

# The Effect of Chemical Composition, Density and Cellular Structure on the Dynamic Mechanical Response of Polyolefin Foams

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## ABSTRACT

Dynamic mechanical analysis has been applied to a collection of polyolefin foams with different chemical compositions and densities and manufactured from different routes. The effect of different foam characteristics, such as density, polymer morphology and cellular structure on the dynamic mechanical response is analysed. The way in which this technique can be used to obtain information about the polymer morphology of the foam is presented. In addition, examples of the use of this technique in studying specific problems are illustrated.

## INTRODUCTION

Dynamic mechanical analysis (DMA) is a powerful technique for the characterisation of polymers and polymer systems<sup>(1,2,3)</sup>. The main reason is related to the ability of this procedure to obtain information about the viscoelastic character of the material that, as is well known, is one of the key properties of polymer systems. As a consequence, by using this method, it is also possible to characterise the microstructure through the study of its viscoelastic relaxations. The preceding ideas are also appropriate for foams; therefore dynamic mechanical analysis should be an effective tool for the characterisation of polymer foams. However, as far as we know, no detailed studies on the effect of the structure on dynamic mechanical behaviour of polyolefin foams have been published

It is widely accepted that the physical properties of a given foam depend on many factors<sup>(4,5)</sup>. A possible functional dependency would be:

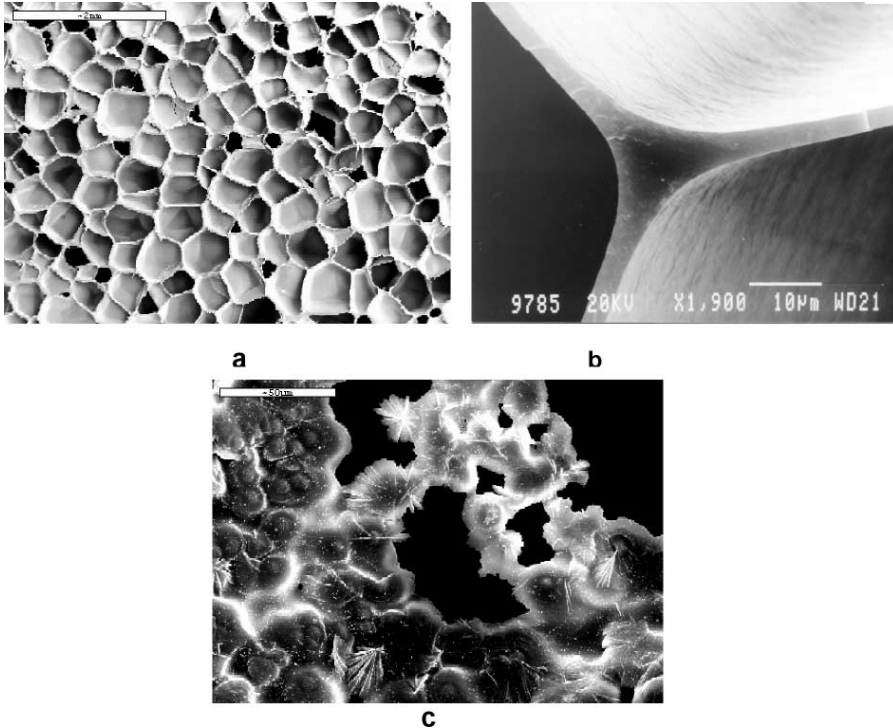
$$X=X(\rho, CC, CS, PM, GE, \text{etc.}) \quad (1)$$

where X is any physical property, CC is the chemical composition,  $\rho$  is the foam density, CS is the cellular structure, PM is the polymer morphology and GE is the kind of gas enclosed.

It is appropriate to bear in mind the range in which the previous factors can be changed for polyolefin foams. On the one hand, the chemical composition of commercial polyolefin foams is mostly based on polymers such as: different types of polyethylenes (low density polyethylene, linear-low density polyethylene, high density polyethylene, metallocene polyethylenes), ethylene vinyl acetate copolymers with varying vinyl acetate content, polypropylenes and blends of these materials. On the other hand, the foam density ranges between 15 and 300 kg/m<sup>3</sup> for commercial materials. The structure and morphology of semicrystalline foams have to be studied at three different levels (Figure 1). The cell size distribution and anisotropy of the cellular structure is observed in low magnification micrographs (Figure 1a). From these micrographs, important parameters such as the mean cell size, the anisotropy ratio, the possible rest of foaming agent, and the overall cell shape are usually obtained. In order to obtain a more detailed characterisation of the cellular structure, it is necessary to determine the mean cell wall thickness and the mass fraction in the struts. Micrographs with higher magnifications are used for this purpose (Figure 1b). Finally, and taking into account the semicrystalline structure of polyolefin foams, it is appropriate to consider a third structural level: the morphology of the base polymer. The shape and size of the constituent elements of this morphology at the micrometry scale can be estimated by using micrographs as showed in Figure 1c. This micrograph corresponds to a cell wall after etching. Finally, 12 months after production it can be established that the gas enclosed by the cells is mainly air<sup>(6)</sup>, this is due to the diffusion of the gas used during production.

