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Comparative Study on Mechanical and Magnetic Properties of Porous and Nonporous Film-shaped Magnetorheological Nanocomposites Based on Silicone Rubber

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Abstract— this paper presents a comparative study on mechanical and magnetic properties of two sets, each including five samples of film-shaped magnetorheological nanocomposites (MRNCs) based on RTV silicone rubber and nano-sized carbonyl iron particles (CIPs). One set of sample was prepared by polymerization of silicone rubber with CIPs and silicone oil, while the other set obtained by filling the ammonium bicarbonate (NH4HC3), CIPs and silicone oils. Both set of samples were manufactured under isotropic condition and their microstructures was characterized by XRD and EFSEM. Porosity characteristics was measured by displacement method and porosity image analysis was applied using ImageJ and Origin Pro Software. The mechanical tensile tests was conducted using Gotech tensile strength tester and the density of samples was observed experimentally and estimated theoretically. The magnetic properties of MRNCs were practically determined using VSM test. Plateau stress induced by the applied magnetics fields and MR effects was determined. Through fabrication of film-shaped MRNCs, the samples deflections was measured against applied magnetic fields .The comparative investigation results show that porosity improve the mechanical and magnetic properties of MRNCs and porous MRNCs will be the good candidate for miniature and flexible gripper's jaws.

Index Terms—Carbonyl Iron, MRNCs, Porosity, Silicone Rubber

I. 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 property enhancement for MR family composites as MR micro composites, MR elastomers and MRNCs, the different ideas have applied and different research works has conducted.

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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 noted that MRNCs exhibit higher zero-field stiffness and absolute MR effect [1]; While the another one points to the mechanical property 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, 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. It is known that porous polymer 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, two set of 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 comparative study between porous and nonporous MRNCs through mechanical and magnetic properties measurements and analysis.

II. EXPERIMENTAL

A. Preparation of MRNCs

The necessary ingredients for the production of the MRNCs samples are 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).

The MRNCs samples are film-shaped, 0.80 mm thick and 40 mm in diameter. Two samples of the same composition are produced: Nonporous MRNCs using hot press, Porous MRNCs using vacuum oven.

The fabrication process of Nonporous MRNCs includes five steps:
(1) Dispersion of nano-sized CIPs in the silicone oil, (2) Dispersion of CIPs-silicone oil solution in the liquid-state silicone rubber, (3) Mixing by ultrasonic mixer, (4) Curing of the mixture under isotropic condition,(5) Moulding the mixture using hot press.

The fabrication process of Porous MRNCs includes three steps as reported in [ref.11]:
(1) The NH_4HCO_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◦C for 3h without external magnetic field.

By curing the samples, the NH_4HCO_3 could be decomposed into NH_3, CO_2 and H_2O; and the porous MRNCs are formed.

The composition of MRNCs samples are listed in table I and table II.

Table I Compositions of Nonporous MRNCs Samples (wt. %)

Sample	Carbonyl Iron	Silicone Rubber	Silicone Oil	NH4HCO4
MRNCs 10%	10	89.75	0.25	0
MRNCs 20%	20	78.50	0.50	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

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

Sample	Carbonyl Iron	Silicone Rubber	Silicone Oil
MRNCs 10 %	10	89.75	0.25
MRNCs 20 %	20	79.50	0.50
MRNCs 30 %	30	69.25	0.75
MRNCs 40 %	40	59.00	1.00
MRNCs 50 %	50	48.75	1.25

Figure 1 shows the prepared flexible porous MRNCs samples using vacuum oven.

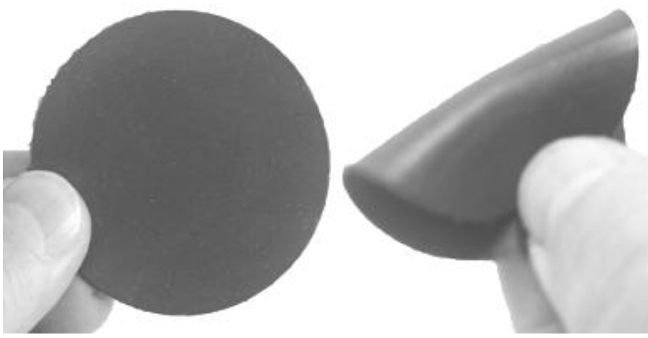

Fig.1 Fabricated film-shaped porous MRNCs sample (40%)

B. Structure and Morphology Characterization

The structure of MRNCs samples were analyzed by X-ray diffractometer (Xpert MPD). The patterns were run with Cu Kα 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 and 3 show the microstructures of porous and nonporous MRNCs samples. The Porosity characteristics were measured by displacement method [12] and porosity image analysis was applied using Image J [13] and Origin Pro [14]. The FESEM micrographs shows that the CIPs are homogeneously distributed in the silicone rubber. In addition, it can be seen from figure 3 that the ammonium bicarbonate content influence

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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 NH4HCO3 content.

