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Mechanical behaviour of magnetic Silly Putty: viscoelastic and magnetorheological properties

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Abstract
In this work the mechanical and viscoelastic properties of the magnetic Silly Putty are investigated. The Silly Putty is a non-Newtonian material whose response depends on the rate at which it is deformed. For a rapid deformation it behaves as an elastic solid while over a relatively long time scale, the polymer molecules can be untangled and it flows as a fluid. The purpose of this paper is to study the behaviour of this material firstly under a quasi-static compression and shear loading and secondly under a dynamic shear loading. The Silly Putty under study presents a volume fraction of ferromagnetic particles. Hence, both quasi-static and dynamic stress are coupled with several values of magnetic field in order to assess the influence of the magnetization on the mechanical and viscoelastic properties of the material. The approach adopted in this work followed the Design of Experiment method so that evaluating the influence of the variables and their interactions on the system response is possible. The results highlight a strong dependence on the deformation rate while the influence of the magnetic field is weak especially under dynamic shear tests in which the viscous components are predominant.

Keywords
Magnetorheological elastomer, characterization, dynamic complex modulus.

1. Introduction

Silly Putty is the commercial name of a material produced by Dow Corning Corporation (Dow Corning 3179 dilatant compound) based on silicone polymers which display unusual physical properties. Silly Putty is made of 65% poly-dimethilsiloxane (PDMS), 17% silica (crystalline quartz), 9% thixatrol ST (castor oil derivative), 4% dimethyl-siloxane (hydroxy-terminated polymers with boric acid), 1% glycerine and 1% titanium dioxide. Its unusual flow characteristics are mainly due to the PDMS, a viscoelastic material. Viscoelasticity is a property of materials that exhibits both viscous and elastic characteristics depending on the deformation rate. Sometimes the relationship between shear stress and shear rate is not linear, viscoelastic materials may be characterized either as dilatant fluid or pseudoplastic fluid. Dilatant or shear-thickening materials are mixtures that exhibit an increase in apparent viscosity as the shear rate increases (e.g., suspensions of corn starch in water). When at low shear rate, the repulsive particle-particle interactions keep the particles in an ordered, layered, equilibrium structure. However, at shear rate elevated above the critical shear rate, the shear forces pushing the particles together overcome their repulsive interactions and forcing them out of their equilibrium position. Many theories

