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Injection molding of magneto-sensitive polymer composites

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ABSTRACT

Magneto sensitive parts made of a thermoplastic elastomer reinforced with iron microparticles were prepared by imposing a magnetic field during injection molding. In particular, an aluminum mold was designed to host an electro-magnet able to apply a magnetic field during the injection of the material into the mold and its subsequent solidification. Samples obtained in the presence of magnetic field were characterized by a peculiar, aligned distribution of iron particles along the magnetic field lines, and columnar structures of variable length were obtained. The mechanical characterization showed that the samples in which the iron particles were aligned had higher modulus compared to samples in which the particles were randomly dispersed. The magnetic field induced an anisotropic structural reinforcement that imparted to composite samples a magnetostrictive feature, namely the capability to sense the magnetic field and to react with a shape change under the application of the magnetic field.

Keywords

Injection molding, magnetic field, composites, mechanical characterization, magnetostriction

1. Introduction

Magneto-sensitive polymer composites are smart materials made by embedding magnetic particles, micrometric or nanometric in size, into a polymeric matrix. In presence of a suitable magnetic field, particles align themselves along magnetic field lines leading to an anisotropic structural reinforcement. This alignment confers to the material the characteristic of being "smart" in the sense it can react to an external stimulus by changing its properties in the desired way.

In the scientific literature, several magnetorheological (MR) composites based on polymers and aligned magnetic particles (MP) have been proposed [1]. Magnetorheological composites are smart materials able to quickly, continuously and reversibly respond to the application of a magnetic field external stimulus with a change in their properties [2-7]. These smart materials can be fluid or solid [8-10]. MR fluids exhibit a field-dependent yield stress and are conventionally used for vibration control and suspensions in the automotive field. Bastola et al. in 2017 [11] developed a new type of hybrid magnetorheological elastomers (H-MREs) as a combination of an MR fluid and an elastomer matrix by means of a 3D printing technology. MR solids are usually prepared by mixing magnetic particles in viscous precursors of the solid and, unlike the fluids, retain their shape after the molding process. They have field-dependent stiffness and can be used in adaptive tuned variable-stiffness devices, soft actuators and artificial muscles [12, 13]. They include magnetorheological gels [14-16], foams [17-20] and elastomers. In the last decades several authors focused their attention on magnetorheological elastomers (MREs), materials obtained by embedding micro-sized magnetizable particles in an elastomeric matrix during the crosslinking process [21, 22]. The presence of a magnetic field during the production of the elastomer object induces interactions between particles that promote an anisotropic ordered configuration formed by particle chains aligned along the field direction. Anisotropic MR elastomers with special chain structure of magnetic particles have attracted the interest of the researchers and materials have been fabricated based on different matrices, i.e. silicone elastomers, hard natural or synthetic rubber and polyurethane [23-27].

In 2003 Farade and Benine analyzed mechanical and magnetic properties of particle-filled silicon rubber produced by aligning the embedded particles in the elastomer by the application of a magnetic field when the cross-linking of the resin took place [22].

Guan et al. in 2008 [28] prepared and analyzed MRE consisting of carbonyl iron particles displaced in a silicon rubber matrix. They proved that magnetostrictive effect (shape variation of such materials during the application of a magnetic field) increases on increasing the volume fraction of particles. Magnetostriction generally increases with increasing magnetic field and orientation of particles influences the magnetostrictive performance significantly. In 2010 Wu et al. [29] investigated the influences of carbonyl iron particle content on the microstructure, thermal, mechanical and MR properties of PU MREs. They found that the chain-like structures, obtained by alignment of the particles according to the magnetic field lines, becomes denser as the iron content increases. Furthermore, they demonstrated that the orientation of carbonyl iron particles can greatly enhance the compressive strength of the composite, but the tensile properties of PU MREs decrease. In 2017 Perales-Martínez et al. [30] studied the rheological and mechanical properties of magnetorheological elastomer based on silicone rubber with different contents of carbonyl iron micro-particles. They found that the enhancement of the mechanical properties was achieved with the adding of 20% of carbonyl iron particles to the silicon rubber matrix, obtaining up to 111% of additional stretch and 95% of increment in tensile strength compared to that of the bare material.

Until now, to these authors' knowledge, only magneto-sensitive elastomers produced by curing the magnetizable particles/elastomeric matrix mixtures in presence of a magnetic field have been proposed [31]. In this work, the combination of magneto-sensitive elastomers concept to the injection molding process is proposed. In fact, among polymer processing technologies, injection molding allows large scale productions of 3D articles. The idea behind this work involves the use of injection molding technology in order to make profit of its main advantage: the ability to give arbitrary and complex shapes to the artifacts. In this way, if the polymer viscosity is low enough, magnetic particles can be aligned by the application of an external magnetic field. Gradients of mechanical properties inside the injection molded parts can be obtained depending on the intensity and the magnetic field direction. To this aim, a special mold has been designed and a preliminary rheological study has been made in order to choose the elastomer suitable for the purpose.

