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The aim of this work is to fabricate and investigate nickel nanowires (NiNWs) as a novel magnetorheological material and determine how the aspect ratio of these magnetic particles influences its magnetic properties. The latest methods for synthesizing NiNWs and nickel nanospheres (NiNSs) are presented and the corresponding magnetorheological fluids (MRF) are obtained. Materials were characterized so that the properties of NiNWs could be compared to NiNSs. As different size NiNWs were fabricated, their saturation magnetization values increased as the size increased. Moreover, MRF containing NiNWs processed shear stress 15 times as strong as the one with the same volume of NiNSs, although the saturation magnetization of NiNWs was smaller than NiNSs. MRF containing magnetic particles with more saturation magnetization and smaller coercivity usually has a stronger MR effect. Our result is interesting, and further finite element simulations were utilized to analyze the possible mechanisms. The simulation indicated that the large aspect ratio of NiNWs helped to align the particles into columns and also caused the magnetized direction of particles to deviate from the direction of the applied field, thus restoring the torque and achieving a large shear stress. Furthermore, MRF with higher fraction of NiNWs has a more stable suspension, and NiNWs disperse much better than NiNSs with the same volume.

Disciplines

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A Novel Nickel Nanowire based Magnetorheological Material

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Abstract

The aim of this work is to fabricate and investigate nickel nanowires (NiNWs) as a novel magnetorheological material and determine how the aspect ratio of these magnetic particles influences its magnetic properties. The latest methods for synthesizing NiNWs and nickel nanospheres (NiNSs) are presented and the corresponding magnetorheological fluids (MRF) are obtained. Materials were characterised so that the properties of NiNWs could be compared to NiNSs. As different size NiNWs were fabricated, their saturation magnetisation values increased as the size increased. Moreover, MRF containing NiNWs processed shear stress 15 times as strong as the one with the same volume of NiNSs, although the saturation magnetization of NiNWs was smaller than NiNSs. MRF containing magnetic particles with more saturation magnetization and smaller coercivity usually has a stronger MR effect. Our result is interesting, and further finite element simulations were utilized to analyze the possible mechanisms. The simulation indicated that the large aspect ratio of NiNWs helped to align the particles into columns and also caused the magnetised direction of particles to deviate from the direction of the applied field, thus restoring the torque and achieving a large shear stress. Furthermore, MRF with higher fraction of NiNWs has a more stable suspension, and NiNWs disperse much better than NiNSs with the same volume.

Keywords: nanowires; nickel; magnetorheological; MRF; enhanced MR effect

1. Introduction

Magnetorheological fluid (MRF) is a type of smart material, it was discovered in 1948 and until [1], has undergone significant amount of development. The physical properties of MRF such as shear stress can be controlled continually by an external magnetic field such that the increased shear stress can reach several orders of magnitude [2]. Under an external magnetic field this material has a fast and reversible transition from a liquid phase to a solid-like state, a process known as the "magnetorheological (MR) effect". [3] This behaviour allows MRF to have many potential applications in mechanical systems such as transportation, safety engineering, civil engineering, and one of its most famous applications, the MRF damper for controlling the structural response. [4-6] Conventional MRFs commonly consist of ferromagnetic spherical particles and a non-magnetic and low viscosity medium. Besides the sphere-based MRFs, magnetic particles with other shapes have been fabricated while developing MRF because the morphology and particle size play important roles in the properties of MRFs [7]. Furthermore, magnetic materials other than carbonyl iron have also been exploited; scientists have developed magnetic particles with a wide range of shapes, such as cobalt fibres [8], FeSiB wires [9], iron rods [10], iron plates and flakes [7, 11, 12], in an effort to improve the stability of MRF. Within these researches, one dimensional (1D) nano-sized magnetic particles, especially nanofibers, have attracted a great deal of attention because of their ability to enhance the yield stress and sedimentation [13]. MRF with good dispersion stability has attracted a lot of attention because additives which increase stability usually decrease the shear strength of MRF. [14, 15] It would be good to find stable MR suspensions which do not need additives to exploit a stronger yield stress. MRF with 1D nano-sized magnetic particles are reported to have this desired stability, so it would be worthwhile trying to develop more of these types of magnetic particles. . de Vicente *et. al.* [16] synthesized out magnetite nanorods, Antonel *et. al.* [17] obtained $CoFe_2O_4$ nanowires, and Nagtu and Bell [13, 18] generated iron nanowires, etc. MRF based on iron nanowires were fabricated and compared with MRF based on spheres [13], and a comparison between cobalt nanowire microrod and microspheres has also been reported [19]. However, the challenge is wire is much wider than spheres, and this difference between width and diameter creates more interference in simulation. Furthermore, templates and Teflon-lined autoclave are commonly used when fabricating these one-dimensional magnetic particles, and they would limit the production of more magnetorheological materials due to the high cost and arduous conditions. To overcome these gaps, we have been looking for a facile and simple approach to synthesize large amounts of NiNWs to better study 1D MR materials. Although a facile method for synthesizing large amounts of nickel nanospheres (NSs) has been developed for a more precise comparison to NiNWs, the NiNSs obtained are almost 200 nm, which is much larger than the particle for ferrofluid defined as 5-15 nm [20], so the samples containing NiNWs or NiNSs should be considered as MRFs. In the preliminary work, NiNWs and NiNSs with an average diameter that is similar in size to the mean width of NiNWs have also been obtained, and the particles characterised. The comparisons of corresponding MRF samples were also characterised to provide more information about how their shape affects the MR effect. Simulations were utilised to confirm that the geometric effect of NiNWs' extreme aspect ratio caused their enhanced MR effect.

