-static cyclic tests both in compression and in tension

| Batches | Foam's phase | Test mode | Mean values | | |
|---|---|---|---|---|---|
| | | | Poisson's ratio | Tangent modulus (kPa) | Energy dissipation per cycle (mJ/cm$^3$) |
| A | 1st auxetic | Compression | −0.11 | 61.3 | 0.21 |
| | | Tension | −0.17 | 49.9 | 0.06 |
| | Returned | Compression | 0.15 | 118.2 | 0.11 |
| | | Tension | 0.35 | 203.9 | 0.2 |
| | 2nd auxetic | Compression | −0.14 | 30 | 0.39 |
| | | Tension | −0.16 | 37.9 | 0.29 |
| B | 1st auxetic | Compression | −0.14 | 80.4 | 0.24 |
| | | Tension | −0.23 | 76 | 0.09 |
| | Returned | Compression | 0.25 | 167.4 | 0.07 |
| | | Tension | 0.3 | 220.2 | 0.18 |
| | 2nd auxetic | Compression | −0.12 | 35.3 | 0.5 |
| | | Tension | −0.19 | 43.3 | 0.24 |
| C | 1st auxetic | Compression | −0.12 | 88.5 | 0.25 |
| | | Tension | −0.24 | 81 | 0.08 |
| | Returned | Compression | 0.32 | 147.8 | 0.06 |
| | | Tension | 0.55 | 258.5 | 0.17 |
| | 2nd auxetic | Compression | −0.12 | 25.2 | 0.47 |
| | | Tension | −0.17 | 36.1 | 0.24 |
| D | 1st auxetic | Compression | −0.08 | 50.3 | 0.16 |
| | | Tension | −0.14 | 36.2 | 0.05 |
| | Returned | Compression | 0.35 | 212.2 | 0.07 |
| | | Tension | 0.46 | 287.1 | 0.15 |
| | 2nd auxetic | Compression | −0.11 | 24 | 0.38 |
| | | Tension | −0.14 | 28.9 | 0.21 |

Each data is the mean from the five specimens

different compression ratios of 3:1 and 2:1, respectively) are shown. All the results acquired from the quasi-static cyclic tests both in compression and in tension are shown in Table 2.

For the 1st auxetic phase, the samples belonging to batch C (compression ratio of 2) show a mean value of Poisson's ratio statistically significant and tangent modulus 63 and 95%, respectively, higher compared to the ones from batch D (compression ratio of 3). Equally, no significant difference was found comparing batches B and C (grey versus blue). The only noticeable, but statistically significant, trends are between the Poisson's ratio and tangent modulus between batches A and B (32 and 40%, respectively), and A and D (27 and 29%, respectively). For the 2nd auxetic phase, the most important difference was identified between batches A and D (A 20% bigger than D), whereas no significant variations were found among the other batches.

In the returned state, batches C and D (blue foam) show a mean value of Poisson's ratio and tangent modulus 62 and 28%, respectively, higher than the analogous values identified for batches A and B (grey PU-PE foam). Batch D shows the mean highest values for Poisson's ratio (0.35 and 0.46), and tangent modulus (212.2 and 287.1 kPa), both under tensile and compressive loadings.

Figures 5 and 6 describe the behaviour of the Poisson's ratio versus the energy dissipation for specimens belonging to all four batches in each different phase. In particular, Fig. 5a shows a comparison between batches A–B, and Fig. 5b from batches A–D. A similar comparison can be made between Fig. 4a, b, where batches C, B and D are concerned. No significant differences in terms of energy dissipation were found between samples from different native bases belonging to the 1st and 2nd auxetic phases when imposing the lower axial compression (B and C). However, batch A showed an energy dissipation mean value which was 28 and 15% higher than the one from batch D (grey versus blue, respectively) in the 1st and then 2nd auxetic phases. Comparing treatments within native foam classes, differences in energy dissipation of 22 and 8% (A versus B) and 57 and 20% (C versus D) were found.