2. Materials and methods

A polyolefin elastomer, Engage 8402 supplied by DuPont Dow Elastomers (Midland, Michigan, USA) was adopted in this work. Engage is an ethylene-octene copolymer that performs well in a wide range of thermoplastic elastomer applications. The properties of the adopted polymer are reported in Table 1.

Table 1

Properties of Engage 8402 as provided by the supplier**.**

Property	**Method**	**Unit**	**Typical value**
Melt Flow Index (190°C/2.16 kg)	ASTM D-1236	dg/min	30
Total Crystallinity	-	%	34
Melt Temperature	DSC Melting Peak (Rate 10°C/min)	°C	96
Glass Transition Temperature	DSC Inflection Point	°C	-44
Flexural Modulus (2% Secant)	ASTM D-790	MPa	69.9
Density	ASTM D-792	g/cm^3	0.902

Carbonyl Iron Powder CIP SQ supplied by Basf (Ludwingshafen, Germany) was adopted as filler in this work. These particles have performed well for magneto-sensitive polyurethane foams [31]. The main properties of the adopted Carbonyl Iron Powder are: minimum Fe content

99.5 %, maximum C content 0.05 %, maximum C content 0.22 %, mean particle size lower than 10 microns.

2.1 Masterbatch preparation and Rheological measurements

A masterbatch with 50 % by weight of iron microparticles (particle size -325 meshes, assay 97%), supplied by Sigma Aldrich (Saint Louis, Missouri, USA) was produced with Engage 8402 by means of a twin screw extruder (type PTW100 from Haake Thermo GmbH, Germany). Subsequently, in order to obtain a compound with 20 % by weight of iron particles, the masterbatch was diluted with neat polymer directly in the injection molding machine. The extrusions were conducted at 160°C, while the die temperature was set at 130°C. Rheological properties were studied by means of a Haake Mars rotational rheometer (Thermo Haake GmBH, Germany), rheological tests were performed on the neat Engage 8402 and Engage 8402+50% Fe by weight (10% by volume) to select the best processing conditions. These tests were carried out at different temperatures (160 °C, 180 °C and 200 °C) thus obtaining the dependence of the complex viscosity, G' and G" on the oscillation frequency.

2.2 Injection molding

A 70-ton Negri-Bossi injection molding machine (CANBIMAT 65/185, from Negri-Bossi SpA, Italy) with a screw diameter of 25 mm and L/D=22 was used in this work. Clamping unit of the injection molding machine consists of an aluminum mold expressly designed to host an electromagnet which generates a magnetic field in the cavity (Figure 1).

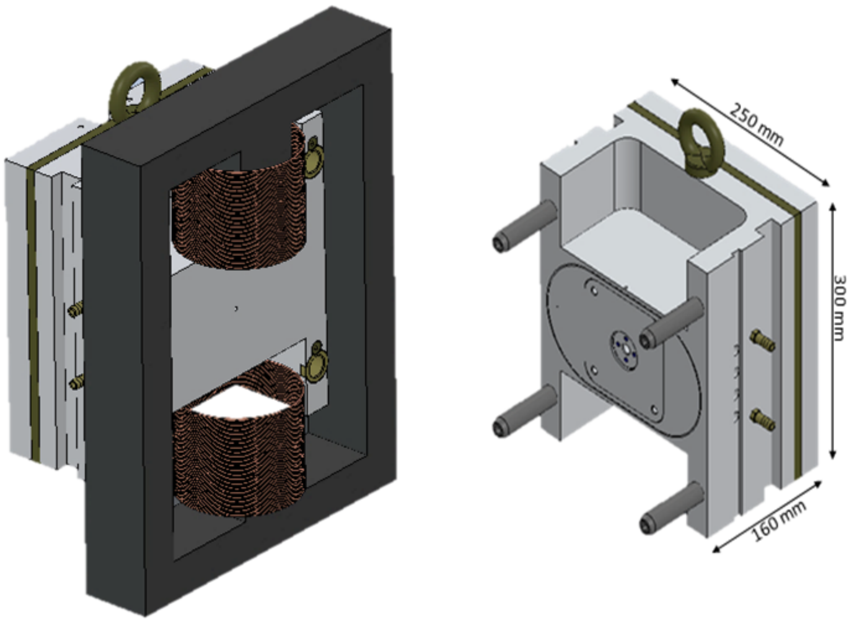
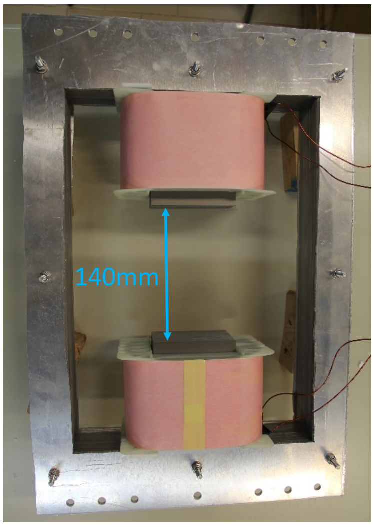
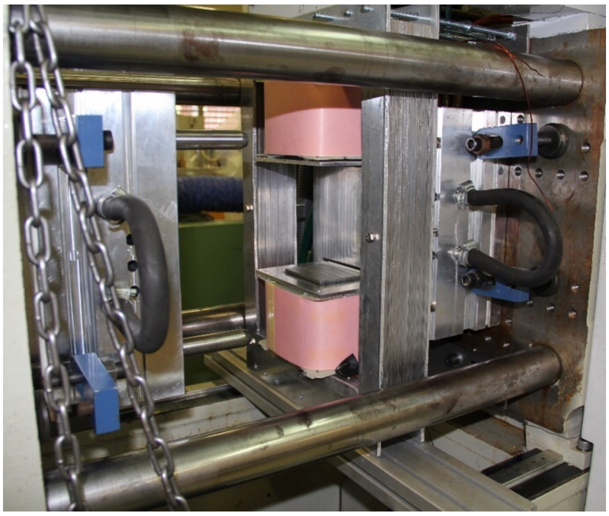