C. 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.

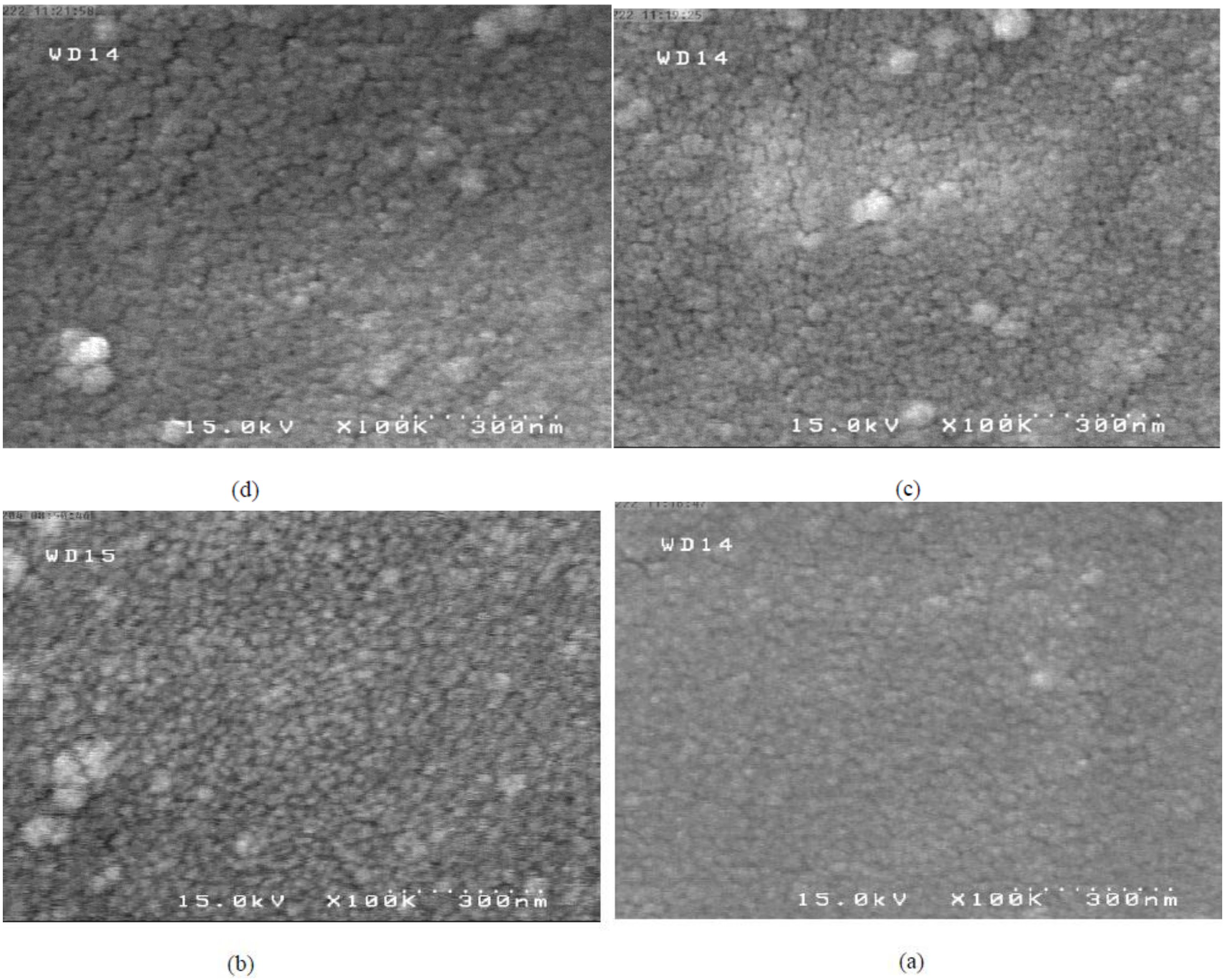
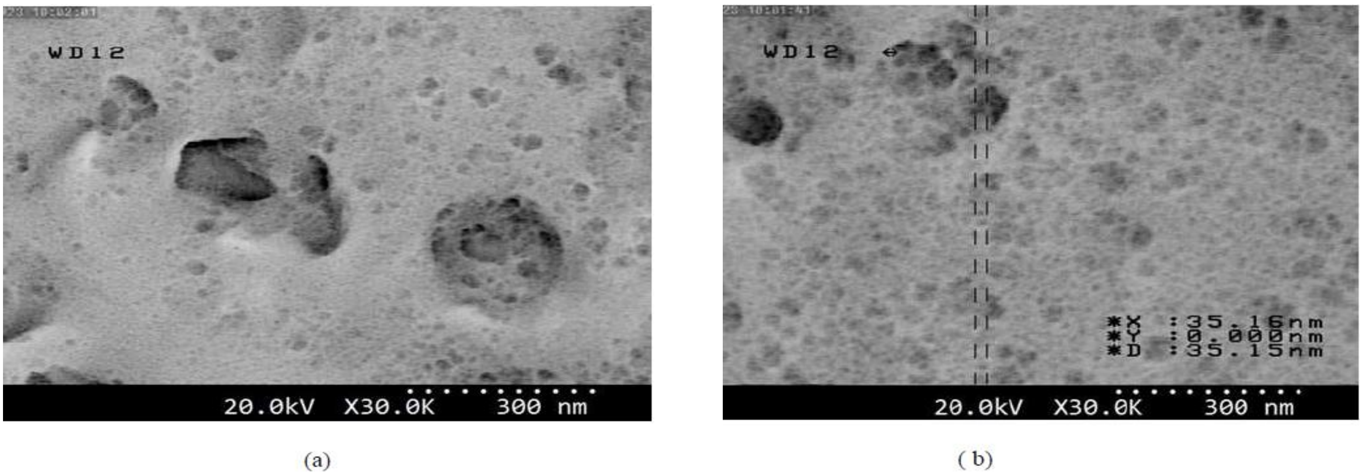
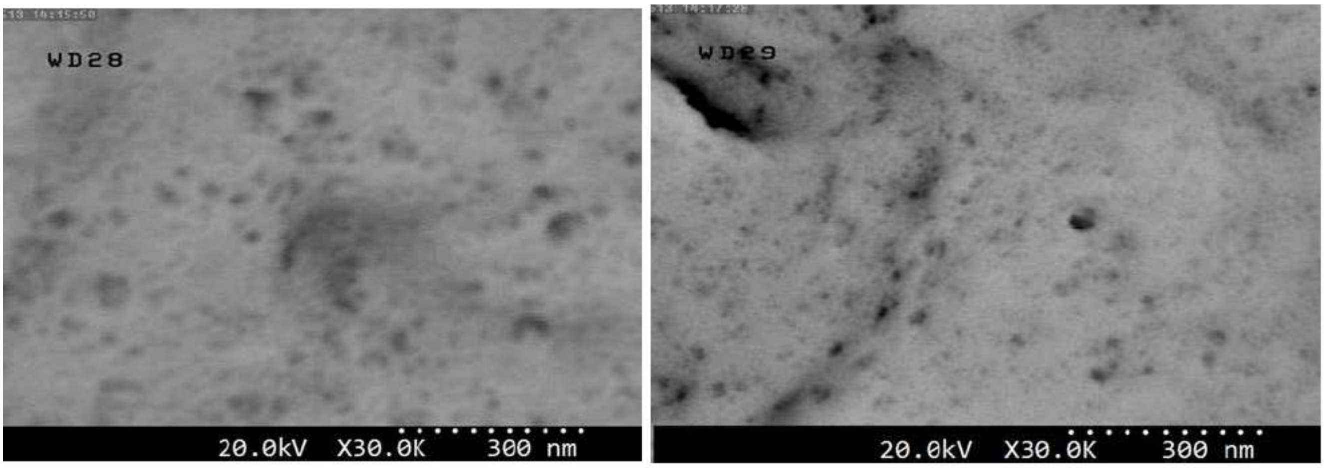