The previous structural description gives an impression of the complications associated with the study of these intricate materials. Consequently, powerful analytical techniques, such as differential scanning calorimetry (DSC), scanning electron microscopy (SEM), thermo-gravimetric analysis (TGA), Raman and Infrared spectroscopies, and dynamic mechanical analysis (DMA) should be used. In this work, several aspects in which DMA can help the scientist or the engineer in the understanding of the foam properties or in the selection of foams for a given application are presented. The following cases are considered:

**Figure 1** The three levels of the structure of a semicrystalline foam, a) Cellular structure, b) cell walls and strut, c) morphology of the base polymer in a cell wall (electron beam perpendicular to the wall surface)



- 1) The effect of chemical composition on the dynamic mechanical response is described. The  $\alpha$  and  $\beta$  relaxations, the glass transition, and the maximum service temperature are evaluated. The matrix morphology is studied in terms of the polymer relaxations.
- 2) The effect of the type of cellular structure (opened or closed cells) is investigated.
- 3) The effect of different foam characteristics such as density, cellular structure and matrix polymer morphology on the dynamic properties is reported.
- 4) Dynamic mechanical analysis is used to evaluate the damping capabilities of several foams.

## EXPERIMENTAL

The DMA equipment (Perkin-Elmer DMA7) was calibrated according to the recommended procedures using the manufacturer's software. The storage modulus ( $E'$ ), loss modulus ( $E''$ ) and loss tangent or  $\tan \delta$  were obtained in compression. Two important reasons justify this choice. Firstly, most of applications of foams cause them to be loaded in compression (packaging, cushioning, structural parts), and secondly other typical geometries for polymers, as for example bending, are not adequate due to several experimental problems. Experiments in bending are difficult due to the possible indentation of the foam at high temperatures or loads.

Experiments were performed at 1 Hz frequency, between  $-40^{\circ}\text{C}$  and  $125^{\circ}\text{C}$  and with a heating rate of  $5^{\circ}\text{C}/\text{min}$  in order to obtain the dynamic mechanical properties as a function of the temperature. The applied static strain and dynamic strain were chosen in the low strain range (2% static strain and 0.11% dynamic strain). The plate diameter was 10 mm and the test specimens were prepared in a cylindrical shape with the same diameter. Three experiments were carried out for each material. The standard deviation of these measurements was  $\pm 7\%$  for the storage and loss modulus and  $\pm 5\%$  for the  $\tan \delta$ .

Commercial crosslinked polyolefin foams kindly provided by Sekisui Alveo BV (Roermond, the Netherlands), Zotefoams Plc. (Croydon, UK) and Microcel S.A. (Burgos, Spain) were used in this study. Most of the foams were crosslinked closed cell materials, however non-crosslinked foams and opened cell foams were also considered. A description of the main characteristics of the foams is given in each section. More detailed information about these materials can be found in previous publications<sup>(7,8,9)</sup>.

## DEFORMATION MECHANISMS

The main factors that should be taking into account when the dynamic mechanical response of a given foam is analysed are:

- 1) The bending of the cell edges and stretching of the cell faces are the main mechanisms that control the response in the low strain range, therefore the behaviour of the foams is similar to that of the base polymer<sup>(10,11)</sup>.

- 2) The gas pressure inside the closed cells is an important mechanism that, especially at high temperatures and low densities, influences the dynamic mechanical response of closed cell foams<sup>(12)</sup>.
- 3) Previous works have established that polymer foams have a non-linear dynamic mechanical behaviour<sup>(10,13)</sup>.
- 4) The fluid flow through the structure influence on the dynamic mechanical response<sup>(15)</sup>. This mechanism is especially important in opened cell foams.

## RESULTS

### 1. Effect of Chemical Composition

In this section the dynamic mechanical behaviour of foams of similar densities is compared, obtaining information about the matrix polymer morphology of each material. Two important factors have to be taken into account<sup>(12)</sup>. First, due to the possible effect of the gas pressure, it is necessary to compare foams of similar densities, i.e. similar gas content, and second due to the non-linear behaviour, the experiments have to be performed with the same static and dynamic strains. The main characteristics of each material (supplied by Sekisui Alveo BV) are given in Table 1.

**Table 1 Main characteristics of the foams used for the study of the effect of the chemical composition. LDPE, low-density polyethylene, LLDPE, linear-low density polyethylene, HDPE, high-density polyethylene**

Foam	Density (kg/m <sup>3</sup> )	Relative density	Chemical Composition	Average Cell size (µm)	Melting Point (°C)	Crystallinity (%)
NA85	85	0.092	100% LDPE	258	109.1	42.3
NL89	89	0.097	50% LDPE+ 50% LLDPE	223	107.8	41.4
NT105	105	0.110	50% LDPE+ 50% HDPE	222	125.3	52.4
NEE86	86	0.093	10% LDPE+ 90% EVA	242	88.3	30.9

The foams have similar densities and average cell sizes; the main difference between them is the chemical composition and, as a consequence, the morphology of the base polymer. These foams are based on LDPE (NA85), EVA (NEE86), a blend of LDPE and LLDPE (NL89) and a blend of LDPE and HDPE (NT105). As can be observed in Table 1, while the material which contains HDPE has the highest melting point and degree of crystallinity, the material based on EVA has the lowest crystalline content and melting of this phase. The other two foams have intermediate values of these characteristics. It is well known that HDPE polymers present almost linear chains which can crystallise to a great extent, however vinyl acetate reduces the regularity of the chains and consequently the degree of crystallinity and melting point. Moreover the addition of this monomer increases the amorphous and interfacial contents.

