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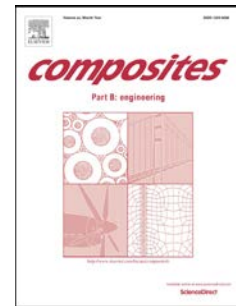
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# A review on magneto-mechanical characterizations of magnetorheological elastomers

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## Abstract

Magnetorheological elastomers (MREs) are a class of recently emerged smart materials whose moduli are largely influenced when exposed to an external magnetic field. The MREs are particulate composites, where micro-sized magnetic particles are dispersed inside a non-magnetic polymeric matrix. These elastomers are known for changing their mechanical and rheological properties in the presence of a magnetic field. This change in properties is widely known as the magnetorheological (MR) effect. The MR effect depends on a number of factors such as type of matrix materials, type, concentration and distribution of magnetic particles, use of additives, working modes, and magnetic field strength. The investigation of MREs' mechanical properties in both off-field and on-field (i.e. the absence and presence of a magnetic field) is crucial to deploy them in real engineering applications. The common magneto-mechanical characterization experiments of MREs include static and dynamic compression, tensile, and shear tests in both off-field and on-field. This review article aims to provide a comprehensive overview of the magneto-mechanical characterizations of MREs along with brief coverage of the MRE materials and their fabrication methods.

**Keywords;** Smart Materials, MR elastomers, 3D printing, magneto-mechanical characterization, MR effect,

Testing protocols; *\*Corresponding authors:* [anil.bastola@hzg.de](mailto:anil.bastola@hzg.de) (Anil K. Bastola),

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## List of Nomenclatures

MR	:	Magnetorheological
MREs	:	Magnetorheological Elastomers
MRFs	:	Magnetorheological Fluids
MRPs	:	Magnetorheological Plastomers
3D	:	Three dimensional

Vinyl Rubbers  
 Epoxidized Natural Rubber  
 Interpenetrating Polymer Network  
 High Temperature Vulcanizing  
 Room Temperature Vulcanizing  
 Magnetic Nanoparticles  
 Carbon Black  
 Carbon Nanotubes  
 Body Centered Cubic  
 Face Centered Cubic  
 Dynamic Mechanical Analyzer  
 Particle Volume Concentration

Isotropic  
 Anisotropic  
 Ampere  
 Kilo Ampere/meter  
 miliTesla  
 Tesla  
 Volume  
 Weight  
 Micrometer  
 Kilo Pascals  
 Mega Pascals  
 Newton  
 Parallel  
 Normal

Off-field : Absence of a magnetic field  
 On-Field : Presence of a magnetic field  
 CIPs : Carbonyl Iron Powders  
 PDMS : Polydimethylsiloxane  
 PU : Polyurethane  
 NR : Natural Rubber  
 VR : ENR : IPN :  
 HTV : RTV : MNPs  
 : CB : CNTs :  
 BCC :  
 FCC : DMA :  
 PVC : : Iso :  
 Aniso :  
 A :  
 kA/m mT : T :  
 Vol. : Wt. :  $\mu\text{m}$  :  
 kPa : MPa : N :

|| :  
⊥ :  
@ : at

## 1. Introduction

Magneto-rheological (MR) materials belong to the category of smart materials, and their rheological and mechanical properties can be changed with respect to an externally applied magnetic field. An MR material can be a fluid, a gel, or a solid-like elastomer [1-3]. Depending upon the type of matrix material and **magnetic particles**, MR fluid [3], ferrofluid [4], **ferrogels** [5, 6], MR foam [3], and MR elastomer [3] can be distinguished. The magnetorheological fluids (MRFs) and magnetorheological elastomers (MREs) are the two main branches of the MR materials. In MR fluids, magnetic particles are suspended in a carrier fluid such as silicone oil, whereas magnetic particles are locked in a place within a polymeric matrix in the case of MREs. They undergo rheological and mechanical changes when an external magnetic field is applied. MR fluids are known for a large stress enhancement, whereas, MR elastomers are typically known for changing their moduli under the magnetic field. The concept of MR materials was introduced by Rabinow [1] in 1948, wherein he demonstrated the MR effects in the case of MRFs only. On the contrary, MREs do not share a long history as MRFs. The introductory research on MREs was conducted after a few decades in 1983 by Rigbi and Jilken [7].

Apart from the MREs and MRFs, MR plastomers (MRPs) are another emerging category of MR materials that exhibit promising properties to develop smart materials and structures and demonstrate higher MR effects than MREs and lower sedimentation than MRFs [8-12]. The first MRP was reported in 2011 [9]. MRPs are known for showing plastic properties at room temperature and higher MR effects than MREs due to highly mobile magnetic particles. However, MRPs possess very low initial moduli and are not suitable materials for applications such as vibration isolators and absorbers. Yet, MRPs are potential candidate materials in the field of flexible, stretchable, and conductive sensors and actuators, for example, on-off switches [12, 13].

Magneto-rheological elastomers (MREs) are multi-functional materials with manifold characteristics, in which the mechanical properties such as stiffness, natural frequency, and damping capacity can dynamically be changed with respect to an external magnetic field. Every now and then the magnetic field responsive elastomeric materials are referred as magneto-active polymers, magneto-active elastomers, magnetosensitive

elastomers, and magneto-rheological elastomers [14-24]. However, in a recent survey based on the publications over the last two decades, Pelteret and Steinmann [14] found that the magnetorheological elastomers (MREs) is the most widely used name to refer to the magnetic-responsive polymers. So, this review also uses the term 'MREs' throughout the article. As demonstrated in Figure 1, over the last twenty years starting from 2002, the interest in MREs' has been increased significantly. The early investigations of MREs were on obtaining small strain properties such as the change of storage modulus and the natural frequency, which have been extensively reported in [7, 15, 16, 19, 25]. On the other hand, research focused on MREs as smart materials by studying magnetostrictive behavior can be found in the literature [26]. Shape changing capacity determines the magnetostrictive behavior, Diguel et al [27] demonstrated a maximum stretch of 10% for an MRE. However, such shape-changing behavior is not as popular as property-changing behavior such as a change in modulus/stiffness. Thus, the property-changing behavior of MREs has been extensively investigated in recent years [15, 16, 28]. The popular property-changing behavior of MREs includes the changes in storage/loss modulus, stiffness, natural frequency, damping capability as well as complex viscosity. Therefore, MRE is a natural candidate material in which the stiffness or modulus tunability is required, for example, in vibration absorbers/isolators and sandwich beams in various engineering applications.

The investigation of the mechanical properties of MREs both in the presence and in the absence of a magnetic field is essential to use them in real engineering applications. The common magneto-mechanical characterizations of MREs include static and dynamic uniaxial and biaxial compression, tensile, and shear tests in both off-field and on-field conditions. On the other hand, studies devoted to the fatigue properties of MREs can also be found in the literature [29-31]. In addition, the deformation property of MREs exposed to a magnetic field has also been reported [32]. An optical method, known as digital holographic interferometry, can be applied to analyze the morphology transformation of the MREs. It was found that an MRE sample goes under contraction and stretch deformation when it is exposed to the magnetic field [32].

One of the promising ways to enhance the properties of MREs in both off-field (no magnetic field applied) and on-field (magnetic field applied) is the addition of magnetic and non-magnetic additives. Several additives materials such as plasticizers, carbon-based additives, and magnetic nanoparticles have been considered. Additives help prevent the accumulation of magnetic particles and aid the compatibility of the matrix material with the magnetic particles; thus, additives enhance both off-field and on-field properties. Common additives such as mineral oils, Phthalate esters, and silicone-based natural esters were considered as early as 2003 [33] and are continuously increasing [34-38]. On the other hand, the addition of particulate additives such as carbon black (CB), carbon nanotubes (CNTs), and magnetic nanoparticles (MNPs) was considered starting from 2008. In 2008, Chen et al [39] used the CB in MREs for the first time, while the use of another category of carbon-

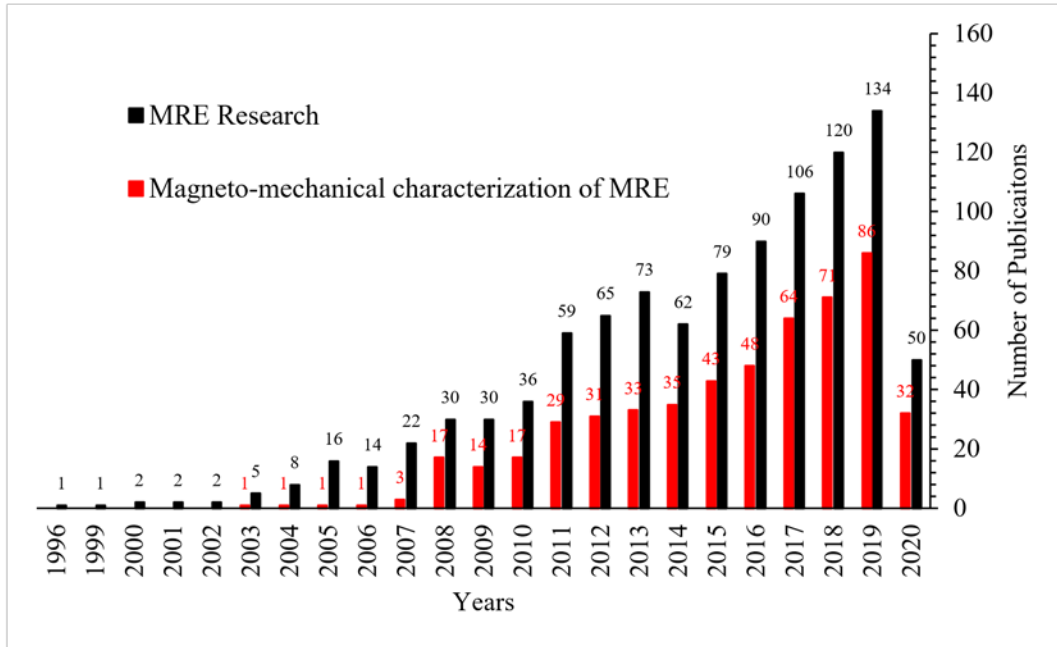
based additives, CNT, was reported in 2011 by Li et al [40]. Since then, the consideration of carbon-based additives is continuously increasing [37, 41]. Magnetic additives were used for the first time in 2013 by Landa et al [42]. They reported the use of Nickel-based nanoparticles and nanochains. Other types of magnetic additives based on iron oxides have also been considered and are reported in recent studies [28, 43, 44].

A number of research works devoted to the mathematical and phenomenological modeling of MREs' are extensively available in the literature. Jolly et al [2] in 1996, modeled the MRE behavior for the first time, whereby MREs were also treated similar to MR fluids. The approach is widely known as the particle interaction-based model, which is further investigated by Ivaneyko et al [45-47]. Multiscale-based approaches are another class of successful modeling schemes to obtain microscale information in the MRE composites [48-55]. Furthermore, continuum-based models are popular tools to understand magneto-elastic responses [52, 56-66], magneto-viscoelastic phenomena [67-69], and temperature-dependent responses of MREs [70-76]. These mathematical models are summarized in a recent article by Cantera et al [77]. These modeling approaches offer an in-depth understanding of the MREs' behavior subjected to various loading conditions as well as help to predict the behavior of MREs for long-term use.

The most common applications of the MREs include vibration absorbers, vibration isolators, dampers, and shock absorbers for vehicles [15, 16, 78-86]. Another type of application of MREs composites is sandwich structures, where an MRE acts as a smart core in the structures such as sandwich beams [87-92]. More recently, MREs receive significant attention in other applications such as sensors, for example, MRE-based microelectromechanical systems (MEMS) magnetometer, magneto-resistor, flexible tactile sensor/display [93-96], flexible microfluid transport system [97], and peristaltic pump [98] and even as a shape memory polymer [99]. For the advancement on MRE materials and systems, refer to a recent topical review [100]. In all the above-mentioned applications, MREs subject to the different loading conditions such as shear load, compressive load, tensile load or even biaxial loadings in both off and on-field conditions (i.e., absence and presence of a magnetic field). Similarly, the mechanical properties of MREs over time (e.g., fatigue properties) are important features that need to be investigated for the sustainability of the MRE-based devices for long-term applications.

MREs offer specific responses categorized as the alteration of elastic and viscous properties in a time domain, for example, the change in storage/loss modulus, stress, strain, and complex viscosity as well as the alteration in electrical resistance and capacitance. Hence, it is noteworthy to investigate the mechanical properties of MREs in detail. Having said that, the existing reviews [15, 16, 37, 77, 100-104] reported on the advancements of the MREs only focus on the modifications of MRE materials, their potential applications, and constitutive modeling. Nonetheless, as can be seen in Figure 1, every year more than 50% of the MRE based research

works focus purely on magneto-mechanical characterizations using various experimental techniques. The mechanical properties of MREs play a crucial role in successful applications such as vibration absorbers or isolators. Therefore, it is strongly believed that there is a growing demand to report only mechanical properties and characterization methods for the MREs. In this review, magneto-mechanical characterizations and MR effects of MREs are comprehensively presented and discussed.



**Figure 1.** Publications on the magneto-mechanical characterizations of MREs compared with various aspects of MRE-based research since 1996, Engineering Village © as per 15 May 2020, (Compendex database only). All Scopus archived documents (Journal articles, Conference proceedings, and books) are considered. The keyword “*magneto-mechanical*” is used to filter the research works that are focused on the magneto-mechanical characterizations of MREs.

**Table 1.** A comprehensive chart summarizes all major experimental investigations on the mechanical properties of MREs. MRE constituents, types, and testing methods are summarized.

