

# Magneto-mechanical behavior in polyurethane foams containing oriented FeCo microparticles: A competition between bending and stretching effects

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## ABSTRACT

Soft magneto-active composites are of particular interest in areas of biomedical applications, wearable sensors and soft robotic. In this context, this work presents a deep study of how the incorporation of Fe<sub>70</sub>Co<sub>30</sub> microparticles into foam polyurethane composites affects its magnetostrictive response. The composites were fabricated in both foam and foam film-like structures, resulting from free or confined expansion, respectively, and with varying percentages and orientations of magnetic microparticles. All of the fabricated foams have been optically, magnetically, mechanically and thermally characterized. The inclusion of magnetic microparticles had negligible impact on thermal stability and degradation behavior. In contrast, mechanical and magnetic properties were highly sensitive to both the concentration and alignment of the particles. Magnetostriction behavior revealed a competition between bending and stretching mechanisms, governed by the composite's stiffness. At lower particle contents ( $\leq 40$  wt%), bending dominated, producing negative magnetostriction values. As particle content increased ( $> 40$  wt%), the material stiffened, suppressing bending and allowing stretching to prevail, resulting in positive magnetostriction, particularly evident in foam-film composites. These findings demonstrate the tunability of the magnetic response in soft composites through structural and compositional control.

## 1. Introduction

The development of magneto-active materials has experienced a huge growth in the last years due to their relevance in areas such as industrial automation, agriculture, and medical robotics[1,2]. Particularly interesting are the soft magneto-active materials, which have been recently employed in biomedical applications, wearable sensors and soft robotics, among others[3]. Those are composed of a soft polymeric matrix, such as soft elastomers or hydrogels, combined with dispersed magnetic micro or nanoparticles. These materials are able to generate large mechanical deformation under the application of an external magnetic field, providing a remote response and allowing reversible and

complex deformations.

In the particular case of elastomers, their mechanical softness enables a reversible deformation, allowing a shape morphing effect. Among the most employed elastomers are silicones[4], acrylate-based polymers[5] and polyurethanes[6]. Regarding the magnetic filler, NdFeB particles[7] and oxides such as Fe<sub>3</sub>O<sub>4</sub>[8] or CrO<sub>2</sub>[9] have been already used.

Most of the research in soft magneto-active composites has been devoted to improve their magnetorheological response, mainly by modifying the volume, size and distribution of the filled particles[10]. However, the magnetostrictive effect in these materials has not been deeply analysed. Some studies have reported the importance of the

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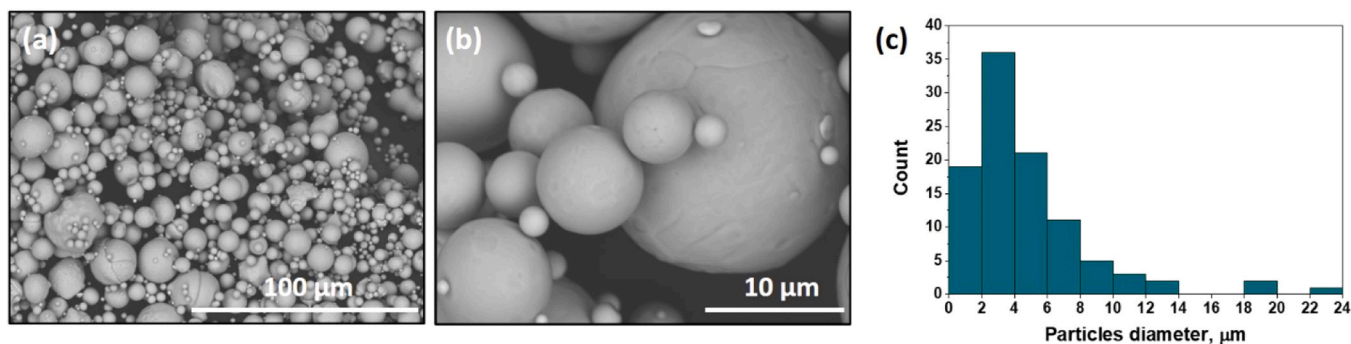


Fig. 1. SEM images of  $\text{Fe}_{70}\text{Co}_{30}$  microparticles (a and b) and their size distribution (c).

particles' shape and size for optimizing the mechanical deformation [11–13]. In the same way, other works have paid attention to the effect of the particles alignment in the polymeric matrix[14], but without providing a full study. More recently, Silva *et al.* reported a giant magnetostrictive effect in low-concentration isotropic and anisotropic  $\text{FeCo-2 V}$  helicoidal particles combined with the polydimethylsiloxane soft elastomer matrix[15].

These magneto-active composites are usually fabricated in a continuous matrix, commonly a silicone structure[16,17]. However, due to the need to find light weighted low-density smart materials, the potential of polymer composites in the form of foams has begun to be recognized and studied, counteracting the high density of monolithic materials[18]. The porosity in magneto-active composites reduces the effective Young's modulus, making the final structure softer[19,20]. Magnetic foams have been already used in several application such as microwave absorption composites [21] or energy generating systems [22]. Among the matrixes used in foams, the polyurethane is the most common one[23–25], due to its good shape memory as well as high energy and shock absorption properties[26]. The magnetostrictive characteristics of polyurethane foams filled with magnetic particles, such as carbonyl iron particles (CIP)[27], have been previously investigated. However, the influence of particle orientation on the magnetostrictive properties of these structures has received relative little attention. Most existing studies have primarily focused on their magnetorheological behaviour, however, some studies have already shown that structures with anisotropies can exhibit different responses depending on the orientation of the magnetic field [28]. Exploring the magnetostrictive effect in foam-like structures could reveal new insights into the material's behavior under magnetic fields, which is crucial for optimizing their performance in various applications.

By investigating how the orientation of the particles influences the magnetically induced deformation in these porous materials, we can enhance the design of advanced composites with tailored functionalities. Such materials could be pivotal in developing next-generation sensors, actuators, and smart hybrid materials, expanding the potential of magnetostrictive technologies in industrial and technological domains.

In this work, magneto-active composites formed by high-magnetization  $\text{Fe}_{70}\text{Co}_{30}$  microparticles embedded in a polyurethane matrix are investigated in a foam-like structure. In order to have a broader understanding of the magnetic, mechanical, and structural properties in the composites, we have explored two different routes for their fabrication. In the first approach, the foam-like structure with magnetic particles has been freely grown, forming a magnetic foam-like composite. In a second approach, the composite has been fabricated in a restricted volume, avoiding the free expansion of the foam, leading to a compressed magnetic foam film-like composite. These two distinct methodologies will enable us to assess the influence of structural confinement and shape on the composite's overall performance, offering valuable insights into their potential applications in flexible,

magnetically-responsive devices. The properties of the fabricated composites have been analysed as a function of the particle content percentage, as well as a function of their relative orientation. We have observed a competition between bending and stretching effects in the material when particles attempt to align with the magnetic field. This results in both negative and positive values of magnetostriction, is being highly dependent on the mechanical properties of the material. These properties, in turn, arise from varying concentrations and orientations of the magnetic  $\text{Fe}_{70}\text{Co}_{30}$  microparticles. We believe that these findings underscore the critical role of particle orientation and percentage in tailoring the magnetostrictive response of foam-like composites. This understanding opens up new perspectives for designing and optimizing foam-based magneto-active materials for a range of applications, from sensors and actuators to adaptive structural components.

