



## Mechanical and Magnetic Properties of Porous Magnetorheological Nanocomposites (MRNCs)

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### ABSTRACT

This paper deals with the study on mechanical and magnetic properties of Magnetorheological Nanocomposites (MRNCs) based on room temperature vulcanized (RTV) silicone rubber and nano-sized carbonyl iron particles (CIPs). The five samples were prepared using ammonium bicarbonate ( $\text{NH}_4\text{HCO}_3$ ), CIPs, silicone oils and silicone rubber. All samples were manufactured under isotropic condition and their microstructures were characterized by x-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM). Porosity characteristics were measured and porosity image analysis was applied through ImageJ and Origin Pro Software. The mechanical tensile tests were conducted using Gotech tensile strength tester. The magnetic properties of porous MRNCs were practically determined using VSM test. Plateau stress induced by the applied magnetic fields and MR effects was determined. Upon successful fabrication of MRNCs, the samples' deflections were measured against applied magnetic fields. The findings show that porosity increases the flexibility and relative magnetic permeability of MRNCs and porous MRNCs will be the desired candidate for miniature and flexible actuators.

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### 1. Introduction

Magneto-Rheological Nanocomposites (MRNCs) are the new category of smart magnetorheological materials which are sensitive against applied magnetic fields. MRNCs consisting of nano-sized CIPs embedded in a silicone rubber matrix, display a variety of interesting properties to design the flexible actuators [1-4].

MRNCs have a good prospect for engineering applications. A very interesting application that will benefit from the specific characteristics of this smart composite is the miniature devices as well as grippers [5]. According to the literature and aiming the flexibility enhancement for MR family composites as

MR micro composites, MR elastomers and MRNCs, the different ideas have been applied and different research works have been conducted. Continuing the efforts, fabrication of porous MRNCs is targeted to reach to the flexible, lightweight and sensitive dynamic structures.

The authors are aware of only a few reports of MR composites containing nano-sized CIPs and Iron particles. In one instance, a brief report is discussing on MR nanocomposites with 1 wt. % of MWNTs in the silicone rubber and 20 vol. % of iron. In this work, the researcher characterized the dynamic mechanical behaviour of MRNCs through shear test, and notes that MRNCs exhibit higher zero-field stiffness and absolute MR effect [1]; while the other one points to the mechanical property

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improvement using micro and nano-sized CIPs and iron oxide powder without any clear data summary[2].

The Author's previous works[3-5] were focused to fabricate a light-weight and flexible nonporous MRNCs for miniature gripper applications using laser ablated nano powder of CIPs. The magnetic property values of manufactured MRNCs were determined lower than current reported work and the different fabrication technologies have been tested as hot press, chemical vacuum vaporization and laser beam moulding. Generally, in magnetorheological composites based on silicone rubber, the increase in particle percentages considering a constant magnetic field, causes the increase in deflection and magnetic permeability values [6]. The deflection of MR composite when exposing to a uniform magnetic field is dependent on the distribution of particles inside the composites and results from complex magnetic interaction between particles and mechanical interaction between particles and rubber-like matrix[7]. Besides that, The MR elastomer composites show the mechanical properties enhancement during the tensile test. Y. Wang et al [8] developed the MR elastomers based on immiscible silicon rubber via co-solvent method and noticed that MR elastomers had a higher tensile strength and lower elongation at break than that of MR elastomers based on pure silicone rubber.

In some MR elastomers composite category containing CIPs, RTV silicone rubber and silicone oil, the CIPs by itself has a measurable impact on dynamic mechanical property[9] whilst in MR nanocomposites based on MWNTs and liquid state silicone rubber, the higher magnetic field-induced increase the dynamic properties[10].

Recently, two research works reported on porous magnetorheological composites. In the first detailed work, the performance of porous magnetorheological elastomers were evaluated regarding stress-strain relationships and sensitivity against to external magnetic fields[11]. By exposing the porous MREs to the magnetic field, the shear storage moduli is obtained lower and the higher MR effect is achieved. The second study was focused on magnetostriction and Hall Effect of porous magnetorheological composites and concluded that magnetostriction and hall voltage show the nonlinear behaviours and hysteresis loops during the magnetic field applications [12].

It is known that porous polymer nanocomposites result in materials with good flexibility and lower

modulus. To get light-weighted MRNCs with proper deflection, better MR effect, higher flexibility and magnetic permeability, the porous MRNCs based on silicon rubber matrix is fabricated without applying magnetic fields. The MRNCs samples are provided in five different weight percentage categories as 10%, 20%, 30 %, 40 % and 50 % of nano-sized CIPs and characterized by FE-SEM and XRD.