2. Experimental details

2.1 Materials

Nickel (II) chloride hexahydrate ($NiCl_2 \cdot 6H_2O$; 99.9%) and ethylene glycol (EG; 99.8%), hydrazine monohydrate ($N_2H_4 \cdot H_2O$; 98%) were purchased from Sigma-Aldrich, and silicone oil (viscosity 10 mm^2s^{-1}) was purchase from Beijing Sihuan-Antong. Ultrapure deionised water (DI; millipore water systems) with a resistivity 18.2 $M\Omega cm^{-1}$ was used throughout the work. All the chemicals were used as received, without any further purification.

2.2 Preparation of the MRF

The method for synthesizing NiNWs was developed from a previous work [21] where, in a typical process, 1 mL 1M $NiCl_2$ aqueous solution is added into 200 mL EG, and then heated to 100 ^oC. This solution is mixed with 2 mL $N_2H_4 \cdot H_2O$ and kept at 100 ^oC for around 30 min until a dark gray product eventually floated on the surface. The NiNWs are washed with water by magnetic decantation, and dried with a freeze dry system (FreeZone 12, Labconco, United States). Other size NiNWs were also synthesized at a reaction temperature of 70 ^oC for 2 hr and 140 ^oC for 20 min, respectively.

Ni nanospheres were obtained by a new approach deferred from NiNWs. 3 mL 1M $NiCl_2$ aqueous solution was added into 200 mL EG, which was then heated to 100 ^oC. This solution is mixed with 6 mL $N_2H_4 \cdot H_2O$, stirred at 1200 rpm and heated to 100 °C for around 100 min. The washing and drying process is the same as NiNWs.

The MRF samples are prepared by dispersing NiNWs or nickel nanospheres into silicone oil.

2.3 Characterizations

SEM images of NiNWs and NiNSs are obtained with JEOL JEM6390 SEM. The average length and width of NiNWs were calculated based on 50 individual nanowires, and it equalled the average diameter of NINSs. To investigate the behaviour of NiNWs, some NiNWs were dried on a silica substrate under weak magnetic field, while others were dried without it. VSM measurements of magnetic particles were carried out by LakeShore 7300 VSM at room temperature and with a maximum magnetic field of 10 kOe. An Anton Paar MCR301 magnetorheometer was used to characterize the MRF. The sedimentation test was carried out by mixing the MRF samples and then keeping them steady near a ruler for observation.

2.4 Simulations

The nearest neighbour interactions of NiNWs and NiNSs were modelled using COMSOL Multiphysics 4.3, a commercial finite element method software package, and the physics module of Magnetic Fields, No Currents was utilized, in which the magnetostatic set of equations

$$\mathbf{H} = -\nabla V_m$$
$$\nabla \cdot \mathbf{B} = 0$$
$$\mathbf{B} = \mu_0(\mathbf{M} + \mathbf{H})$$

were solved numerically. In the simulations, the NiNWs and NiNSs were the average sizes obtained from the measurements. Periodic boundary conditions were applied in the x and y directions in the simulations.

3. Results and discussions

The different conformations of NiNWs with and without a magnetic field were investigated with SEM. (**Figure 1**) When there is no external field, the NiNWs tended to be randomly dispersed in silicone oil and formed a network-like structure, but once a weak magnetic field is applied the NiNWs preferred to align along the direction of the field. However, the NiNWs were not completely aligned along the direction with the field due to the solid frictions between NiNWs; this interaction benefits the MR property of NiNWs because interfibre friction could also contribute to the yield stress [22].