## 2. Experimental section

### 2.1. Materials

The polyurethane based magnetic foam composites were prepared by using the commercial polyurethane foam Polyfoam F3, purchased from Ferroca-Polytek.

Magnetic metallic microparticles of composition  $\text{Fe}_{70}\text{Co}_{30}$  were synthesized by using the gas-atomization technique. The FeCo alloy shows the highest known magnetization values in the Fe-rich region [28, 29], and as it will be further discussed, the  $\text{Fe}_{70}\text{Co}_{30}$  microparticles used in this work show a saturation magnetization value of  $230 \text{ Am}^2/\text{kg}$ . Due to the fabrication process, these microparticles show a nice, almost perfect spherical morphology. Scanning electron microscopy (SEM, Hitachi TM3000) images of these microparticles are represented in Fig 1 (a) and (b), as well as the estimated size distribution in Fig. 1(c).

### 2.2. Preparation of composites

The neat foam was prepared as instructed by the manufacturer. The polyol and isocyanate components were mixed in a 2:1 ratio. A total of 1.5 g of material was prepared for the various tests, consisting of 1 g of polyol and 0.5 g of isocyanate. The magnetic composites were prepared in the same way, but with the addition of the corresponding amount of  $\text{Fe}_{70}\text{Co}_{30}$  microparticles to the polyol component to achieve 15, 30, 40, 50 and 65 %wt of microparticles content. The maximum microparticle content was established by the point at which the distribution of microparticle lines became non-uniform, with visible agglomeration occurring at higher concentrations, as will be discussed later.

The composites were prepared using two different routes:

1. **Foam-like structure (MF: magnetic foam).** This structure was obtained by mixing both components in a glass vial, allowing the foam to expand freely in the vertical direction. For the preparation of magnetic composite samples, the corresponding amount of  $\text{Fe}_{70}\text{Co}_{30}$

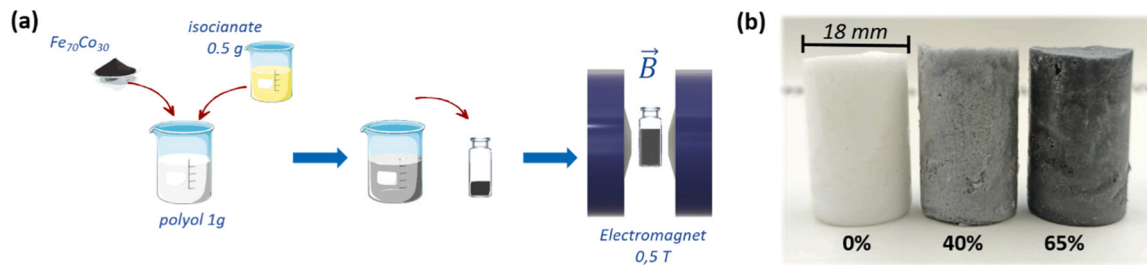


Fig. 2. Diagram of the employed procedure to fabricate the foam-like structure (a). Picture of foam-like composites with 0, 40 and 65 %wt of  $Fe_{70}Co_{30}$  microparticles (b).

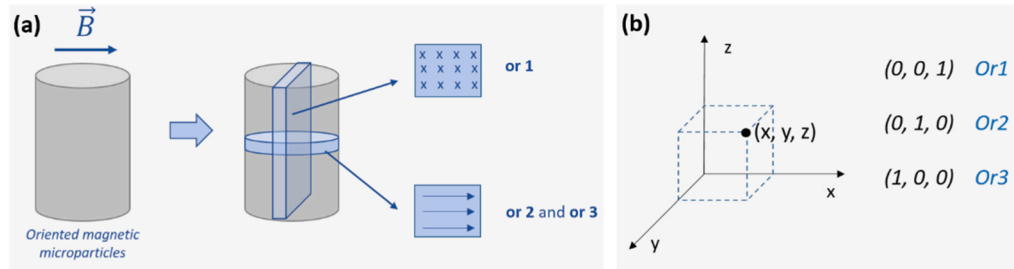


Fig. 3. Diagram of the cutting planes used to obtain the different sample orientations for MF (a). Scheme of the reference coordinate system (b).

Table 1

Summary of all the MFs fabricated with polyurethane matrix and  $Fe_{70}Co_{30}$  microparticles.

Sample	% wt of $Fe_{70}Co_{30}$	Orientation of $Fe_{70}Co_{30}$
$MF_{0\%}Or0$	0	-
$MF_{10\%}Or1$	10	
$MF_{30\%}Or1$	30	
$MF_{40\%}Or1$	40	Or 1
$MF_{50\%}Or1$	50	
$MF_{65\%}Or1$	65	
$MF_{65\%}Or0$	40	Or 0
$MF_{40\%}Or2$	40	Or 2
$MF_{40\%}Or3$	40	Or 3

Table 2

Summary of the porous polymer films fabricated with polyurethane matrix and  $Fe_{70}Co_{30}$  microparticles.

Sample	% wt of $Fe_{70}Co_{30}$	Orientation of $Fe_{70}Co_{30}$
$MFF_{0\%}Or0$	40	Or 0
$MFF_{10\%}Or1$	10	
$MFF_{30\%}Or1$	30	
$MFF_{40\%}Or1$	40	Or 1
$MFF_{50\%}Or1$	50	
$MFF_{65\%}Or1$	65	

microparticles was pre-mixed with the polyol component. For composites with oriented particles, the vial was promptly placed inside the poles gap of an electromagnet, which generated a 0.5 T magnetic field in the transverse direction of the vial, as shown in Fig. 2(a). This magnetic field intensity was tested beforehand and found sufficient to ensure nanoparticle alignment during foam composite formation. For non-oriented composites, the application of the external magnetic field was removed. After 10 min, the vial was broken to extract the composite. Some of the obtained materials are shown in Fig. 2(b).

Once the composite is extracted, different orientations are obtained by cutting the cylindrical structure along different planes: a transverse cut through the centre of the cylinder and a longitudinal cut along its

length. These cuts will be used to study the effect of the magnetic field on the samples applied with different orientations. This procedure is graphically described in Fig. 3. The cutting direction had no impact on the randomly oriented particles (Or 0). Fig. 3(b) illustrates the coordinate system used to define orientations of particles. As an example, Or1 is aligned with (0, 0, 1), Or2 with (0, 1, 0) and Or3 with (1, 0, 0). Table 1 summarizes the prepared composites, along with the corresponding quantities of  $Fe_{70}Co_{30}$  microparticles.