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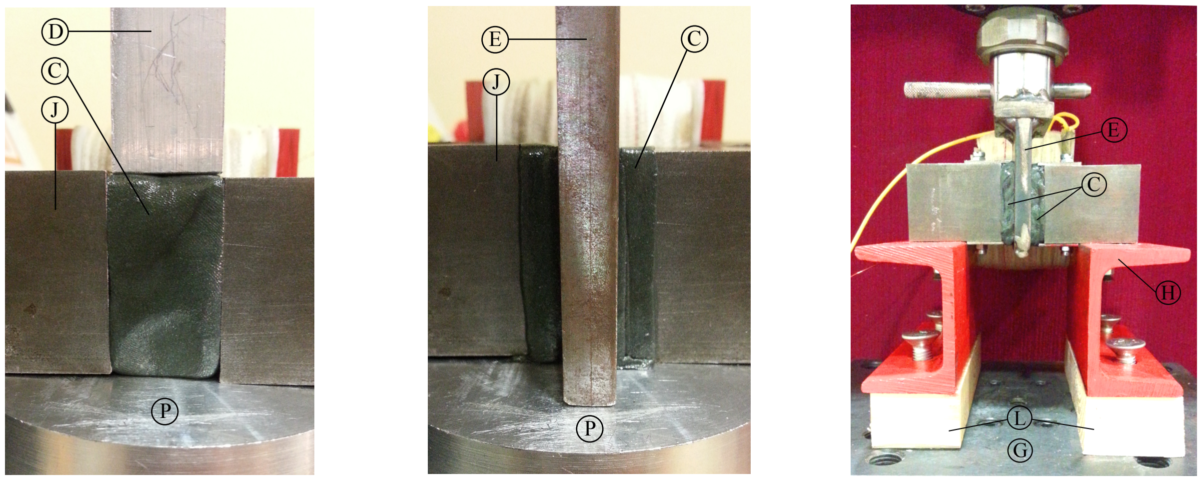
are behind this phenomena (Boersma et al. 1990) like the formation of hydroclusters at high shear rates which drive particles into close proximity leading to an increase in energy dissipation and consequently to a higher viscosity. This theories though are mainly related to the shear thickening effect and seems not quite suitable for the Silly Putty, which shows (Cross 2012a) a shear thinning effect as the shear ratio increases. The higher molecular weight PDMS has a characteristic relaxation time, defined by the time that a random walk allows the chain to relax from the stretched state through thermal vibration (Askeland et al. 2010). Moreover, due to the boric acid there are also transient boron cross-links arising from associating boron linkages. These cross-links act to give the Silly Putty a behaviour more similar to an elastic solid than a liquid. However, since these cross-links are dynamic, the material is not permanently locked in place and can consequently flow under long time scale stress (Osswald and Menges 2012). Despite the fact that mechanical and viscoelastic properties of Silly Putty are well known in a qualitative sense, finding quantitative measurement of these properties is difficult. In scientific literature a few studies were carried out to determine the viscoelastic properties of PDMS-based elastomers (Li et al. 2010, Niu et al. 2007, Tian et al. 2013) and Silly Putty (Cross 2012a,b). Cross (2012a) subjected the material to a rapid deformation by dropping different masses at different heights onto one end of a Silly Putty cylinder, thus he was able to assess the deformations and the elastic forces at different speeds rates. Cross studied slow deformations by compressing a cylinder of Silly Putty in a testing machine at a fixed rate ranging from 200 mm/min to 1000 mm/min. He found an increase in the force by about a factor of 3, at any given displacement, when the compression rate increased by a factor of 5. Other tests have shown that the mechanical properties of Silly Putty are temperature dependent. Testing the material up to failure at 30°C, leads an elongation at break of 200% higher than that which is obtained at 20°C. Even though the mechanical and viscoelastic properties has been previously considered in other works, the magnetorheological properties are almost unknown. In (Tian et al. 2013) four isotropic PDMS based magnetorheological elastomer samples with different percentages of iron particles were fabricated. Steady state and dynamic tests such as strain amplitude sweep and angular frequency sweep were used to test the magnetorheology of PDMS magnetorheological elastomers and the results could be applied also to the magnetic Silly Putty. The steady state tests showed that the increase of iron particles in the sample would lower the viscoelastic linear range of magnetorheological elastomers (MREs). The dynamic and magnetic field intensity sweep test proved that the samples with higher iron weight fraction show higher initial storage and loss moduli and also higher magnetorheological effects. A similar approach was adopted in (Padalka et al. 2010) where the stiffness and damping properties of MRE composites were investigated. The samples were filled with 10wt% of Fe, Co and Ni nanowires and were tested under normalized strain amplitude of 1, 2, 3%, cyclic deformation frequency of 1 Hz and magnetic flux density of 0, 0.1 and 0.2 T. In the following sections quasi-static and dynamic tests on magnetic Silly Putty (Enterprises 2013) are described and the results are provided in terms of significant variables involved in the process.

2. Materials and methods

2.1. Experimental set-up

Two different testing machines were used to carry out respectively the quasi-static and dynamic tests. For the quasi-static tests, we used the universal testing machine Galdabini Sun 500, equipped with two different fixtures to perform both compression and shear tests. Since magnetic Silly Putty has an elastomeric nature due to PDMS, overcontraining would lead to extremely high stresses, due to its poisson's ratio close to 0.5. Hence, the material was confined only by the yoke of the magnetic system (Figure 1(a)). The force was measured by a load cell located onto the head of the testing machine. The magnetic field was provided by a coil powered by a direct current supplied using a stabilized TTI power supply system. The values of the deformation rates, of the magnetic fields and the time of application of the latter are described in the Experimental Plan subsection. Since the maximum speed that can be achieved using the Galdabini machine is 1000 mm/min, in order to reach higher deformation rates needed for the dynamic tests, the universal testing machine MTS Mini Bionix 858 was used. The MTS provided the instantaneous force-displacement trend. By means of the MTS Flextest control software, it was possible to set up the characteristics of the input command (number of cycles and the sampling frequency).



(a) (b) (c)

Figure 1. Experimental set up: quasi-static compression (a), shear (b) and dynamic (c).

The approach adopted for dynamic shear tests resembles the dynamic mechanical analysis (DMA)(Menard 2008, Swallowe 1999). DMA is a technique used to evaluate and characterize materials mainly used for studying the viscoelastic behaviour of polymers. A sinusoidal stress is applied and the strain in the material is measured, allowing one to determine the dynamic complex modulus. When a sample is subjected to a sinusoidal oscillating stress, it responds with a similar strain wave, provided the material stays within its elastic limits. When the material responds to the applied wave in a linear way, an in-phase, elastic response is seen, while a viscous fluid gives an out-of-phase response with energy dissipation. Silly Putty falls in between these two behaviours. Equation (1)(Menard 2008) describes the maximum values of strain (γ_0) and stress (τ_0) at the peak of the sinusoidal input wave, δ is the phase lag due to the extra time necessary for molecular motions and relaxations to occur.