Bearing in mind the similar density and cellular structure of these four foams, it seems clear that the main differences in the dynamic mechanical response should be explained in terms of the chemical composition and matrix polymer morphology.

As was mentioned previously, the behaviour of the  $\tan \delta$  curve in the low strain range and at low temperatures is due to the response of the cell walls. Therefore, in order to understand the experimental behaviour of the samples it is essential to be familiar with the response of the base material.

Materials based on polyethylene or ethylene copolymers present three main relaxations, designed by the symbols  $\alpha$ ,  $\beta$  and  $\gamma$ . In measurements performed at low frequencies, the  $\gamma$  relaxation, usually identified as the glass transition of these materials, is observed between  $-150^{\circ}\text{C}$  and  $-120^{\circ}\text{C}$ , the  $\beta$  one is detected in the temperature range between  $-30^{\circ}\text{C}$  and  $10^{\circ}\text{C}$ , and the  $\alpha$  relaxation is seen between  $30^{\circ}\text{C}$  and  $120^{\circ}\text{C}$ . Our experiments were performed in the temperature range between  $-40^{\circ}\text{C}$  to  $125^{\circ}\text{C}$ , therefore attention was focused on the  $\beta$  and  $\alpha$  relaxations.

It has been reported that at low temperatures the  $\beta$  relaxation of continuous solid polyethylenes and ethylene copolymers can be detected as a peak in the loss modulus curve or as a shoulder in the  $\tan \delta$  curve<sup>(14)</sup>. Furthermore, it is well known that this relaxation results from motions of chain units located in the interfacial region, i.e. the zone between crystalline and amorphous part of the polymer. Its existence is not

universal in the different types of polyethylenes, being conditioned by the presence of an interfacial content higher than about 7%<sup>(15,16)</sup>. On the other hand, the  $\alpha$  relaxation can be observed at higher temperatures as a wide peak in the  $\tan \delta$  curve or as a shoulder in the  $E''$  curve<sup>(14)</sup>. The  $\alpha$  relaxation can be associated with the crystalline part of the polymer; in fact the intensity of this relaxation increases as the crystalline content rises<sup>(14,15)</sup>. The temperature of the peak has been associated with the thickness of the lamellae<sup>(15,17)</sup>.

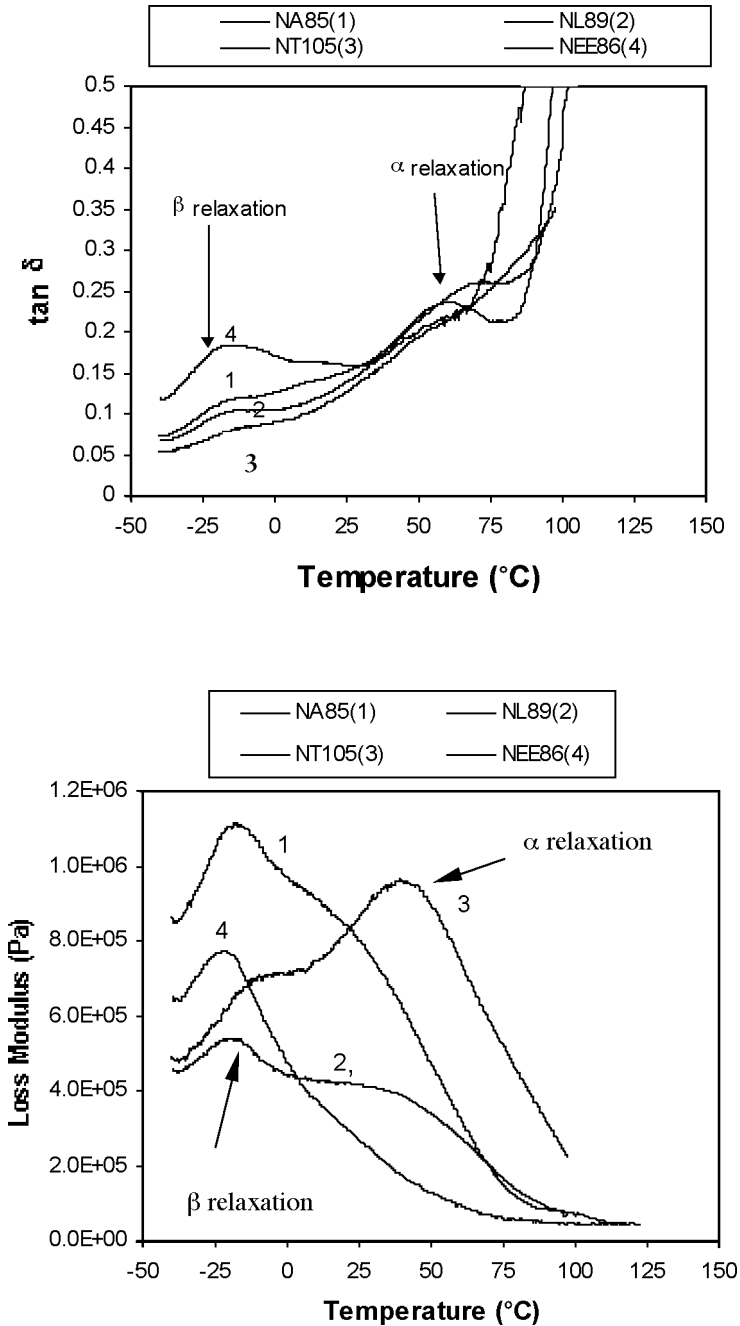
A typical example of the viscoelastic response of foams with different chemical compositions is shown in Figures 2a and 2b. The well-known viscoelastic relaxation for each type of polymer can be observed.