2. **Foam Film-like structure (MFF: magnetic foam film).** For this second structure, the polyol and the appropriate amount of microparticles were added to a beaker and manually mixed with a spatula, followed by the addition of isocyanate. The mixture was then poured into a Teflon-coated mold, covered with a Teflon sheet, and sandwiched between two non-magnetic metal plates. That is, in this setup

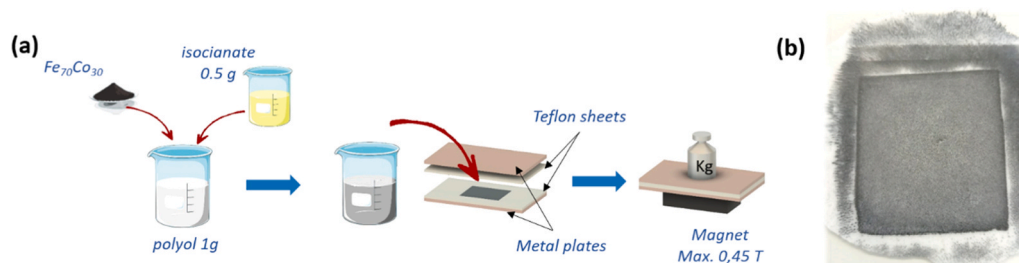
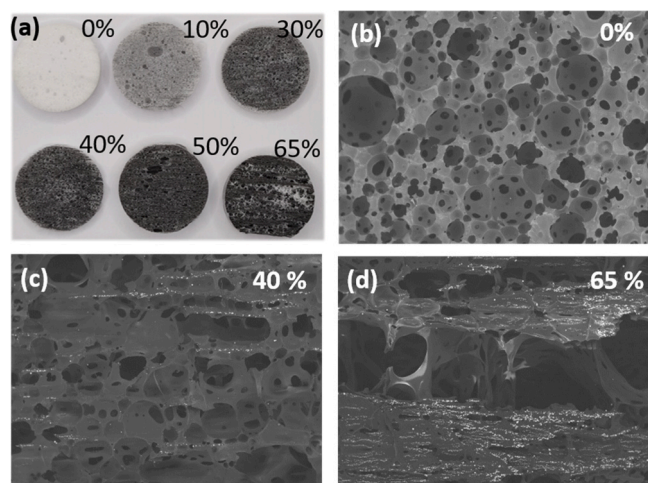


Fig. 4. Diagram of the employed procedure to fabricate the porous polymer film (a). Picture of foam film-like composites with 40 %wt of  $Fe_{70}Co_{30}$  microparticles (b).



**Fig. 5.** View of oriented MF composites for 0, 10, 30, 40, 50 and 65 % wt content of  $\text{Fe}_{70}\text{Co}_{30}$  (a), and SEM images of the composites with 0, 40 and 65 % wt of  $\text{Fe}_{70}\text{Co}_{30}$  microparticles (b, c and d, respectively). All SEM images are shown at the same scale.

the material was confined and did not expand freely. The structure was placed on a magnet generating a perpendicular (to the film plane) 0.45 T field, and a non-magnetic weight was placed on the top to maintain pressure and prevent the material from expanding. A non-magnetic weight of approximately 5 kg was used, which was sufficient to apply the necessary pressure to prevent the material from expanding and to ensure it conformed to the thickness of the Teflon mold. This procedure is graphically described in Fig. 4. After 10 min, the composite was removed from the mold. In Table 2, the prepared MFF composites are summarized, along with the corresponding quantities of  $\text{Fe}_{70}\text{Co}_{30}$  microparticles.

### 2.3. Characterization

After the fabrication of the composites, they were optically, magnetically, mechanically and thermally characterized by using scanning electron microscopy, hysteresis loop and magnetostriction measurements, mechanical tests and thermogravimetric analysis, respectively.

To study the morphology of the samples, scanning electron microscopy (SEM, JEOL JSM-7000F) was performed using an accelerating voltage of 5 kV. The samples were fractured after immersion in liquid nitrogen for cross-sectional analysis.

A SDT Q600 (TA-Instruments) thermobalance was employed to evaluate the thermal stability of the prepared composites with varying concentrations of magnetic microparticles. Thermogravimetric analysis (TGA) was carried out under nitrogen atmosphere and heating up from 25 to 800 °C at a rate of 10 °C · min<sup>-1</sup>. The initial degradation temperature ( $T_i$ ) was determined at the point where the mass loss reached 2 wt %, while the maximum degradation temperature ( $T_d$ ) for each degradation phase was derived from the minimum of the first derivative curves. TGA experiments were conducted in duplicate.

Compression tests (Metrotec FTM-50) were carried out by employing a 20 N cell at room temperature with a speed of 0.5 mm/min. Samples were prepared with an initial thickness of 5 mm and a diameter of 1.2 cm. The Young's modulus was calculated by linear regression from the slope of the stress-strain curves in the linear deformation zone and the plateau stress from the stress value in the plastic region.

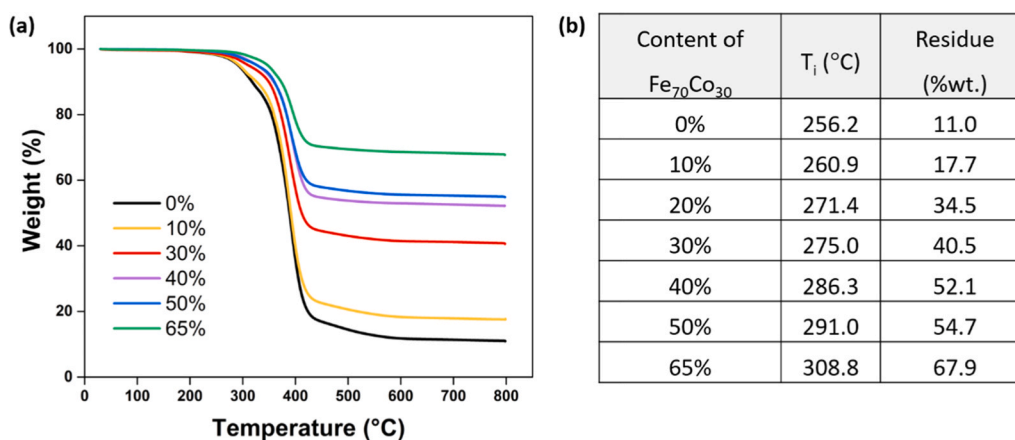
Room-temperature  $M(H)$  hysteresis loop measurements were carried out using a vibrating sample magnetometer (VSM), with an applied external magnetic field ranging between -1.7 and 1.7 T. Magnetostriction measurements  $\lambda(H)$  were performed by using rectangle shaped strain gauges with a gauge factor of 2.075 and a resistance of 120 Ω. These were connected to a Wheatstone bridge working in half-bridge configuration, and including a passive gauge as reference. The strain gauges were glued on the composites using a M-Bond 600 adhesive (purchased from Micro-Measurements).

Dynamic Mechanical Thermal Analysis (DMTA) was performed in bending mode on a DMA 1 from Mettler Toledo. Magnetic foam films (MFF), with samples dimensions approximately 1 mm thick, 5 mm wide and 30 mm long, were measured using the dual cantilever clamp. Magnetic foams (MF), with samples dimensions approximately 4 mm thick, 6 mm wide and 20 mm long, were measured using the single cantilever clamp. Both measurements were performed at room temperature (25 °C) and 1 Hz frequency measuring the force necessary to deform the sample 900 μm (in steps of 10 μm).