The main contribution of current work is concerning to the brief study of porosity role, and pore interaction on operational behaviour of porous MRNCs through mechanical and magnetic properties measurements and analysis.

## 1. Experimental Procedure

### 2.1 Preparation of MRNCs

The necessary ingredients for the production of the MRNCs samples are laser ablated nano-sized CIPs (23-35 nm) of Russian Sintez CIP Ltd, silicone oil and ammonium bicarbonate of Sigma-Aldrich, and 704 RTV silicon rubber of Liyang Co. (China) with a porosity of 1.75%.

The MRNCs samples are film-shaped, 0.80 mm thick and 40 mm in diameter. Five samples are produced using vacuum oven. The fabrication process [11] of Porous MRNCs includes three steps as:

- 1) The  $\text{NH}_4\text{HCO}_3$  is immersed with silicone rubber and then mixed with the CIPs and stirred in a beaker for about 20 min at room temperature.
- 2) The mixture was placed in a vacuum oven to remove the air bubbles and then packed into an aluminium mould.
- 3) The mixture was cured at a temperature of  $100^\circ\text{C}$  for 3h without external magnetic field.

By curing the samples, the  $\text{NH}_4\text{HCO}_3$  could be decomposed into  $\text{NH}_3$ ,  $\text{CO}_2$  and  $\text{H}_2\text{O}$ ; and the porous MRNCs are formed. Figure 1 shows the prepared flexible porous MRNCs samples using vacuum oven. The composition of MRNCs samples are listed in table 1.

Table 1. Compositions of Porous MRNCs Samples (wt. %)

Samples	Carbonyl Iron	Silicone Rubber	Silicone oil	$\text{NH}_4\text{HCO}_3$
MRNCs 10 %	10	89.75	0.25	0
MRNCs 20%	20	78.50	0.5	1
MRNCs 30%	30	67.25	0.75	2
MRNCs 40%	40	56.00	1.00	3
MRNCs 50%	50	44.75	1.25	4



Fig.1: Fabricated film-shaped porous MRNCs sample (40%).

### 1.2. Structure and Morphology Characterizations

The structure of MRNCs samples were analysed by X-ray diffractometer (Xpert MPD). The patterns were run with Cu K $\alpha$  radiation at 35 kV and 25 mA. The MRNCs samples were cut into small pieces as 3 mm x 3 mm x 0.8 mm and Field Emission Scanning Electron Microscopy (FESEM) of cut surfaces was performed with an S-4160 electron micro-analyzers at an accelerating voltage of 15-20 kV depending on samples. Samples were gold-sputtered in order to visualize the dispersion clearly.

Through the observation of the microstructure, information about the CIPs and pore distribution in the silicone rubber matrix was obtained. Figures 2 show the microstructures of porous MRNCs

samples. The Porosity characteristics were measured by displacement method [13] and porosity image analysis was applied using Image J [14] and Origin Pro [15].

The FESEM micrographs shows that the CIPs are uniformly distributed in the silicone rubber. In addition, it can be seen from figure 2 that the ammonium bicarbonate content influence on the structure of the porous MRNCs samples. Through addition of ammonium bicarbonate to the samples, different size of pores form in the silicone rubber and the number of pores has an increasing trend with increasing  $\text{NH}_4\text{HCO}_3$  content.

### 1.2. Mechanical Property Measurement

Tensile tests were performed on a Gotech Tensile Strength Tester in according to the ASTM D638 and the elastic modulus along with elongation at breaks and tensile strength were obtained for five porous MRNCs samples. All the measurements were made at room temperature.

### 1.3. Magnetic Property Measurement

The magnetization of the samples was measured at room temperature using a Vibrating Sample Magnetometer (VSM; 9600-1 LDJ) in a maximum applied field of the saturation magnetization and relative permeability were determined.