Fig. 1. Sketch of the Aluminum mold with the custom electromagnet.

Aluminum is chosen because it does not interfere with the magnetic field and allows to preserve the spatial distribution of the magnetic field lines, in order to exploit them to properly align the reinforcing particles. The magnetic field time profile was optimized to properly move the iron particles in linear aggregates just before the polymer consolidation during cooling in the mold. The electromagnet (Figure 2), fixed to the stationary platen of the injection molding machine, consists of two coils of copper wires windings (copper wire diameter 1.4 mm, tolerance 2 mm, winding resistance is 6.7 Ohm) connected to a power supply (EA-PSI 8360-10 T) and a laminated magnetic core to form an “iron circuit” for the magnetic field lines. The dimensions of the electromagnet are a trade-off among magnetic performance, maneuverability and safe operations of the injection molding machine, as shown in Fig. 2b.



a



b

Fig. 2. a) Custom Electromagnet with a 140 mm airgap; b) aluminum mold hosting the electromagnet

The magnetic core is laminated in order to avoid eddy currents effects. The iron sheets are standard M300-35A ISO. Number of spires and geometry of the magnetic core allow to obtain a magnetic flux of 1.3 mWb.

The cavity with dumbbell geometry is obtained on a circular insert mounted on the moving mold. The dumbbell cavity, 3 mm thick, was dimensioned according to the Type V of ASTM D 638-03. The rotation of the circular insert allows to orient the dumbbell sample at different angles with respect to the magnetic field lines (Figure 3).

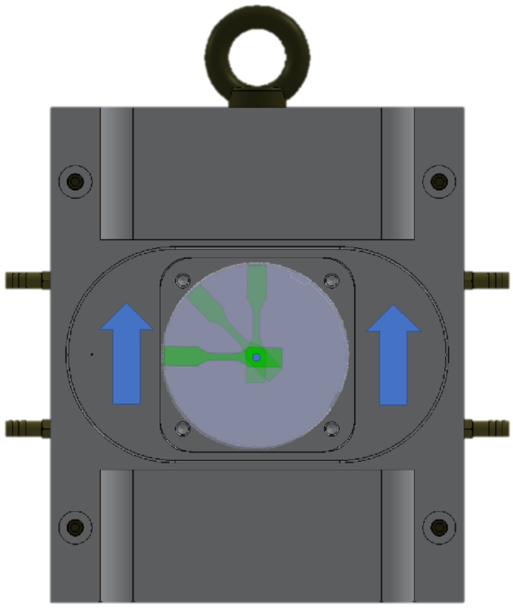


Fig. 3. Moving mold with dumbbell geometry obtained on a circular insert. The blue arrows refer to the direction of the MF given by the custom electromagnet.

This would lead to iron particles aligned with different angles with respect to the dumbbell main axis (which coincides with the flow direction during molding), thus inducing different mechanical and magneto-mechanical properties. In this work, the dumbbell samples were molded with the main axis of the sample oriented at 0 degree, 45 degree and 90 degree with respect to the magnetic field orientation (Figure 3). The samples will be coded according to this feature so that “NoMF” will refer to a sample molded without magnetic field and “MF X°” will refer to a sample molded with the main axis of the dumbbell oriented at X° degrees with respect to the Magnetic Field direction. The experimental conditions adopted during the injection molding process are reported in Table 2.