Matrix material	Magnetic Particle			MRE type	Test type	Reference
	Type	Size	Content			
Silicone oil	CIP	3-4 $\mu\text{m}$	10-30 vol.%	anisotropic	static shear test	Jolly et al 1996 [25]
Natural rubber	CIP	0.5-5 $\mu\text{m}$	27 & 40 vol.%	isotropic & anisotropic	vibration absorber	Ginder et al, 1996 [105]
Silicone rubber	CIP	2 $\mu\text{m}$	5-25 vol.%	anisotropic	static tensile test	Bellan et al, 2002 [106]
Natural, nitrile and silicone rubber	CIP	up to 200 $\mu\text{m}$	-	isotropic	dynamic shear	Lokander et al, 2002 [33]
Silicone rubber	CIP	3 $\mu\text{m}$	27 vol.%	isotropic & anisotropic	free vibration	Zhou, 2003 [107] and 2004 [26]
Silicone rubber and gel	CIP	3.8 $\mu\text{m}$	27 vol.%	anisotropic	compression & tensile	Farshad et al 2005 [108]
Silicone rubber	CIP	4 $\mu\text{m}$	30 vol.%	isotropic & anisotropic	static & dynamic compression	Kallio, 2005[109] and 2007 [110]
Silicone rubber	CIP	6-9 $\mu\text{m}$	35 vol.%	-	compression & tensile	Albanese, 2003 [111], Albanese Lerner, 2005 [112] and 2007 [113]
Silicone rubber	CIP	10 $\mu\text{m}$ + 40 $\mu\text{m}$ ,	30 vol.%	isotropic & anisotropic	shear	Lockette 2008 [114], Jung, 2009 [115]
Silicone/vinyl rubber	CIP, Fe <sub>3</sub> O <sub>4</sub> , Fe-Nd-B	-	-	isotropic & anisotropic	compression & tensile	Abramchuk et al, 2006 [116], Stepanov et al 2007 [117],
PU, NR, silicone rubber,	CIP	$\mu\text{m}$	20-70 wt.%	isotropic & anisotropic	shear	Chen, 2007 [118] & 2008 [119]
PU/silicone	CIP	-	-	isotropic & anisotropic	compression & tensile	Xu, 2010 [120], Liao, 2011 [121] & 2012 [122]
Thermoplastic elastomer	irregular CIP	60 $\mu\text{m}$	-	isotropic & anisotropic	simple shear	Zajac, 2010 [123], Krolewicz, 2012 [124]
Silicone rubber	CIP	$\mu\text{m}$	-	-	shear-compression mixed mode	Du, 2011 [125] and Li, 2012 [126]
Silicone rubber/ PU	CIP	4-5 $\mu\text{m}$ , 7-9.5 $\mu\text{m}$	-	-	compression	Gudmundsson, 2011 [127]
Silicone rubber	CIP + hard magnetic filler	11 $\mu\text{m}$ + 40 $\mu\text{m}$ ,	-	-	compression & shear	Lockette, 2011 [128], Koo, 2012 [129]
Silicone	CIP	2-8 $\mu\text{m}$	-	-	compression & shear	Gordaninejad, 2012 [130]
Silicone rubber	CIP	-	-	isotropic	equi-biaxial & fatigue	Zhou, 2013 [29]
PDMS	CIP	6 $\mu\text{m}$	33, 50 & 70 wt.%	MR brush	compression	Huang et al, 2014 [131]

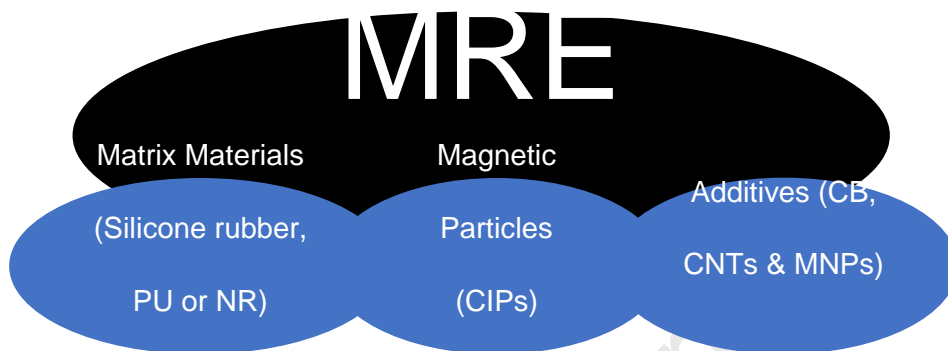


RTV silicone rubber	CIP	6-7 $\mu\text{m}$	20 vol.%	isotropic & anisotropic	equi-biaxial fatigue	Zhou et al, 2014 [30]
NR, stearic acid, paraffin oil	iron sand	0-106 $\mu\text{m}$	0-100 wt.%	isotropic & anisotropic	DMA test	Khimi et al, 2015 [132]
Silicone rubber	CIP	6-7 $\mu\text{m}$	15-35 vol.%	isotropic	equi-biaxial fatigue	Zhou et al, 2015 [133]
NR, stearic acid, ZnO	CIP	5 $\mu\text{m}$	15-25 vol.%	isotropic	shear test	Hegde et al, 2015 [134]
Polychloroprene rubber, ENR	CIP and CB	3.8-5.3 $\mu\text{m}$ CIP	0-40 vol.%	isotropic & anisotropic	tensile & shear	Wang et al, 2015 [135]
PU	Nanoflakes, CIP	6.5 $\mu\text{m}$	60-70 wt.%	anisotropic	shear test	Yu et al, 2015 [136]
PU, IPN	CIP	5-8 $\mu\text{m}$	70 wt.%	isotropic	creep & creep-recovery	Qi et al, 2015 [136]
PU/Butanediol	CIP	5-8 $\mu\text{m}$	50-70 wt.%	anisotropic	shear mode	Ju et al, 2016 [137]
Silicone rubber	CIP	7 $\mu\text{m}$	0-30 vol.%	isotropic	shear mode	Gao et al, 2016 [138]
Thermoplastic	CIP, coated CIP	10 $\mu\text{m}$	38.5 vol.%	isotropic	tensile	Małeckı et al, 2016 [139]
Silicone rubber/ silicone oil	CIP	5-9 $\mu\text{m}$	10-40 wt.%	isotropic	uniaxial tension & rheological	Perales-Martinez et al, 2017 [140]
Silicone rubber	CIP	6-7 $\mu\text{m}$	15-35 vol.%	isotropic	bubble inflation, fatigue	Zhou et al, 2017 [141]
NR	CIP, CB	-	18.3 vol.%	-	bubble inflation, cyclic	Gorman et al, 2017 [142]
Silicone rubber	CIP	3-5 $\mu\text{m}$	70 wt.%	-	compression & tensile	Norouzi et al, 2017 [143]
Silicone rubber	CIP	-	20 vol.%	anisotropic	compression	Harne et al, 2018 [144]
Silicone rubber	CIP	-	20-70 wt.%	-	double lap shear test	Johnson et al, 2018 [145]
Silicone rubber	CIP	3.5 $\mu\text{m}$	-	isotropic & anisotropic	coupled magneto-mechanical	Bodelot et al, 2018 [146]
Bromobutyl rubber	CIP	-	60-70 wt.%	anisotropic	shear	Xu et al, 2018 [147]
NR	CIP	-	0-30 vol.%	isotropic	compression	Agirre-Olabide et al, 2018 [148]



## 2. Materials for Magnetorheological Elastomers (MREs)

The main constituents of an MRE composite are given in Figure 2. An MRE material comprises a nonmagnetic polymeric matrix loaded with magnetizable particles of various fractions and additives. Table 1 summarizes some key MRE composites that have been synthesized over the last two decades, in which research works only focusing on the magneto-mechanical characterizations of MREs have been considered.



**Figure 2.** Main constituents of MREs.

The choice of matrix material highly influences the mechanical properties of MREs such as initial modulus, field-dependent modulus, and MR effect. Several matrix materials have been employed to manufacture MREs, including but not limited to silicone rubbers, vinyl rubbers (VR), polyurethanes (PU), thermosets/thermoplastics elastomers, and natural/synthetic rubbers [3, 15, 16, 37, 107, 110, 149, 150]. However, silicone rubbers are the most widely used materials among other polymers. They have some exotic properties that make them attractive in MRE applications. For instance, silicones are readily available as resin (liquid state), which facilitates a homogenous distribution and easy suspension of the magnetic particles during the synthesis process. Moreover, silicone resins have low viscosities, which help the magnetizable particles to easily move to form chains along the magnetic flux direction for the development of anisotropic MREs. In addition, silicone-based matrix materials are vulcanizable faster at an elevated temperature as well as at room temperature and they are also non-flammable, non-toxic, less dissipative, less temperature-sensitive, and highly deformable [151-153].

Silicone rubbers used to develop MREs are either one-part silicones or two part-silicones. Usually, silicone rubbers used in MREs are heat-curable either at room temperature or at an elevated temperature. Sometimes, two-part silicone is an addition-cured platinum-catalyzed system. A few common types of silicone rubber are elevated temperature vulcanizing or also known as high temperature vulcanizing (HTV) [32] silicone rubbers:

Sylgard 184 ©Dow, MVQ 110-2 from Dong Jue Fine Chemicals China, Gniikhteos Russia, Silgel®612 A/B from Wacker and room temperature vulcanizing (RTV) silicone rubbers [78, 107]: Ecoflex 00–20 from Smooth-On Inc., USA, SS-B6 from silicone solutions, USA, Dow HS II RTV silicone from Gluespec, USA and Elastosil M4644 from Wacker silicones. Similarly, 3D printable silicones include UV curing SS-155 and heat-curable but addition-cured platinum-catalyzed system SS-3006T from Silicone Solution in Ohio, USA [154].

Magnetic particles are field-sensitive components of MREs and are mainly responsible for the magnetic field dependent properties (i.e., MR or magnetostrictive effect). Carbonyl iron powders (CIPs) are the most widely used magnetizable particles. CIPs are considered to be one of the best choices in synthesizing MREs because of their high magnetic saturation ( $>700$  mT), low magnetic remnant, softness, and high magnetic permeability [16, 33]. Furthermore, magnetic particles such as Cobalt, Nickel, and Nd-Fe-B and even  $\text{Fe}_3\text{O}_4$ ,  $\text{CoFe}_2\text{O}_4$ , and iron sands have also been considered to develop MREs [42, 155-159]. In MREs, the spherical CIPs with an average size below  $10\ \mu\text{m}$  have been widely considered. The primary reason for the use of smaller particles is that the smaller particle size offers a higher effective area of interfacial friction between magnetic particles and the matrix materials. Nonetheless, larger particles reaching up to  $100\ \mu\text{m}$  or even higher up to  $200\ \mu\text{m}$  have also been considered [160]. Even the use of bimodal iron and magnetite particles have been demonstrated [17].

Additives are the additional components of MREs. The key contribution of additives in MREs is to enhance the magnetic field-dependent properties, primarily known as the MR effect. Moreover, consideration of additives in MREs also opens a new door for MRE-based applications by offering sensing and actuation capabilities such as a change in resistance and capacitance. In MREs, additives are categorized as nonmagnetic and magneto-active additives. Silicone oils, plasticizers, and carbon-based materials are popular non-magnetic additives while magnetic nanoparticles (MNPs), nanorods such as  $\text{Fe}_2\text{O}_3$  and Chromium-based particles fall under the category of the magnetic additives. The most commonly used additives, also known as plasticizers materials, are silicone oil, mineral oil, phthalate Esters, and silicone/natural-based Esters. Such plasticizers are mixed with matrix materials that help to improve flexibility, flowability, and workability, consequently, to assist the MRE materials' processing.

Carbon-based materials are other emerging non-magnetic additives. The carbon black (CB) is the first type of carbon-based additives. CB improves the electrical conductivity of MREs. The first use of CB was reported in 2008 [39]. The second member of carbon-based additives is carbon nanotubes (CNTs) that gain popularity thanks to their high aspect ratio, high surface to mass ratio, and lightweight. The first use of CNTs was reported by Li et al [40] in 2011. In the same way, the additions of graphite microparticles and graphene nanoparticles have also been used to provide the sensing capability by decreasing the electrical resistance of MREs [161].

**Magneto-active** additives include the nanoparticles based on iron and nickel or cobalt oxides. These nanosized magnetic particles can fill the voids among the micro-sized CIPs and thus offer higher MR effects. In 2009 [162], the use of Fe and Co-based nanowires in MREs was reported for the first time. They demonstrated that the nanowire-based MREs displayed higher modulus compared to MREs with only pure CIPs. Likewise, the use of nano-flakes Fe particles has also been considered to develop MREs: the MR effect (based on the loss factor) was found to be 1.56 times higher when Fe nano-flakes were added up to 6 wt.% [136]. It has also been reported that the MR effect of MREs can also be enhanced by adding rodshaped  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles [44]. Moreover, surface modifications of CIPs are also one of the emerging areas to improve the properties of MREs [163]. MREs made of the surface-modified CIPs exhibit higher MR effects than those with pristine CIPs [163].