$$\gamma = \gamma_0 \sin(\omega t); \qquad \tau = \tau_0 \sin(\omega t + \delta) \tag{1}$$

Using trigonometric relationships and recalling that $\tau_0 = G\gamma_0$, the equation (1b) can be divided into an in-phase component and out-of-phase component, which gives:

$$\tau = \gamma_0 G' \sin(\omega t) + \gamma_0 G'' \cos(\omega t) \tag{2}$$

in which, G', the in-phase component associated with stiffness, is called dynamic storage modulus and represents the ability of the materials to store potential energy and release it upon deformation. G'', the out-of-phase component associated with internal friction, is called dynamic loss modulus and represents the energy dissipation in internal motion (Brown 2002, Boyd and Smith 2007). Subsequently, the dynamic complex modulus can be defined as follows:

$$G^* = \frac{\tau_0}{\gamma_0} e^{i\delta} = \frac{\tau_0}{\gamma_0}(\cos\delta + i\sin\delta) = G' + iG'' \tag{3}$$

2.2. Magnetic system

One of the purposes of this paper is the study of the behaviour of the magnetic Silly Putty under various magnetic fields, thus the design of the magnetic system plays a key role. To increase the efficiency and the accuracy of the applied magnetic field during the experimental tests, particular attention was paid to the magnetic circuit materials and the flux path. On the one hand, in quasi-static compression tests shown in Figure 1(a), both the support plate (P) and the punch (D) are made in aluminium, a paramagnetic material, in order to prevent any disturbance in the magnetic flux through the magnetic Silly Putty (C). On the other hand, in shear mode tests (quasi-static and dynamic) (Figures 1(b), 1(c)) a steel punch (E) was used, so in this way the magnetic flux lines may cross the two layers of Silly Putty (C) without deviations. To avoid any flux loss in the dynamic tests set up, two wooden layers (L) were placed to isolate the support (H) of magnetic yoke from the MTS frame (G)(Figure 1(c)). For both quasi-static and dynamic tests, a magnetic system obtained by cutting a low carbon steel square bar was used. The bar was welded to form the traditional magnetic yoke shape and inserted in a copper coil. The copper coil was made of AWG 22 wire (diameter 0.64 mm) with 1700 coils (Spaggiari and Dragoni 2011, 2013). Even if the maximum allowable current for the wire is 1.2 A, in order to reach the desired magnetic field we used 2 A but for 300 seconds only. In order to prevent excessive heating of the coil we prescribed a cooling of the system every time the coil became too hot. A finite element analysis of the magnetic system was performed by means of FEMM 4.2 software (Finite Element Method Magnetics, (Meeker 2015)). The model of the magnetic core can be considered as a 2D magnetostatic problem (Figure 2(a)). Specifically, FEMM discretized the problem domain using 13043 discrete triangular elements and 6674 nodes. Over each element, the solution is approximated by a linear interpolation of the values of potential at the three vertices of the triangle. With a DC current of 2 A, the required value of 0.2 T of magnetic field is reached as shown in Figure 2(b). In order to verify the values obtained from the simulation, the Hall effect probe of the Gaussmeter Hirst GM05 was used. The experimental measures are in good agreement with the FEMM prediction, the average error is below 5%.

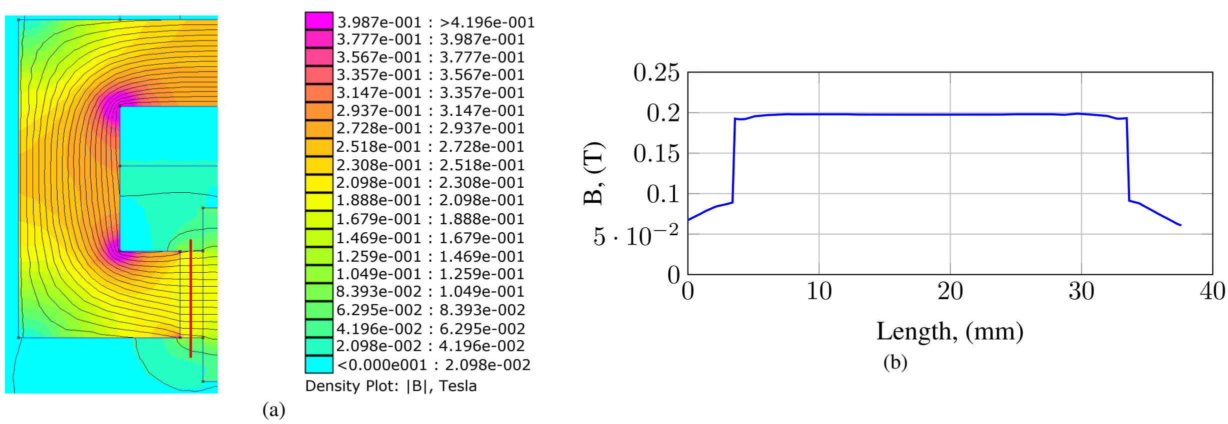


Figure 2. Half view of 2D FEMM Simulation model with magnetic field values (a). Magnetic field through the Silly Putty computed along the vertical red line in (a)(b).