Table 2

Processing condition for injection molding.

Temperature profile [°C] **(increasing distance from the hopper to the nozzle)**	200 - 210 - 220 - 220
Nozzle temperature [°C]	220
Mold temperature [°C]	45
Filling pressure (hydraulic system) [bar]	70
Screw rotation speed [rpm]	100
Back pressure (hydraulic system) [bar]	2
Injection speed [%]	30
Shot size [cm3]	2.3
Cooling time [s]	60
Iron content [% by weight]	20 - 50
Working current [A]	8 - 9
Working voltage [V]	115 - 120
Magnetic field “ON” time [s]	10

During the injection molding process, at the beginning of the injection phase, the power supply is switched on so that the desired magnetic field is generated. The magnetic field is kept active for 10 seconds, in order to allow the orientation of the iron particles within the molten polymer.

2.3 Scanning electron microscopy

After production, the samples were sectioned and analyzed by a LEO-EVO 50 Scanning electron microscope (Zeiss, Germany) in order to observe the spatial distribution of iron particles in the polymeric matrix. The section observed by scanning electron microscopy is the central part of the narrow section.

2.4 Mechanical properties

Mechanical properties of the samples have been tested by a 5kN universal testing machine (LRXplus, Lloyd, United Kingdom). Self-tightening grips (model TH243-20 from DGTS, Italy) have been used for tensile tests with a column speed of 10mm/min. The internal load cells and extensimeter have been used to measure forces and deformations.

3. Results

The rheological properties of neat Engage and Engage 8402+50% Fe by weight are shown in Fig. 4, in which it is possible to note that 50% by weight of iron powder results in increased viscosity of the material. The rheological results obtained at three temperatures (160°C, 180°C, 200°C) have been superposed, using an appropriate shift factor for each temperature, to obtain a master curve. The shift factors (αT) used to make the master curves of pure Engage and Engage+50%$_w$ Fe are reported in Table 3.

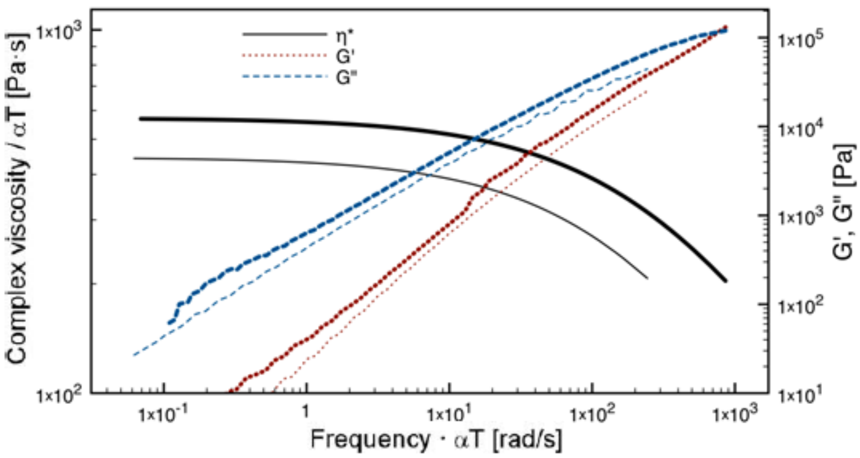


Fig. 4. Rheological properties of neat Engage (thin lines) and Engage 8402+50% Fe by weight (thick lines). Mastercurves at 180°C

Table 3

Shift factors of the mastercurves of pure Engage and Engage+50%$_w$ Fe.

Temperature [°C]	Shift factor Pure Engage	Shift factor Engage+50%$_w$ Fe
160	1.66	1.85
180	1	1
200	0.62	0.69

Samples obtained by injection molding with the application of a magnetic field were observed by SEM in order to verify the degree of orientation of the particles. Figure 5 shows the micrographs of the Engage 8402+Fe 50% by weight obtained by orienting the dumbbell at 0°, 45° and 90° with respect to the direction of the magnetic field. The micrographs on the left show the entire section of the sample, while those on the right side show a magnification of the very central part of the corresponding image on the left.