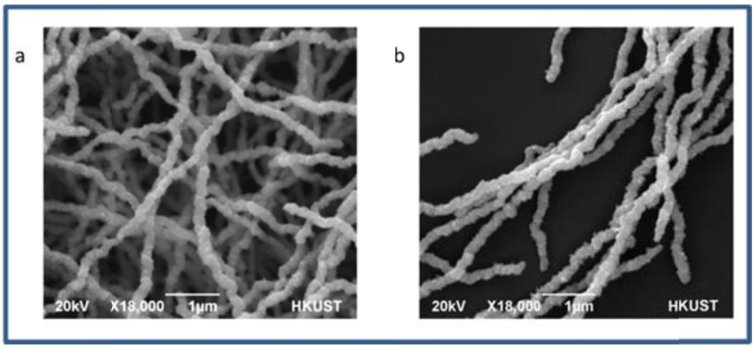

Figure 1. The SEM images of NiNWs (a) without and (b) under a magnetic field.

The SEM images indicate that the average NiNWs was 14.80 ± 3.65 μm long by 190 ± 28 nm wide, which provided a notable and extreme aspect ratio of 78 ± 22. This narrow distribution of size is a rare occurrence in the present MR materials, so it guaranteed a new kind of MRF whose properties could be controlled in a more elaborate range by changing the size of the magnetic particles. At the same time the size and morphology of NiNWs can be controlled by adjusting the reaction temperature (**Figure 2. a-d**); a more detailed discussion of the reaction conditions has been presented in our previous work [21]. A lower reaction temperature generally leads to larger NiNWs (**Figure 2.b**), while a higher reaction temperature leads to a smaller size (**Figure 2.c**). To fabricate the NiNS (**Figure 2.d**) as a comparison to NiNWs means that stirring is required. The NiNSs have an average diameter of 201 ± 45 nm, which is very close to the width of NiNWs. This notable result has never been published, and this method of chemical synthesis could provide NSs with diameters close to the width of NWs.

The magnetic particles needed for conventional MRF should have a large saturation magnetization (M_s) and small coercivity (H_c) to perform efficiently, so the magnetic properties of NiNWs and NiNSs were characterised to investigate the relationship between their morphology and the magnetic properties. (**Figure 2. e, f**) Larger NiNWs should provide larger M_s and H_c that are smaller than the smaller ones, but the larger size led to worse performing suspension. However, larger NiNWs also had larger aspect ratio so it was hard to confirm whether the differences in the M_s and H_c values were due to the changed aspect ratio, so NSs with similar diameter to NiNWs were utilised. According to the hysteretic loop, NSs had much larger M_s and smaller H_c compared to NiNWs, so it could be concluded that the aspect ratio played an important role in the magnetic property, and larger aspect ratio could create magnetic properties which which are unfavourable for MRF. However, the corresponding MRF behaviour was difficult to predict based only on the magnetic information because extra factors also influenced the MR effect.