2.3. Experimental plan

Many experiments involve the study of two or more variables over the response. In general, factorial designs are very efficient for this type of experiments. A factorial design investigates in each trial or replicates all the possible combinations of the factors and determines the influence on one or more responses. A variable is a characteristic under consideration while a level is a peculiar value of the variable. This method focuses on three variables (for each test) which influence the behaviour of the system, it is able to identify the interaction between these variables precisely, and to provide a reliable model to describe the system behaviour. In quasi-static tests, both for compression and shear mode, the variables involved are the compression/shear rate (S) and the applied magnetic field (B). Considering the compression rate and the shear rate, the same values used by Cross (2012a) were adopted, which are divided in three levels, 200 mm/min, 500 mm/min and 1000 mm/min. The values adopted for the magnetic field are 0 mT, 100 mT and 200 mT with a supply current of 0 A, 1 A and 2 A respectively. The formation of the particle chains, and consequently the change in mechanical and viscoelastic properties, is not as quick as for the magnetorheological fluids. This phenomena is caused by the higher viscosity of the PDMS polymer matrix (compared with the oily suspension of magnetorheological fluids) so, different times of application of the magnetic field means a different spatial distribution of the ferromagnetic particles. Hence, as a third variable involved, the time of application of the magnetic field (T) is also considered on three levels: 0 seconds, 150 seconds and 300 seconds. Furthermore, three replicates for each combination of the levels were considered. The completed experimental plan for quasi-static tests is reported in Table 1. Quasi-static tests showed that the influence of magnetic field at 0 and 0.1 T were hardly distinguishable, therefore for dynamic tests were chosen only two levels of magnetic flux density of 0 and 0.2 T (B). Still concerning the dynamic tests, since sinusoidal strains are considered, the variables involved are also amplitude (A) and cyclic frequency of the sinusoidal input (F). The values chosen for the amplitude are 2 mm and 4 mm and cyclic frequency of 4 Hz, 8 Hz and 12 Hz. The number of replicates is the same used for quasi-static tests. Table 2 reports the complete experimental plan for dynamic tests.

Table 1. Experimental plan for quasi-static tests.

Levels	I	II	III
Magnetic Field, B, mT	0	100	200
Def. speed, S, mm/min	200	500	1000
Time, T, sec.	0	150	300
Replicates	3 for each combination		
Experimental Points		27	
Grand total		81	

Table 2. Experimental plan for dynamic shear tests.

Levels	I	II	III
Magnetic Field, B, mT	0	200	
Frequency, F, Hz	4	8	12
Amplitude, A, mm	2	4	
Replicates	3 for each combination		
Experimental Points		12	
Grand total		36	

3. Results and Discussions

3.1. Quasi-static tests

Figures 3(a), 3(b), 3(c) show the experimental results in compression mode at different compression rates (dashed red lines are the tests at 1000 mm/min, dotted blue lines at 500 mm/min and solid green lines at 200 mm/min). The applied displacement is 10 mm. The figures also depict the trends of the forces at three different couples of values of magnetic field applied and time of application, precisely $B = 0$ mT and $T = 0$ s, $B = 100$ mT and $T = 150$ s, $B = 200$ mT and $T = 300$ s. For each graph, the three curves with the same colour and style are referred to the three replicates. The data obtained are consistent with those obtained from the experiment in (Cross 2012a, Nunes 2010). The forces increase by a factor of 3, at any given displacement, when the compression rate increases by a factor of 5. As it can be seen in all the compression tests at 1000 mm/min a force peak occurred at the end of the stroke. This was probably caused by the control unit of the testing machine that was not able to sudden stop the stroke causing an extra force due to the viscoelastic behaviour. From the experimental curves, it is possible to qualitatively evaluate that both the applied magnetic field and the time do not significantly influence the yield stress. A possible reason is that the force direction and the magnetic flux lines are perpendicular to each other. Consequently, the particle chains do not enhance the mechanical properties so that the values of elastic modulus are those of the polymeric matrix only. Figures 3(d), 3(e), 3(f) depict the quasi-static shear results. In this case, the applied displacement is 50 mm. Each graph shows the values of forces as a function of the shear rate. Dashed red lines represent the tests carried out at 1000 mm/min. Dotted blue lines show the tests at 500 mm/min while solid green lines are the tests at 200 mm/min. An increment in the shear strength of the material at constant strain rate is found in shear mode. It is worth noting that in Figure 3(f) one test at 1000 mm/min reached higher forces. This is due to the Silly Putty that remained stuck to the steel punch much longer compared to the other tests. Due to magnetic field, at 200 mm/min going to zero magnetic field to 200 mT with an application time of 300 sec., the maximum τ increases by 57.04%. At 500 mm/min shear rate, the increase reported to τ max is 24.57%, while at 1000 mm/min is 41.48%. The percentage increase is greater when the speed value is at the lowest level because of the nature of the viscoelastic material. Increasing the shear rate leads to a predominant effect of the viscoelasticity over the magnetic effect. It is worth noting that in shear mode, the force of attraction between the magnetic system and the punch is about 4 N, so can be neglected compared to the force exerted by the material.