Fig. 2 FESEM images of nonporous MRNCs (a) 20 %, (b) 30 %, (c) 40% and (d) 50%





(d) (c)

Fig.3 FESEM images of porous MRNCs (a) 20 %, (b) 30 %, (c) 40% and (d) 50%

Five MRNCs samples were tested form both set.

The density of MRNCs samples were determined theoretical through ROI [15] and practical measured using Archimedes' principle [16].

All the measurements were made at room temperature.

D. 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 250 kA/m. From the obtained B-H curve, the saturation magnetization and relative permeability were determined.

The developed MRNCs samples with a size of 40mm x 4 mm x 0.8 mm was placed near to the magnetic field which varied from 0 to 1 kA/m and its displacement observed and recorded through AEC-5509 Gap sensor. The test setup reported in ref.17.

The variation of the plateau stress induced by the applied magnetic field was estimated for the MRNCs samples in according to the relation [18] as

$$\Delta\sigma = \frac{1}{2\mu_0}B^2 - \frac{1}{2\mu_{MRE}}B^2 \quad (1)$$

Where μ_0 and μ_{MRE} are the permeability of vacuum and MRNCs and B the applied magnetic field. the absolute and relative MR effect was calculated using shear modulus achieved in magnetic field and zero field modulus [11, 19]. The absolute and relative MR effect can be expressed as

$$G_{abs} = G_{max} - G_0 \quad (2)$$

$$G_r = \frac{G_{abs}}{G_0} \quad (3)$$

Where Gabs is the absolute MR effect in MPa, Gmax is the maximum shear modulus achieved in a magnetic field, G0 is the initial shear modulus (zero field modulus) and Gr is the relative MR effect.

III. RESULTS

A. Structure and Morphology Characterization

Figure 2 illustrates microstructures and homogeneous distribution of CIPs in the nonporous MRNCs observed from FESEM. The CIPs are uniformly distributed with no big clusters. FESEM images shows that the CIPs average size is about 35nm and are platelets.

The FESEM images showed in Fig. 3 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 prove the mentioned up issues (Table III).