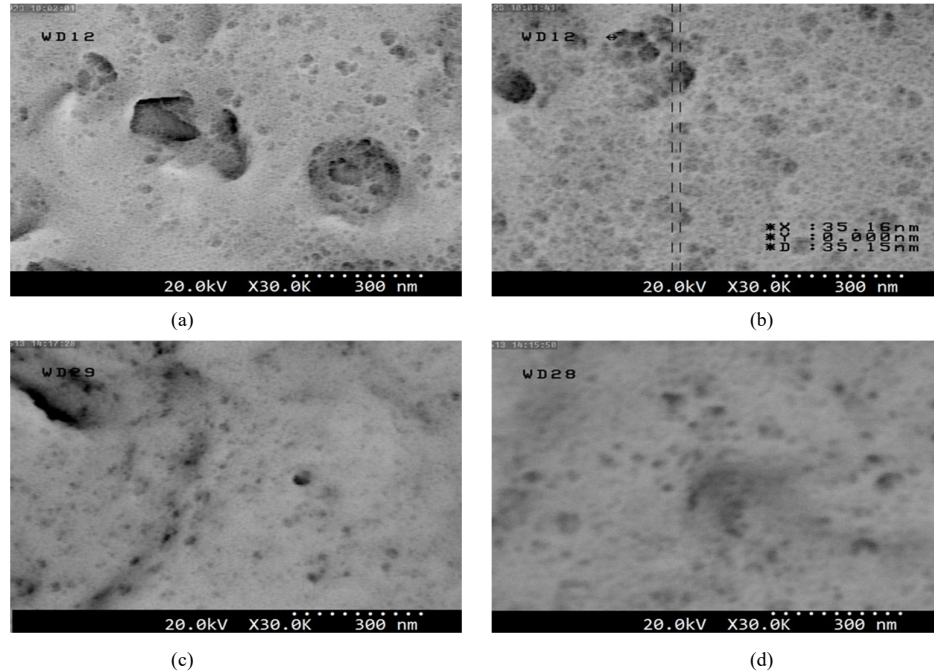


Fig. 2. FESEM images of porous MRNCs at different CIPs content (a) 20 %, (b) 30 %, (c) 40% and (d) 50%

## 2. Results and Discussion

### 3.1. Structure and Morphology

Figure 2 illustrates microstructures of porous MRNCs observed from FESEM. The FESEM images shows that the CIPs average size is about 35nm and are platelets. The FESEM images showed in figure 2 confirm that porous MRNCs with higher contents of CIPs has the lower degree of porosity. The Image analysis through Image J and Origin Pro software agree with the mentioned up issue regarding the CIPs content and degree of porosity (Table 2).

The X-ray spectrums for three MRNCs samples in the range of 0-70 degree are shown in Figure 3. As shown in X-ray spectrums, the characteristic XRD peaks of porous MRNCs was not appeared. In the XRD patterns of the MRNCs, the peak could be hardly detected, indicating the complete exfoliation and of the CIPs in the silicone rubber matrix.

It also noticed that pore size distribution is decreasing while the CIPs contents is increasing in MRNCs (Figure 4). Pore size distribution of MRNCs are confirmed to be mesoporous. As it shown in figure 4, the peak pore size of MRNCs centred between 60 to 85 nm, respectively.

Table 2. Comparison of MRNCs Samples Porosity (%)

Samples	OriginPr o	Image J	Disp. Method
MRNCs 10 %	23.02	23.97	24.25
MRNCs 20%	21.99	22.86	23.02
MRNCs 30%	20.99	21.93	22.14
MRNCs 40%	18.97	19.23	20.01
MRNCs 50%	16.92	17.13	18.08

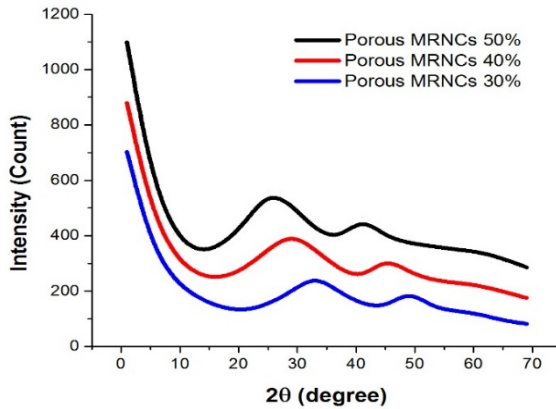


Fig.3. X-ray diffraction profile of porous MRNCs samples.