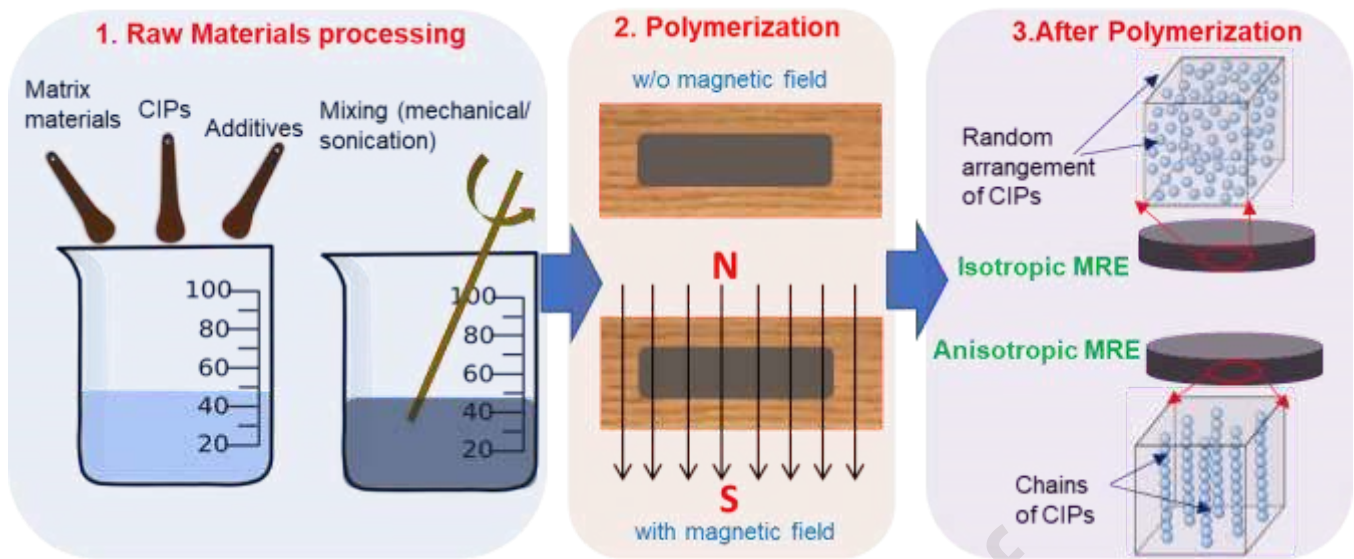
Having said that the CNTs belong to non-magnetic additives, there are a number of works, which have proven that CNTs help in improving the MR effect in addition to the enhancement of zero-field properties of MREs. Therefore, it can also be argued that the CNTs or even other carbon-based materials can be considered as magneto-active additives. For example, Li and Sun [164] reported that the MR nanocomposites exhibited not only higher zero-field stiffness and damping than the conventional MR elastomers, but also a greater magnetic-field-induced increase, or absolute MR effect. They used multi-wall CNTs (MWCNTs) up to 3.5 wt.%, MREs with MWCNTs exhibited a 30% increase in the stiffness compared to MREs without MWCNTs. A similar type of results has also been reported in Aziz et al [165, 166], in which MREs containing the functionalized MWCNTs (COOH-MWCNTs) showed a higher MR effect (13.7% increase) compared to MREs comprising of pristine MWCNTs. In some applications, a high initial stiffness/modulus and damping are demanded but upholding a high relative MR effect is a challenging task and supplementary materials (e.g., a higher CIP content) or efforts (e.g. a higher magnetic field) are required to improve the MR effect. Interestingly, the addition of MWCNTs offers a higher MR effect at a fixed concentration of CIPs. Therefore, it is worthy to consider CNTs as magneto-active additives for MREs and a further investigation is required to understand the physics behind the phenomenon.

### 3. Syntheses of MREs

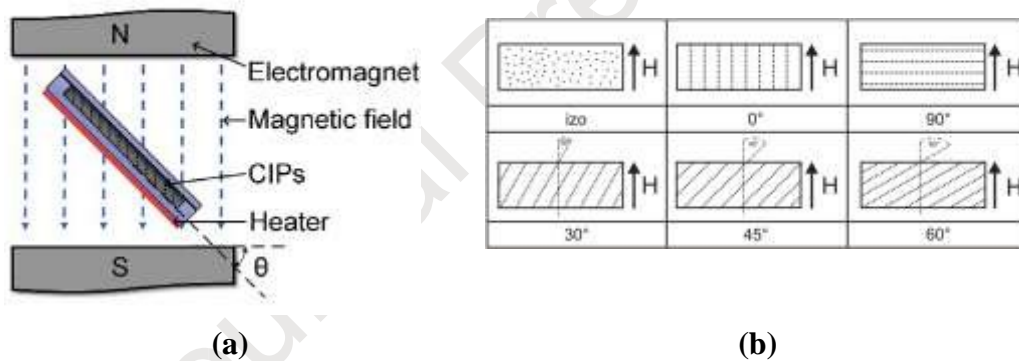
#### 3.1 Conventional Methods

The manufacture of magnetorheological elastomers is similar to a conventional polymer-based material processing technique. Figure 3 depicts a typical conventional manufacturing technique to develop MREs. The commonly adopted matrix material is the silicone rubber, which is initially a fluid. As a result, an MRE can be manufactured simply by mixing a silicone rubber and magnetic particles sometimes by adding supplementary

materials such as carbon black and other additives. The processing of raw materials is usually performed at room temperature and thereafter the mixture is poured into a mold and allowed to cure ranging from several minutes to several hours depending on the polymer curing time.



**Figure 3.** MRE production: a conventional method. It includes three major steps: 1. Raw materials mixing: e.g. mix silicone rubber, CIPs, and additives mostly using a mechanical method and followed by sonication, 2. Polymerization: allow polymerization of the suspension within a mold either in the absence or presence of a magnetic field. 3. After polymerization: two types of MREs are produced based on polymerization techniques.



**Figure 4.** Production of anisotropic MREs with different directions of particle alignments. (a) Example to align magnetic particles in the desired angle in MREs [167]. (b) Example of application of magnetic flux for different forms of anisotropic MREs [168].

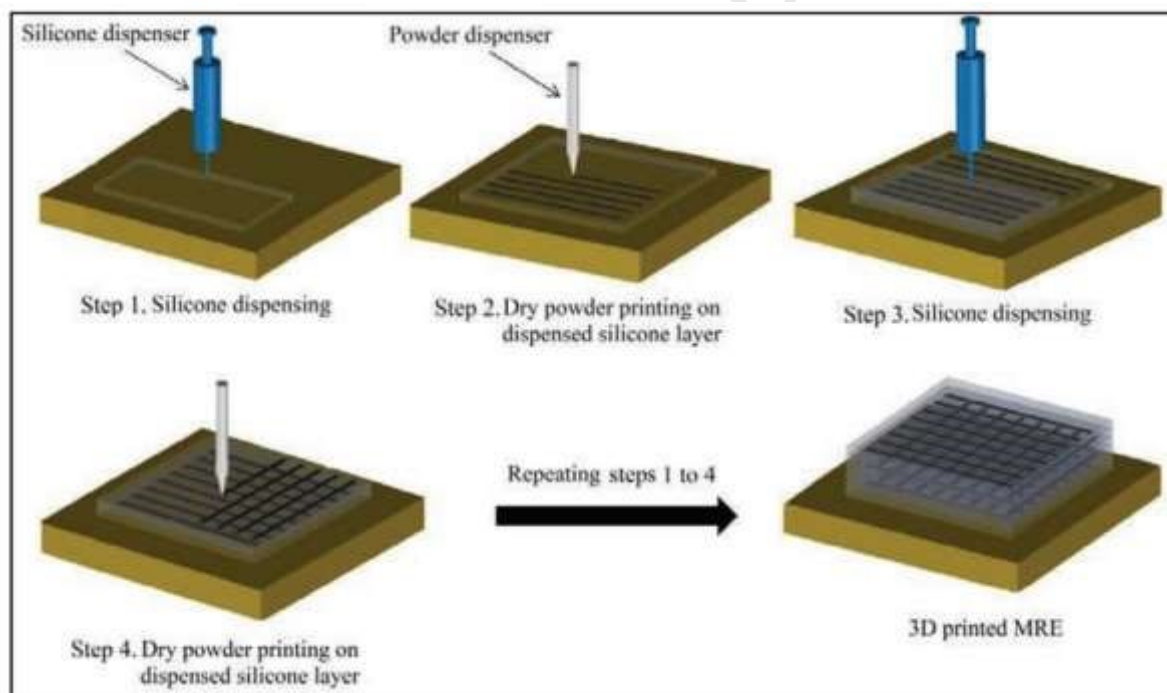
In the conventional methods, the mixture of magnetic particles and the matrix material is crosslinked or cured or vulcanized in a mold. Therefore, magnetizable particles stay locked within the polymeric network after the mixture is completely vulcanized. The mold defines the dimensions such as the thickness and width of an MRE to be developed. On the other hand, of course, desired samples can be obtained by cutting the molded bulk MRE into the right dimension. Typically, the mixing of the raw materials is performed at room temperature

and the crosslinking is achieved at a higher temperature in order to accelerate the curing process. Furthermore, if the matrix material is an RTV (room temperature vulcanization) polymer, the curing can also be performed at room temperature. Sometimes, a magnetic field is applied to configure the CIPs into the direction of the magnetic flux lines. In that case, magnetic particles are aligned and locked in a place, such MR composites are known as the anisotropic MREs. Otherwise, if the mixture is cured in the absence of a magnetic field, isotropic MREs are achieved. Magnetic particles are anticipated to be randomly distributed in isotropic MREs.

In MREs, the magnetic particles can also be arranged in different directions, for example,  $0^\circ$ ,  $30^\circ$ ,  $45^\circ$ ,  $60^\circ$  and  $90^\circ$  [167, 169] as depicted in Figure 4. In 2012, Boczkowska and co-workers [168] investigated the effects of aligning particles in different directions for the first time. They found that the MREs exhibited a higher MR effect when particles were aligned in  $60^\circ$ . In order to align magnetic particles in different directions, a non-magnetic mold containing magnetic suspension is placed in between an electromagnet or set of permanent magnets at a  $45^\circ$  angle or in the desired angle as given in Figure 4. In such a case, the

magnetic flux lines make the CIPs form chains along the magnetic field in the well-defined path. Thereafter, MR suspension is left to be vulcanized to trap the CIP chains along the direction of magnetic flux lines.

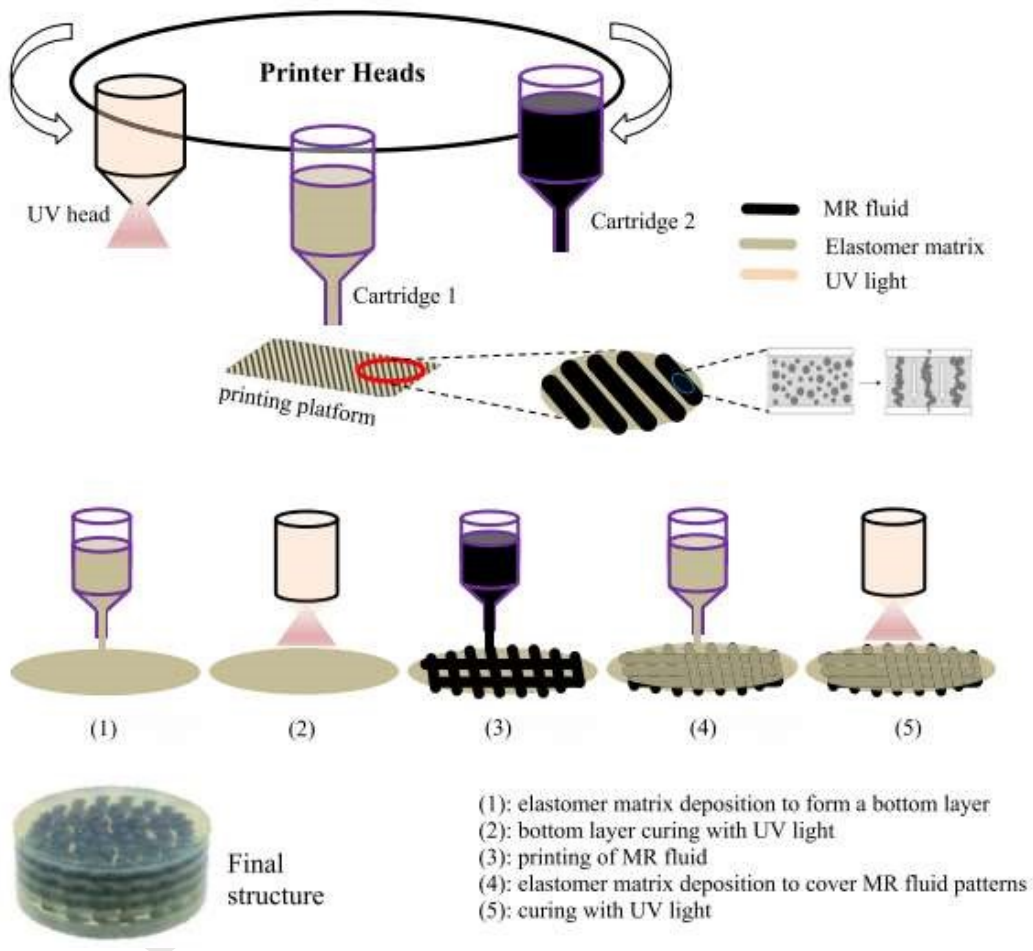
### 3.2 3D Printing Methods



**Figure 5.** A schematic diagram of the integrated dry powder printing to develop MREs, performed by Krueger et al [170], in 2014.