The X-ray spectrums for MRNCs samples were recorded in the range of 0-70 degree, which is shown in Figure 4 for MRNCs 30%, 40% and 50 %. As shown in X-ray spectrums, the characteristic XRD peaks of porous MRNCs was not appeared. In the XRD patterns of the MRNCs (including porous and nonporous), the peak could be hardly detected, indicating the complete exfoliation and uniform dispersion 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 5).the poresize distribution are confirmed to be mesoporous. The peak pore size of MRNCs centered between 60 to 85 nm, respectively.

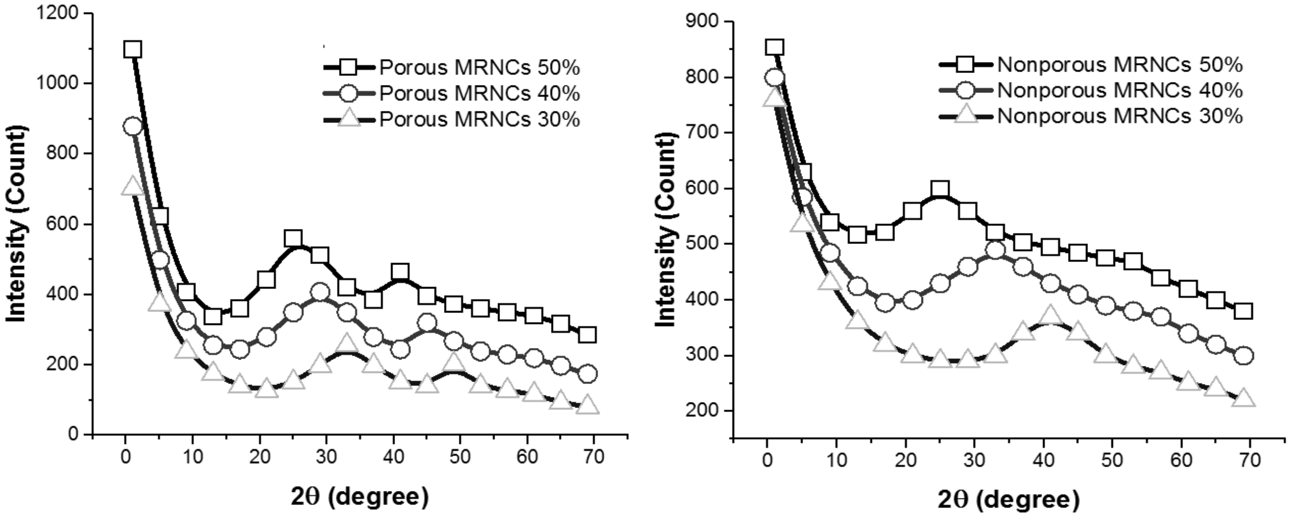


Fig. 4 X-ray diffraction profile of porous and nonporous MRNCs samples

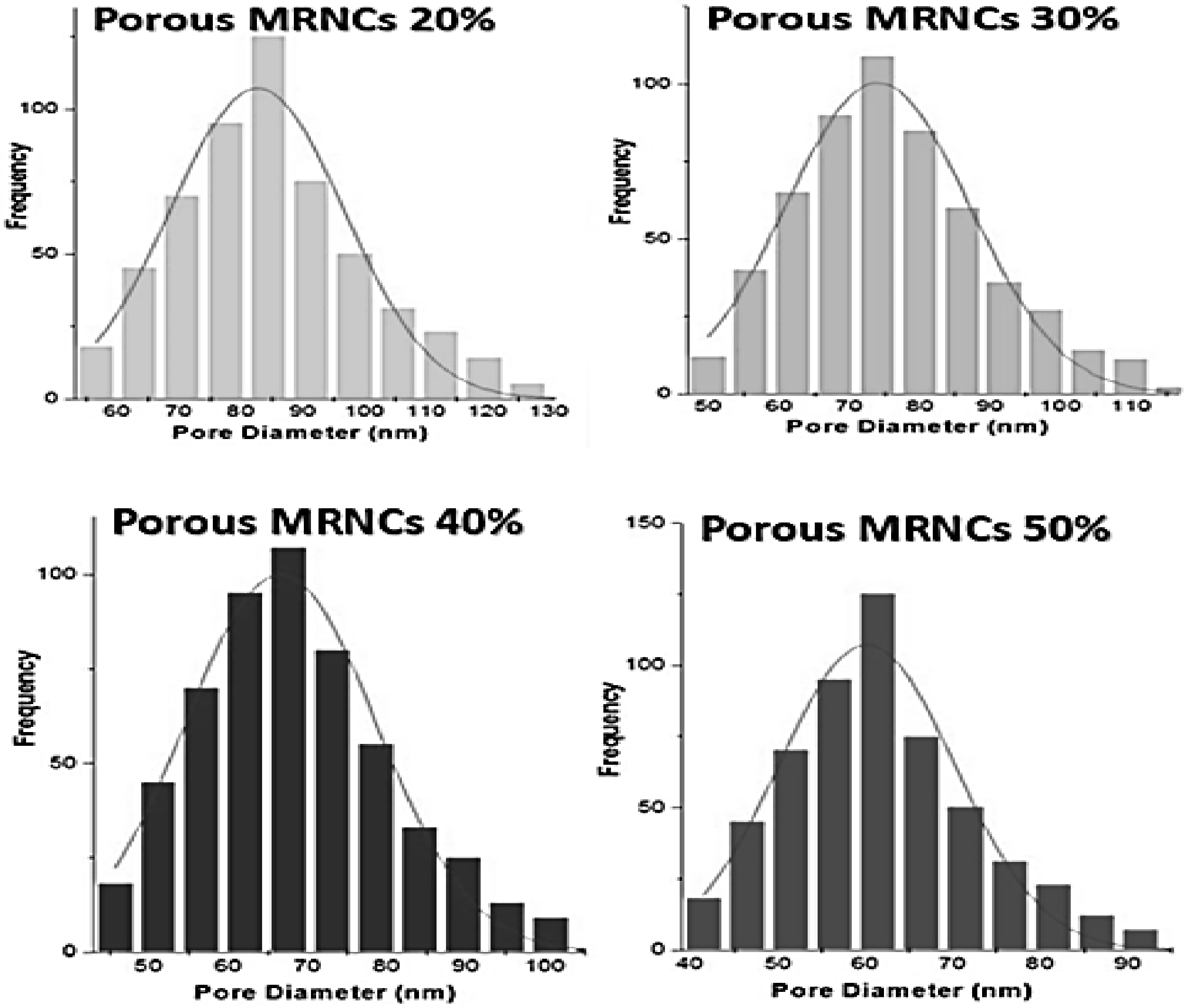


Fig. 5 Pore distribution of porous MRNCs samples using image analysis

Table III Comparison of MRNCs Samples Porosity (%)

Samples	Porosity (%)		