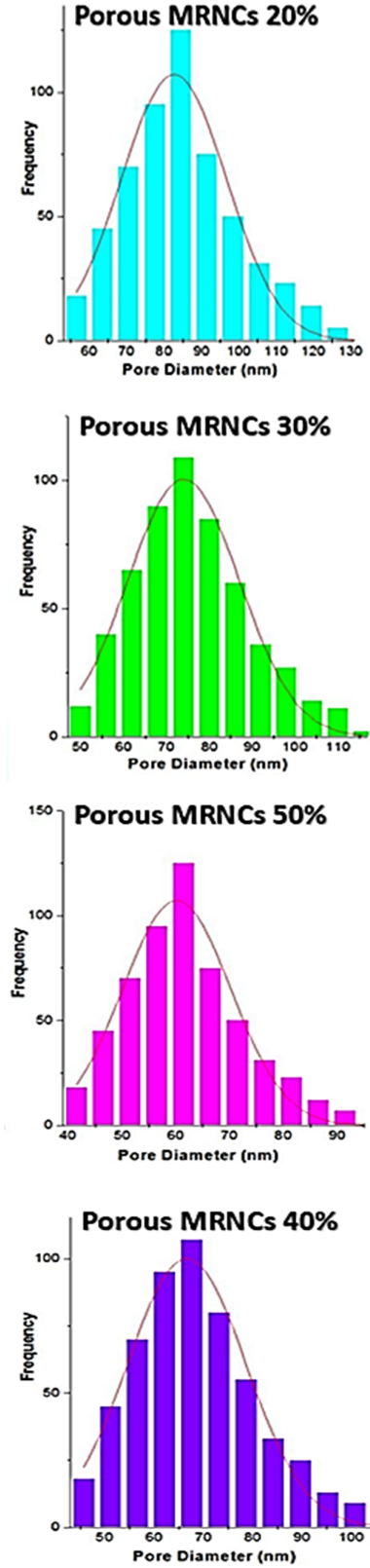


Fig. 4. Pore distribution of porous MRNCs samples using image analysis.

### 3.2 Mechanical property

The tensile stress-strain curve and tensile strength variations against CIPs loading are plotted in figures 5 and 6 respectively and elastic modulus derived for each set of samples and concluded in table 3. As expected result, the elastic modulus was decreased for porous MRNCs rather than nonporous MRNCs.

### 3.3 Magnetic property

The B-H curve obtained from VSM test is shown in figure 7. As the MRNCs are belonging to the soft family of magnetic materials (i.e. magnetorheological elastomers), the hysteresis has lowest rate. Saturation magnetization and relative permeability was determined and denoted in Table 4. Based on the observed data analysis from B-H curve, the porous MRNCs show the higher saturation magnetization and relative permeability rather than nonporous ones. The higher relative permeability of porous MRNCs is beneficial for enhancing pressure sensibility cycle of MRNCs during the piezoresistivity reaction mode. The higher relative permeability also causes a relatively rapid magnetic attraction which facilitates its sensitivity against applied magnetic field, handling desired deflection and contributes to an improvement of the effective and overall structure deformation for subsequent applications.

Table 3. Modulus of elasticity for porous MRNCs sample

Samples	E (GPa)
MRNCs 10 %	0.022
MRNCs 20%	0.033
MRNCs 30%	0.042
MRNCs 40%	0.053
MRNCs 50%	0.071

Table 4. Magnetic properties for MRNCs samples

Samples	Saturation (T)	Relative Permeability - $\mu_r$
MRNCs 10 %	0.81	2
MRNCs 20%	1.02	2.48
MRNCs 30%	1.45	3.59
MRNCs 40%	1.93	4.57
MRNCs 50%	2.38	5.58

Table 5. MR effects values for porous MRNCs samples

Samples	Absolute	Relative
MRNCs 10 %	0.034	2.16
MRNCs 20%	0.099	5.66
MRNCs 30%	0.318	15.9
MRNCs 40%	0.689	28.82
MRNCs 50%	1.309	43.12

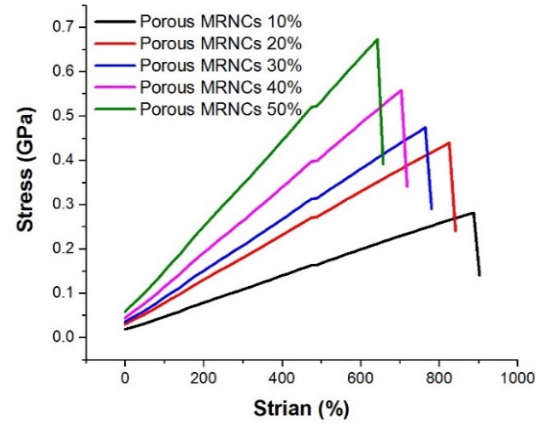


Fig. 5. Stress-Strain curves of porous MRNCs

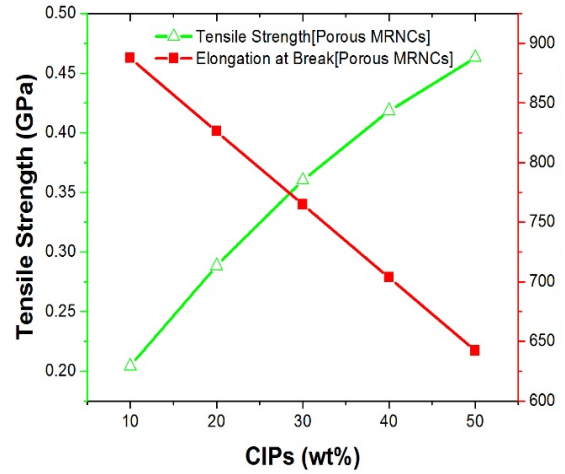


Fig. 6. Tensile Strength and Elongation at break changes vs. CIPs loading of porous MRNCs samples.