Even though anisotropic MREs offer higher MR effects compared to their isotropic counterparts, it is highly challenging to uniquely distribute the magnetic particles in the desired fashion without applying a magnetic

field [171]. One of the possible ways to configure magnetic particles inside an elastomeric matrix is to use the recently emerged 3D printing or additive manufacturing methods for the fabrication of MREs. With the help of some advanced 3D printing processes, the magnetic particles can be configured accurately and precisely within the matrix materials in the desired fashion without applying a magnetic field. In 2014, Krueger et al [170] attempted for the first time to make 3D printed MREs using a dry powder dispenser, see Figure 5. However, the printing process was unsuccessful. The dry powder printing had a very poor flowability, resulting in several problems such as discontinuous printing lines, weak bonding between a silicone layer and the dry powder, and scattering of the printed patterns. Afterwards, no further studies dealing with the development of MREs via 3D printing can be found in the literature by the same group or by others using the dry powder printing technique or any other techniques.

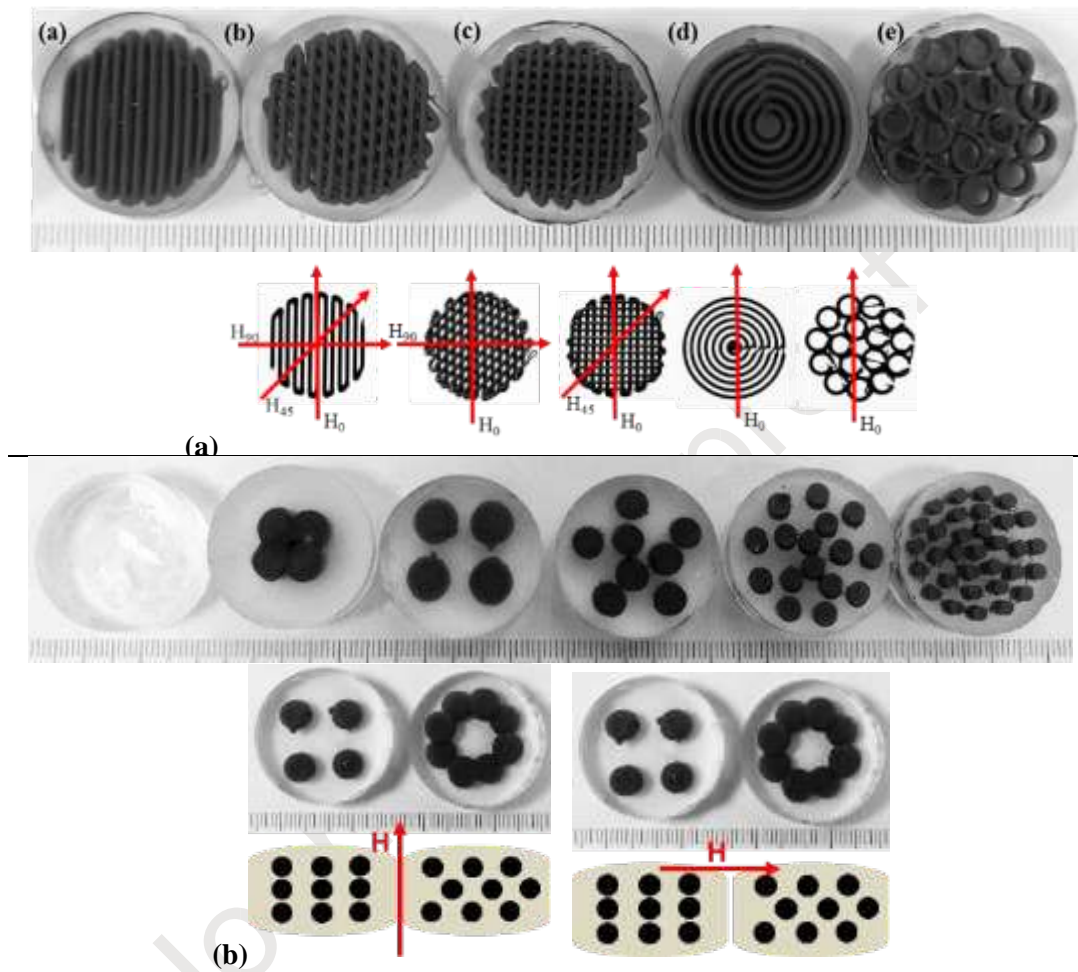


**Figure 6.** Schematic illustrations for the printing of hybrid MREs via extrusion-based 3D printing performed by Bastola et al [172].

In 2017, Bastola et al [172, 173] successfully presented a 3D printing method to develop hybrid MREs, where MR fluid filaments were 3D printed within a soft elastomeric matrix. To achieve such 3D printing, a multi-material 3D printing process was employed, whereby a controlled volume of MR fluid was 3D printed and encapsulated within a polymeric matrix in a layer-by-layer manner. They demonstrated two different types of



3D printed MREs. The selection of printing inks defines the final structures of the 3D printed MREs. 3D printing with MR ink out of room temperature vulcanizing polymer produces the solid MR structure inside the matrix material while the printing with non-vulcanizing MR ink produces the structures where the ink remains fluid and encapsulated inside the matrix material. Figure 6 provides the schematic diagram of the 3D printing technique to fabricate hybrid MREs performed by Bastola et al [172, 173]. They have successfully demonstrated the change in elastic and damping properties as well as anisotropic behavior of 3D printed MREs by applying a magnetic field. In addition to 3D printing, Bastola et al [172, 174] have also used a conventional method to develop core-shell hybrid MREs. Core-shell hybrid MREs were developed by encapsulating bulk of MR suspension within a core made up of a soft elastomer.

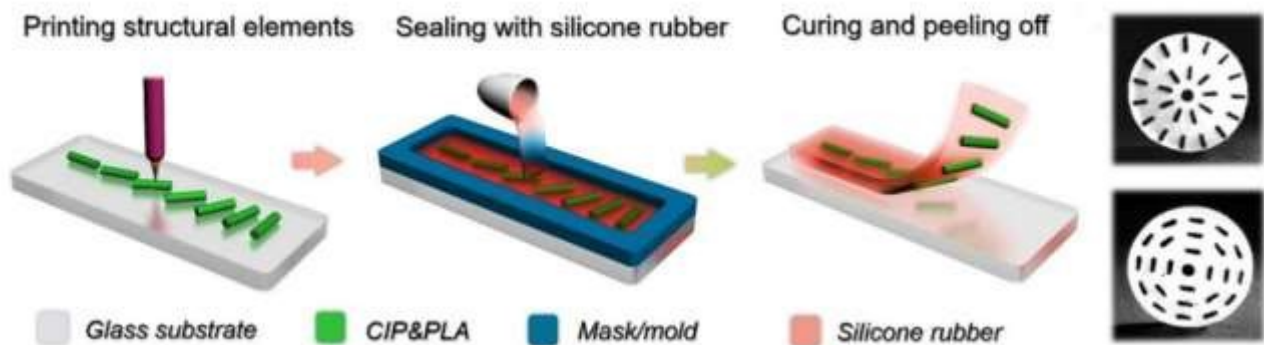


**Figure 7.** 3D printed MREs developed by Bastola et al [ 175, 176]. (a) Line patterned 3D printed MREs, change in direction of the applied magnetic field with respect to the printed filaments. (b) Dot patterned 3D printed MREs, change in direction of the applied magnetic field with respect to the printed dots.

Furthermore, Bastola et al [175, 176] used a 3D printing method to develop line patterns with the help of a continuous printing while the dot-pattern printing is a discontinuous printing. In other words, for the line patterns, the nozzle continuously dispenses the materials in the printing path; while, for the dot patterns, the nozzle only dispenses materials in the specified points, see Figure 7. Developments of unique structures such

as anisotropic line patterns, grid patterns, various dot patterns, and basic crystal structures such as body-centered cubic (BCC) and face-centered cubic (FCC) demonstrate the capability of a 3D printing method. Such a flexible manufacturing process is difficult with other conventional fabrication techniques by solely applying a magnetic field during the MRE fabrication. The line-pattern 3D printed MR elastomer exhibits an anisotropic MR effect when the direction of the applied magnetic field is parallel to the plane of printed filaments. Dot-patterns 3D printed MR elastomers exhibit anisotropic MR effects when the direction of the applied magnetic field is normal to the plane of printed dots. The novel approach for the development of MRE materials using different 3D printing techniques can further be explored. For example, other potentials of 3D printing techniques such as shape memory effects can be studied in the future.

Very recently (2020), the development of shape-programmable magneto-active soft material (MASM) using the 3D printing technique has been also reported by Qi et al [177]. In the process, filaments of MR materials were 3D printed and afterward encapsulated within a silicone rubber using a manual process, see Figure 8. This technique is similar as reported by Bastola et al [172] (Figure 6), but silicone rubber was manually poured into the mold containing 3D printed patterns as given in Figure 8. It should also be noted that the CIPs were mixed with Polylactic acid (PLA), which is already an established 3D printable material using a fused filament fabrication (FFF) technique. The study [177] provides the possibility that the CIPs can be mixed with existing 3D printable materials to develop 3D printed magnetic elastomers. In contrast, Bastola et al [172] demonstrated a new method to make 3D printed MREs using new material combinations by means of rheological modifications.

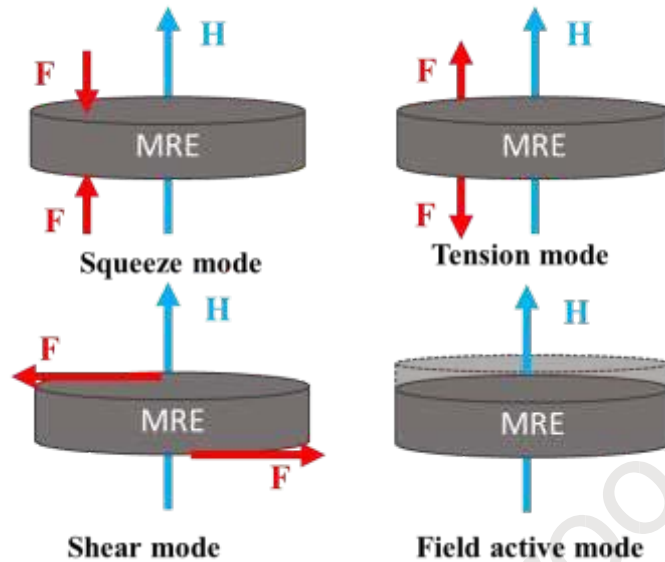


**Figure 8.** Schematic illustrations of 3D printing and encapsulation. 3D printing is employed for manufacturing various magnetic structural elements, which are encapsulated by a silicone rubber (photos of the MASM samples with oriented magnetic structural elements are taken from [177]).

#### 4. Magneto-mechanical characterizations of MREs

There are a number of testing methods available in the literature in order to characterize the magnetomechanical properties of MREs. The most widely used test method is the shear mode and other modes can be uniaxial

compression or tension as well as biaxial tests. All major testing modes are schematically illustrated in Figure 8. The test approaches reviewed in this sequel are from both static and dynamic cases. Usually, a universal testing machine/dynamic mechanical analyzer (DMA) equipped with a magnetic device to apply a magnetic field or laboratory customized setups are used to investigate the various magnetomechanical properties of MREs. MREs are mainly characterized to determine dynamic moduli (i.e., storage and loss moduli), and finally the so-called MR effect.



**Figure 9.** Basic working/operation modes of MREs. F: Force and H: magnetic field strength.

Using the obtained magneto-mechanical properties, the absolute and relative MR effects are measured for the composites. If  $E_0$  is modulus at zero fields and  $E_1$  is the modulus when the magnetic field is applied, then the absolute MR effect is measured as simply the difference between these two moduli, i.e., absolute MR effect =  $E_1 - E_0$ . Moreover, a relative MR effect is also measured which is defined as the relative difference between the two moduli, i.e., relative MR effect =  $(E_1 - E_0) / E_0$ . Note that the relative MR effect is usually

and dynamic modes. A magnetic device developed using either electromagnets or permanent magnets is

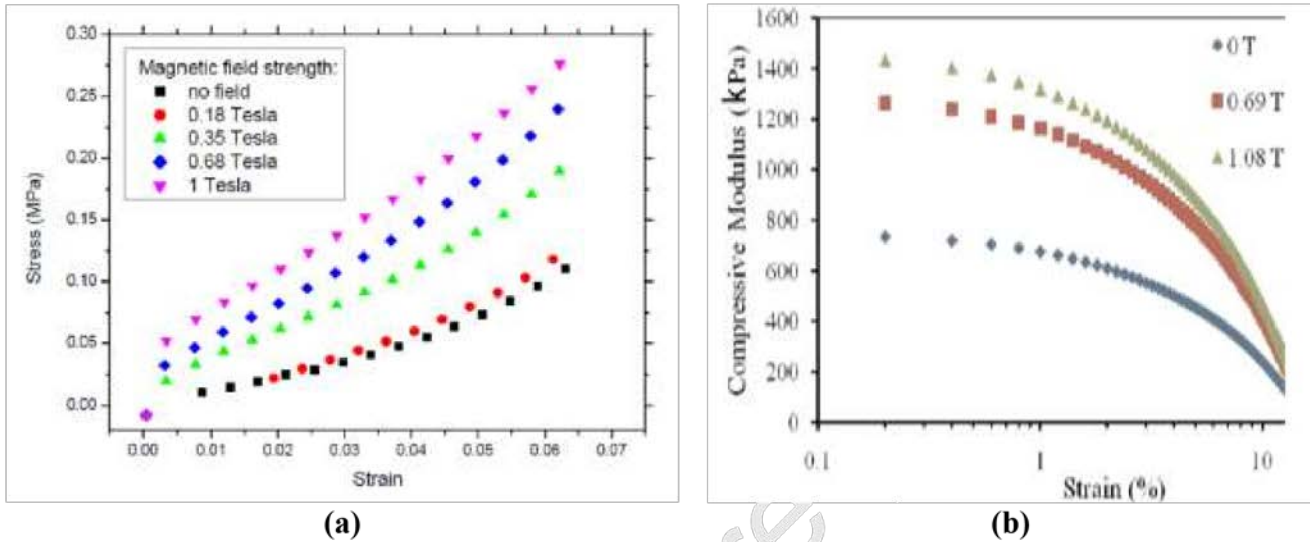
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expressed as percentages.