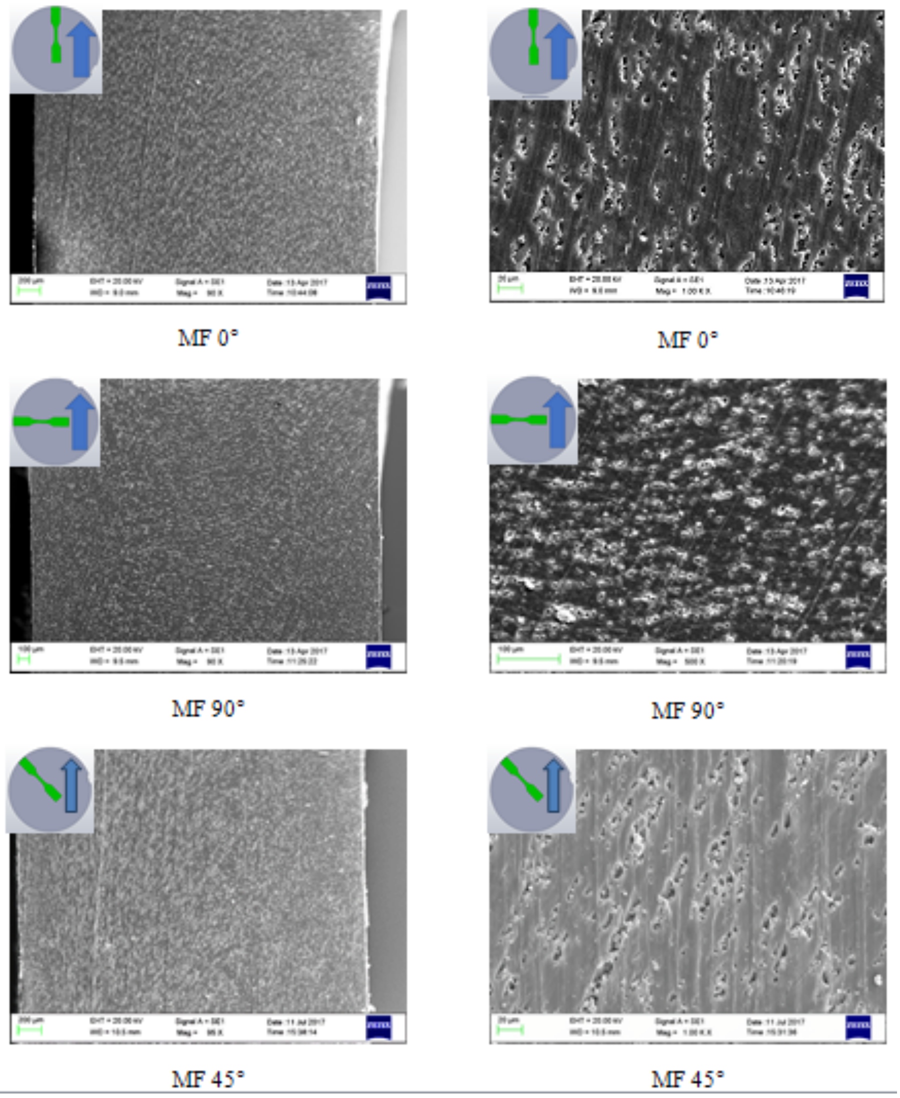


Fig. 5. SEM micrographs of Engage 8402+Fe 50% by weight obtained by orienting the dumbbell at 0°, 45° and 90° with respect to the direction of the magnetic field.

The images confirm that the iron particles are indeed oriented along the magnetic field direction, developing chain-like structures. The spatial arrangement of particles in the three different cases is also clearly visible: the chains are directed along a direction that exactly corresponds to the direction of the magnetic field during molding. Furthermore, the observation of the entire section of the sample allows the identification of distinct zones (Fig. 6): a central zone oriented in the direction of the magnetic field, an outer area that exhibits a random distribution of the particles, a partially oriented intermediate zone. This result may show that, in perspective, it is possible to obtain different orientation of particles and morphology within the same molded part.