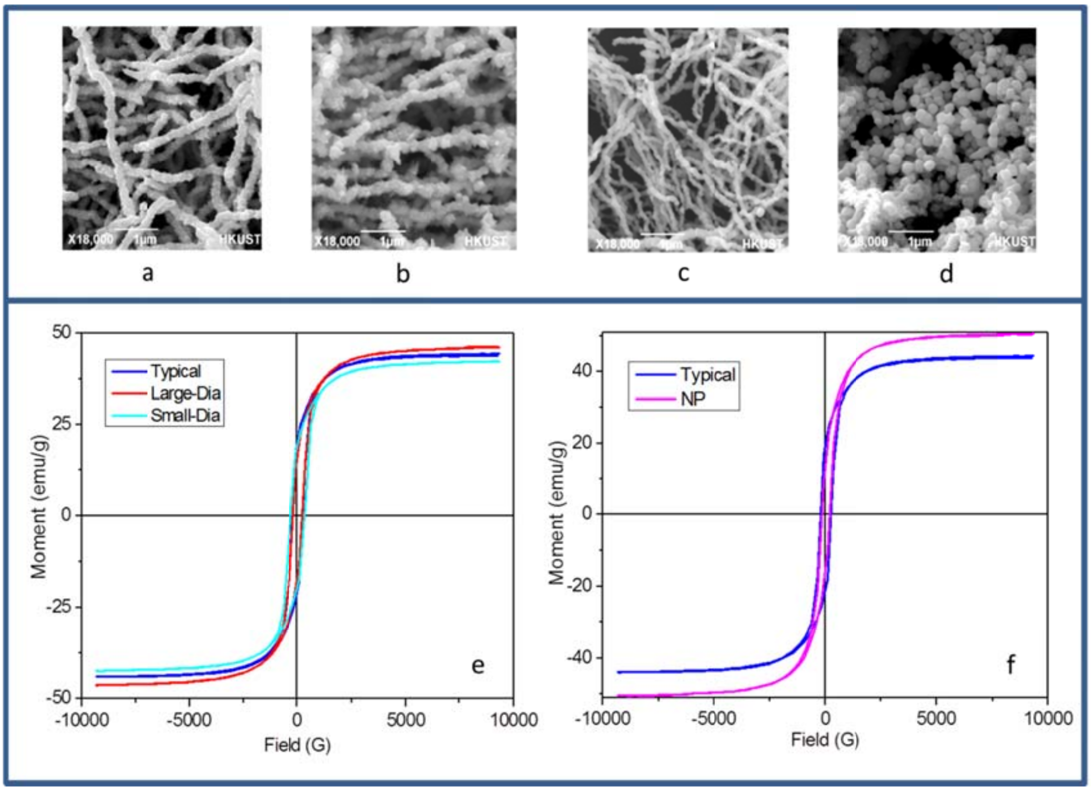

Figure 2. SEM images with the same magnification of (a) typical synthesized NiNWs, (b) NiNWs synthesized at 70 °C, (c) NiNWs synthesized at 140 °C, and (d) NiNSs. Hysteretic loops at room temperature of (e) typical NiNWs compared to other size NiNWs and (f) NiNWs compared to NiNSs.

To investigate the property of NiNWs as a composite of MRF, 5 MRF samples were prepared for characterization; 4 contained 1.5 vol%, 3 vol%, 4.5 vol%, and 9 vol% NiNWs, and one had 3 vol% NSs. They are labelled NiNW-MRF and NiNS-MRF according to their particle species, respectively. The viscosity and shear stress of the samples was measured with a magnetic field strength increasing linearly from 0 to 660 mT at a constant and almost steady shear rate of 0.01 s^{-1}. (**Figure 3.a**) For 1.5 vol%, 3 vol%, 4.5 vol%, and 9 vol% NiNW-MRF samples, the corresponding values of off-state shear stress were 9.97, 25.1, 29.7, and 56.7 Pa. In a 660 mT of magnetic field, the 1.5 vol%, 3 vol%, 4.5 vol%, and 9 vol% NiNW-MRF samples respectively had shear stress values of 39.1, 139, 193, and 400 Pa. The result indicates that the off-state viscosity would increase significantly e as the percentage of

NiNWs increases, which should be considered a drawback of particle loading, but as the volume of NiNWs increases, the shear stress under field also increased significantly; this means that as more NiNWs are loaded, the MR effect is larger. Although further studies would be carried out to determine the optimal loading volume of MRF to achieve ae balance between off-state viscosity and a larger MR effect, 3 vol% was selected as a standard volume to compare NiNW-MRF and NiNS-MRF because the following test focused on how the particle aspect ratio influenced the MRF. In a 660 mT magnetic field, the shear stress in NiNW-MRF was more than 14 times larger than the value of NiNS-MRF, which was only 9.07 Pa. The off-state shear stress of NiNS-MRF was as low as 7.29 Pa, which is similar to the corresponding value of 1.5 vol% NiNW-MRF. Note that even 1.5 vol% NiNW-MRF had more than 3 times stronger shear stress than NiNS-MRF in a 660 mT field. These results suggest that the aspect ratio of magnetic particles should serve an important role in having a better MR performance of NiNWs. This is consistent with previous published works where, as the concentration or aspect ratio of particles increased, the suspension would have increased viscosity in a magnetic field [18, 23].

Since MRF are expected to have mechanical applications, it was necessary to investigate the thixotropy of the samples. The shear stress and viscosity tests were processed with a logarithmic shear rate increasing from 0.1 to 200 s^{-1} at constant magnetic field strengths of 0, 110, 220, 330 mT. Considering the concentration of NiNWs, the higher loading amount of NiNWs supported MRF with larger thixotropy; for example, in a 110 mT field, 1.5 vol% NiNW-MRF had a shear stress of 13.5 Pa and a shear rate of 60 s^{-1} and 17.2 Pa with 200 s^{-1}, while 9 vol% NiNW-MRF had 106 Pa and 139 Pa respectively. To compare the 3 vol% NiNW-MRF and 3 vol% NiNS-MRF, in a 330 mT field and a shear rate of 60 s^{-1}, the shear stress of NiNW-MRF was 75.2 Pa while the NiNS-MRF was only 8.28 Pa. Moreover, the NiNS-MRF had curves undulating stronger than NiNW-MRF, which might be caused by the poor stability of NiNSs suspended in silicone oil. To summarise, the better stability of NiNW over NiNS allows NiNW to be a novel MR material.