	Origin Pro	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

Table IV Comparison of MRNCs modulus of elasticity

Samples	Young's Modulus (GPa)	
	Non-Porous	Porous
MRNCs 10%	0.06022985	0.02204587
MRNCs 20%	0.090344776	0.03287985
MRNCs 30%	0.120459701	0.04218293
MRNCs 40%	0.150574626	0.05353998
MRNCs 50%	0.180689551	0.07011847

Type	Sample	Satuartion (T)	Relative Permeability-μ_r
Non-Porous	MRNCs 10%	0.72	1.88
	MRNCS 20%	0.84	2.1
	MRNCs 30%	1.34	3.43
	MRNCS 40%	1.68	4.23
	MRNCs 50%	2.18	5.26
Porous	MRNCs 10%	0.8	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

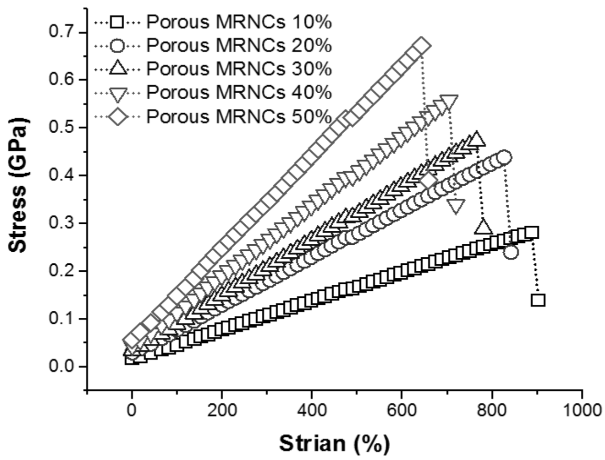
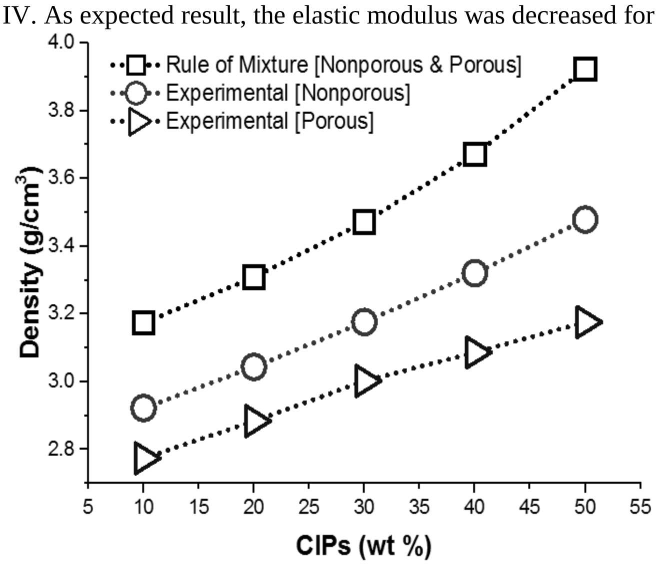


Table V Comparison of magnetic properties for MRNCs samples

B. Mechanical Property Measurement

The density measurements of porous and nonporous MRNCs shows that porous MRNCs has the lower density. The comparison between theoretical and experimental measurements presented in figure 6.