## 4.1. Uniaxial Compression Test

The compression test is one of the most popular methods to investigate the properties of MREs both in static and dynamic modes. A typical result of static compression is given in Figure 10. Figure 10(a) shows the results of the stress-strain curves as performed by Kallio [109], which were obtained at a strain level of 6.5% by applying a magnetic flux density up to 1 T (Tesla). Figure 10(b) shows results of compressive modulus versus strain at various magnetic flux densities and up to 20% strains as obtained by Gordaninejad et al [130]. Both stress and modulus versus strains at various magnetic flux densities are used to report the compressive behavior of MREs. The experimental results demonstrated that the moduli are increasing with the increase of the applied magnetic field.

Table 2 provides a few results from the compression mode of deformations as summarized based on MRE types, test conditions, and MR effects. As can be seen from Table 2, the testing conditions such as particle concentration, magnetic flux density, and the direction of the magnetic flux are different among different studies. Thus, a direct comparison of different studies is inappropriate. Particle concentrations ranging from 4.45% to 33% by volume can be observed. Similarly, the absolute MR effect reported varies from 38.9 kPa to 5.5 MPa. The highest relative MR effect reported is 223% by Abramchuk et al [116]. In order to obtain the MR effect, the modulus at zero magnetic fields and the final modulus upon the application of a magnetic field are used. The stiffness modulus considered in most of the studies is the tangent modulus.



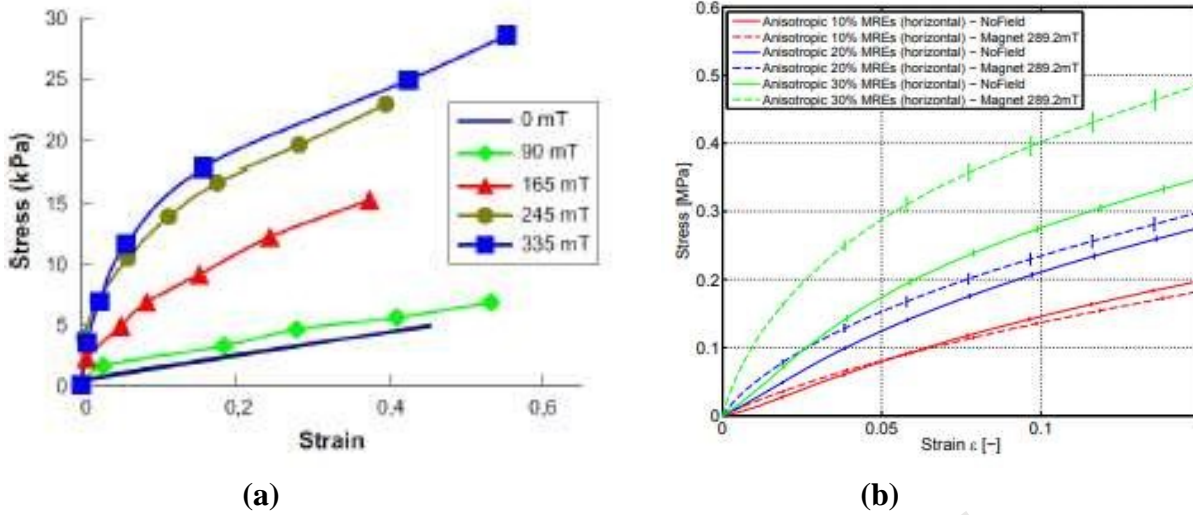
**Figure 10.** Results of uniaxial compression tests performed by (a) by Kallio [109] and (b) by Gordaninejad et al [130].

**Table 2.** Experimental results of a wide range of uniaxial compression tests on MREs. The summary is based on types, concentrations of MREs, and testing conditions with the maximum absolute and relative MR effects.

Study	Materials		Test conditions		MR effect	
	PVC	Type	Strain level	Magnetic flux and direction	Absolute	Relative
Kallio [109]	30 %	Aniso @1000 mT	6.50%	700 mT $\perp$ loading $\parallel$ alignment	1.75 MPa	100%
Verga et al [178]	5.45%	Aniso @400 mT	40%	100 mT $\parallel$ loading $\parallel$ alignment	32kPa	58%
Abramchuk et al [116]	9.20%	Iso	30%	230 mT $\perp$ loading	38.9 kPa	223%
Boczkowska et al [179]	33%	Aniso @ 100 mT	30%	300 mT	80 kPa	4.50%
Gudmundsson et al [127]	27%	Aniso	15%	700 mT $\parallel$ loading $\parallel$ alignment	5.5 MPa	120%
Gordaninejad et al 2012 [130]	23.90%	Aniso @1000 mT	20%	710 mT $\parallel$ loading $\parallel$ alignment	550 kPa	73%
Schubert and Harrison [180]	30.00%	Aniso @400 mT	50%	450 mT $\parallel$ loading $\parallel$ alignment	3.65 MPa	111%

## 4.2. Uniaxial Tensile Test

Tensile test setups are very similar to a compression test; the only difference is that an MRE sample is stretched instead of compression. Typical results of tensile tests are given in Figure 11. As shown in Figure 11, in a tensile test, when a magnetic field is applied, the increase in the total stress is much more pronounced within a small strain region. It has also been observed that the MR effect decreases rapidly with the increasing strain levels in the tensile tests.



**Figure 11.** Results of uniaxial tensile tests performed by (a) Stepanov et al [117] and (b) Schubert and Harrison [180].

Table 3 summarizes a set of experimental investigations on MREs under tensile testing protocols. In the literature, experimental results up to 100% strains are available. However, as expected, the MR effect is higher at a low strain. This is due to the fact that during a mechanical stretching, the distances among magnetic particles are increased that results in reduced interactions between them. The maximum relative MR effect of 3000% has been reported by Stepanov et al [117], which was mainly observed at a low strain level. Recently, in 2019, Hernández et al [181] performed tensile tests on isotropic MREs by varying CIP concentrations ranging from 20% to 70 % (by wt.%) and found that when a 52 mT of magnetic flux density is induced on the material samples, the one with 63 wt.% of CIPs shows the maximum MR effect. However, the relative MR effect reported by Hernández et al [181] is still lower compared to the MR effect reported by Stepanov et al [117]. This is because of the difference in a zero  $\sigma$ -field modulus of MRE samples and testing conditions.

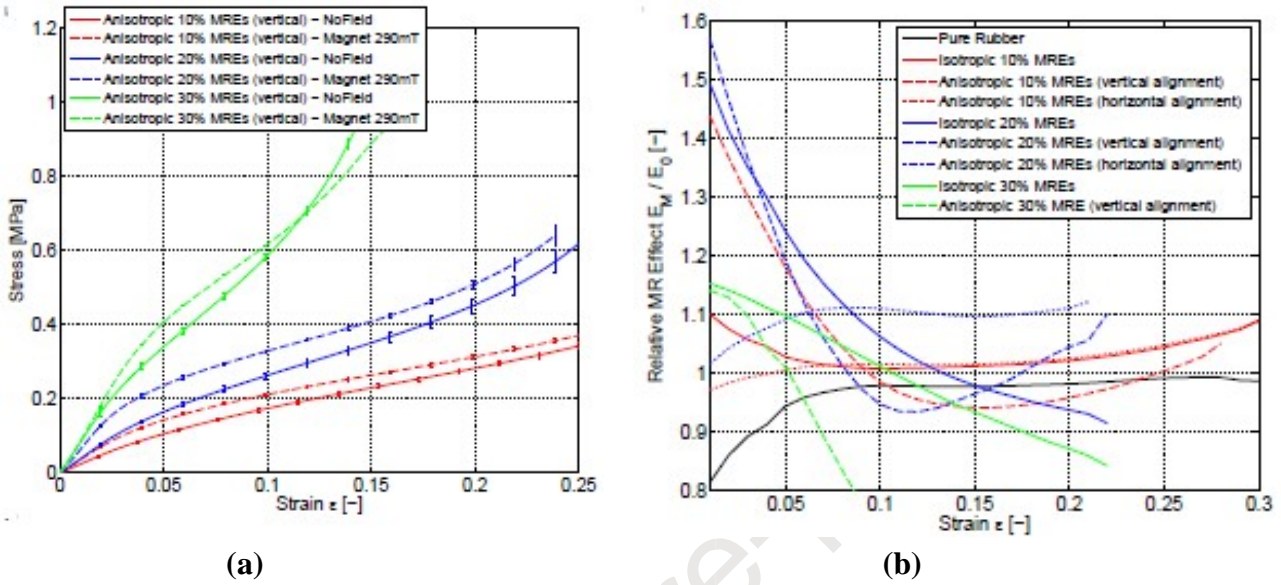
**Table 3.** Experimental results of a couple of investigations conducted on MREs under a uniaxial tensile mode. Summary is based on types, concentrations of MREs, and testing conditions with maximum absolute and relative MR effects.

Study	Materials		Test		MR effect	
	PVC	Type	Strain level	Magnetic flux and direction	Absolute	Relative
Bellan et al [106]	15%	Aniso @ 250 mT	10.00%	150 mT $\parallel$ loading $\parallel$ alignment	-	-
Stepanov et al [117]	37%	Iso	60%	335 mT $\perp$ loading	400 kPa	3000%
Schubert et al [180]	30%	Aniso @400 mT	50%	289 mT $\parallel$ loading $\parallel$ alignment	12.17 MPa	284%
Mordina et al [182]	10% wt. %	Aniso @200 mT	1%	1089 mT	9.6 kPa	7%
Hernández et al [181]	63 wt. %	Iso	-	64 mT	65 kPa	118%

### 4.3. Simple Shear Test

The shear mode of testing is one of the most popular modes that has been adopted to characterize MRE properties. This fact can also be supported from the applications point of view, as a magneto-responsive composite is subjected to the shear loading in most of the MRE-based devices such as in vibration isolators and absorbers [15, 16]. Shear tests can be of two types; single lap shear and double lap shear. Test conditions are also similar to that of compression/tensile testing. However, strain levels and strain rates cannot be as high

as that of tensile tests (strain level can be higher than 100% in tensile tests). A widely known shear experimental result performed by Schubert and Harrison [180] is given in Figure 12. Therein, the MR effect was found to be higher at smaller strain levels and decreased with the increasing strain.



**Figure 12.** Results of the pure shear test performed by Schubert and Harrison [180]. (a) Stress-strain results at various concentration of fillers, and (b) relative MR effect versus strain where the MR effect was obtained with the help of the tangent moduli.

Table 4 summarizes a few investigations on MREs via the shear tests. Yet again, we can see that the testing conditions and the outcome are not exactly similar. The maximum absolute MR effect of 2.05 MPa by Schubert and Harrison [180] while 750% relative MR effect have been achieved by Stepanov et al [117].

**Table 4.** Results of a few experimental investigations via shear testing on MREs. The summary is based on types, concentrations of MREs, and testing conditions with maximum absolute and relative MR effects.

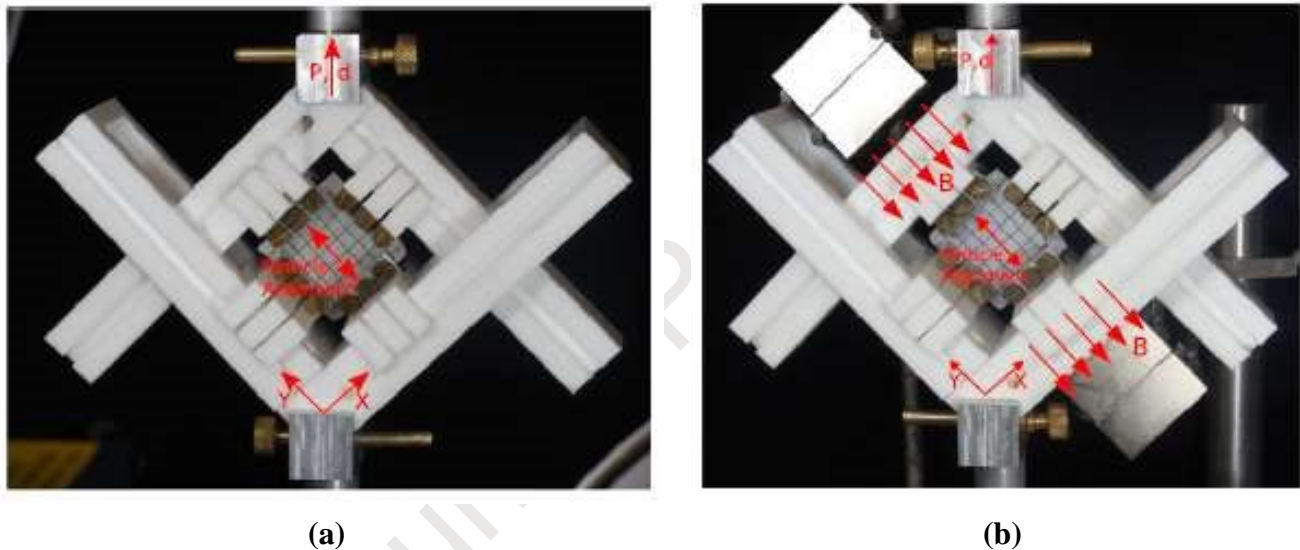
Study	Materials		Test		MR effect	
	PVC	Type	Strain level	Magnetic flux and direction	Absolute	Relative
Shen et al [183]	25%	Aniso @ 400 mT	12%	395 mT $\perp$ loading $\parallel$ alignment	327 kPa	64%
Stepanov et al [117]	24%	Aniso @ 400 mT	20%	80 mT $\perp$ loading	150 kPa	750%

Yu and Wang [184]	33%	Aniso	120%	47.9 mT $\parallel$ alignment	1.75 MPa	25%
Zajac et al [123]	35%	Iso	13%	163 mT $\perp$ loading	-	70%
Hu et al [185]	24%	Iso	100%	400 mT	125 kPa	500%
Gordaninejad et al [130]	24%	Aniso @ 1000 mT	15%	700 mT $\perp$ loading $\parallel$ alignment	130 kPa	37%
Schubert and Harrison [180]	20%	Aniso @ 400 mT	25%	290 mT $\parallel$ loading $\parallel$ alignment	2.05 MPa	57%

#### 4.4. Equi-Biaxial Test

In 2016, Schubert and Harrison [186] performed equi-biaxial tests for both isotropic and anisotropic MREs with the application of a magnetic field for the first time. A bespoke test rig was designed to conduct the equi-biaxial test, as shown in Figure 13. The magnetic field can be applied in two different directions either parallel or perpendicular to the direction of the particle alignments of an anisotropic MRE. The relative MR effect up to 74% at a magnetic flux density of 67.5 mT was reported for the anisotropic MRE when the applied magnetic field is parallel to the chains of particle alignment.