The  $\beta$  relaxation is observed in all the materials under study, because all of them have a high LDPE content, and as consequence and interfacial content higher than 7%. While the NA, NT and NL foams have a similar  $\beta$  relaxation, the intensity of this relaxation is higher for the NEE foam, which is associated with a higher interfacial content in the EVA copolymers.

The  $\alpha$  relaxation is not visible for the NEE foam, which is due to its low crystalline content. The position of the  $\alpha$  peak is different for the NL and NA foams. Both materials present a similar degree of crystallinity, however the NL foam has its  $\alpha$  peak at higher temperatures than the NA foam, which is related with the different crystalline structure of these two types of materials. The NL foam seems to have thicker lamellae. The  $\alpha$  peak is not clearly observed for the NT foam in the  $\tan \delta$  curve, but it appears as an intense peak in the loss modulus curve, which can be explained in terms of the high crystallinity of this material. The maximum service temperature of these foams is also shown by the previous tests, this is the temperature at which the  $\tan \delta$  rises.

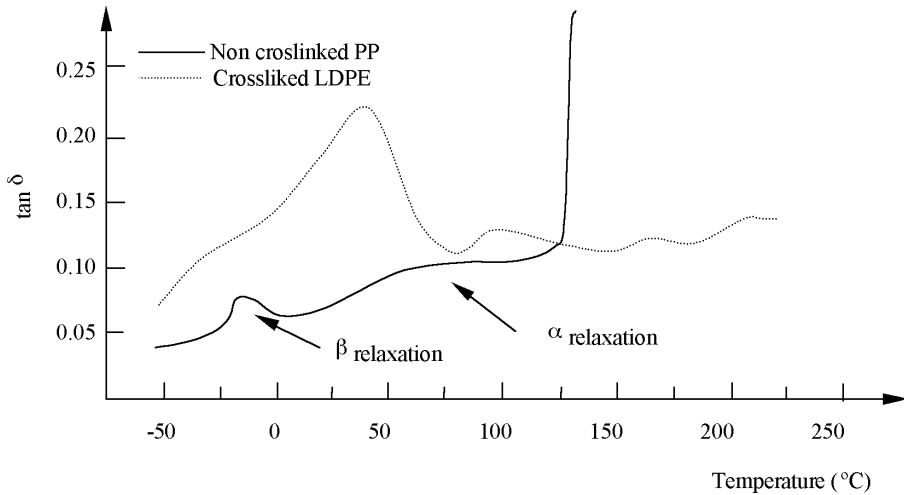
Figure 3 shows the  $\tan \delta$  curve of two polyolefin foams based on different polymers, the first one is a crosslinked LDPE foam. The second one is a non-crosslinked polypropylene (PP) foam. This last material presents the two main relaxations of PP in the range under study<sup>(18,19)</sup>, the glass transition at approximately 10°C and the  $\alpha$  relaxation at 110°C. It is also necessary to mention the different behaviour of the foams after the melting of the crystalline phase. The crosslinked material reaches a constant value of the  $\tan \delta$ , however the  $\tan \delta$  of the non-crosslinked foam increases dramatically. Consequently, dynamic mechanical analysis can be used to identify if a foam has been crosslinked during processing.

**Figure 2 Dynamic mechanical response of foams with different chemical composition and similar densities. a)  $\tan \delta$ , b) loss modulus**





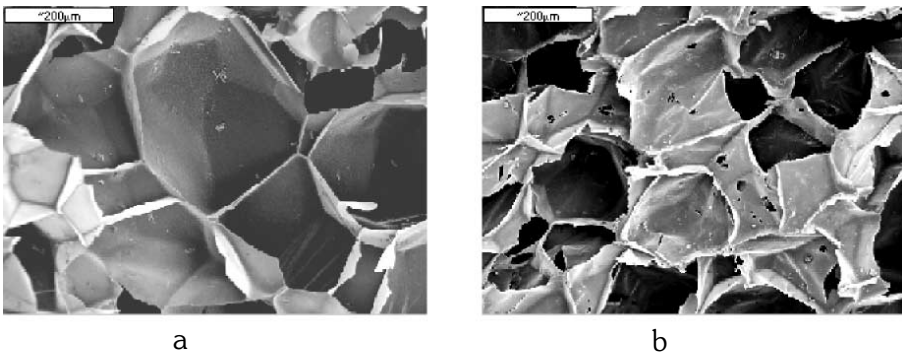
**Figure 3 Dynamic mechanical response of two foams with different chemical compositions, one of the foams is a non-crosslinked polypropylene foam and the other is a crosslinked polyethylene foam**



## 2. Effect of Opened Cell Content

Two LDPE foams produced by using a compression moulding process<sup>(9)</sup> (supplied by Microcel S.A.) were considered for this analysis. Both materials were produced from the same LDPE grade having a matrix polymer with similar properties. The micrographs of these foams are shown in Figure 4. The first one is a crosslinked closed cell foam of 25 kg/m<sup>3</sup> density (0.027 relative density) with isotropic cells of polyhedral shape.