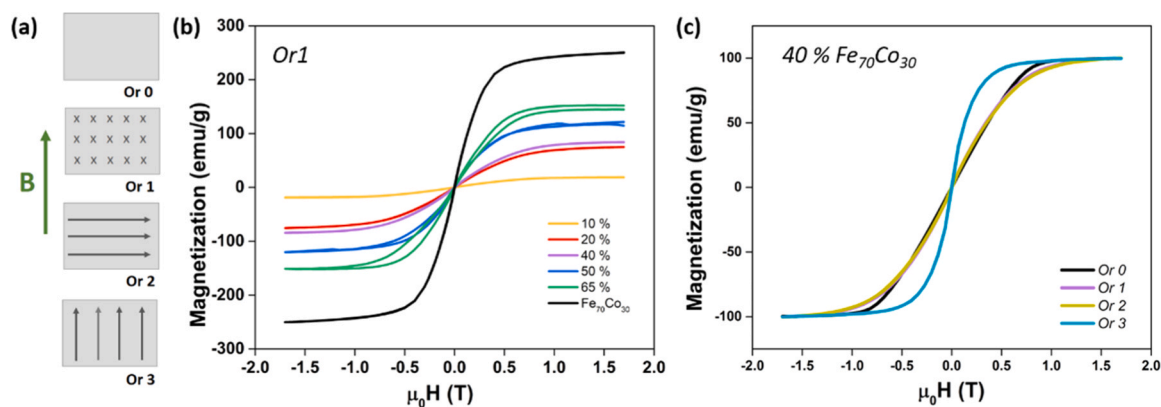
## 3. Results and discussion

### 3.1. Magnetic foam (MF)

As previously mentioned, different MF composites were prepared with different percentages of microparticles and orientations. The obtained composites were firstly characterized in terms of their morphology. Fig. 5 shows samples of the fabricated MF with different percentages of  $\text{Fe}_{70}\text{Co}_{30}$  magnetic particles, prepared under the application of a magnetic field. The circular disks shown in Fig. 5(a) result from a transverse cut through the centre of the cylindrical material, which correspond to the planes used to study the effect of the applied



**Fig. 6.** TGA measurements of the MF composites for different  $\text{Fe}_{70}\text{Co}_{30}$  content (a). Table with initial degradation temperature ( $T_i$ ) and temperature at the maximum weight loss rate ( $T_d$ ) values for the foams, along with residual weight percentages. (b).



**Fig. 7.** Scheme of the orientation of the magnetic field during hysteresis loop measurements in relation to the alignment of the magnetic microparticles (a). Hysteresis loops of MF<sub>Or1</sub> for 10, 30, 40, 50 and 65 % content of Fe<sub>70</sub>Co<sub>30</sub> as well as the raw microparticles (b), and of MF with 40 % content of Fe<sub>70</sub>Co<sub>30</sub> with different alignments of the magnetic microparticles (c).

magnetic field in orientations Or2 and Or3.

As it can be observed, the particle orientation process was successful, as the magnetic microparticles lines are clearly identified, even to the naked eye. The orientation was confirmed through SEM analysis, with images for composites containing 40 and 65 % wt of microparticles shown in Fig. 5(c) and (d), respectively. However, it can be also appreciated the aggregation of the microparticle chains for the 65 % wt content, which is not observed at lower percentages. This aggregation could impact the material's performance.

Further, the materials were characterized regarding to their degradation temperatures. The thermal characterization of the composites was performed through TGA measurements for the MF with different percentages of magnetic microparticles. The results are shown in Fig. 6.

The thermogravimetric analysis reveals two primary stages of mass loss, associated with degradation processes: the first occurs between 290°C and 310°C, and the second, takes place between 350°C and 450°C. As reported in the literature[30], these stages correspond to the breakdown of the hard and soft segments of the polyurethane (PU) chains, respectively. Table presented in the Fig. 6(b) summarizes the initial decomposition (T<sub>i</sub>) and the weight percentages of the residue remaining at the end of degradation (% residual weight).

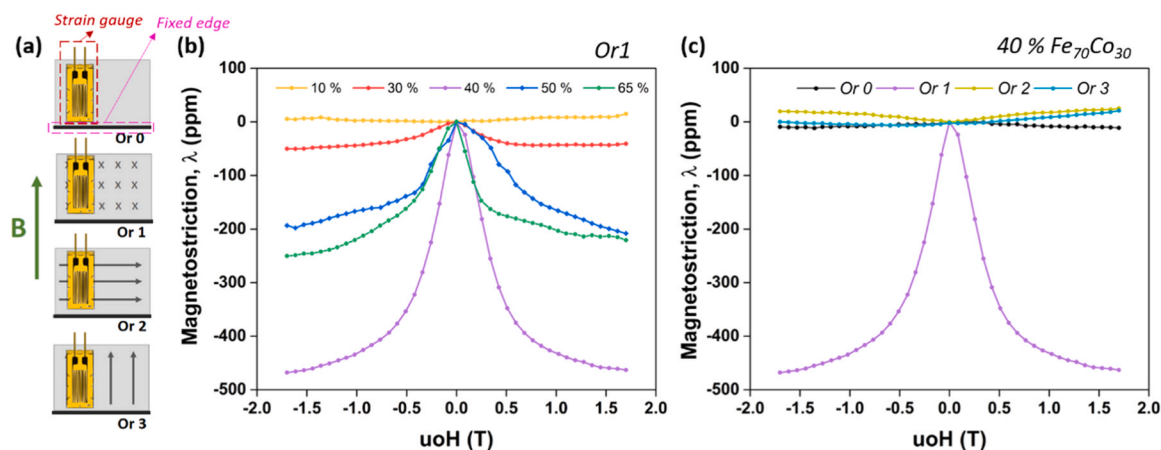
The initial degradation temperature (T<sub>i</sub>) increases as the microparticle content rises and the polymer content decreases, indicating that the particles do not accelerate the material's degradation. In any case, they improve its stability. The temperature at which the maximum weight loss rate (T<sub>d</sub>) occurs remains approximately 390°C across all samples,

regardless of microparticle concentration. Conversely, the final residual mass increases with a higher percentage of Fe<sub>70</sub>Co<sub>30</sub> magnetic particles, as they do not degrade even at temperatures as high as 800°C[31].

The magnetic properties of the composites were evaluated by measuring the magnetic hysteresis loops and magnetostriction curves. The magnetic field was applied with the orientation (1, 0, 0). The magnetic hysteresis loops of samples with different particles percentage, with orientation Or1 (0, 0, 1), as well as of pure Fe<sub>70</sub>Co<sub>30</sub>, are represented in Fig. 7(a).

As expected, the saturation magnetization of the MF increase as the percentage of the Fe<sub>70</sub>Co<sub>30</sub> content does, reaching a maximum value 144 emu/g for the MF of 65 % wt of microparticle, being the coercivity negligible. It can be noted that for the 50 % wt and 65 % wt samples, branches of the respective hysteresis loops do not overlap. Since this behaviour only appears at these high microparticles content percentages, it may be attributed to movement of the sample caused by the applied magnetic field rather than to a structural property. This displacement is much more pronounced at higher percentages, as the sample tends to respond more strongly to the applied field. This movement alters the sample's orientation in favour of the field direction, resulting in a curve that reaches saturation earlier than expected. Consequently, the effect of the orientation of the MF with respect to the applied magnetic field was studied for the MF of 40 %wt content of microparticle. Those results are represented in Fig. 7(c).