The mean values of the energy dissipation for batch A were 24 and 41% larger than the analogous ones for batches B and D, respectively. Batch B also showed energy dissipation values 9% higher than the ones related to C, which in turn dissipated 5% more energy under cyclic

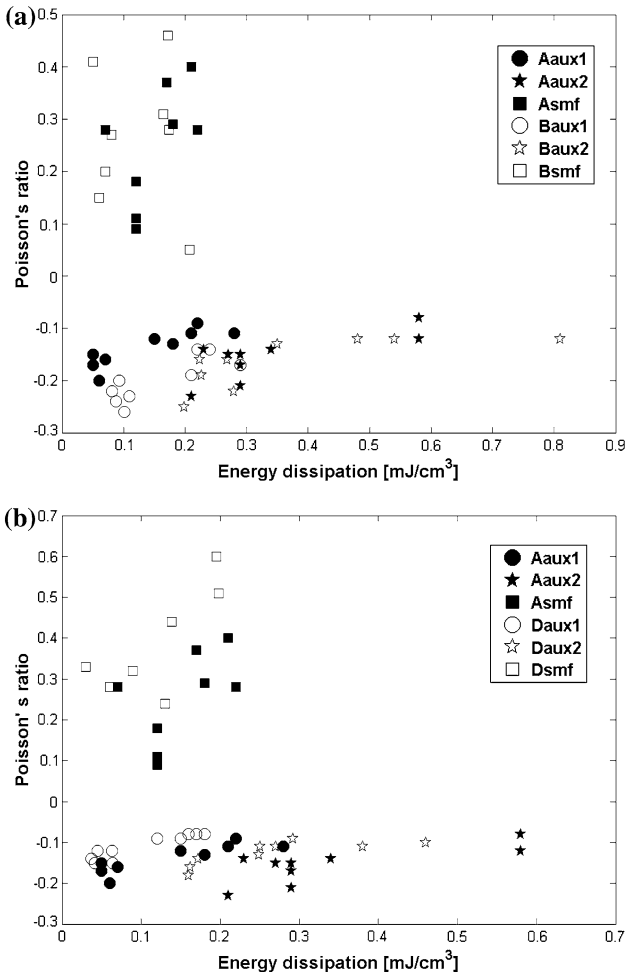


**Fig. 5** Dissipation of energy versus Poisson's ratio resulted from compressive and tensile loadings in the 1st and 2nd auxetic (aux1 and aux2) and returned (smf) phases of batches A–B (**a**) and A–D (**b**)

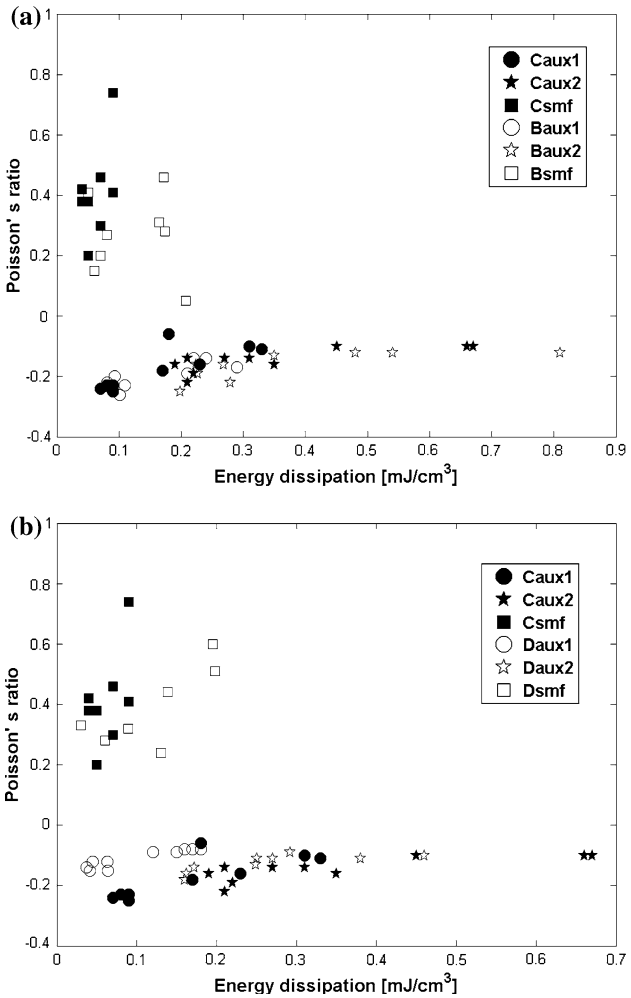


**Fig. 6** Dissipation of energy versus Poisson's ratio resulted from compressive and tensile loadings in the 1st and 2nd auxetic (aux1 and aux2) and returned (smf) phases of batches B–C (**a**) and C–D (**b**)

loading than the samples belonging to D. More specific details concerning the results of Poisson's ratio, tangent modulus and energy dissipation are reported in Table 2.