**Figure 4 SEM images of a) closed cell foam produced by a compression moulding process and b) semi-opened cell foam produced by a compression moulding process**



The second one is a crosslinked semi-opened cell foam of 34 kg/m<sup>3</sup> density (0.037 relative density) in which the cells have small voids. Taking into account the similar composition and density of both foams, the differences in the DMA response have to be understood in terms of their different cellular structure.

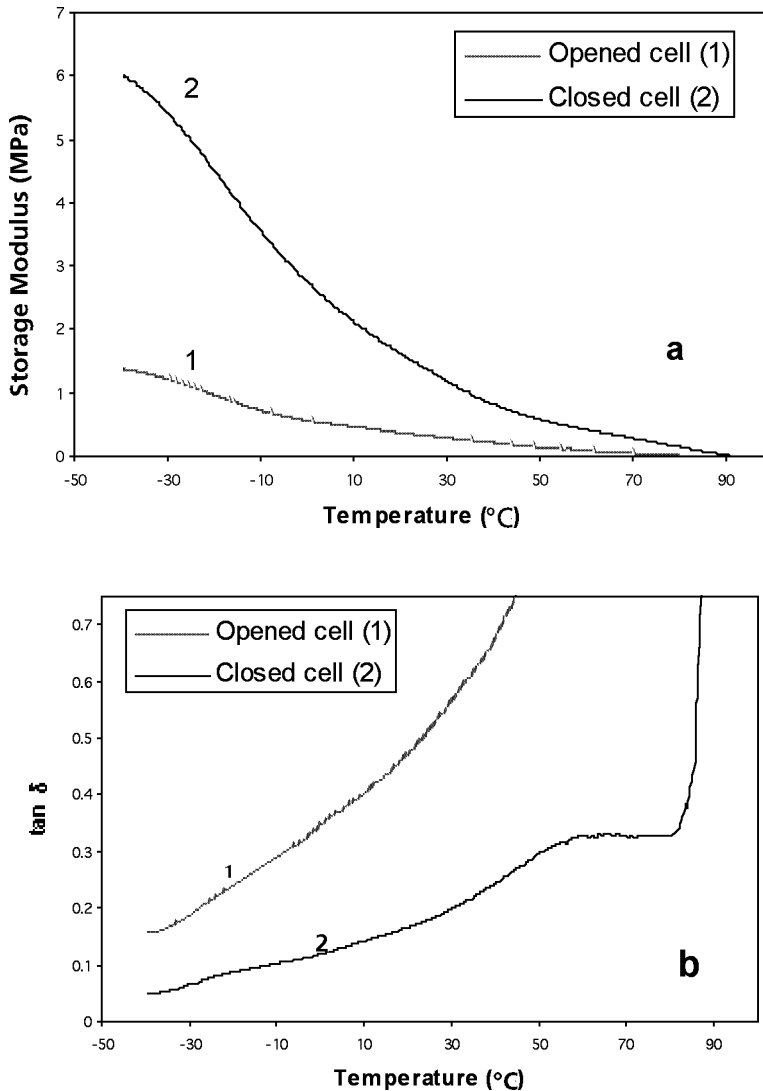
As can be observed in Figures 5a and 5b, the dynamic mechanical response of both materials is totally different. The closed cell foam presents a higher storage modulus and a similar  $\tan \delta$  curve to that is observed for continuous LDPE samples. However, the opened cell foam has a lower storage modulus and a higher  $\tan \delta$ , which increase significantly with temperature. The deformation mechanisms for this last material seem to be different, the lower storage modulus should be related to the non-regular shape of the cells and the presence of broken cell walls, both effects reducing the stiffness of the cellular structure. Moreover, the fluid can flow through this foam during the dynamic experiment: this additional energy loss mechanism increases the  $\tan \delta$  values.

### 3. Effect of Cellular Structure and Matrix Polymer Morphology

The product code, measured density, cell size, melting point and crystallinity of the materials under study in this section are summarised in Table 2. These foamed samples are crosslinked closed cell low-density polyethylene foams manufactured by a high-pressure nitrogen gas solution process, and have been kindly provided by ZoteFoams Plc<sup>(20)</sup>.

Foams	Density (kg/m <sup>3</sup> )	Relative density	Cell size (µm)	Cell wall thickness (µm)	Fraction of material in the struts	Melting Point (°C)	Crystallinity (%)
LD15	16.7	0.018	194	1.4	0.22	106.0	41
LD18	22.5	0.025	880	5.8	0.21	108.5	42
LD24	24.6	0.027	312	1.9	0.16	108.6	44
LD29	30.8	0.034	528	4.2	0.24	108.5	43
LD33	32.0	0.035	424	3.6	0.28	105.8	44
LD60	58.5	0.064	773	10.3	0.24	109.0	42
LD70B	69.5	0.076	528	6.0	0.35	106.6	41
Solid polymer	910	1	—	—	—	112.8	42

**Figure 5 Dynamic mechanical behaviour of the foams manufactured in a compression moulding process. a) Storage modulus, b)  $\tan \delta$**



The density ranges between 17 and 70 kg/m<sup>3</sup> (0.019 and 0.077 relative density), and the cell size between 194 and 880  $\mu\text{m}$ . The solution process allows producing materials with fixed density and different cell sizes by changing industrial process parameters<sup>(8,20)</sup>. Due to this reason there is no a relationship between density and cell size for the foams under study.