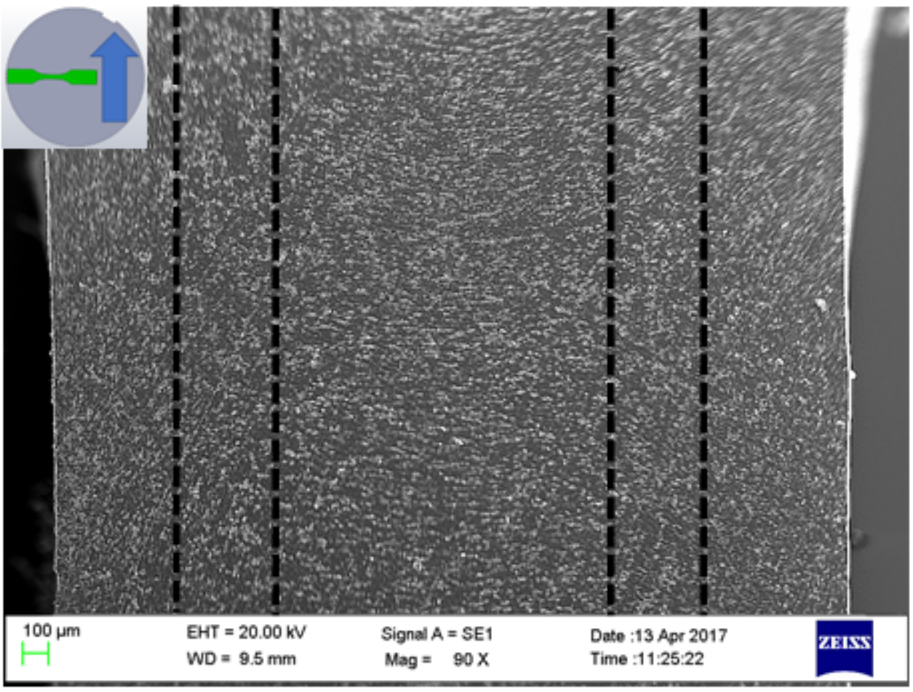
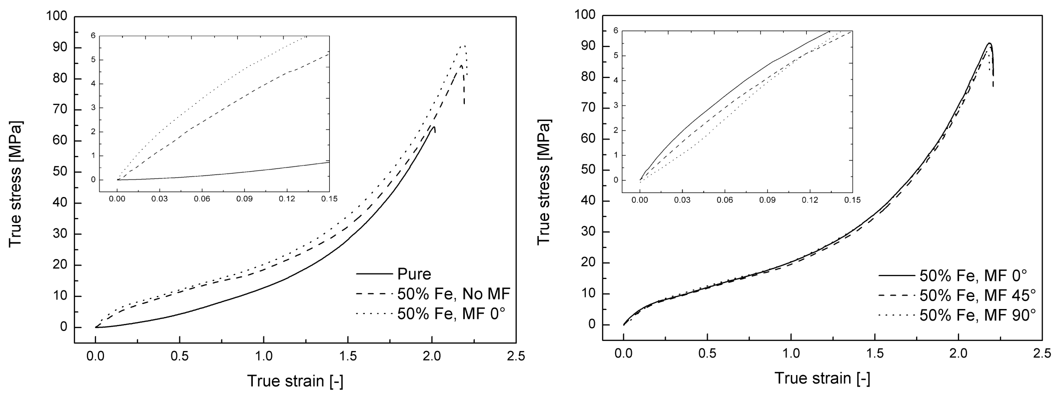


Fig. 6. Engage 8402+Fe 50% by weight, MF 90°: subdivision of the section into an oriented central zone, an outer area with random distribution of the particles, a partially oriented intermediate zone.

This spatial arrangement of particles is closely related to the temperature profile of the polymer melt inside the cavity. In particular, since the solidification proceeds from the sample surface toward the core, the internal layers remain at high temperatures for a longer time. The low temperature of the polymer melt in the outer area implies high viscosity, and thus the action of the magnetic field is not sufficient to allow an orientation and the particles are distributed without a particular order within the polymer.

The presence of aligned particles in chain form can also be detected by placing the molded sample, free to move, under a magnetic field. In fact, when the magnetic field is activated, the sample instantly respond by moving and stopping in a position where the particle chains inside are perfectly aligned with the applied magnetic field lines, similarly to a compass needle. The position that the samples take under a magnetic field indirectly confirms the effective alignment of the iron particles along the designed angle. Also NoMF samples instantly respond to the application of a magnetic field, but the direction is always that of the main flow during molding (namely MF 0°). This phenomenon is probably due to the fact that, even in absence of magnetic field during the foam injection, the iron particles can assume a certain anisotropic distribution, hence some degree of orientation, due to the flow.

The mechanical properties of samples were characterized in tension mode, in order to evaluate the stiffness and the strength as function of the reinforcement spatial distribution. Figure 7 shows the stress-strain curves obtained by tensile test on pure Engage, Engage with 50% Fe by weight without magnetic field (50% Fe, No MF) and Engage with 50% Fe oriented at 0°. Since the values of strain are considerable, the curves are expressed in terms of "true stress" and "true strain". In particular, if L is the current length of the gage, L_0 is its initial value, P is the load and A_0 is the initial cross-sectional area, the "true strain" was calculated as $\varepsilon = \ln(L/L_0)$ and the "true stress" as $\sigma = P/A_0 \, L/L_0$.



a b

Fig. 7. Stress-strain curves of pure Engage, Engage with 50% Fe by weight without magnetic field (50% Fe, No MF) and Engage with 50% Fe oriented at 0° (50%Fe, MF 0°) (a); stress-strain curves of Engage with 50% Fe oriented at 0°, 45° and 90° (b).