From this Fig. 7(c), it is clear that the way the saturation magnetization is reached is consistent across all samples. As expected, the



**Fig. 8.** Scheme of the orientation of the magnetic field during hysteresis loop measurements in relation to the alignment of the magnetic microparticles and the piezoelectric strain gauge (a). Magnetostriction curves of MF Or1 orientation for 10, 30, 40, 50 and 65 %wt content of Fe<sub>70</sub>Co<sub>30</sub> (b), and for MF 40 %wt content with different orientations (c).

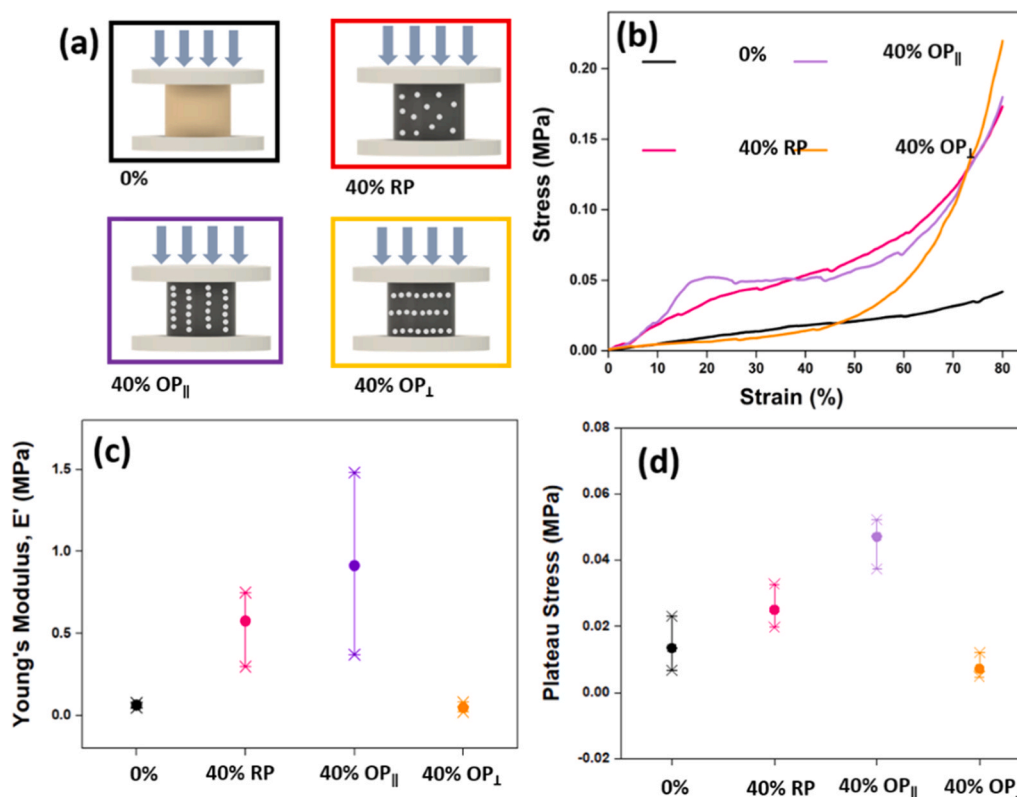


Fig. 9. Scheme of the force direction and microparticle chain orientation during compression tests of MF with 40 wt% magnetic particles (a). Representative stress-strain curves from the compression tests (b). Young's modulus in the elastic region (c), and plateau stress in the plastic region (d).

samples aligned with the applied magnetic field (*Or3*) require the lowest value of applied magnetic field to reach saturation, about 0.7 T. Among the other orientations, the MF with randomly oriented particles (*Or0*) saturates at the applied magnetic field of 1.0 T, while the ones with orientations *Or1* and *Or2* saturate about a magnetic field of 1.4 T. These measurements evidence that these composites are highly anisotropic magnetic materials, since their hysteresis loops depend strongly on the direction of the applied field. Chains of magnetic particles exhibit a single magnetic easy axis aligned with the chain direction, resulting from strong dipolar interactions between Fe<sub>70</sub>Co<sub>30</sub> microparticles within the chain. Although the individual particles are magnetically isotropic, the collective dipolar coupling induces anisotropy at the chain level, which dominates the magnetic response.

Fig 8(b) and (c) show the measured magnetostriction curves of the MF under two different conditions: (b) varying Fe<sub>70</sub>Co<sub>30</sub> content, with magnetic microparticles aligned perpendicularly to both the applied magnetic field and the magnetostriction measurement direction (*Or1*); and (c) a fixed Fe<sub>70</sub>Co<sub>30</sub> concentration of 40 %, while the orientation of the applied magnetic field varies. The sample is fixed along one of its edges, as shown in Fig. 8(a).

The analysis of magnetostriction curves for MF foam with *Or1* reveals a clear relationship between the Fe<sub>70</sub>Co<sub>30</sub> particle content and the magnetostrictive response. At low particle concentration of 10 wt%, magnetostriction values are low and positive. As the concentration increases, the absolute magnetostriction value also increases but becomes negative, indicating material compression. For the MF<sub>30%</sub> *Or1* sample, the value reaches -55 ppm and a peak near -500 ppm is observed for the MF<sub>40%</sub> *Or1*. However, when the microparticle concentration rises further (to 50 and 65 wt%), the absolute magnetostriction value decreases but remains negative (around -200 ppm). Building on these observations, the influence of magnetic particle orientation on the composite's magnetostrictive response was further investigated for the MF<sub>40%</sub> sample (Fig. 8(c)). The MF with randomly oriented particles

(*Or0*) does not exhibit any significant deformation. In contrast, the MF with orientation *Or2* and *Or3* exhibits a positive magnetostriction. In the case of *Or2*, it reaches a value of 30 ppm. Finally, MF with *Or1* orientation, as previously discussed, shows the highest negative deformation levels, nearly -500 ppm.

This observed behaviour can be explained as a competing effect between the alignment of the particles with the magnetic field through polymeric foam stretching, and their alignment due to material bending. The compression undergone by the MF (reflected in a negative magnetostriction) can be associated with a bending mechanism of the sample, while the stretching is expected to result from the torque exerted on the chains of magnetic particles embedded within the polymer foam, as they attempt to align with the applied magnetic field. These competing mechanisms have to be directly correlated with the material's mechanical behaviour. Thus, seeking for a better understanding of the observed phenomenon, the samples were deeply analysed with respect to their mechanical properties through both compression and DMA measurements.

The results of the compression tests performed on various foam samples are presented in Fig. 9. These tests were conducted on samples with the same microparticle concentration (40 wt%) but different orientations (see Fig. 9(a)). Foam like structures compression tests are typically characterized by three distinct regions [34]. The first region exhibits linear elastic deformation. During the second region, the porous structure collapses due to plastic bending. Finally, the third region involves plastic deformation, where the pore walls start interacting as porosity is minimized.