The Tensile tests results plotted in figures 7and 8 respectively and elastic modulus derived for each set of samples and concluded in table IV. As expected result, the elastic modulus was decreased for porous MRNCs rather than nonporous MRNCs.



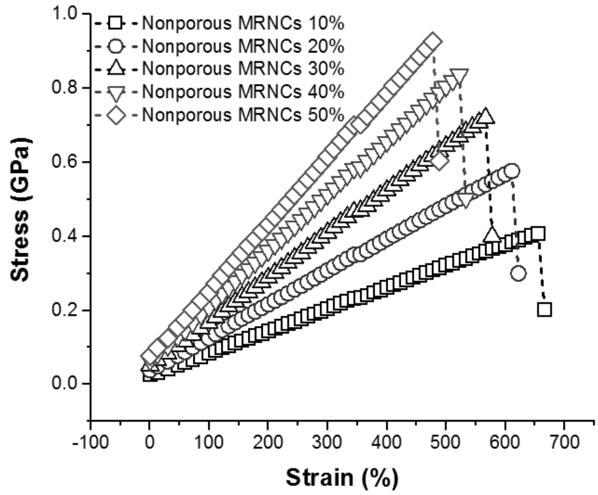


Fig. 6 Density changes CIPs loading in MRNCs

C. Magnetic Property Measurement

The B-H curve obtained from VSM test (Fig.9). As the MRNCs are belonging to the soft family of magnetic materials, the hysteresis has lowest rate. Saturation magnetization and relative permeability was determined and denoted in Table V. the porous MRNCs show the higher saturation magnetization and relative permeability. The estimation results of plateau stress induced by the applied magnetics fields (depicted in figure 10) shows that the plateau stress has a higher values for porous MRNCs. The calculated MR effect values summarized in table VI. An enhancement is remarked in the reported values for porous MRNCs samples.The MRNCs samples deflections against magnetic field noted and graphed in figure 11. The graphed data show that porous MRNCs are more sensitive against applied magnetic fields

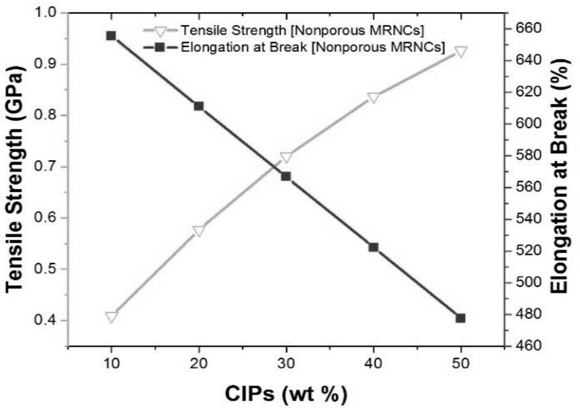


Fig. 7 Stress-Strain curves of porous and nonporous MRNCs samples

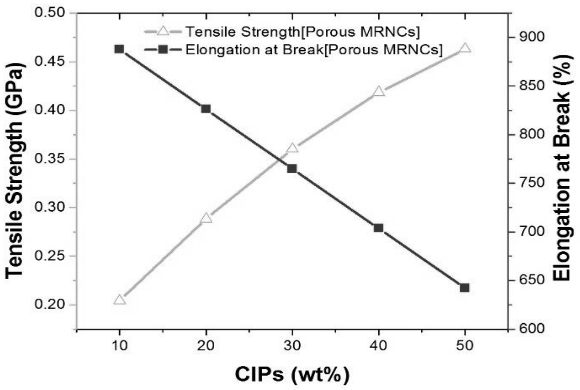


Fig.8 Tensile Strength and Elongation at break changes vs. CIPs loading of nonporous and porous MRNCs samples

Table VI Comparison of MR effects for MRNCs samples

Samples	Nonporous		Porous	
	Absulute MR Effect	Relative MR Effect	Absulute MR Effect	Relative MR Effect
MRNCs 10%	0.033072	2.064481	0.034707	2.166547
MRNCs 20%	0.063202	3.594081	0.099637	5.666009
MRNCs 30%	0.25087	12.52809	0.318497	15.90529
MRNCs 40%	0.57012982	23.834859	0.689515	28.8258793
MRNCs 50%	1.07398589	35.36461	1.3095299	43.1206922