Equi-biaxial tests for MREs were also performed by Zhou et al [29] and Gorman et al [142, 187] using bubble inflation test setups. Furthermore, these groups have investigated the fatigue behavior of MREs, which will be discussed in Section 4.5.



**Figure 13.** The biaxial test setup used in Schubert and Harrison's study [186], (a) An anisotropic MRE without any magnetic field (b) An anisotropic MRE with a magnetic field in  $y$ -direction parallel to the particle chains. ( $p$  is vertical load and  $d$  is the displacement).

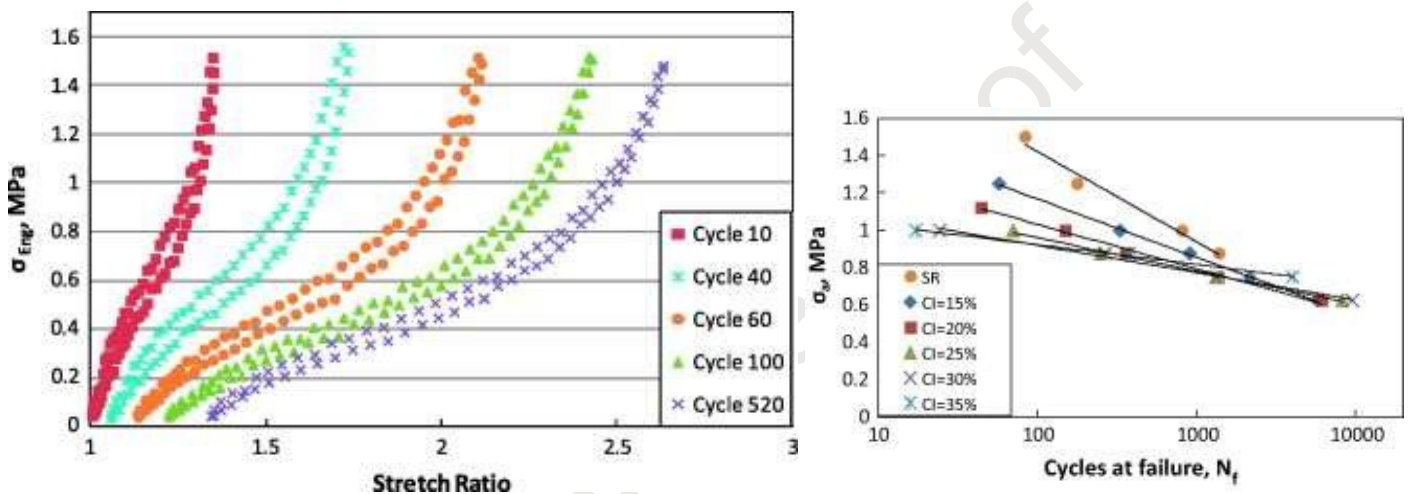
#### 4.5 Fatigue Test

Fatigue strength of MREs is one of the most critical mechanical properties that need to be investigated to efficiently deploy them in real engineering systems such as in high dynamic loadings experienced by machine parts. One of the first experimental works on the fatigue properties of MREs has been reported by Zhou et al



[29] in 2013 and some results from the fatigue test are given in Figure 14. They have performed equi-biaxial tests on an MRE by creating a bubble inflation method without applying a magnetic field. Initially, they investigated the fatigue behavior of MREs in the absence of a magnetic field. However, the same research group [133, 141] further investigated the same behavior of MREs varying the concentration of CIPs. It was reported that the fatigue life decreased with the increasing strain amplitude and the modulus decreased with an increasing number of cycles. The MRE material failed in a range suggesting a limiting value of complex modulus for the material between 1.22 MPa and 1.38 MPa regardless of the CIP contents and the magnitude of the stress amplitude.

In 2017, Gorman et al [142, 187], investigated the fatigue behavior of MREs in the presence of a magnetic field. They have performed the fatigue study of MREs in both uniaxial and biaxial test methods. Firstly, Gorman and co-workers reported that, in both types of tests, the largest MR effect was observed for low strain levels. Secondly, they reported that it is not difficult to provide a direct comparison between uniaxial and biaxial data. However, the experimental trend follows the other experimental studies like an increase in the modulus with an increasing magnetic field.



**Figure 14.** (a) Stress softening behavior in an engineering stress-controlled fatigue test for selected cycles (stress amplitude = 0.75 MPa, 20 vol.% CIPs, and (b) S-N curves for MREs with various CIPs concentrations, performed by Zhou et al [29, 141].

#### 4.6. Dynamic Test

The dynamic tests include both compression/squeeze mode and shear mode. A dynamic test aims to study the viscoelastic behavior of MRE as related to excitation frequency, strain amplitude, and magnetic field strength. Researchers have adopted two ways for dynamic characterizations of MREs: the first one is to study MRE samples using a dynamic mechanical analysis (DMA) or rheometer [10, 71, 188-198], while other groups

of researchers study the dynamic behavior of MREs via a vibrational analysis [107, 145, 172, 175, 199, 200]. In the second approach, mostly the force vibration testing is adopted to study the dynamic behavior of MREs.

#### 4.6.1 Dynamic Mechanical Analysis

A linear viscoelastic model can describe the behavior of MREs in the viscoelastic region with a small strain amplitude. When the viscoelastic material is subjected to a sinusoidal loading, the strain is either in the lagging phase or the leading phase with the stress. The applied instantaneous stress can be expressed as a sinusoidal function of the maximum stress amplitude  $\sigma_o$ :

$$\sigma = \sigma_o \sin(\omega t + \delta) = \sigma_o \sin \omega t \cos \delta + \sigma_o \cos \omega t \sin \delta \quad (1)$$

where,  $\omega$  is the angular frequency,  $t$  is time and,  $\delta$  is a phase angle between strain and the stress. Equation (1) can be expressed as:

$$\sigma = \gamma_o (G' \sin \omega t + G'' \cos \omega t) \quad (2)$$

where,  $\gamma_o$  is the maximum strain amplitude,  $G'$  is the storage modulus and  $G''$  is the loss modulus. The storage and loss moduli represent the ability of an elastomeric material to store and to dissipate the energy of distortion, respectively. Normally, these two moduli are expressed as a complex quantity. The complex modulus  $G^*$  of material and loss modulus of the viscoelastic material are expressed as follows;

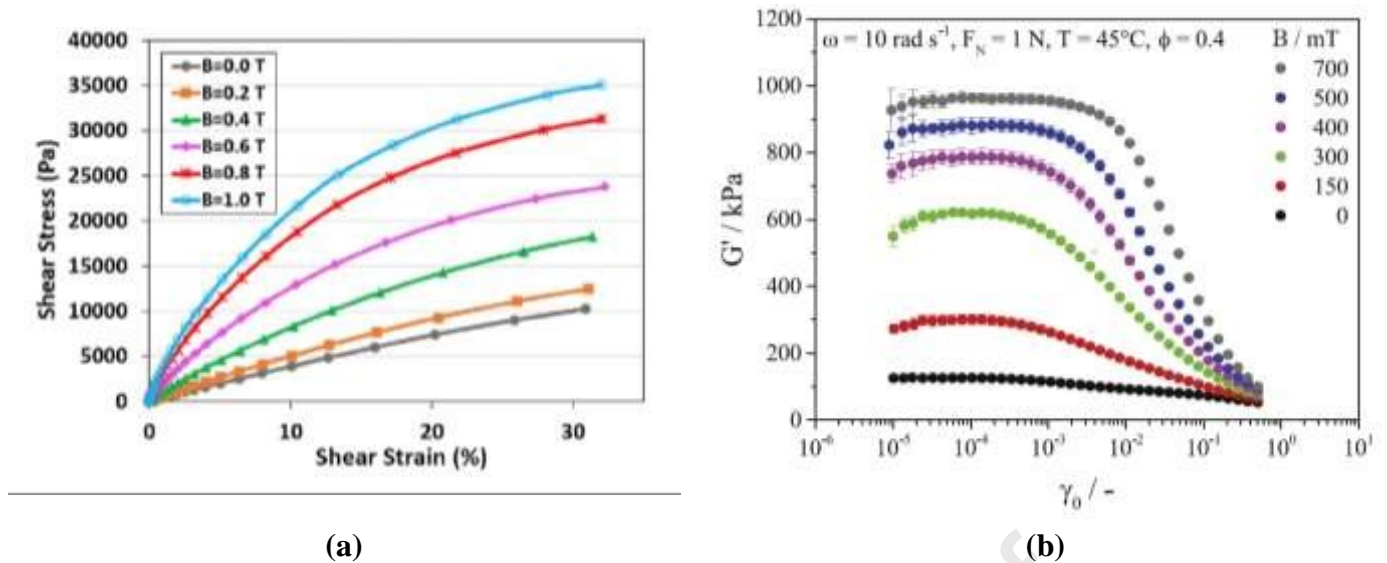
$$G^* = G' + iG'' \quad (3)$$

$$\tan \delta = \frac{G''}{G'} \quad (4)$$

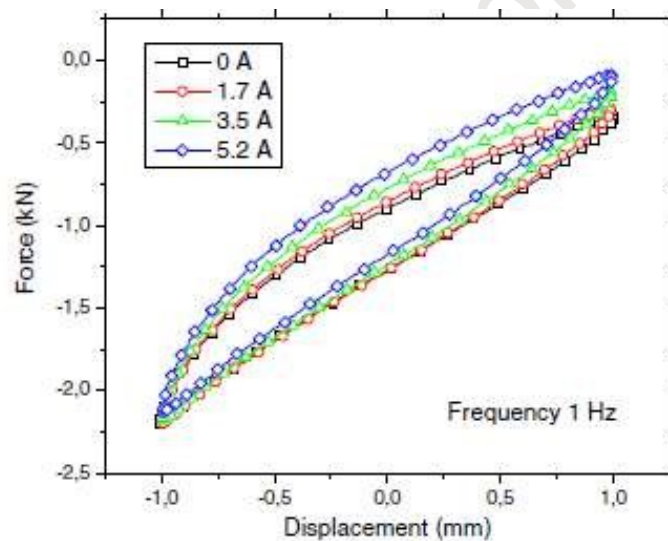
where,  $\tan \delta$  is a loss tangent angle/loss factor. For MREs, using rheology, responses of  $G'$  and  $G''$  can be studied using various tests such as amplitude sweep, frequency sweep, temperature sweep, and magnetic field sweep. Experimental conditions of different types of sweeps are exhaustively presented in an article by Li and Nakano [171].

The experimental results of a rheological test are given in Figure 15. The stress-strain curves are similar to those that are obtained via a simple shear test. The advantage of the rheological or DMA test is that the instrument comes with an extremely compact magnetic device, which offers a high range of magnetic fields (>1000 mT). Whereas in a simple shear test performed using universal testing instruments, the magnetic field strength completely depends on a bespoke test setup. Thus, mathematical modeling and comparative study become a difficult task for the studies performed using a bespoke test setup. Several commercially available instruments such as Anton Paar (Austria) and TA instruments (USA) rheometers offer very good control of

the magnetic field during actual tests. Thus, such rheological studies facilitate the modeling of MRE behavior with much more reliable data [71, 188, 189, 201, 202]. Walter et al [189, 203] have performed a number of systematic studies to provide the dynamic characterization of MREs with the use of Anton Paar rotational rheometry. The rheological measurement data are further used to model the behavior of MREs [189].



