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13. ABSTRACT (Maximum 200 Words) This effort was an investigation on the preparation of a novel smart structure with tunable damping and stiffness properties. This structure was a Magneto-rheological (MR) fluid filled volumetrically stiff cylinder. The MR fluid contained magnetic particles with various particle sizes ranging from nanometers (15-20 nm) to several microns (45 microns). An application of an external magnetic field would then alter the rheology of the MR fluid, thereby resulting in variation in stiffness and damping properties of the cylinder. Fluids were prepared with various amounts of solids loading and filled in a tube. The behavior of the struts containing a mixture of nano and micron sized iron powders were strongly influenced by the weight fraction of the large particles.				
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1. Project Objective

The primary objective of this effort was to investigate the preparation of a novel smart structure with tunable damping and stiffness properties. This structure was a Magneto-rheological (MR) fluid filled volumetrically stiff cylinder. The MR fluid contained magnetic particles with various particle sizes ranging from nanometers (15-20 nm) to several microns (45 μm). An application of an external magnetic field would then alter the rheology of the MR fluid, thereby resulting in variation in stiffness and damping properties of the cylinder.

2. Rationale

The main concept that was investigated in this research was the magneto-rheological (MR) effect in a fluid enclosed by a radially stiff cylinder. An MR fluid is a suspension of a magnetic powder (e.g. iron) in a fluid (e.g. hydraulic oil). In the absence of a magnetic field, the MR fluid behaves essentially like a Newtonian fluid with zero yield stress. In the OFF state, as soon as a shear stress is applied to the fluid, the fluid begins to flow. As magnetic field is applied, the particles in the MR fluid form particle chains between the poles of the magnetic field. These particle chains resist local shear stress, so that the fluid will flow only when the local shear stress exceeds a yield stress.

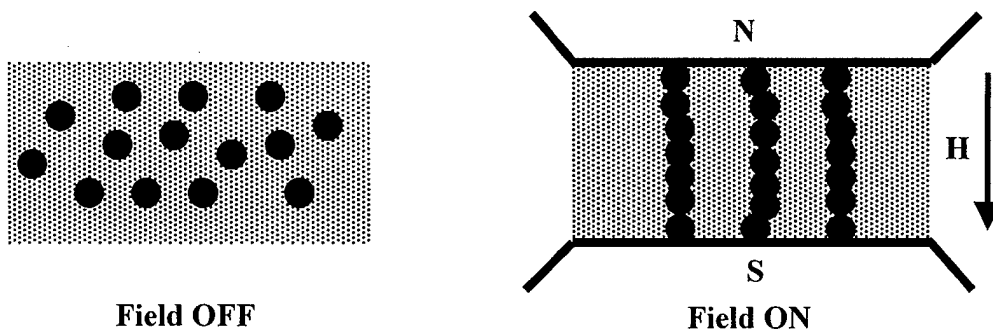


Figure 1: An illustration of the Magneto-Rheological Effect

This effect is manifested as a large field dependent or controllable Coulomb or surface friction effect in the fluid and can be exploited in powder compacts with their porosity filled with fluid, where magnetic field is applied to the compact to enhance the Coulomb or surface friction. The preliminary design of this controllable structure is a thin-walled tubular container fabricated from a composite material such as graphite epoxy. The container is filled with a powder consisting of a micron-size magnetic powder, along with a nano-sized magnetic powder, so that a bimodal powder distribution minimizes porosity. The container is vibrated to reduce porosity of the compact to be as small as possible. The powder container is then pressurized to squeeze out adsorbed gases in the fluids. This powder-filled composite tube then acts like a structural member with extensional stiffness and bending stiffness because: (1) during bending or extension, the powder in the compact exhibits strong surface friction due to the large sliding surface area, (2) the stiffness can be controlled via the pressurization level in the tubular structure and (3) magnetic field can be applied to the powder/fluid mixture to significantly increase the sliding friction between the particles.