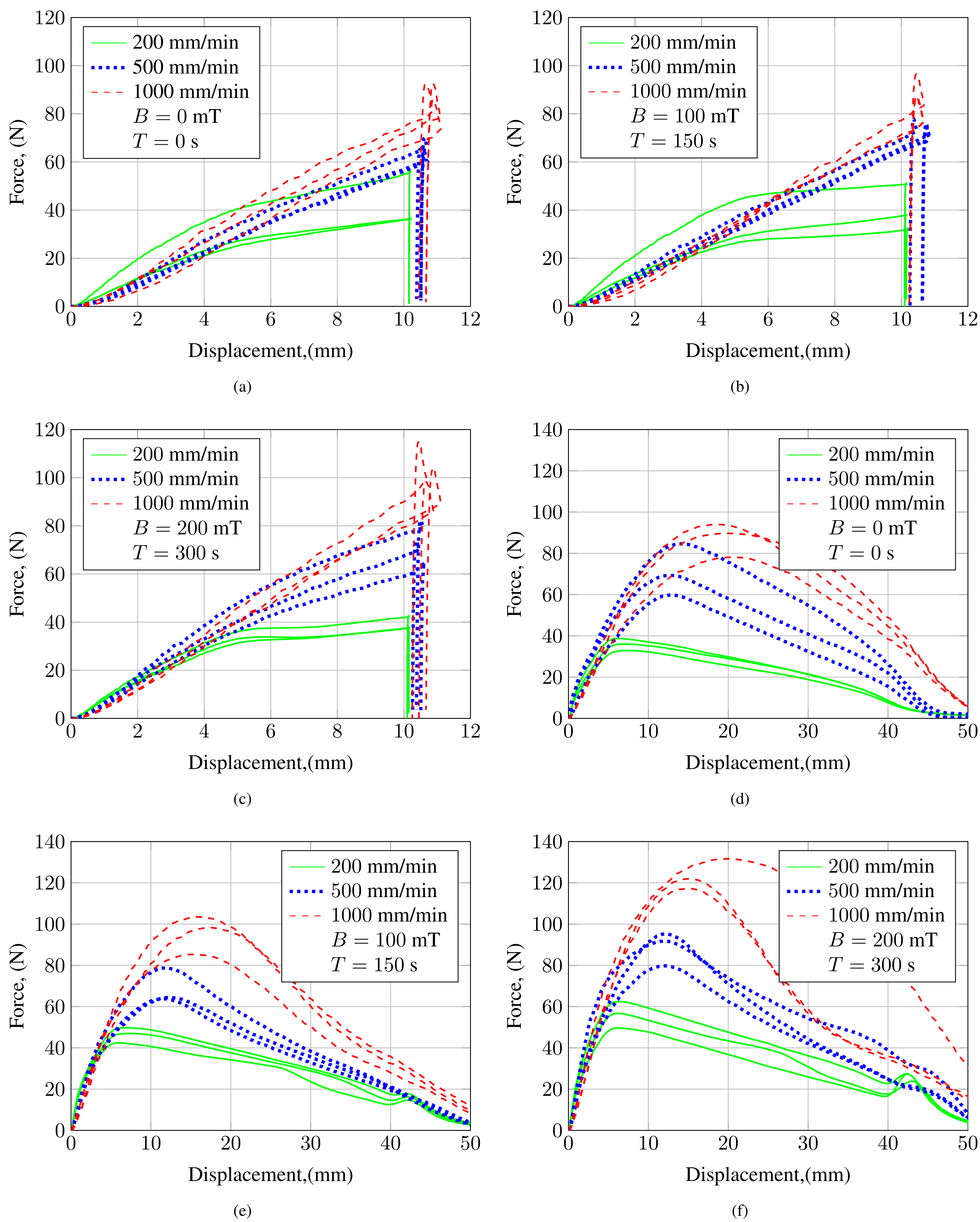


Figure 3. Quasi-static results: Compression tests, Force-Displacement at $B = 0$ mT, $T = 0$ s (a), $B = 100$ mT, $T = 150$ s (b), $B = 200$ mT, $T = 300$ s (c). Shear tests, Force-Displacement at $B = 0$ mT, $T = 0$ s (d), $B = 100$ mT, $T = 150$ s (e), $B = 200$ mT, T= 300 s (f).