Fig. 9 shows a strong dependence of the mechanical behaviour exhibited by the composites on particle orientation. Samples without particles and randomly oriented particles (RP case) show the typical behaviour of foam structures described before. Nevertheless, the addition of the Fe<sub>70</sub>Co<sub>30</sub> microparticles increases the stiffness of the material in the initial elastic region (Fig. 9(c)) and enhances the plateau stress in

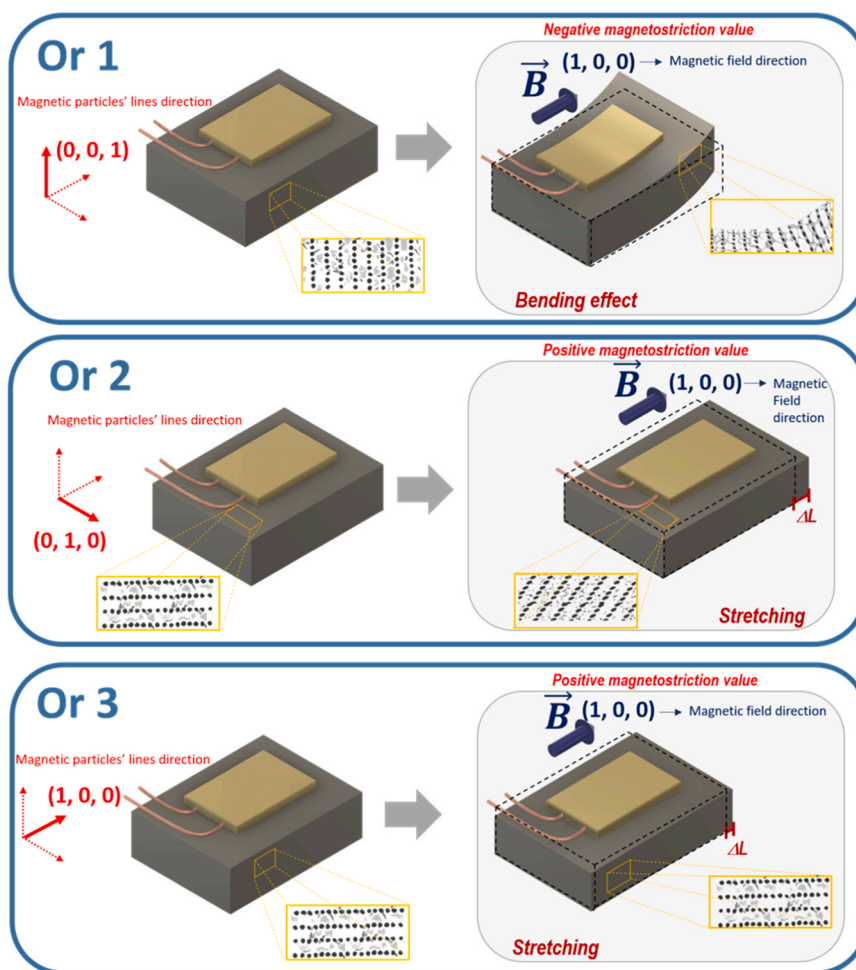


Fig. 10. Illustration of the bending effect (negative magnetostriction) and stretching process (positive magnetostriction) showed by MF under the application of a magnetic field in the samples with magnetic microparticles's line in the direction Or1 (a), Or2 (b) and Or3 (c).

the plastic region (Fig. 9(d)), indicating an overall reinforcement effect. For samples with particles aligned in the direction of the applied force (OP<sub>||</sub> case), the three distinct regions are even more pronounced. Initially, the samples are stiffer due to the alignment of particle chains with the applied force. As the structure collapses, it transitions into region 2, where plastic deformation occurs, resulting in a plateau with a higher stress than the samples without particles and with randomly oriented particles. Finally, as the foam cells begin to collapse and interact, the stress increases again, entering region 3. In contrast, for samples with particles oriented perpendicularly to the force direction (OP<sub>⊥</sub> case), the material appears to immediately enter region 2, as there is little to null resistance at the beginning. While region 1 is likely present, its contribution is minimal and may be neglected, as the stress values are much lower compared to the other cases or to the values observed in region 3 for the parallel orientation.

To the light of the previously obtained data and plausible explanation, the importance of particle orientation in the mechanical properties of foam composites, and consequently in their magnetostrictive response to magnetic fields, becomes evident.

Particles with the same orientation as the magnetic field (Or3) exhibit little to no deformation. Since the particle chains are already aligned with the field, they do not exert any torque on the polymer matrix to reorient themselves. For particles oriented along the Or2 direction, the attempt to align with the magnetic field induces stretching within the film, which results in positive magnetostriction. Conversely, for particles aligned along the Or1 direction, alignment with the applied magnetic field is more easily achieved through bending rather than

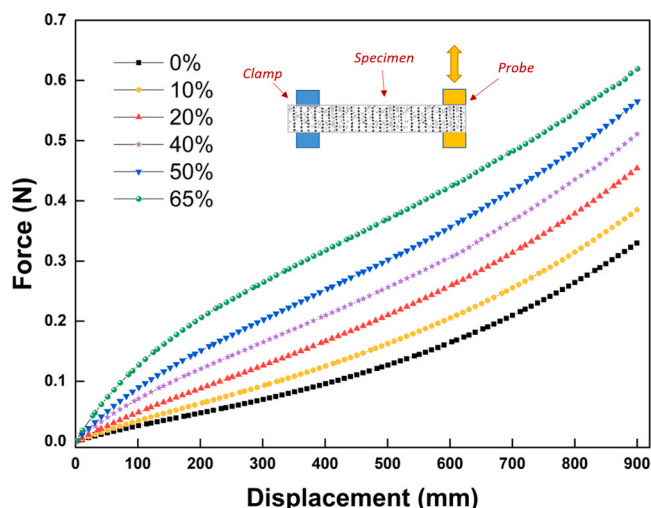


Fig. 11. DMA results obtained in single cantilever bending mode for MF with magnetic microparticles aligned along Or1 and varying particle concentrations.

stretching. This is due to the orientation of the particles, which mechanically favor bending as the dominant realignment mechanism. When the two-layered specimen bends, compressive stress acts on the magnetostrictive material, causing it to shrink. This behavior is observed because, at

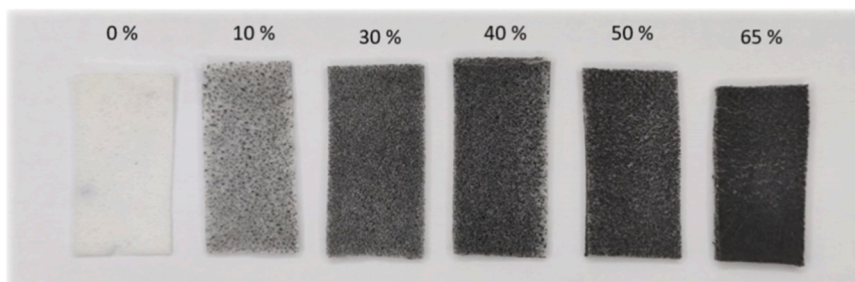


Fig. 12. Image of the obtained MFFs for the different  $\text{Fe}_{70}\text{Co}_{30}$  concentrations.