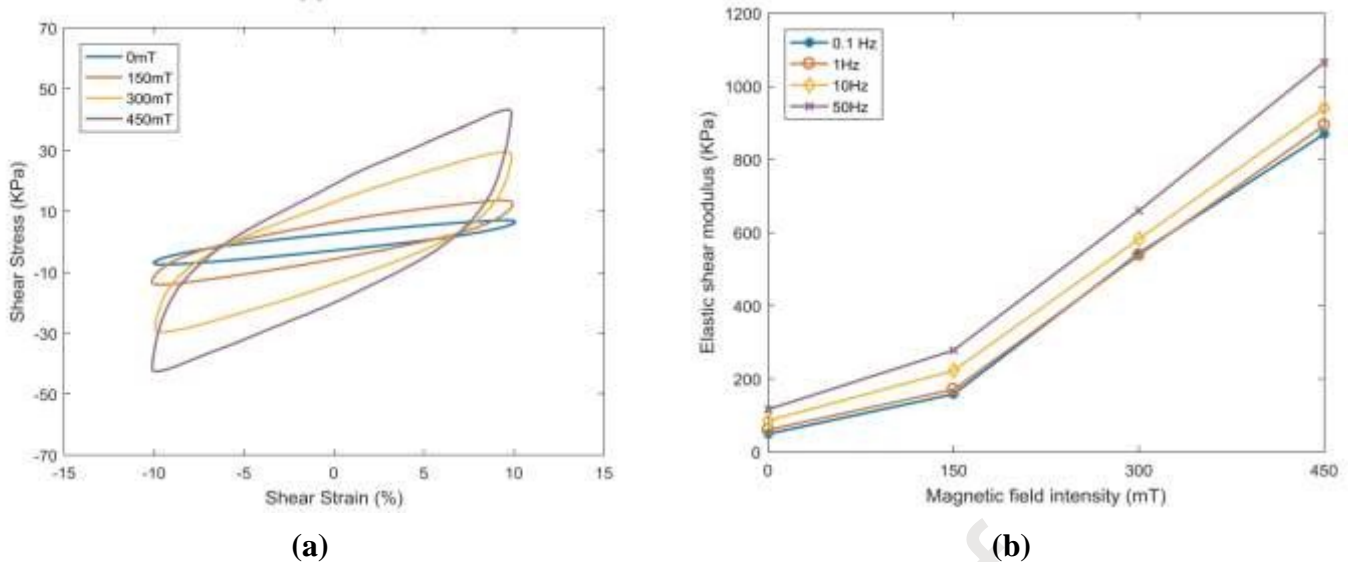
**Figure 15.** Experimental results of rheological measurements: (a) Shear stress vs. strain for different levels of magnetic flux densities at 0.1 Hz oscillation frequency as performed by Khanouki et al. [188], and (b) Storage modulus versus strain amplitude at various magnetic field strengths obtained by oscillatory strain sweep measurements as conducted by Walter et al [189].



**Figure 16.** Dynamic force-displacement loops with increasing current to the electromagnet at 1 Hz of sinusoidal frequency, performed by Kallio et al [110].

An example of dynamic compression testing on MRE using a bespoke test setup is given in Figure 16. Force-displacement loops with an increasing magnetic field can be recorded as shown in Figure 16, was performed by Kallio et al [110]. Such hysteric cycles can also be expressed as stress-strain loops [204, 205]. The hysteric loops provide the viscoelastic information of MREs such as storage modulus, loss modulus, or loss factor.





**Figure 17.** Experimental results of the dynamic shear tests performed by Dargahi et al [ 206]. (a) Stress-strain loops at 10 Hz and at various magnetic field intensities, (b) Storage modulus versus magnetic field intensity at various excitation frequencies.

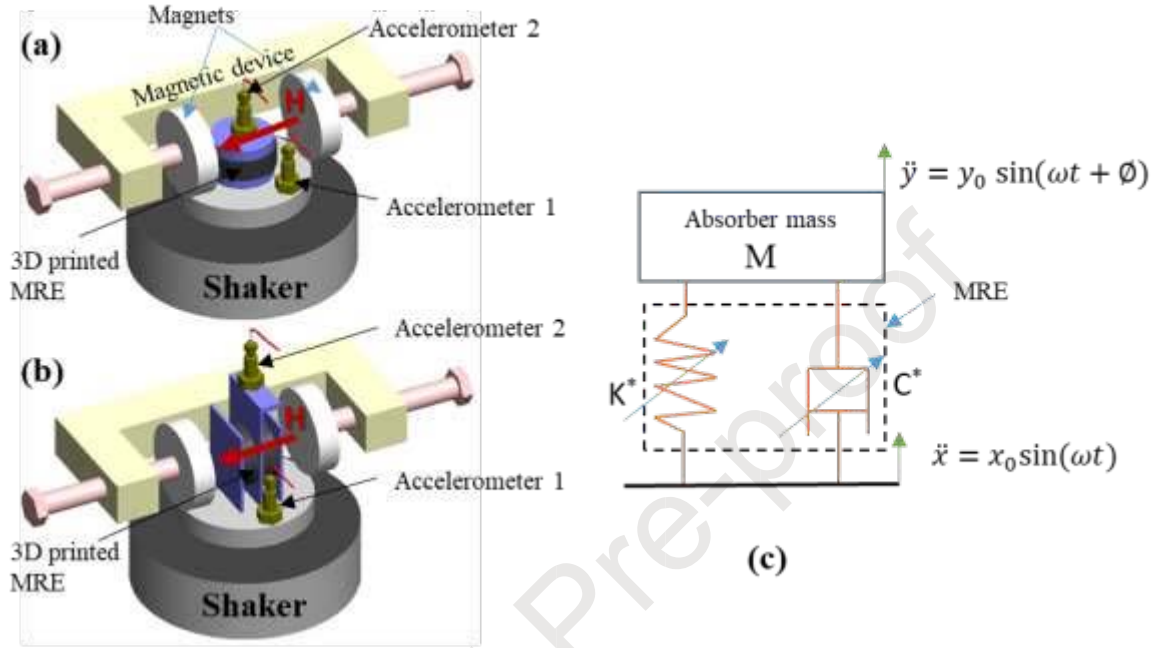
In the shear mode, the stress-strain or force-displacement loops also look similar as in Figure 15, see Figure 17(a). An example of elastic modulus obtained for an MRE in a shear mode is given in Figure 16 which is obtained by Dargahi et al [ 206]. The slope of the stress-strain loop is generally considered as the storage/elastic modulus. The area under the stress-strain loop provides the damping capability/loss modulus for the MREs. In a dynamic test, the general trend is that the moduli increase with the increasing magnetic field strength and excitation frequency while decrease with the increasing strain levels.

#### 4.6.2 Forced Vibration Analysis

Typically, in the potential applications of MREs such as vibration absorbers and isolators, the range of loading frequency can widely vary [15, 16]. However, using a DMA or a rheometry, the testing frequency is limited, usually less than 100 Hz [196, 206-208]. Thus, other types of technologies are needed in order to characterize MREs in a wide frequency range. According to Smit et al [209], the testing frequency can be as high as 6000 Hz using a forced vibration technique. The frequency range of interest can easily be obtained by tuning the thickness of the samples ( $h$ ) and the absorber mass ( $m$ ). In forced vibration testing, a single degree of freedom system is developed where the MRE sample acts as a spring element. Accelerometers are only the sensors used in vibration tests. Hence, test setups are relatively smaller and easily adaptable for use in small temperature-controlled chambers [209] as well as magnetic field-controlled systems needed for MREs [107, 145]. In 2003,

Zhou GY [107], reported the first vibration testing method to obtain the shear properties of MREs, in which he used the free vibration testing method.

A typical experimental setup used in the forced vibration test is given in Figure 18. It mainly consists of a shaker, accelerometers, and a magnetic field generator. The shaker provides excitation signals. Furthermore, it can control the range of strain amplitude and frequency. The MR elastomer system forms a single degree of freedom (DOF) system. Two accelerometers are used; one records the excitation signal from the shaker and other records the response signal of the MRE system. The magnetic field generator can either be developed using a permanent magnet or an electromagnet. The magnetic field can be applied either normal or parallel to the direction of vibration.



**Figure 18.** Experimental setups for the forced vibration. (a) MR elastomer in a squeeze mode operation, (b) MR elastomer in a shear mode of operation, and (c) a schematic representation of the single degree of freedom system with the base excitation [172].

In the forced vibration testing, for a single degree of freedom system, the natural frequency ( $\omega_0 = 2\pi f_0$ ) of a single DOFs system can simply be calculated as:

$$\omega_0 = \sqrt{\frac{K}{M}} \quad (4)$$

where,  $K$  and  $M$  is the stiffness and the mass of the single DOF, respectively. When the damping element is presented, the natural frequency becomes the damped natural frequency ( $\omega_d$ ) and generally damping element has very less fluctuation of the natural frequency as:

$$\omega_d = \omega_0 \sqrt{1 - \xi^2} \quad (5)$$

where,  $\xi$  is the damping ratio and defined as: the ratio of actual damping ( $C$ ) to critical damping, ( $C_c$ ) value of the material as;

$$\xi = \frac{C}{C_c} = \frac{C}{2m\omega_0} \quad (6)$$

The magnitude and phase transmissibility of a single DOF vibrational system can be expressed as:

$$T = \sqrt{\frac{1 + (2\beta\xi)^2}{(1 - \beta^2)^2 + (2\beta\xi)^2}} \quad (7)$$

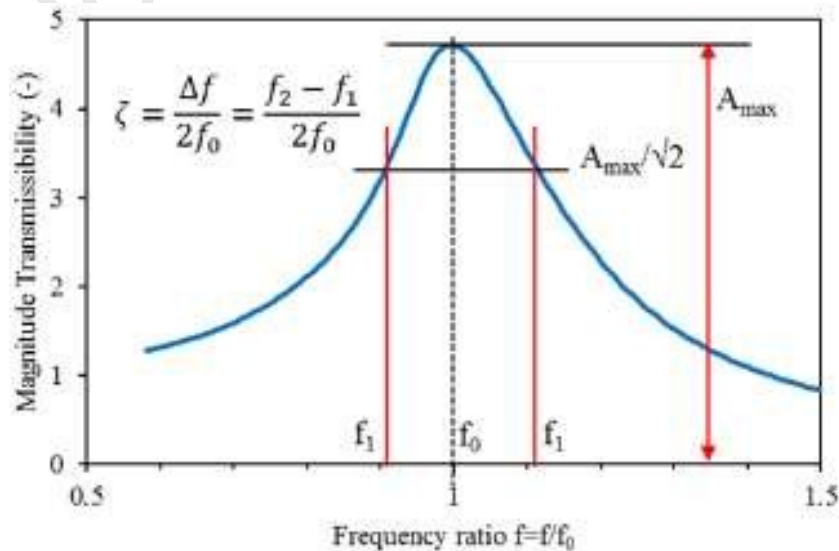
$$\delta = \tan^{-1} \frac{-2\xi\beta^3}{1 - \beta^2 + (2\beta\xi)^2} \quad (8)$$

where,  $\xi$  is the damping ratio,  $\beta = \frac{\omega}{\omega_0}$  or  $\frac{f}{f_0}$  is the frequency ratio between the excitation frequency and the natural frequency of the system.

With the forced vibration technique, the frequency-response curve of the samples can be constructed through a frequency sweep by measuring the excitation and response amplitude of the vibration. The typical frequency response of a single degree of freedom is shown in Figure 19. In the figure, the magnitude transmissibility is obtained as a ratio of the response signal and the excitation signal, while  $A_{max}$  is the maximum displacement at the resonance frequency. As the transmissibility amplitude reaches the maximum value, the phase delay between the base and mass becomes  $90^\circ$ . Under such a condition the corresponding excitation frequency becomes the natural frequency. Therefore, the stiffness of the system can be obtained using Equation 9, while; the damping ratio can be obtained as follows:

$$\zeta = \frac{\Delta\omega}{2\omega_0} = \frac{\Delta f}{2f_0} = \frac{f_2 - f_1}{2f_0}$$

where,  $\Delta\omega$  or  $\Delta f$  is the frequency difference when the response amplitude is equal to  $A_{max}/\sqrt{2}$  response amplitude as illustrated in Figure 19.



max

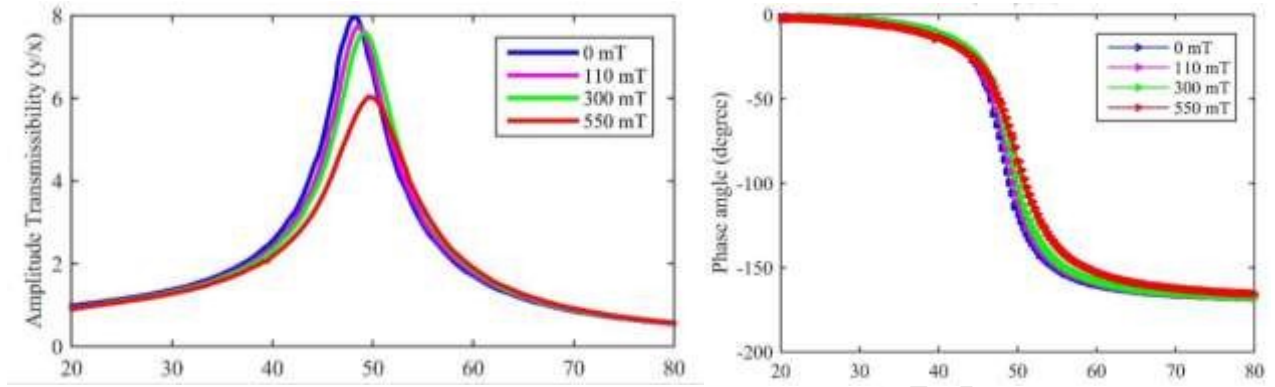


(9)

 $max/\sqrt{2}$  of the peak

**Figure 19.** A frequency response curve of a moderately damped single degree of freedom system is illustrated. For the definition of symbols, readers are referred to the text.