3.2. Dynamic tests

The MTS control software provides the applied displacement and force response as a function of time. The force data were first averaged in a single cycle then divided by the contact areas to obtain the shear stress values. Next, in order to calculate the complex modulus and damping, the phase angle (or loss modulus) is needed. Accordingly, the strain rate curves along with shear stress curves were considered as a function of their associated phase angles (Eq. 1-3). Figure 4 shows the phase lag. The dotted red curve and the solid blue curve represent the calculated shear strain and shear stress respectively. As it can be seen, the shear stress curve is out of phase respect the shear strain which confirm the well known fact that the magnetic Silly Putty has a viscoelastic behaviour. The difference between the angles in which the maximum peaks of stress and strain occur is the phase angle δ. Once loss factors (δ) were found, the complex dynamic modulus and the equivalent damping were calculated using equation 3.

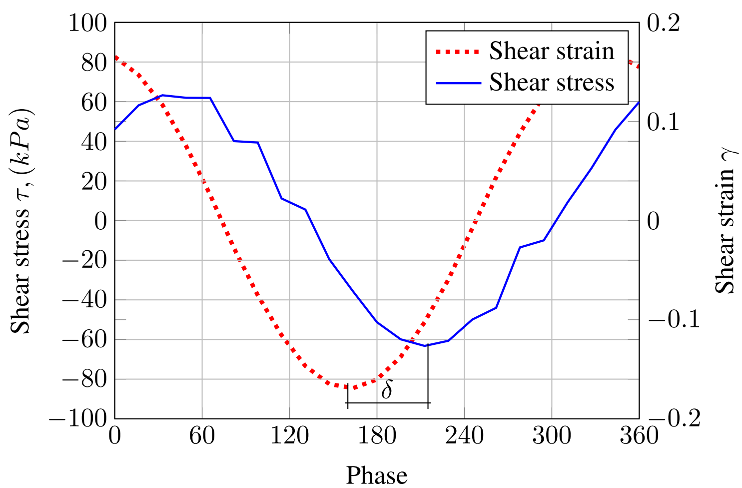


Figure 4. Phase lag between shear stress and shear strain.

Figure 5(a) shows the experimental values of forces as a function of the deformation speed. The forces at speed values below 50 mm/min are those previously retrieved from the quasi-static shear tests, in which the strain rate values were lower. The experimental forces increase as a power function of the deformation speed, which is an effect of shear-thinning fluids. Figure 5(b) depicts the trend of the loss factor calculated as $\tan\delta = \frac{G''}{G'}$. The increasing in frequency by a factor of 3 causes a decreases of the damping capacity by 57.3 %. This is due to the stiffening of the material as the frequency increase, that decreases its ability to dissipate energy. Figures 5(c) and 5(d) represent the dynamic loss modulus and dynamic storage modulus as a function of frequency. The higher the frequency, the stiffer the material. In fact, the time constant related to the shear rate is smaller than the time relaxation which is the time needed for molecules to relax after applying a shear stress. Consequently, even the loss modulus decreases by 65.7 % because the material responds more elastically when the frequency increases.

3.3. Analysis of Variance

Figure 6(a) shows the so called Half-Normal probability plot and is provided by Design Expert 8.0 (Stat-Ease 2015). This is a graphical tool that exploits the estimated effects to assess which factors are important. Since the half normal line starts at the origin, this produces a scale for detection of significant outcomes (Anderson and Whitcomb 2007) which are immediately detected at a glance. The X-axis represents the standardized effect associated with each factor considered (Table 1). The greater the standardized effect, the higher the influence of the variable on the response. The Y-axis represent the half-normal probability associated with each effect. The solid line interpolating the points represents the error of the test considering