The melting point and degree of crystallinity are around 108°C and 42% respectively, typical values for low-density polyethylene.

A previous work<sup>(21)</sup> has shown that these materials have an isotropic cellular structure in which the cells have a constant shape for all the densities. In fact, the cellular structure can be described by using the equation:

$$\phi(1 - f_s) \frac{\rho}{\rho_s} = C\delta$$

where  $\Phi$  is the mean cell size,  $f_s$  is the mass fraction in the struts,  $\rho$  is the foam density,  $\rho_s$  is the polymer base density,  $\delta$  is the mean cell wall thickness and  $C$  is a constant which depends on the cell shape. This constant takes a value of 3.89 for this collection of foams.

The storage modulus ( $E'$ ) and  $\tan \delta$  as a function of the foam density for different temperatures are shown in Figure 6. The following results can be inferred from the previous figure:

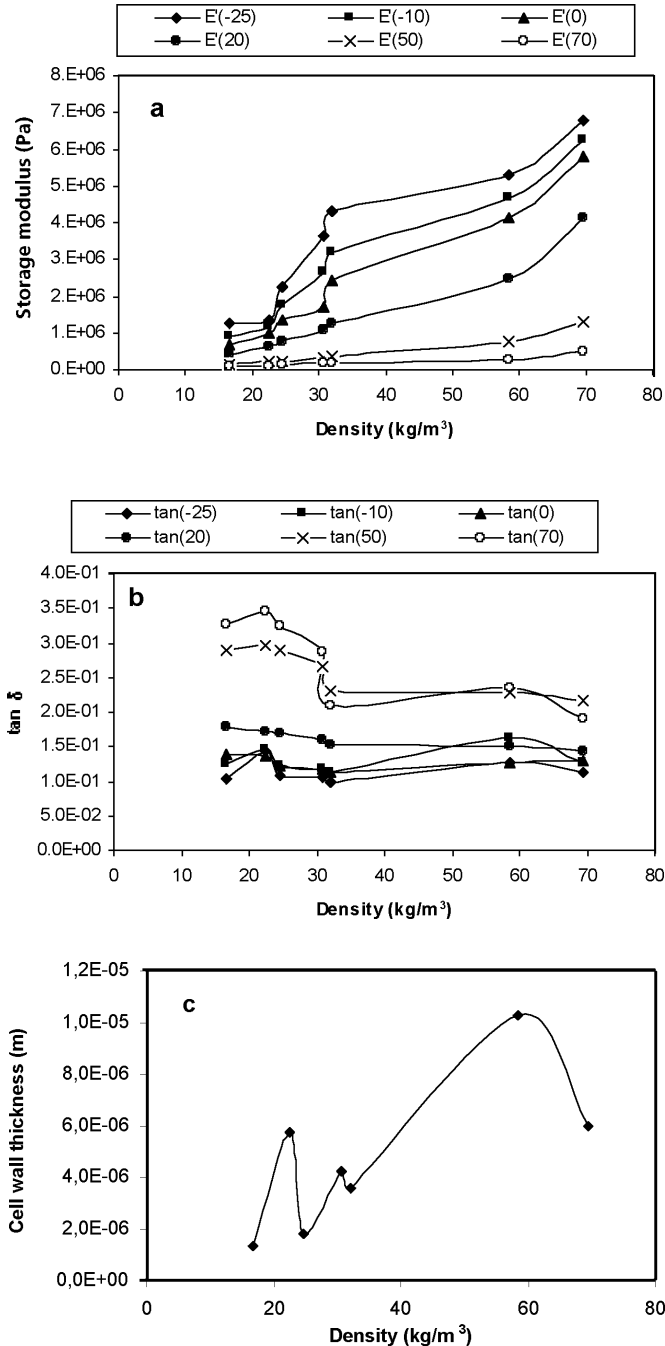
- a) The storage modulus increases with density, in the temperature range under study (Figure 6a).
- b) While at temperatures above 25°C the  $\tan \delta$  decreases with density (Figure 6b), at lower temperatures (in the range of the  $\beta$  relaxation) the shape of the  $\tan \delta$  curve versus density is similar to that of the cell wall thickness versus density (Figure 6c).

The storage modulus increases with density as a consequence of the change in the dimensions, and consequently change of the mechanical resistance of the elements that build the cellular structure when the density changes.

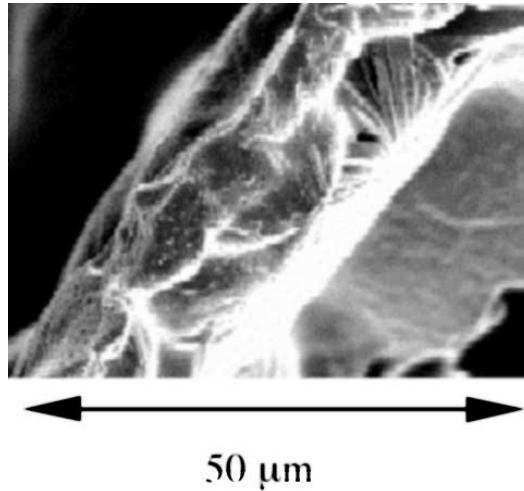
In order to analyse the behaviour of the  $\tan \delta$ , a detailed analysis of the morphology of the cell walls was needed. A permanganate etching was performed for this purpose<sup>(22)</sup>.