The slope of the initial linear portion of the stress-strain curves represents the Young’s modulus of the tested material. Figure 8 shows the Young’s modulus of pure Engage, Engage with 20% and with 50% Fe by weight.

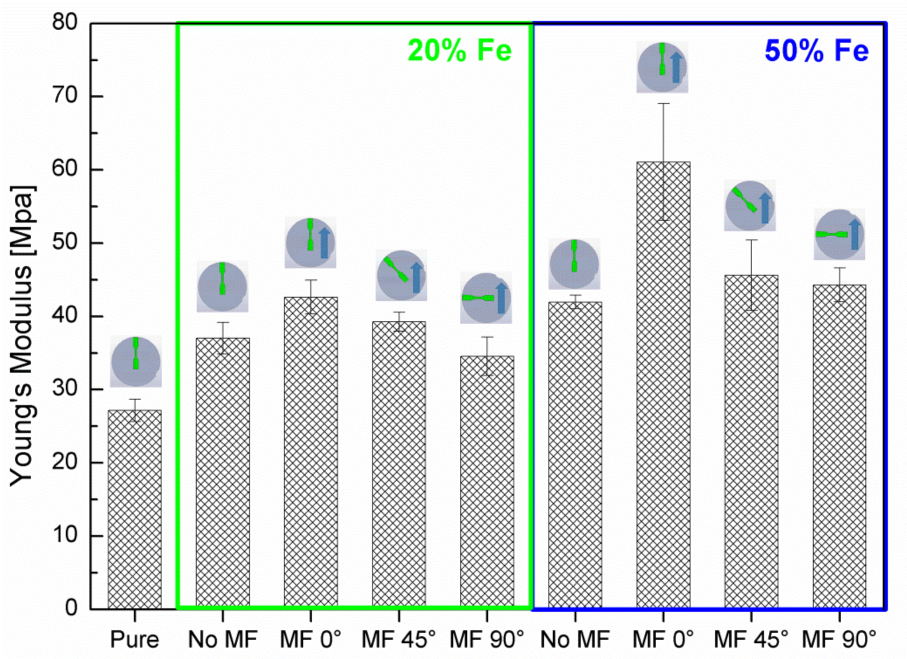


Fig. 8. Young’s modulus of pure Engage and Engage with 20% and 50% by weight of iron particles oriented at 0°. 45° and 90° with respect to the magnetic field direction.

Results without the application of any magnetic field and with magnetic field at 0°, 45° and 90° with respect to the flow direction are reported. It is possible to observe that the addition of iron particles increases the Young’s modulus, and that it is higher in case of magnetic field at 0° with respect to the flow direction. In particular, the Young's modulus of Engage with 50 wt% and MF at 0° is 33% higher than that measured on NoMF sample with the same iron content.

Magnetostrictive behaviour

Samples injection molded at 45° with respect to the magnetic field direction were subjected to further tests, in order to evaluate their smart “active” behavior. In particular, the custom electromagnet was used and two MF 45° samples were positioned in the center of the magnetic field and blocked on one side. When the magnetic field is activated, the two samples tend to deform by aligning the chain-like structures along the direction of the magnetic field, thus inducing a shape change. Samples are deformed upwards or downwards depending on the actual direction of the particles inside them (see the scheme reported in Fig. 9, which shows the response of samples with 20 wt% and 50 wt% of Fe under a MF set at 45°). It is possible to observe that the samples with 20 wt % of Fe undergo an almost imperceptible deformation (Fig. 9, left), so the particle orientation is not enough to induce visible deformation. Vice versa, the samples with 50 wt% of Fe (Fig. 9, right) undergo a visible deformation due to the tendency of the chain-like structures to align with the magnetic field lines. In particular, they tend to bend to move apart in one case (top pictures in Fig. 9) or to get closer in the other case (bottom pictures in Fig. 9). This proved that the presence of chain-like structures induces a magnetostrictive-like behavior in the injection molded samples, phenomenon applicable in valves or pumps design.