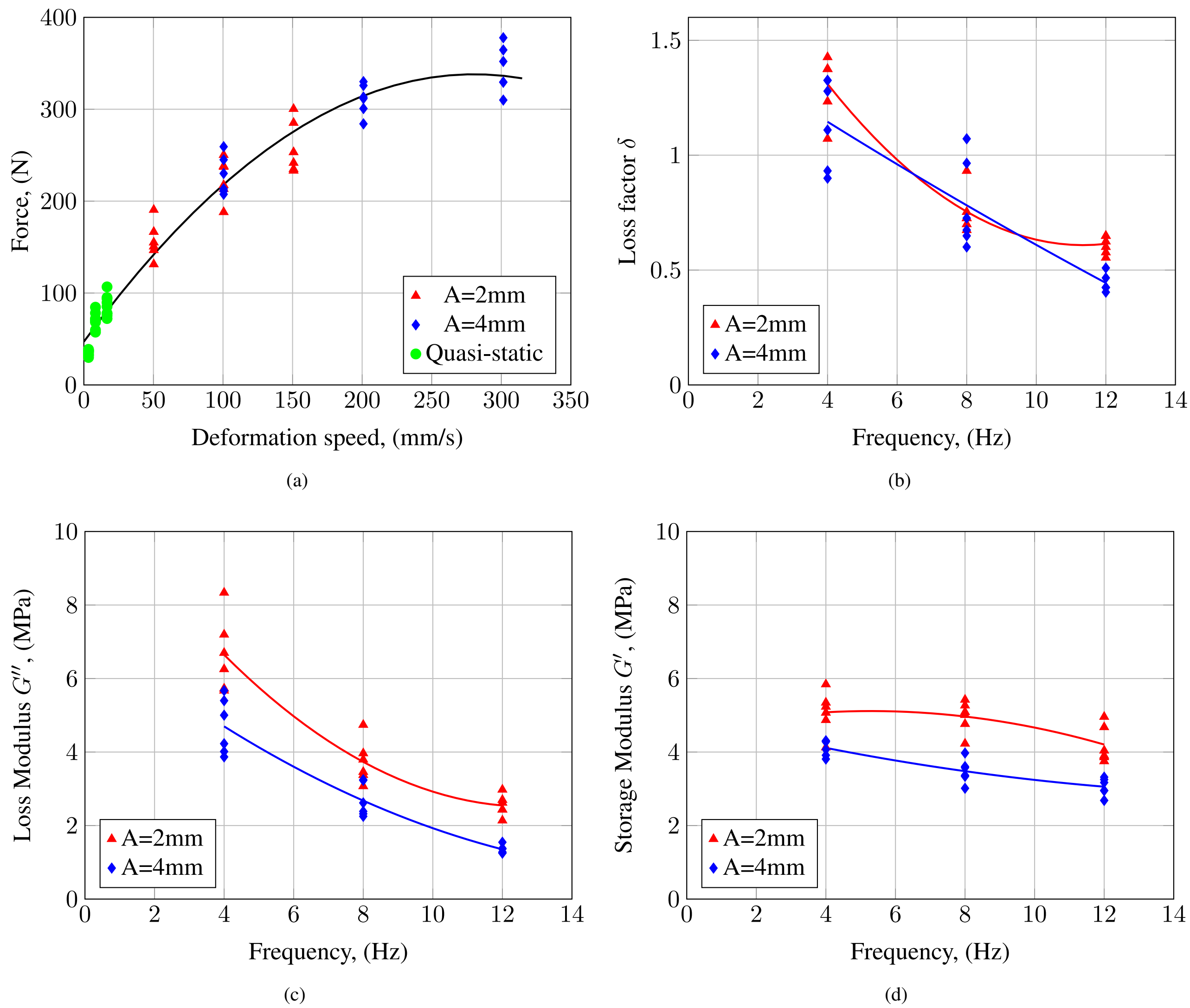


Figure 5. (a) Experimental forces vs deformation speed. Dynamic shear results: (b) Loss factor, (c) Loss modulus and (d) Storage modulus for 2 mm amplitude (red triangles) and 4 mm amplitude (blue diamonds).

all replicates (given by the non influent interactions between the variables). The triangles are an expression of the sum of errors, which is calculated by the software. Since both magnetic field (B) and time of application (T) are superimposed on the error line of the normal distribution of the experimental error (stochastic distribution), analysis of variance (ANOVA) demonstrates that these variables do not influence the process. The compression rate point (S) fall off the error line so represents the only factor that significantly affect the tests (Figure 6(a)). Contrary to quasi-static compression tests, figure 6(b) shows that the magnetic field becomes significant as well as the shear rate. In order to assess the interaction between the magnetic field and the test mode, another analysis of variance was developed. In fact, performing a statistic analysis including as a variable also the type of test (compression mode or shear mode) confirmed that a significant influence of the interaction exists between the tests mode and the strain rate. As shown by the half-normal plot, the design of experiment technique for the dynamic tests (Figure 6(c)) revealed a weak influence of the magnetic field, because of the low percentage by weight of ferromagnetic particles which is about 3.3%. Moreover, there is another important difference between magnetic Silly Putty and magnetorheological elastomers. Usually, during the polymerization of magnetorheological elastomers, a magnetic field that aligns the ferromagnetic particles along the induction lines is applied. Thus, the material is strongly anisotropic, stiffer along the chains direction and softer in the other directions. Conversely, magnetic Silly Putty is an isotropic material because the ferromagnetic particles are dispersed randomly in the polymeric matrix. Given the fluid-like nature of the PDMS it is not possible to obtain a magnetorheological anisotropic Silly Putty. This study is expected to deepen and spread the knowledge of the behaviour of magnetorheological elastomers like magnetic Silly Putty. Moreover, this will help in seeking new applications that take advantage of the properties of these materials and to develop novel field controlled MREs.