Figure 7 shows the etched section of a foam cell wall (the electron beam was parallel to the cell wall surface). Morphological units initiate on both sides of the cell wall. The radiating fibrils of 2  $\mu\text{m}$  thickness approximately, seem to be groups of lamellar crystals, together with amorphous inter-layers. The fibrils initiate on the cell wall surface and grow radially from

**Figure 6** Dynamic mechanical behaviour of Zotefoams as a function of the density for different temperatures. a) Storage modulus, b)  $\tan \delta$ . c) cell wall thickness as a function of density



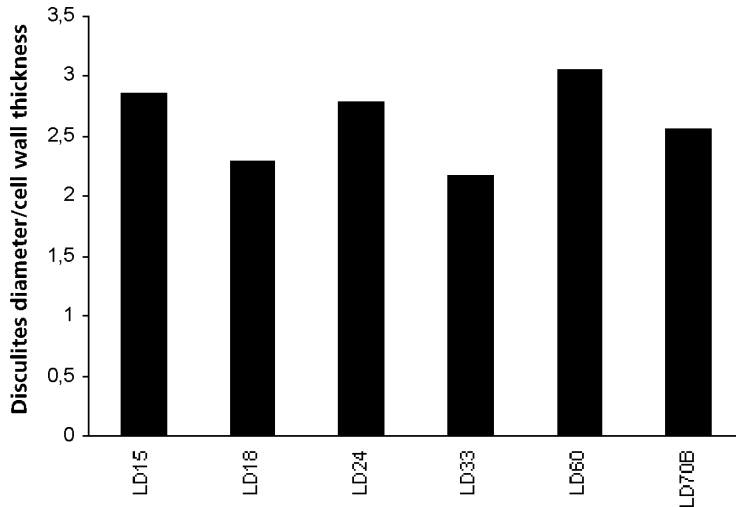
**Figure 7 Cell wall after etching (electron beam parallel to the cell wall surface)**



the nucleus. The growth direction it is initially in the plane of the cell wall to cell gas surface, but finally it becomes almost perpendicular to the surface, when they reach the face mid-plane. The ‘disculites’ meet others growing from the other side of the wall, and complete their growth. The final shape of the disculite is approximately circular on the initial face surface. This morphology is different to the typical spherulites generally observed for LDPE crystallised from the melt. A remarkable result is obtained if the estimated size of the disculites divided by the cell wall thickness is evaluated (Figure 8). A constant value of about 2.5 is obtained, therefore it can be established that the size of the disculites depends on the size of the environment where they grow.

The preceding information allows a more detailed analysis of the DMA results shown in Figure 6b. At low temperatures, in the range of the  $\beta$  relaxation, the dynamic response of the foams is mainly due to the cell walls and struts response. The possible effect of the gas pressure reduction at these low temperatures would not be significant because of the high stiffness of the cell walls. Therefore, the dissimilar response of foams would be a consequence of their different matrix polymer morphology. As was previously mentioned, the interfacial content of the matrix polymer is the responsible for the changes in the  $\tan \delta$  curve; the results suggest that the chain morphology in the interfacial zone is also

**Figure 8 Dyscullites diameter divided by the cell wall thickness for the LD foams**



affected by the cell wall thickness. In summary, it could be assess that the dependency of the  $\tan \delta$  on cell wall thickness would have its source in the different morphology of the base polymer for foams with different cell wall thickness.

On the other hand, at high temperatures, the stiffness of the matrix polymer decreases; as a consequence the gas pressure inside the cells, which increases with temperature, would have an influence on the dynamic response. Because of this additional effect the  $\tan \delta$  curve does not show a clear trend as a function of the cell wall thickness.

#### **4. Evaluating the Damping Capabilities of Foams**

Dynamic mechanical analysis has long been used to analyse the damping capabilities of continuous solid polymers<sup>3,23</sup>. In this section, this technique is used to study the damping of several foams.

Two foamed sheets (TVS<sub>30</sub>, 80 kg/m<sup>3</sup> density (0.087 relative density) and TVS<sub>70</sub> 77 kg/m<sup>3</sup> density (0.084 relative density) were produced in a semi-continuous process based on a chemical blowing agent<sup>(7)</sup>. Blends of a LDPE and a copolymer of polystyrene and vinyl polyisoprene with a 20% polystyrene (VS copolymer) were used. The TVS<sub>30</sub> material is a blend of

LDPE/VS polymer with a 70% by weight of LDPE and the TVS<sub>70</sub> foam is a blend of LDPE/VS polymer with a 30% by weight of LDPE. The TVS<sub>0</sub> foam (62 kg/m<sup>3</sup> (0.067 relative density) was manufactured from a 100% LDPE sheet. All the mechanical measurements were performed in a perpendicular direction to the sheets. The three foams under study presented a similar cellular structure with average cell sizes of 203 μm for the TVS<sub>70</sub>, 216 μm for the TVS<sub>30</sub> and 235 μm for the TVS<sub>0</sub>.

The LDPE/VS blends were designed for damping of low frequency vibrations. Therefore, an important point to consider is their dynamic mechanical response, i.e. the loss tangent at the temperatures and frequencies of a possible final application.