3. Technical Objectives

- Synthesis of iron nanopowders using the Microwave Plasma Synthesis unit available at Materials Modification, Inc.
- Characterization of synthesized powders for composition by XRD and particle characteristics using particle size analysis and Transmission Electron Microscopy (TEM)
- Preparation of a MR fluid filled strut for the purpose of investigating the stiffness-damping characteristics
- Investigation of the effects of changing the volumetric ratio of nano- to micron-sized particles

4. Experimental Details and Results

4.1 Synthesis of Iron Nanopowders

The Microwave Plasma Synthesis unit (Nanogen™) available in-house was used for the synthesis of iron nanopowders. Vapors of iron pentacarbonyl were fed into the head of the plasma chemical reactor, which houses an argon plasma, created by dissociation, and recombination of argon molecules due to interaction with the microwaves. Iron pentacarbonyl, $\text{Fe}(\text{CO})_5$, breaksdown into iron and carbon monoxide by the following reaction:



Microwave plasma synthesis utilizes microwave energy to generate plasma by ionization, dissociation and recombination of gas molecules. The resulting high temperature vaporizes the precursors, which promotes chemical reactions at the molecular level in the presence of microwaves. These vapors are then rapidly quenched to form nanopowders. Figure 2 is a schematic diagram of the microwave plasma synthesis equipment available at MMI. Iron pentacarbonyl was used as a precursor for synthesizing the iron nanopowders. The experimental procedure is detailed below.

Vapors of iron pentacarbonyl were fed directly into the plasma zone, wherein the pentacarbonyl dissociated into iron and carbon monoxide. Argon gas was used to carry the powders into the plasma chamber, where rapid heating of the powders resulted in its dissociation. The microwave process parameters for synthesizing nanocrystalline iron are listed in Table 1.

Table 1. Microwave process parameters for synthesis of nanopowders

Magnetron Power	0-6KW
Magnetron Frequency	2450 MHz, CW
Waveguide	Rectangular
Plasmatron/Applicator	Quartz wall, water cooled brass chamber
Plasma Gas	Argon, Feed rate 2.0-4.0 m ³ /hour
Carrier Gas	Nitrogen, Feed rate 0.3-1.0 m ³ /hour