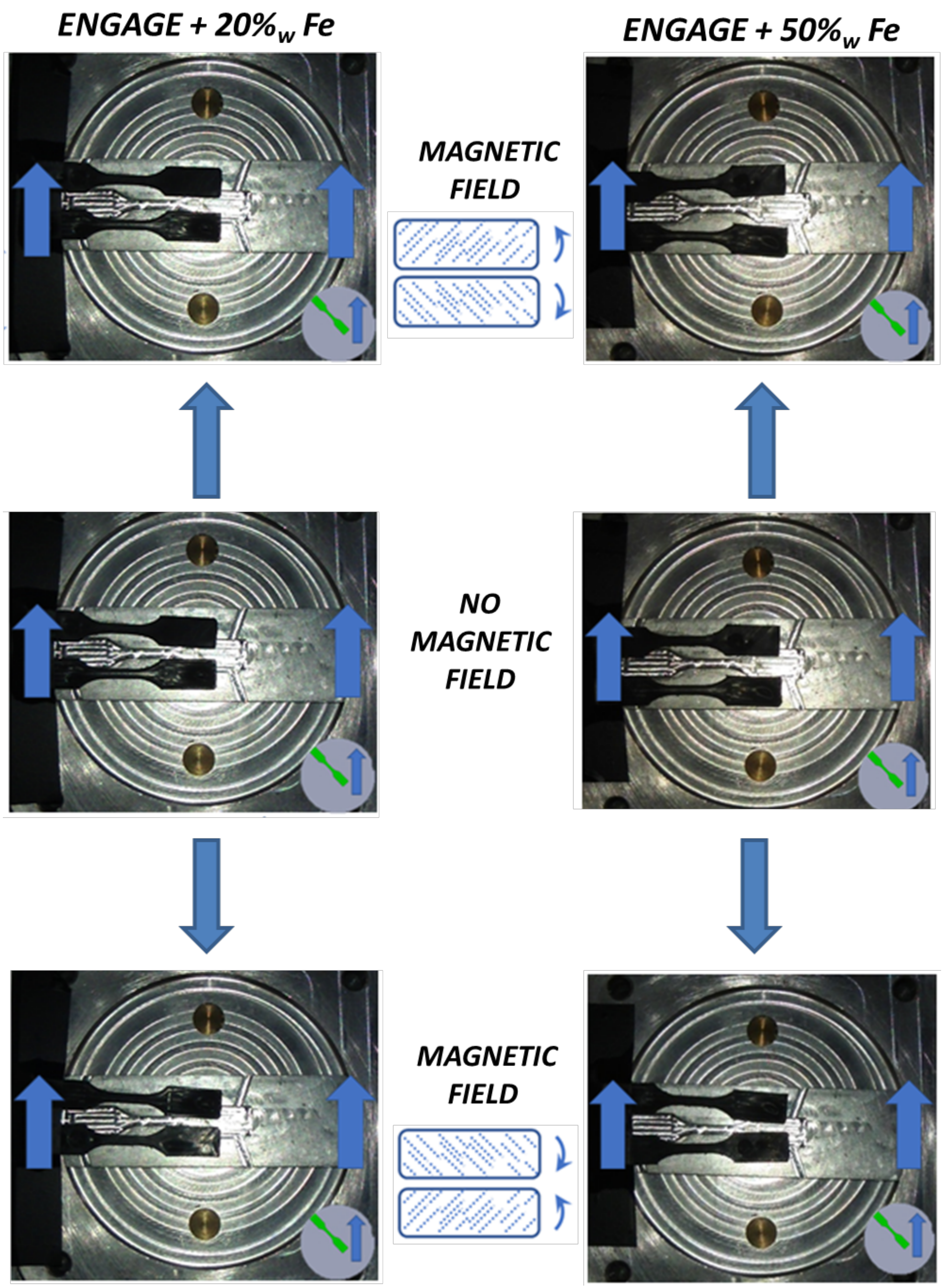


Fig. 9. Magneto-sensitivity tests on samples of Engage+20% Fe by weight (left) and Engage+50% Fe by weight (right) with MF 45°. The schemes indicate the direction of the particles inside the samples.

5. Conclusions

In this work, composite lightweight materials based on a polymeric matrix with embedded magnetic micro-particles have been developed by using a thermoplastic elastomer mixed with iron microparticles.

A new aluminum mold was designed and built in such a way to host an electromagnet, able to impose a magnetic field during the injection of the material into the cavity and its subsequent solidification.

The application of the magnetic field during the injection molding process allowed the formation of columnar aggregates oriented along the magnetic field lines, which were clearly visible by SEM.

These oriented structures make the samples able to orient, if placed in a magnetic field, in the same direction of the magnetic field imposed during injection molding, like the needle of a compass. It was also noticed that the system obtained without the application of a magnetic field also responded to the application of a magnetic field due to some flow-induced orientation in the direction of flow. This phenomenon was interpreted by considering that the iron particles can assume a certain anisotropic distribution, hence some degree of orientation, due to the flow of the polymer after entering the mold. It was observed that the addition of iron particles increases the Young's modulus, which is even higher in case of magnetic field at 0° with respect to the flow direction.

It was verified that the peculiar particle distribution imparted to the samples a magnetostrictive behavior, namely the ability to change shape in presence of an external magnetic field. This property may be exploited in several ways, such for instance in the production of valves or pumps activated in a contactless way by means of a magnetic fields.

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The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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