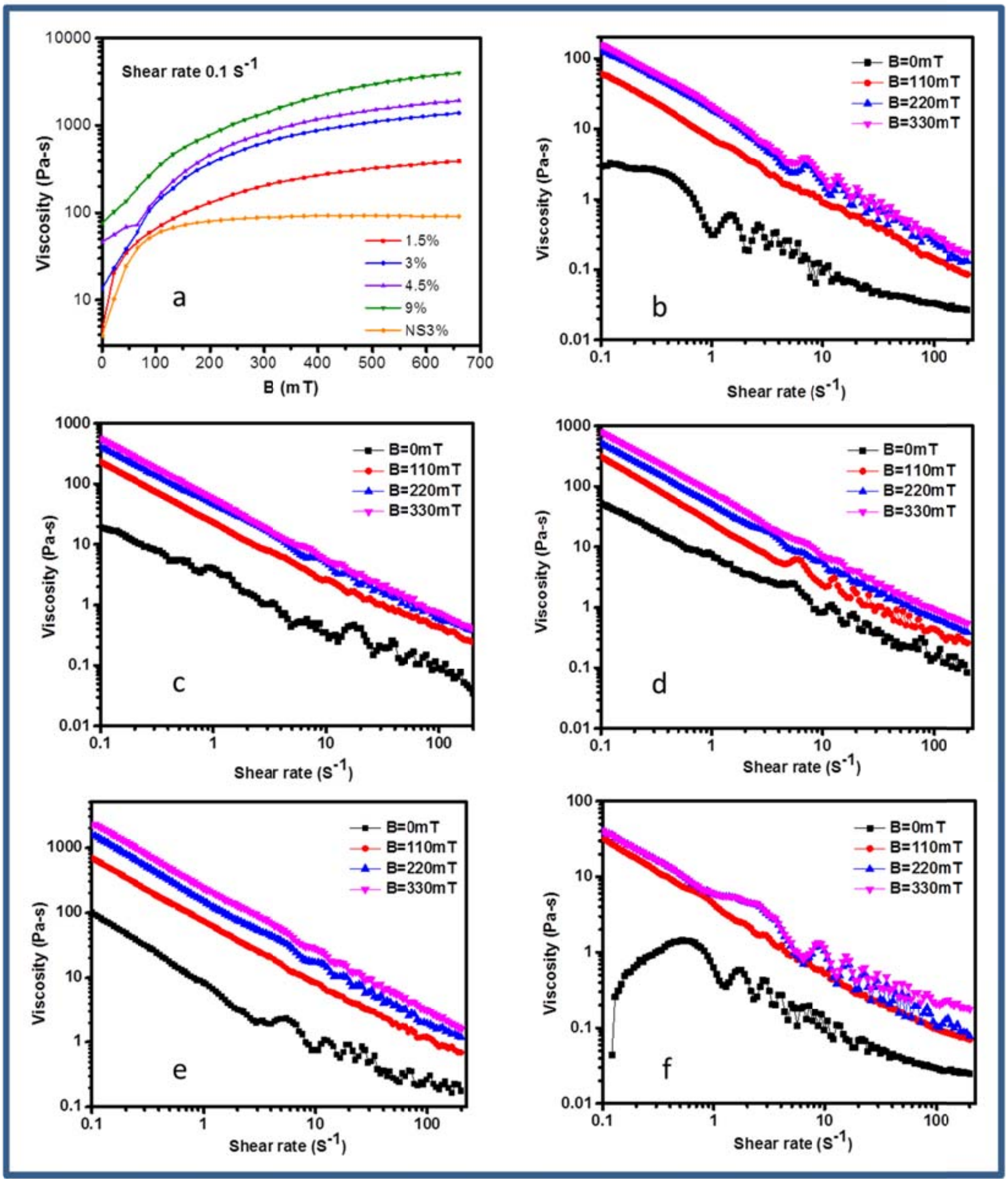

Figure 3. (a) Characterisation of changes in viscosity with varying magnetic fields. The orange colour represents NiNS-MRF, while the remainder are NiNW-MRF samples. The viscosity test of (b) 1.5 vol%, (c) 3 vol%, (d) 4.5 vol%, (e) 9 vol% NiNW-MRF samples and (f) 3 vol% NiNS-MRF with varying shear rate at constant applied magnetic field.