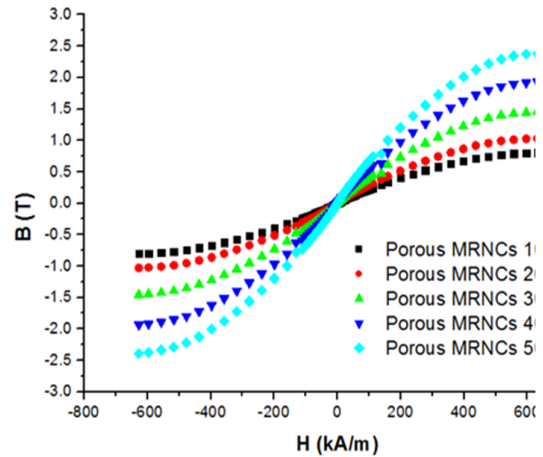


Fig7. B-H curves for porous MRNCs samples.



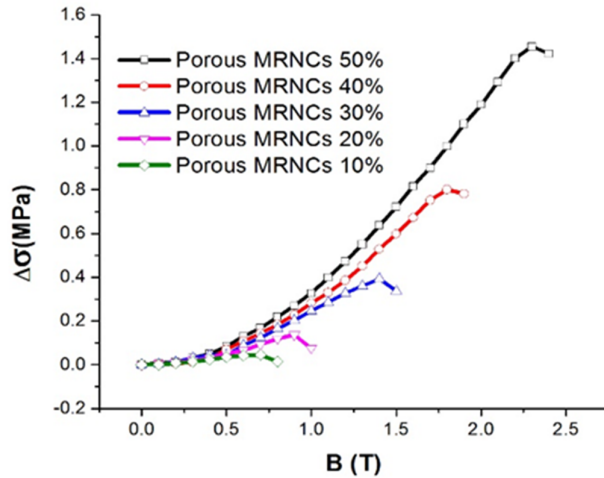


Fig. 8 Plateau stress vs induced magnetic field for porous MRNCs samples.

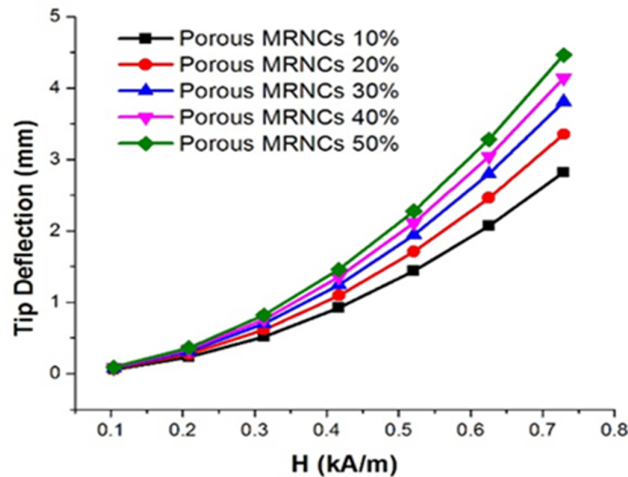


Fig. 9 Experimental measured deflection vs. applied magnetic field for porous MRNCs samples

#### 4. Conclusions

In this paper, porous MRNCs based on silicone rubber was fabricated without using magnetic field to reach to the target design considerations for light-weighted and magnetic-sensitive miniature actuator. A series of desired tests was conducted to study mechanical and magnetic properties porous MRNCs samples. The obtained data was compared with our previously work [3-5]. The lower values of modulus of elasticity in the mechanical property comparison points the flexibility for operational aspects and the higher achieved values of relative permeability in the magnetic property comparison stands for magnetic property

improvement as operational sensitivity and active magnetic responsive. It is observed that by increase in particles loading, the pore size distribution will be decreased and enhancement in particle loading will be disturbed the pore creation in matrix. The porous MRNCs needs more comparative investigations as electrical property and sensibility behaviour. Application of porous MRNCs are suggested for light-weight applications as micro and miniature actuators.

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