The viscoelastic response of these foams is shown in Figure 9. A preliminary analysis of the curves show that the storage modulus decreases when the temperature increases. As expected, the LDPE foam presents the typical behaviour of non-foamed low-density polyethylene (Figure 9b). However, a very intense damping peak at room temperature is detected for the two LDPE/VS blends. This peak has its source on the VS polymer phase. The blend with a 70% content of the VS polymer has a more intense damping peak than the blend with a 30% content. Moreover, the  $\alpha$  and  $\beta$  peaks can be detected for the TVS<sub>30</sub> foam, however the  $\alpha$  relaxation does not appear in the viscoelastic response of the TVS<sub>70</sub> foam.

Above the glass transition, the  $\tan \delta$  increases when the VS polymer content increases. Therefore, the damping capabilities of these foams at room temperature are visibly improved by means of blending. It is also important to point out that the storage modulus decreases when the VS content increases. Under the glass transition, the damping capabilities are not enhanced by increasing the VS polymer content.

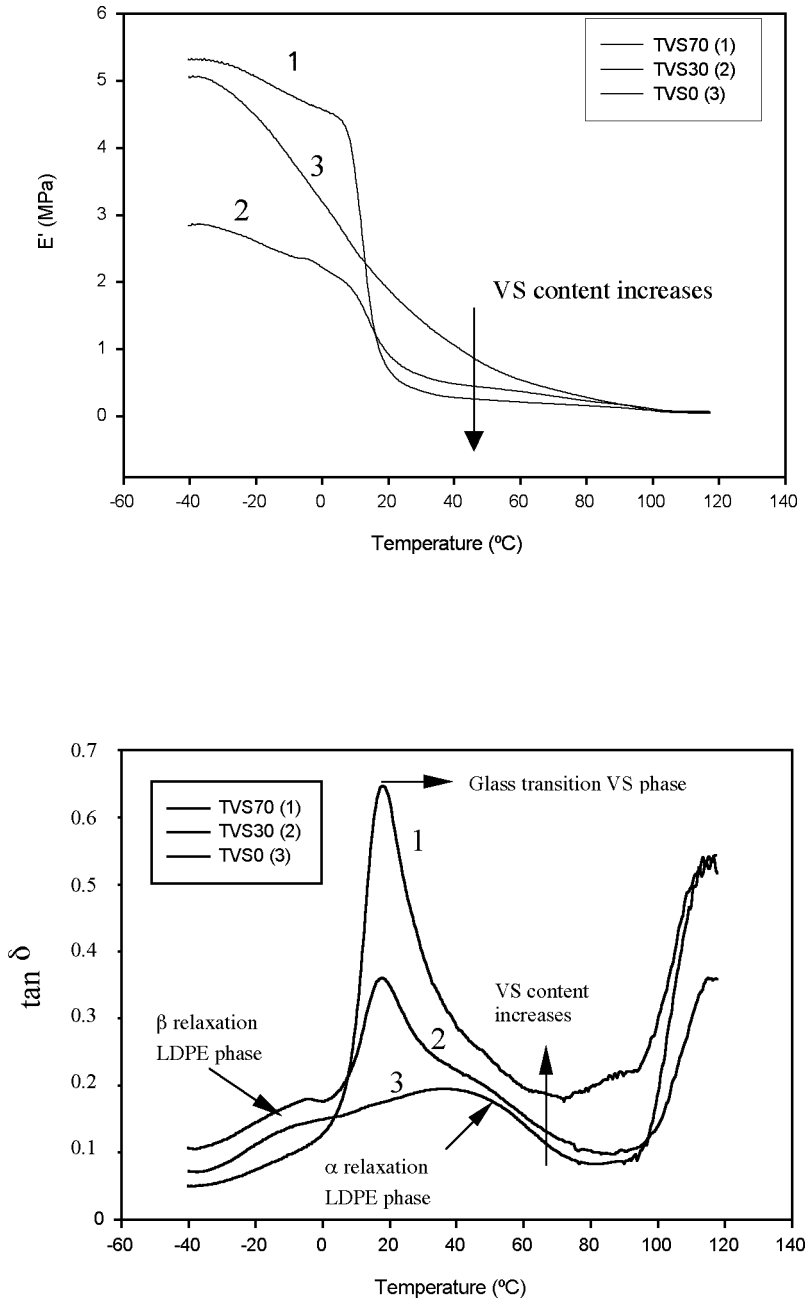
As a conclusion of this brief analysis it is clear that increasing the VS polymer content results in materials with better damping capabilities. However, softer and less resilient materials are produced.

## CONCLUSIONS

The effect of different foam characteristics such as chemical composition, density, type of cellular structure (opened or closed) and matrix polymer morphology on the dynamic mechanical properties response of a wide



**Figure 9 Dynamic mechanical properties of the LDPE/VS foams as a function of the temperature. a) Storage modulus, b)  $\tan \delta$**



collection of polyolefin foams has been analysed. Some notable results have been obtained:

- 1) From the analysis of the behaviour in the  $\alpha$ ,  $\beta$  relaxations information on the matrix polymer morphology can be inferred. In addition, from the response in the melting zone, information on crosslinking can be deduced.
- 2) Materials with opened cells have a completely different dynamic mechanical response, characterised by a higher  $\tan \delta$  and lower storage modulus.
- 3) The matrix polymer morphology in the foams seems to be influenced by the cellular structure and as a consequence the dynamic mechanical response of foams with distinct cellular structure is different.
- 4) With a similar method to that used for continuous polymers, dynamic mechanical analysis can be employed to analyse the damping capability of foams.

### ACKNOWLEDGEMENTS

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