With sedimentation, the samples were shaken thoroughly just before being observed, and photos were taken during this process. (**Figure 4**) The suspension ratio was calculated as one minus the result, which was the height of the supernatant fluid divided by the total height of the MRF. Since the observation occurred by measuring the parameters with a ruler, the graphic result is just a cursory reference for presenting the comparison of stability. NiNW-MRF samples were much more stable than the NiNS-MRF; in fact the stability of MRF could be improved considerably by tuning the shape of the magnetic particles. [24] MRF with magnetic spherical microparicles was prone to sedimentation [18], and our MRF containing spherical nanoparticles also had poor stability. However, the MRF samples with NiNWs were very stable, particularly when the NiNW-MRF had a lower volume fraction. With higher volume fraction of NiNWs, the MRF had a more stable suspension, possibly due to the enhanced NiNW-to-NiNW interactions at higher concentrations. The fact that particles tend to settle is a serious challenge in MRF, which is why different additives have been added to improve stability, and while the NiNW-MRF had the potential to solve the sedimentation problem without adding additional components to MRF.

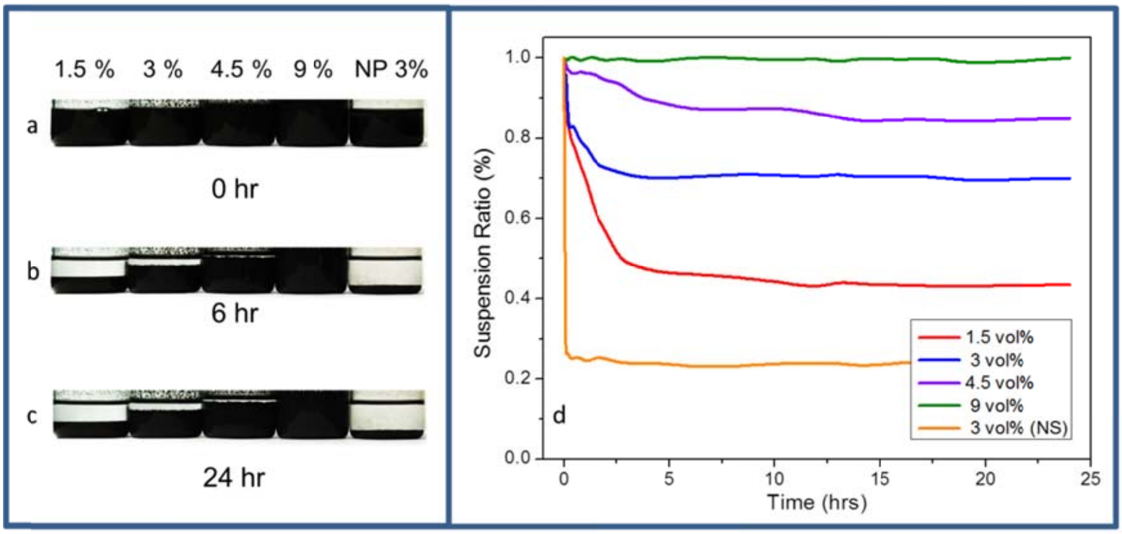

Figure 4. Photos of MRF samples during sedimentation (a) at the beginning, (b) 6 hrs later, and (c) 24 hrs later. (d) Graph of suspension ratio changes within 24 hrs.