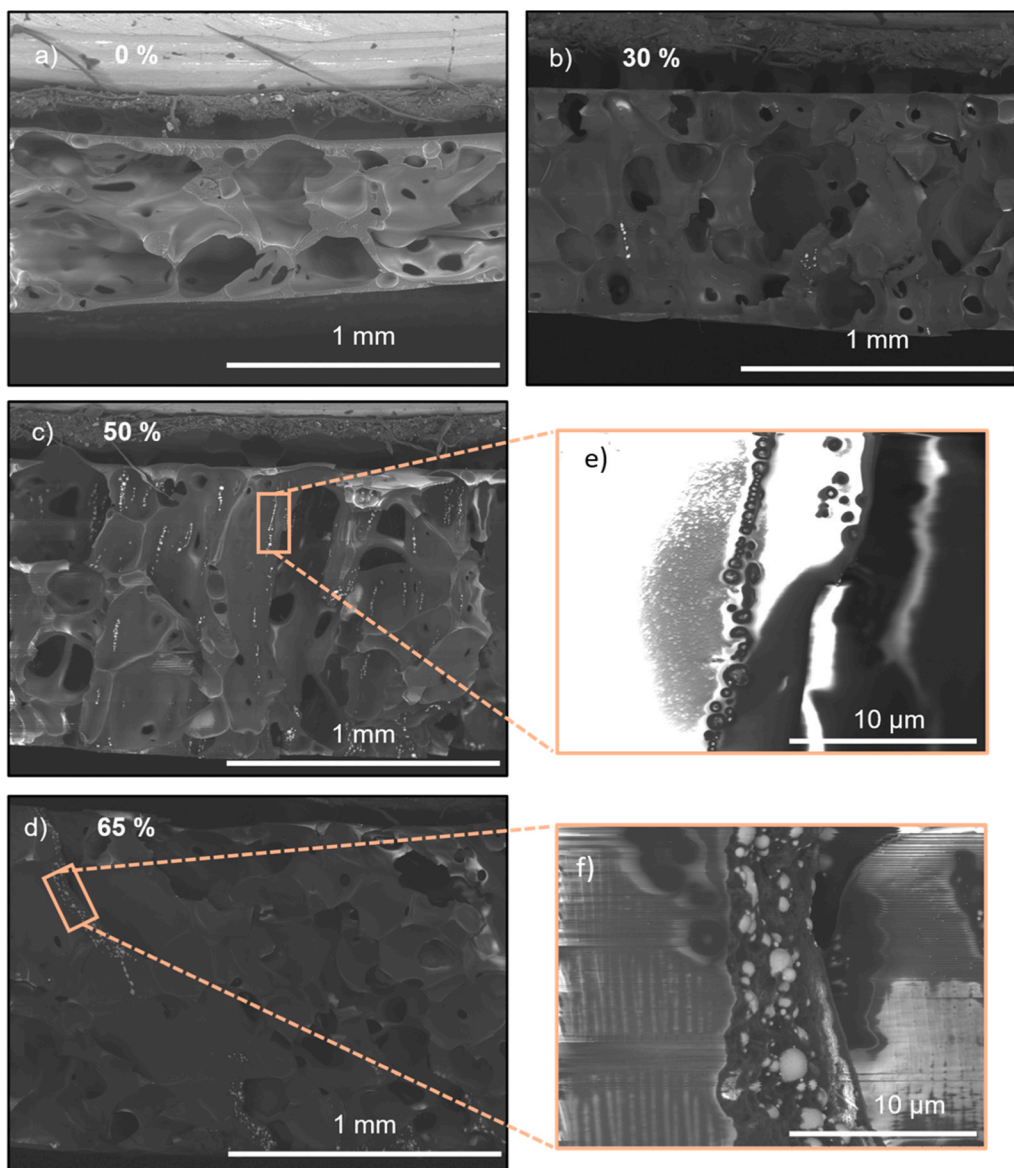


Fig. 13. SEM images obtained for the MFF with different microparticle content: 0, 30, 50 and 65 %, represented in (a), (b), (c) and (d), respectively. (e) and (f) are magnified views of specific areas from images (c) and (d), respectively.

the studied concentrations, the mechanical properties of the polymer matrix still support such deformation modes, as will be further discussed in the following sections. A schematic representation of this behavior is shown in Fig. 10.

Next essential step turns out to be the study of the effect of particle concentration in detail. This analysis was carried out with DMA

technique and using a single cantilever clamp, aiming to understand how the introduction of particles affects the material's bending capability.

DMA measurements (see Fig. 11) indicate that the addition of magnetic microparticles enhances the stiffness of the magnetic foam. When the particle concentration ranges between 10 % and 40 %wt, the

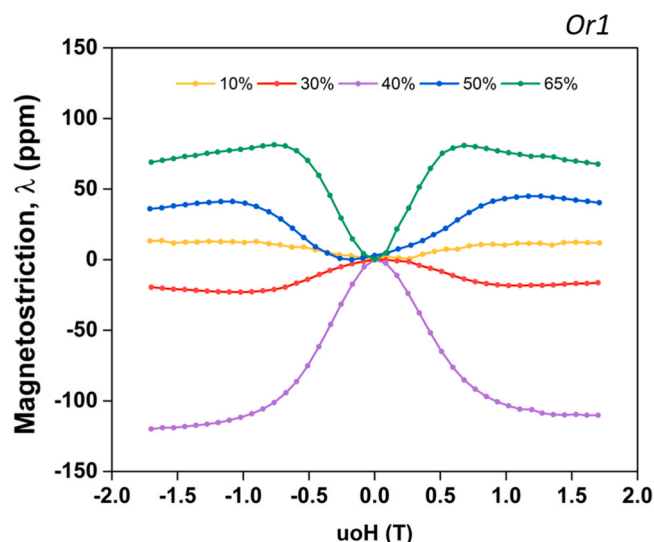


Fig. 14. Magnetostriction curves of MFF with  $\text{Fe}_{70}\text{Co}_{30}$  microparticles oriented along the  $Or1$  direction, for 10, 30, 40, 50, and 65 wt% concentrations.

material remains sufficiently flexible to favour the alignment of particles with the magnetic field through bending rather than direct stretching. This bending behaviour contributes to the observed negative magnetostriction. However, as the concentration increases to 50 %wt and especially to 65 %wt, the material becomes significantly stiffer, reducing its ability to bend. Consequently, the absolute values of magnetostriction decrease at these higher concentrations, although the magnetostriction remains negative.

### 3.2. Magnetic foam films (MFF)

Finally, a new set of magnetic composites was prepared using with the foam film structure (MFF): following the procedure previously described in Section 2, MFFs were prepared with varied  $\text{Fe}_{70}\text{Co}_{30}$  content (10, 30, 40, 50 and 65 %wt). Fig. 12 shows the obtained MFFs for the different microparticle content.

SEM images showed in Fig. 13 reveal the high percentage of porous surface in all these materials, confirming the expected outcome. Additionally, it is observed that with increasing concentrations of oriented microparticles, up to 50 %wt, the particles align in chains along the direction of the applied field (Fig. 12(b) and (c)). However, at higher concentrations, the microparticles begin to agglomerate, forming thicker dendrite-like structures and resulting in a less homogeneous material (Fig. 12(d)). These results agree with the observations made with MF, where similar Composites with higher amount of  $\text{Fe}_{70}\text{Co}_{30}$  particles exhibited phase segregation between the polymer and magnetic filler component of the hybrid material.