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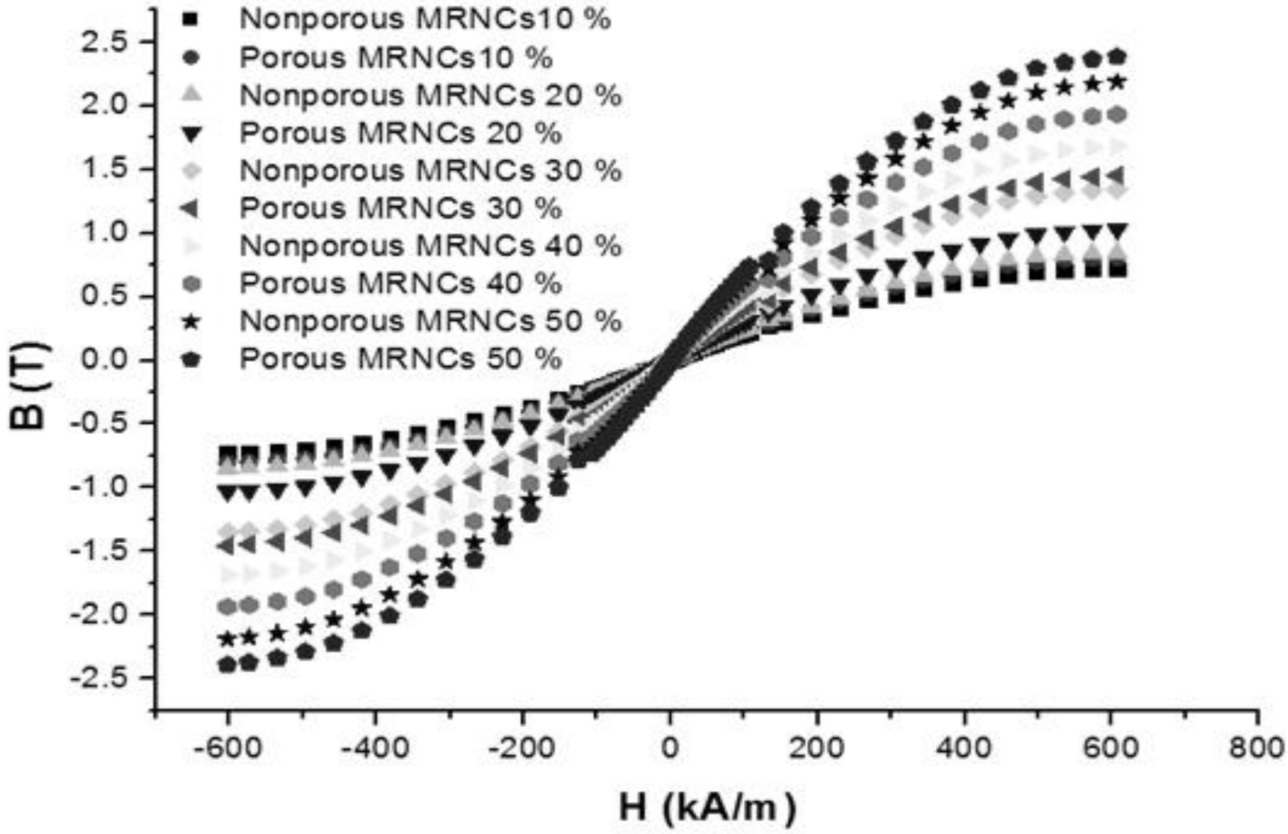