To understand why NiNW-MRF has a stronger shear stress than NiNS-MRF, even though NiNWs has a smaller saturation magnetization (**Figure 2**), the shear stress of 3 vol% NiNW-MRF and 3 vol% NiNS-MRF were calculated. Since the carrier fluid is silicone oil, which is not elastic and cannot resist the shear stress, the shear stress of MRF before flow could occur was caused by the suspended NiNWs or NiNSs. Therefore, their nearest-neighbor interaction was modelled with COMSOL Multiphysics and their sizes were set as the average value obtained from the measurements. (**Figure 2. a, d**) The NiNWs and NiNSs were assumed to align into columns under applied magnetic field and the tilted angle between the columns and the applied magnetic field after shear deformation is denoted as θ, which was also the shear strain [22]. The magnetic hysteresis loops were averaged and then utilised to model the magnetisation of the NiNWs and NiNSs, respectively. The gap between the two nearest tilted NiNWs or NiNSs were assumed to be 1 nm and their interactions under an applied magnetic field were simulated and the shear stress was then calculated by volume averaging the shear stress contributed by one of the NiNW or NiNS [22, 25]. Periodic boundary conditions were applied in the simulations. (**Figure 5.a**) The shear stress calculated under B = 110 mT suggests that NiNW-MRF yields at $\theta = 43°$ and NiNS-MRF yields at $\theta = 38°$, beyond which the stress-strain is indicated by dashed lines as flow begins and the model becomes inappropriate. The calculated shear stress of NiNW-MRF is 2.1 times larger than NiNS-MRF under B = 110 mT, while in the experiment it is about 7 times larger (**Figure 3. c, f**). This discrepancy could be attributed partly to our assumption that all particles aggregate into columns under an applied magnetic field, but in a real system under a magnetic field, it is impossible for all the particles in the suspension to align into columns, so there could be isolated particles which do not belong to the columns and do not contribute to the overall shear stress, especially at lower concentrations [26]. As a result, the real concentration will be smaller than the simulated one which assumes that all the particles aggregate into columns (**Figure 2. a, d**). In fact, as the SEM images show, a NiNW could touch with remote NiNSs owing to its extreme aspect ratio, which implies the NiNWs are better at attracting each other and aggregating into columns under an applied magnetic field than NiNSs which could only touch their nearest neighbours. This phenomenon could also explain why NiNW-MRF disperses better than NiNS-MRF since the extreme aspect ratio of NiNWs led to strong interactions between one NiNW and many other NiNWs, including the remote ones, while one NiNS could only interact with several surrounding NiNSs. Therefore, it is much more difficult for NiNWs to become isolated particles than NiNSs and hence more difficult to settle in the suspension owing to their attraction to many other NiNWs. (**Figure 5.b**) The yield stress of NiNW-MRF and NiNS-MRF under varying magnetic fields was calculated and indicated that the yield stress of NiNW-MRF was always more than NiNS-MRF. This suggests that the increased shear stress of NiNW-MRF was due to the extreme aspect ratio of NiNWs, which had a geometric effect. Note also that only the nearest-neighbor interactions between NiNWs or NiNSs were considered in the simulations. However, the remote interactions for NiNWs should contribute to the shear stress, and to the discrepancy with our calculated ratio between the yield stress of NiNW-MRF and NiNS-MRF being smaller than the experimental ones.

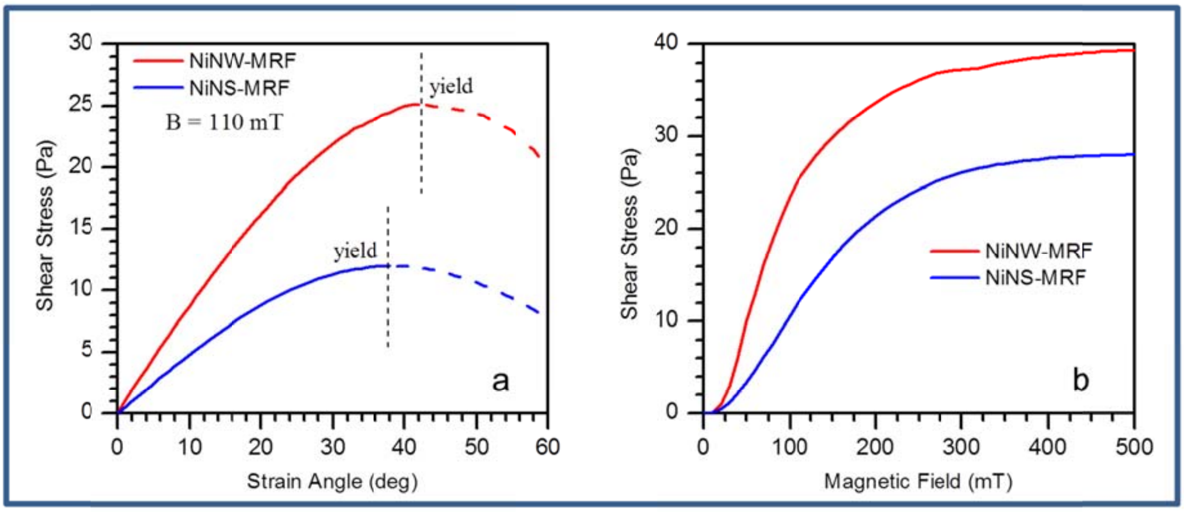

Figure 5. Calculated shear stress under an applied magnetic field. (a) Stress-strain curves of the 3 vol% NiNW-MRF and 3 vol% NiNS-MRF under an applied magnetic field B = 110 mT. NiNW-MRF yields at $\theta = 43°$ and NiNS-MRF yields at $\theta = 38°$, beyond which the stress-strain curves beyond yield are indicated by dashed lines since flow can begin and the model becomes inappropriate. (b) Yield stress of 3 vol% NiNWs and 3 vol% NiNSs under varying applied magnetic field **B**.