Magnetostriction measurements were carried out in MFF with magnetic microparticles in  $Or3$  orientation, as the magnetostriction results for MF was maximum at that particular orientation. The results for the different concentrations of  $\text{Fe}_{70}\text{Co}_{30}$  magnetic filler are shown in Fig. 14.

As it happened for the MF, the magnetostriction curves for MFF are highly dependent on  $\text{Fe}_{70}\text{Co}_{30}$  microparticle content. At low concentrations about a 10 %wt, magnetostriction values are low (~12 ppm) and positive. As the microparticle concentration increases, magnetostriction becomes negative, but with higher absolute values, indicating material compression. For filler concentrations between 30 % and 40 % wt, the magnetostriction reaches values about -20 and -118 ppm, respectively.

However, for concentrations above 50 %wt, the magnetostrictive values become positive, suggesting the concurrence of a stretching process. For the concentration of 65 %wt of microparticles, the magnetostriction reaches a value of + 80 ppm. Once again, it appears to

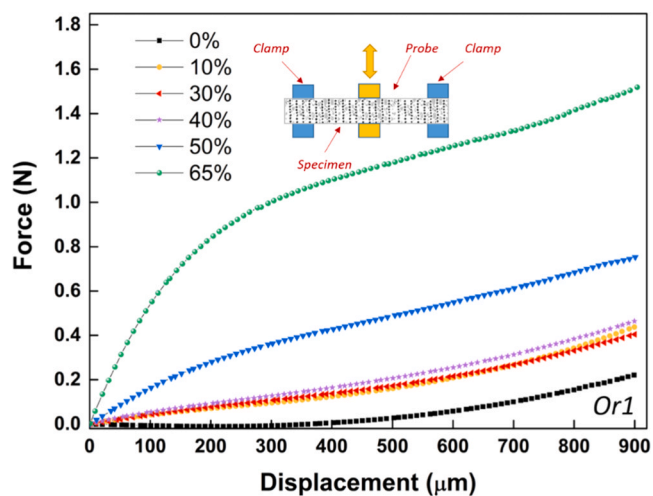


Fig. 15. DMA results obtained in dual cantilever bending mode for MFF with magnetic microparticles aligned along  $Or1$  and varying particle concentrations.

be a competition between two mechanisms: the alignment of the magnetic particles with the magnetic field through polymeric foam stretching which leads to positive magnetostriction values, vs. their alignment via material bending, which causes local film compression and results in negative magnetostriction. The mechanical behaviour will ultimately determine which effect is ruling, for each composite.

DMA measurements using a dual cantilever clamp (see Fig. 15) reveal that the incorporation of magnetic microparticles increases the rigidity of the foam film. For particle concentrations between 10 % and 40 %wt, the material remains flexible enough to favour bending effect over direct stretching, leading as a whole to the observation of a negative magnetostriction value. However, as the concentration reaches 50 and particularly at 65 %wt, stiffness increases significantly. At these highest concentrations, bending is largely suppressed, and the alignment of magnetic particles with the applied field primarily occurs through the stretching of the polymeric foam. This is due to the magnetic torque suffered by the particles[32] as they attempt to align with the field, inducing stress in the polymer matrix and resulting in a total, global positive magnetostriction value. This transition from bending-driven to stretching-driven behaviour is a direct consequence of the increasing stiffness, as confirmed by DMA tests. A graphic scheme of these two different effects is presented in Fig. 16.

## 4. Conclusions

In this study, we analyzed the magnetic response of soft magnetic polyurethane foams loaded with different amounts and orientations of  $\text{Fe}_{70}\text{Co}_{30}$  magnetic microparticles. The observed final structure of the prepared material is clearly dependent on whether the foam was allowed to expand freely (MF) or was confined within a restricted volume (MFF). Composites were prepared and characterized in terms of their morphological, thermal, mechanical, and magnetic properties.

It has been found that the introduction of magnetic microparticles does not significantly affect the thermal properties of the material, with no significant changes recorded in the thermal degradation processes.

On the other hand, both magnetic and mechanical properties are greatly affected by the amount and orientation of the microparticles, being that magnetic particles chains present a single magnetic easy axis oriented along the chain axis.

The magnetostriction tests are highly dependent on both the orientation and concentration of microparticles. There is a competing effect between the bending and stretching of the material as particle chains attempt to align with the magnetic field. Bending leads to material compression, resulting in negative magnetostriction values, while

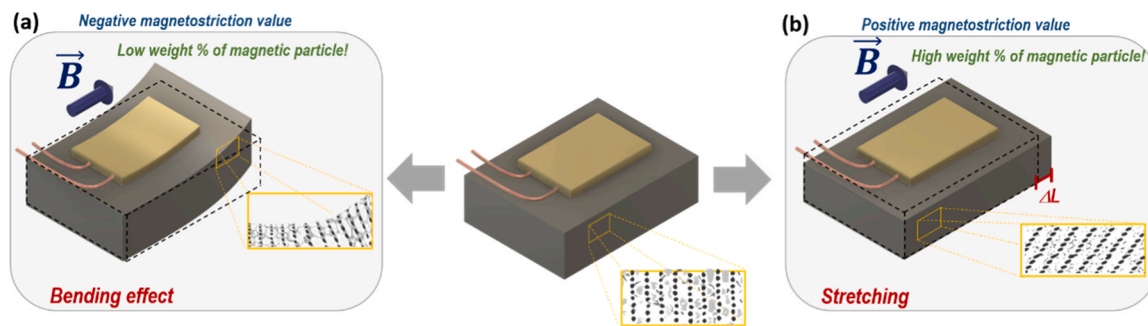


Fig. 16. Illustration of the bending effect (negative magnetostriction) (a) and stretching process (positive magnetostriction) (b) showed MFF under the application of a magnetic field.

stretching causes expansion, leading to positive magnetostriction.

The outcome of this competition is strongly influenced by the mechanical properties of the material. For particle concentrations up to 40 %wt in the composite, the bending effect dominates. However, beyond this concentration, the material becomes more rigid, inhibiting bending and, in some cases, making stretching the dominant deformation mechanism.

As a result, in magnetic foams (MF), the absolute value of magnetostriction decreases (−450 ppm to −200 ppm) as  $\text{Fe}_{70}\text{Co}_{30}$  concentration increases from 40 to 65 wt%. This reduction is attributed to increased stiffness, which makes bending less effective. The effect is even more pronounced in magnetic foam films (MFF), where magnetostriction shifts from a negative value of about −100 ppm at 40 %wt particle concentration to a positive value of + 80 ppm at 65 %wt. In this case, stretching becomes the dominant deformation mechanism.

#### CRedit authorship contribution statement

**J. Gómez-Hurtado:** Writing – original draft, Investigation. **A. Lasheras:** Writing – review & editing, Supervision, Conceptualization. **Vilas-Vilela J. L.:** Funding acquisition. **Ana Catarina Lopes:** Writing – review & editing, Supervision, Conceptualization. **Bou-Ali M. M.:** Funding acquisition. **J. Gutiérrez:** Funding acquisition. **Laza J. M.:** Investigation. **J. Berasategi:** Investigation.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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#### Data availability

Data will be made available on request.

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