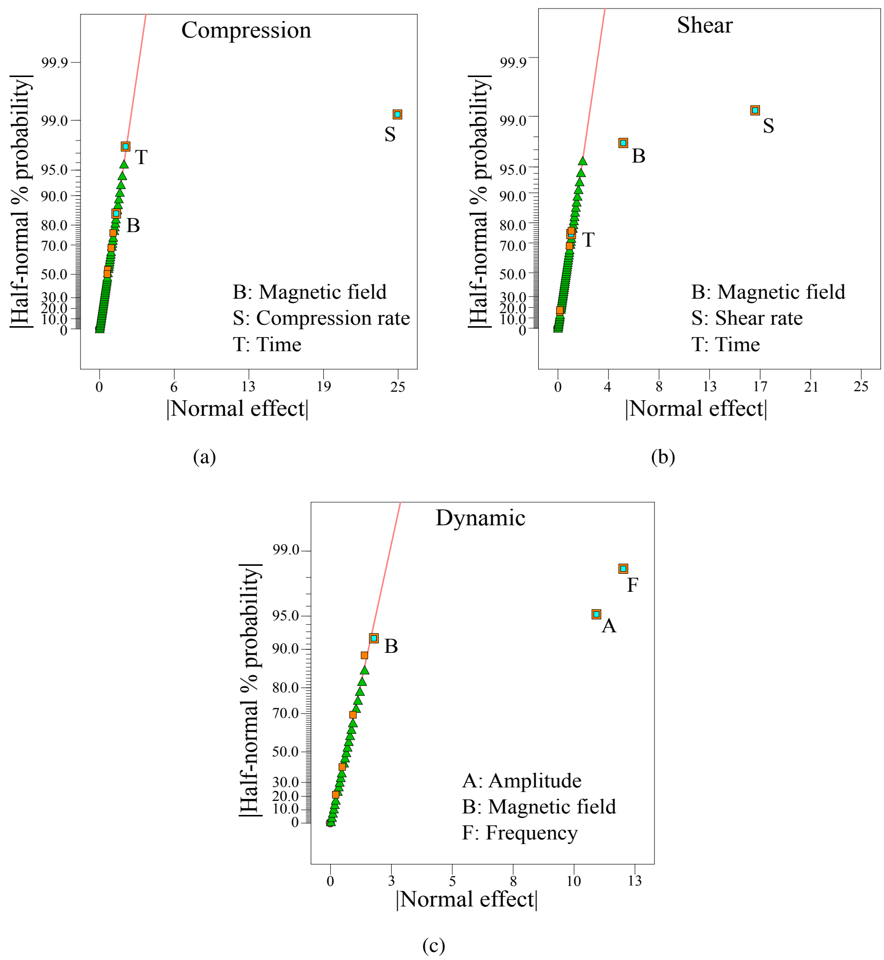


Figure 6. ANOVA for quasi-static compression (a) and shear tests (b) and dynamic tests (c).

4. Conclusions

This work studies the viscoelastic and magnetorheological behaviour of the commercial magnetic Silly Putty. Several tests are carried out, firstly quasi-static compression and shear stress are applied on a sample of Silly Putty and the force response is measured. A magnetic field is also applied in order to evaluate the interactions between the mechanical and magnetorheological inputs. The results show that the forces retrieved in compression mode are not affected by the magnetic field, due to the perpendicularity between the directions of the applied force and the magnetic flux lines. Conversely, in shear mode, especially at slow shear rate (200 mm/min), applying magnetic field increases the maximum shear stress up to 50%. Moreover, a dynamic shear tests was performed on Silly Putty under higher deformation rate, always coupled with a magnetic field. A sinusoidal inputs were used, so that the changes of the damping properties and dynamic complex modulus can be accounted for. The results highlighted the pseudoplastic behaviour of this material whose force response increases with frequency. With an increase in frequency by a factor of 3, the damping capacity and the dynamic loss modulus decrease respectively by 57.3% and 65.7%. This is due to the stiffening of the material as the frequency increase, which lowers its ability to dissipate energy. The design of experiment technique revealed a very weak influence of the magnetic field, especially at higher frequency, where the viscoelastic component is predominant.

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