We plotted the calculated cross-section of the magnetic fields at the yield point for NiNWs and NiNSs under B = 110 mT, respectively (**Figure 6**), but unlike the NiNSs, the magnetic field around NiNWs is slightly weak so the magnetisation field of the NiNWs deviates from the direction of the applied magnetic field. However, the magnetisation field of the NiNSs generally aligns with the direction of the applied magnetic field and only deviates from it near the gap. As a result, the direction of the magnetic moment **μ** of a NiNW would deviate from the direction of the applied magnetic field **B**, but the direction of magnetic moment **μ** of a NiNS could only deviate slightly from the direction of the applied magnetic field **B**. Therefore, the NiNWs could undergo a larger restoring torque $\mathbf{T} = \boldsymbol{\mu} \times \mathbf{B}$ and the shear stress of NiNW-MRF becomes stronger under shear deformation owing to this sharp contrast in magnetisation which originated from the extreme aspect ratio of NiNWs.

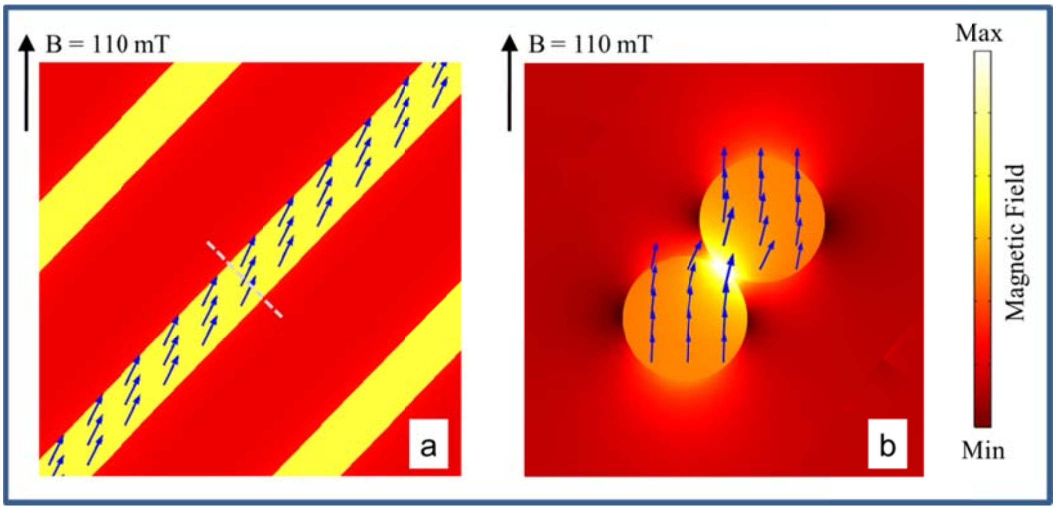

Figure 6. Calculated cross-section of magnetic fields at yield point for (a) NiNWs and (b) NiNSs under applied magnetic field B = 110 mT. The direction of the applied magnetic field is indicated by the black arrows. The gray dashed line indicates the gap between neighbouring NiNWs. The blue vectors are the magnetisation field **M** of the particles.

4. Conclusion

In this work NiNWs were synthesized and investigated as a novel magnetorheological material, and NiNSs were synthesized for the purpose of comparison. SEM images were collected and the size distributions of products were narrow, which means high uniformity. The magnetic hysteresis loops were measured and NiNWs had a smaller saturation magnetisation M_s and larger coercivity H_c relative to NiNSs. However, when the particles were dispersed into silicone oil, the NiNW-MRF had much stronger shear stress than the NiNS-MRF. At the same volume fraction, NiNW-MRF has 15 times the strength of shear stress and much better stability than NiNS-MRF; these advantages even existed when NiNWs was at lower volume fraction. This superior performance was investigated using finite element simulations, and the results suggest that the large aspect ratio of NiNWs should play an important role in enhancing the performance of MR; this promising MR effect allows NiNWs to become a novel MR material for further study and applications.

Acknowledgments

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Conflicts of Interest

The authors declare there is no conflict of interest.

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