## Discussion

In Figs. 1 and 2, the nature and temperature of the first transition at −53 and −52 °C are consistent with assignment to the glass transition ($T_g$) of the polyether block [23]. The second-order transition observed at 114 °C, presumably, corresponds to the $T_g$ of the PU block, although we could find no report of such a transition in the literature. It should be noted here that in the absence of a melting transition prior to 200 °C, it is the presence of this process that results in the auxetic properties of the foam being retained at room temperature. Repeating the experiment on the modifications of this foam afforded essentially identical thermograms, precluding further insight into the observed mechanical differences. Without further experimental evidence regarding chemical changes during the auxetic conversion process, we cannot say how this is brought about, except to that it seems the polymer structure is not altered. Although no synthetic information was available for the grey foam, the identification of virtually identical thermal behaviour to the blue foam (Fig. 1 versus Fig. 2) was suggestive of similar chemical composition. As was also the case with the blue foam, further modification of the grey material resulted in no discernable change in the DSC thermograms, which exhibited glass transitions at −53 and 114 °C. Clearly the heating of both samples above the upper $T_g$ is required in order to process the materials into the auxetic phase.

From a qualitative point of view, it can be seen in Figs. 3 and 4 the closeness of batches A and D, and B and C in terms of tangent modulus–Poisson's ratio. Specimens processed with lower axial compressive ratios tended to

reach higher negative Poisson's ratios in magnitude and higher values of tangent modulus when compared to foams manufactured with a larger compression ratio. For the 1st auxetic phase, the spread of the values of the measured quantities indicates the importance of the different axial compression adopted, whereas for the 2nd auxetic phase the results group in more tightly packed clusters, showing a great consistency of results. The specimens in the returned phases, that represent the main novelty of this study, show a larger spread of results than the ones belonging to the auxetic states and, in the present data, it is difficult to identify clusters. However, a quasi-linear and monotonic dependence of the Poisson's ratio versus tangent modulus is clearly described in Fig. 4b, concerning the comparison between batches C and D.

The results contained in Table 2 show also that Poisson's ratio and tangent modulus are generally higher for compressive rather than under tensile loading, though this was not statistically significant [20]. Also the energy dissipation data show the same trend in the 1st and 2nd auxetic states, whereas an opposing and consistent behaviour could be observed in the returned status.

Comparing specimens made from the same native medium, with similar manufacturing characteristics, in the 1st auxetic phase, and tested under cyclic tensile loading [19], shows they fit well in the range of published data. In particular, the 2G specimen shows the same characteristics of specimens contained in batches A and D, whereas the 5G samples have similar mechanical properties of those belonging to batches B and C. Specimens 2G and 5G show also Poisson's ratios of −0.16 and −0.29, tangent modulus of 50 and 90 kPa and energy dissipation of 0.14 and 0.08 mJ/cm$^3$, respectively. These values are consistent with the results related to batch A (−0.17, 49.9 kPa and 0.06 mJ/cm$^3$) and batch B (−0.23, 76 kPa and 0.09 mJ/cm$^3$). Conversely, since the novelty of the results, no comparisons can be made from the specimens in the returned and 2nd auxetic states.

It should be noted that since values of Poisson's ratio greater than 0.5 were found in a few specimens belonging to both conventional and returned foams it is clear that there is significant anisotropy in the native material used to carry out the experiment.

## Conclusions

This study has shown that the effect of the manufacturing process and shape memory polymer properties of two different PU-based open cell foams on the production of auxetic (negative Poisson's ratio foams) are assessed. Through the re-heating and auxetic manufacturing process, it has been possible to produce two new types of open cell foams with different mechanical characteristics, specifically in terms of stiffness and energy dissipation. The compression ratio imposed during processing to auxetic has been shown to be the most important parameter affecting the mechanical performance of the foams, for example, in the 1st auxetic phase the Poisson's ratio, tangent modulus and energy dissipation differed significantly between specimens manufactured with different axial compressions. The difference caused by the axial compression was significantly reduced for samples belonging to the 2nd auxetic state.

Finally, no significant differences were found between specimens made using the same manufacturing parameters but belonging to different original media. This result is supported by the thermal analysis that shows both these PU-based foams had very similar properties. Heating to a temperature above the upper $T_g$ was identified here as the most significant parameter for the conversion of the conventional foam into an auxetic material.

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