Fig.9 Comparative B-H curves for MRNCs samples

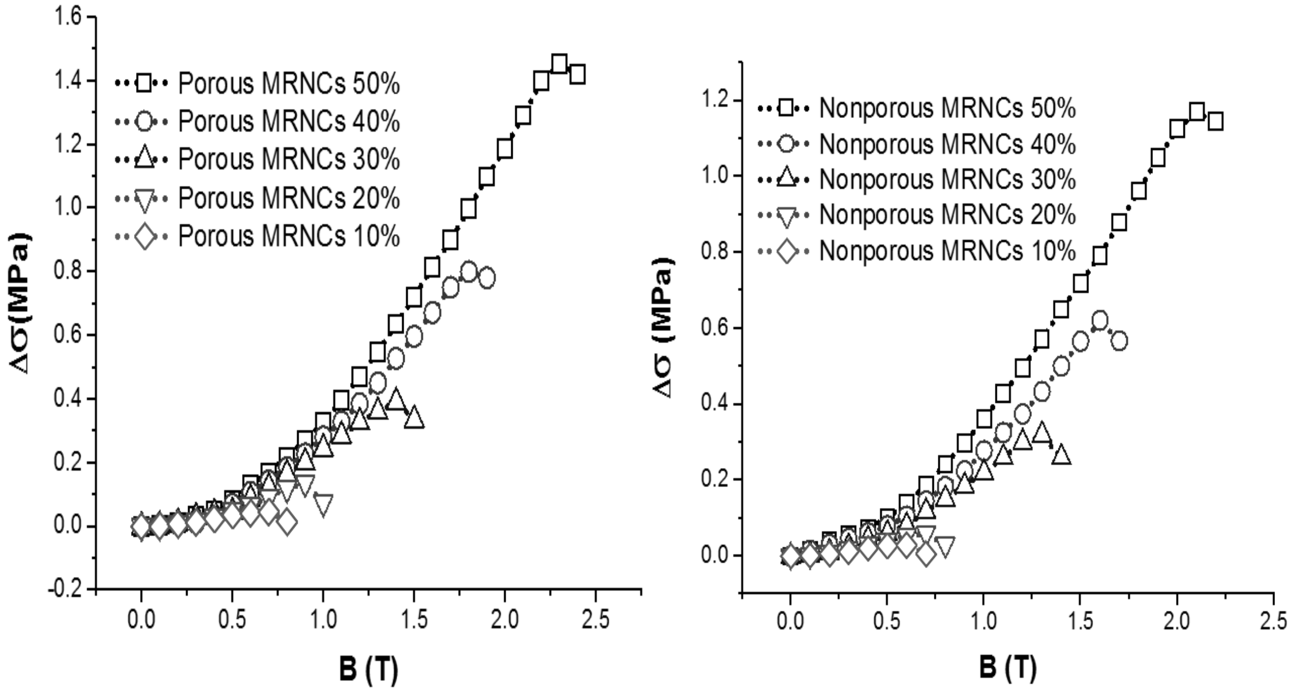
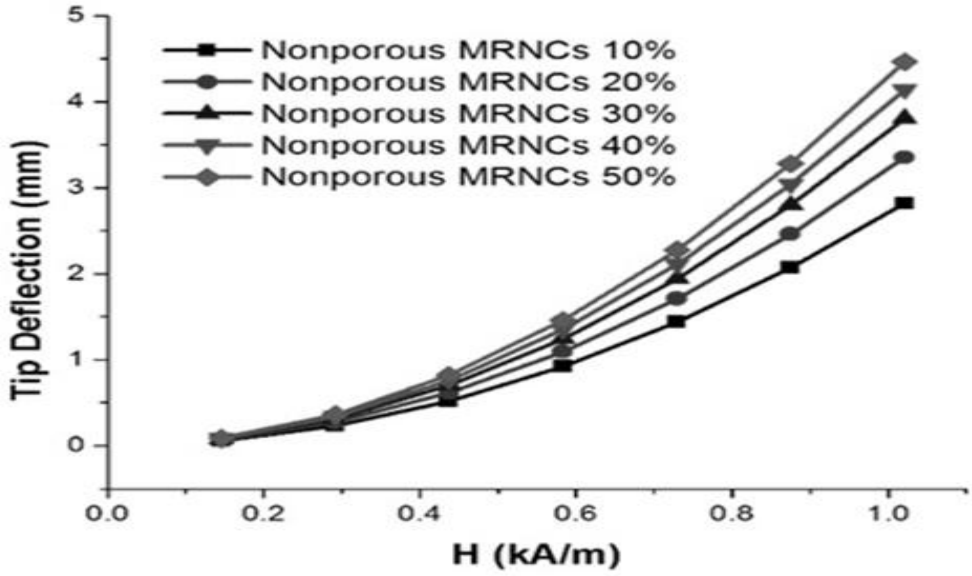


Fig. 10 Plateau stress vs induced magnetic field for nonporous and porous MRNCs samples



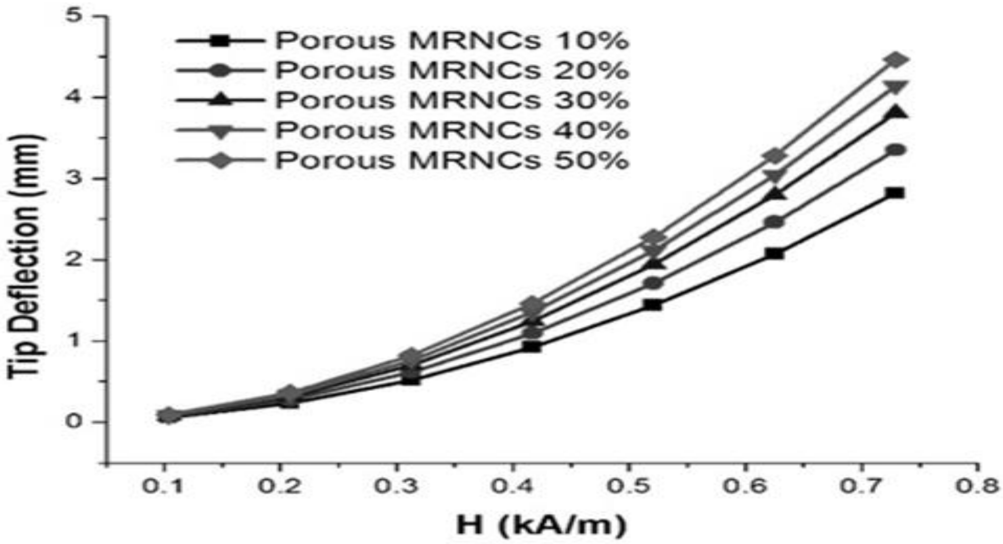


Fig. 11 Experimental measured deflection vs. applied magnetic field for nonporous and porous MRNCs samples

IV. CONCLUSIONS

In this paper, two set of MRNCs based on silicone rubber were fabricated without using magnetic field to reach to the target design considerations for light-weighted and magnetic-sensitive miniature gripper jaws. A series of desired tests was conducted to study and compare the enhancement of mechanical and magnetic properties between nonporous and porous MRNCs samples. 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 behaviors. Application of porous MRNCs are suggested for light-weight applications as micro and miniature actuators.

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