A typical response of the forced vibration testing is given in Figure 20. The peak amplitude of the magnitude transmissibility curve indicates the natural frequency and using Equations 1, 2, and 3 stiffness, damping ratio, and damping coefficient can be obtained. Typically, the increasing magnetic field increases the natural frequency and thus the stiffness of MREs.



**Figure 20.** Magnitude and phase transmissibility versus excitation frequency of an MRE shear mode at various magnetic flux densities [210].

Furthermore, the following stress-strain and force-stiffness relationships, and equation of motions can be used to obtain storage and loss modulus of MREs using the forced vibration analysis [145, 209]

$$\sigma = G^* \gamma$$

$$F = k_e^* (y - x)$$

$$y + \frac{k_e^*}{m} (y - x) = 0$$

where, star (\*) indicates a complex variable,  $\ddot{y}$  is the acceleration and  $y$  is the displacement of the absorber mass ( $m$ ),  $k_e^*$  denotes the stiffness of MRE,  $x$  is the displacement of the shaker base,  $G$  is the elastic modulus of the MRE,  $\sigma$  represents the compressive stress,  $\gamma$  represents compressive strain, and  $F$  is the

(10)

(11)

(12)

force applied to the MRE sample.

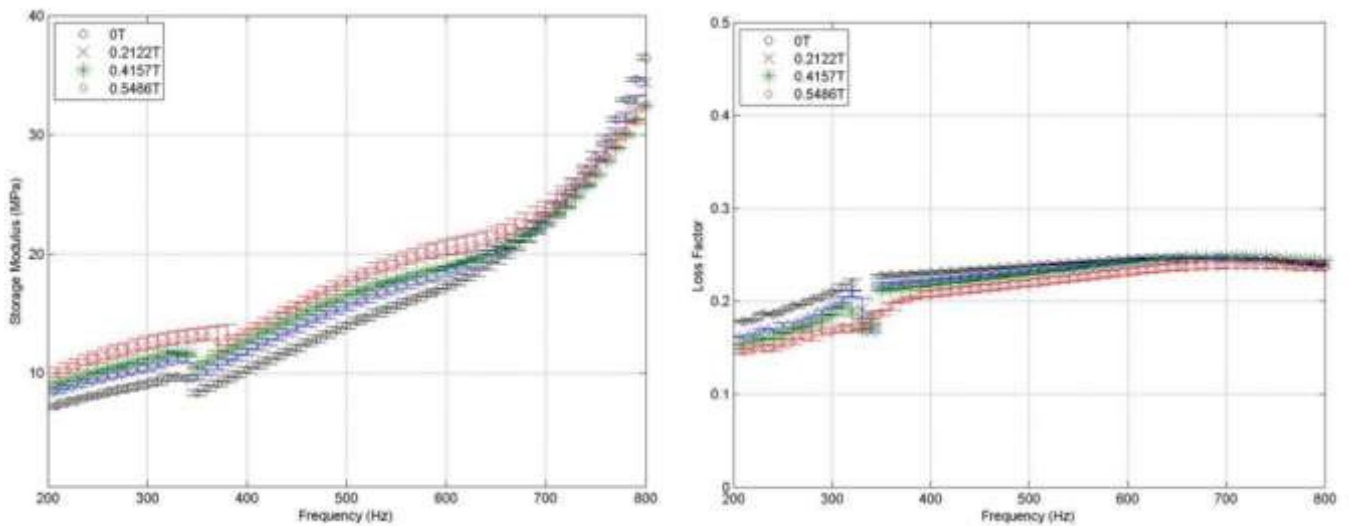
An MRE system is expected to have a variable natural frequency as the stiffness of an elastomer can be changed with the excitation frequency. For the frequency sweep, the above equations can be deployed to obtain the loss factor first and then obtain the natural frequency and finally the storage modulus of the MREs as follows [145, 209]

$$\delta = -\frac{\tan(\varphi)}{\frac{T}{\cos(\varphi)} - 1} \text{ for } \varphi > 0 \quad (13)$$

$$f_n = f \sqrt{\frac{\tan(\varphi) - \delta}{(1 + \delta^2)\tan(\varphi)}} \quad (14)$$

$$G' = \frac{mH(2\pi f_n)^2}{A} \quad (15)$$

where,  $T$  is the magnitude of the transmissibility (which is the ratio of output and input accelerations),  $\delta$  is the loss factor,  $\varphi$  is the phase angle of the system,  $f$  is the excitation frequency,  $f_n$  is the natural frequency,  $G'$  is the storage modulus,  $H$  is the thickness, and  $A$  is the area of the MRE sample. Using Equations 13, 14, and 15, the dynamic properties of MREs (storage modulus and loss factor) can be obtained from magnitude and phase transmissibility curves. An example of the storage modulus and loss factor obtained through forced vibration analysis is given in Figure 21, performed by Johnson et al [145]. Recently, in 2020, Bastola et al [175] also used a similar technique to characterize the dynamic behavior of 3D printed MREs over the 100-500 Hz frequency range.



**Figure 21.** Storage modulus ( left) and loss factor (right) at various magnetic flux densities of MREs obtained via forced vibration analysis, performed by Johnson et al [145].

The forced vibration test also shows that the storage modulus of an MRE is dependent on the magnetic flux density, frequency, and acceleration amplitude (strain level). The storage modulus is found to be increased when the strength of the magnetic field or frequency was increased, while the storage modulus was decreased with the increasing strain amplitude.

## 5. Summary and Future Outlook

In MREs, the distribution of magnetic particles influences the MR effect. Magnetic particles (mainly CIPs) can be distributed either randomly (i.e., isotropic MREs) or in an aligned manner (i.e., anisotropic MREs). For the same concentration of CIPs, the anisotropic configuration of magnetic particles leads to a higher MR effect than that of an isotropic configuration. It should be noted that the magnetic flux direction should be parallel to the direction of particle alignment. In the same way, the percentage content of magnetic particles also influences the MR effect. Usually, either volume fractions (vol.%) or weight fractions (wt.%) are adopted to report the content of magnetic particles, where vol.% can range from 0 to 40% and the wt.% ranges from 0 to 85%. Commonly, both the relative and the absolute MR effects are found to increase with the increasing concentrations of CIPs. It should be noted that too high particle concentration decreases the amount of matrix material. A higher concentration of CIPs could increase the zero-field modulus/stiffness but not the MR effect. Based on the current literature, the optimum concentration of CIPs is about 30% by volume.

Within the current fabrication techniques, controlling the arrangement of the magnetic particles within the matrix material is a non-trivial task as the particles can only be configured in the direction of the magnetic

flux lines. Consequently, a unique configuration is tough to obtain, and it has not been reported to date. However, some potential to develop unique configurations have been reported via various 3D printing techniques. The novel approach for the development of MRE materials using 3D printing methods can further be explored. For instance, other potentials of 3D printing techniques such as shape memory effects, sandwich structures and soft robotics should be studied in the future to take full advantage of such disruptive manufacturing processes. In addition, numerical simulations of the 3D printed MREs can be performed to optimize the MR filament size, orientations and finally the MR effect.

There are a number of testing methods adopted to characterize MRE's mechanical properties, such as uniaxial compression, tension and shear, and multi-axial testing in both the absence and the presence of a magnetic field. Both static and dynamic measurements can be found in the literature. The types of MRE materials (matrix, CIP concentration, isotropic/anisotropic) and the testing conditions (mode, strain level, strain rate, the way of applications, and the strength of magnetic field) and even data analysis differ in each investigation. Thus, a direct comparison is not reliable, and a difficult task, though a certain trend can be developed.

In summary, the following trends are observed in the experimental studies on the MREs:

- MR effects increase with the increase of magnetic particle concentrations (i.e., CIPs).
- Softer matrix materials (with low no-field moduli) lead to higher relative MR effects, but not necessarily the larger absolute MR effects.
- The addition of small quantities of additives such as CNTs and MNPs also increases the MR effect.
- Anisotropic MREs show higher MR effects than that of isotropic MREs. MR effects are the highest when loading and magnetic flux directions are parallel to the chains of particle alignment.
- MRE materials saturate above 700 mT magnetic induction and MR effects do not increase with the increasing magnetic flux.
- Shear and compression modes of deformations are the most popular testing procedures. Compression tests reveal lower MR effects than other deformation modes.
- For static tests, the MR effects are observed to be higher at a lower strain level.
- While characterizing the MRE materials in compressive or tensile loading, the so-called Mullins effect plays a vital role. Therefore, preconditioning is needed to remove the Mullins effect from samples before any actual test.
- The MR effect is higher in low strain regimes. Therefore, when characterizing under different operation modes such as squeeze, tension, and shear modes and when designing applications using MRE materials, a low strain level (< 10%) can be considered.

- Moreover, the MR effect will not be constant over the entire strain range. Thus, stating the strain limits for any MRE test results are highly important.
- Dynamic tests using a rheometer/DMA provide highly reliable data for the modeling of MRE behavior.
- Dynamic modulus increases with the increasing magnetic field strength and frequency and decreases with the increasing strain amplitude.

The types of matrix materials, CIP concentrations, particle configurations, and even the testing conditions (mode, strain level, the way of applications, and strength of magnetic field) differ in each study. Additionally, the absence of a standard method for reporting a magnetic field makes the largest difficulty to replicate previously published MRE test results. Therefore, there is a strong need for making standard protocols for testing of MRE properties both under static and dynamic conditions in the absence and presence of a magnetic field. Moreover, as per currently available literature, obtaining the dynamic properties such as storage modulus and loss factor of MREs using the forced vibration method is highly recommended as this method does not deform the sample and can be performed in a wide frequency range (10-1000 Hz). Similarly, high strain rate dynamic tests can be performed using the Hopkinson pressure bar (SHPB) [211], in which dynamic stress-strain curves as a function of strain rates in the range of  $10^0 \text{ s}^{-1}$  to  $10^3 \text{ s}^{-1}$  can be obtained. As commercially available SHPBs are designed for hard materials like metals, concretes, and ceramics, a modified SHPB is required to test the MRE properties [211].

In the absence of any existing protocols, there is an urgent need for developing standardized methods for MRE material characterizations, device testing, and performance measurements. So far, a widely shared consensus on how to measure the magneto-mechanical properties of MREs is not available even though MRE research is rapidly growing in recent years. However, it is very common for a relatively new but an active field of research. Such being the case, we attempt and provide the following suggestions for the critical need for the standardization of the magneto-mechanical characterizations of MREs.

- Reporting of a magnetic field is mandatory (not only current to electromagnet): the term “magnetic flux density” should be used instead of the magnetic field or magnetic field strength (A/m). The commonly used handy instruments to measure the magnetic field such as Gauss-meter/Tesla-meter provide the value of the magnetic flux density in Tesla (T) or in Gauss (G)
- The reported magnetic flux density should be uniform throughout the sample thickness
- The best way to generate uniform magnetic flux density is by developing a closed magnetic circuit

- There are a few studies devoted to investigating the magnetic permeability of MREs [212-215]. However, there is still a growing need to report accurate values of MREs based on the different volume fractions of CIPs
- There is no standard sample size for the pure magneto-mechanical testing, either in compression, tensile or shear test. Thus it is recommended to follow sample dimensions as per ISO standards
- Compression tests can be based on ISO 7743: diameter of  $29\pm 0.5$  mm and height of  $12.5\pm 0.5$  mm at a compression rate of  $10\pm 2$  mm/min
- Tensile tests can be performed based on ISO 37: Dumbbell sample: gauge section measuring  $16\pm 1 \times 4\pm 0.1$  mm, with a thickness of  $2\pm 0.2$  mm, and with an overall length of 50 mm
- Pure shear tests can be performed based on ISO 1827: sample dimension can be  $4\pm 1$  mm thick,  $20\pm 5$  mm wide, and  $25\pm 5$  mm long, bonded to each of two largest opposite faces
- For material cutting, fixed blades are preferable to moving knife techniques, so as to improve accuracy (see, for instance, the cutters described in ISO 23529)
- Mullin's effect is very crucial for MREs characterizations. Hence, a precondition of all samples is required for at least 3 cycles before actual tests
- Young's modulus or elastic modulus values must be reported using the tangent modulus (not using the secant or linear modulus) at a very low 0-2% strain. Furthermore, a plot of the tangent modulus over the whole range of strain explored would be of interest.
- MR effect should be obtained using a tangent modulus
- For the dynamic test, rheometry can be used. However, rheometer plate size and sample thickness must be well-defined and fixed for MRE society
- Yet again, the standard sample size must be determined for all dynamic tests including customized DMA and forced vibration testing. Even though Gordaninejad et al [130] reported that the mechanical properties of MREs are independent of sample thickness in the case of forced vibration testing, a thin sample must be considered for the dynamic test. A thin sample allows us to generate a uniform magnetic flux density within a small air gap

The rapid growth of MREs research strongly desires for a standard testing protocol. Hence, the MRE research community needs to develop standard testing protocols for MRE materials characterization and performance measurements sooner than later. For example, the standard testing methods developed for dielectric elastomers can be a good reference to start with [216], which was the outcome of an excellent teamwork among the experts in the dielectric elastomers. Moreover, existing modeling approaches must be unified as few models

predict stiffness enhancements of MREs under the application of a magnetic field while some of them assume the stiffness reduction under an applied field [45, 47, 75].

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## **Declaration of conflicting interests**

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

**Journal Pre-proof**

**Declaration of interests**

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.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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