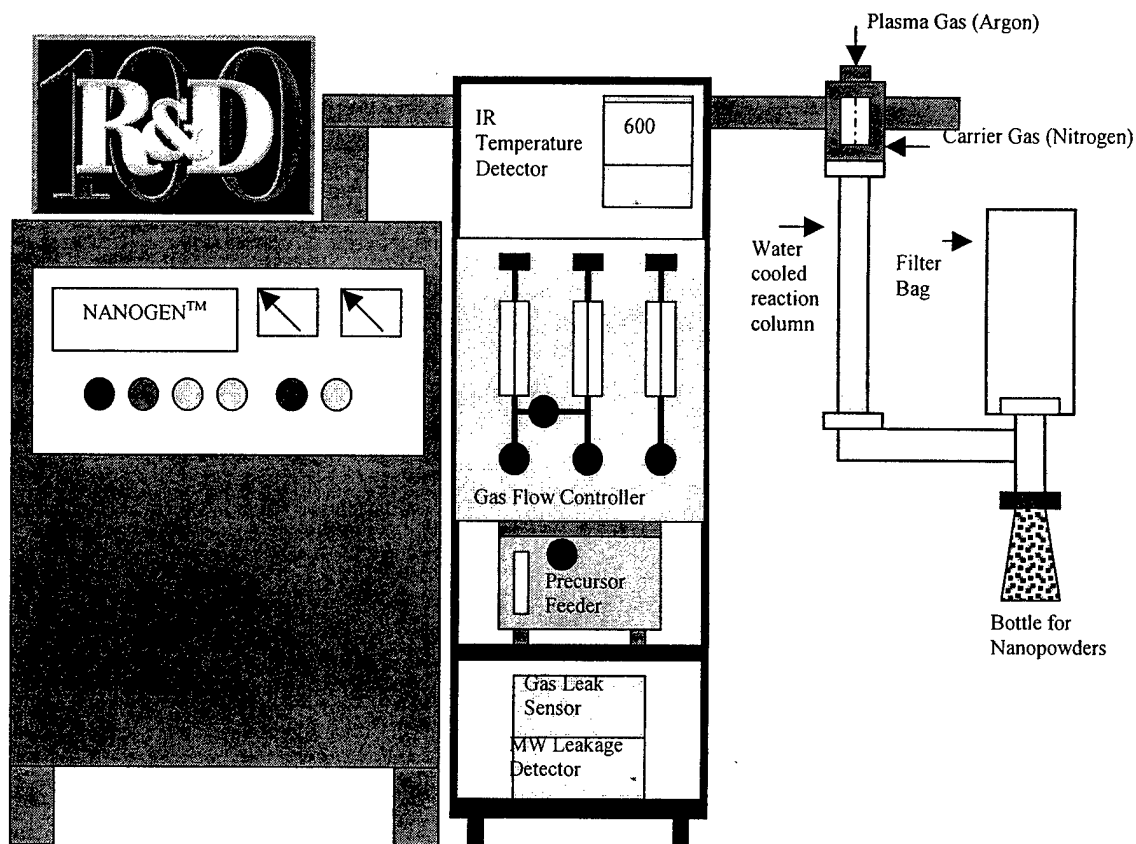


Figure 2. Schematic of the microwave plasma synthesis setup

4.2. Characterization of Iron Nanopowders

Figure 3a shows the XRD of the synthesized iron nanopowders. X-ray scan performed at a lower scan rate (0.8 degrees/min) showed the presence of an impurity phase. This impurity phase (see Figure 3b) was identified as a mixture of gamma-iron oxide (maghemite) and magnetite (Fe_3O_4). Such oxides are present on the surface of nanoparticles and act as a passive layer to prevent further oxidation or pyrophoricity.

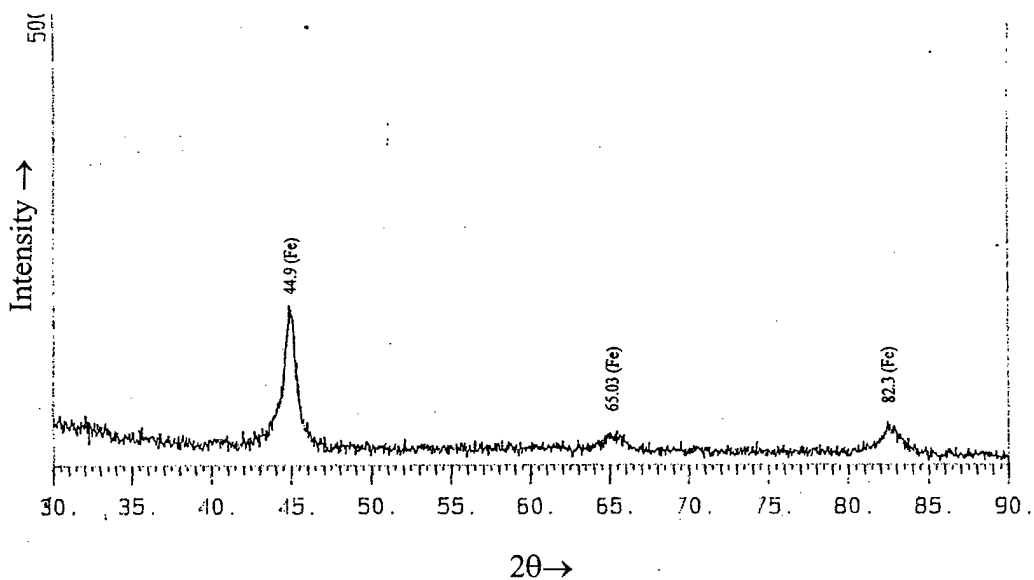


Figure 3a. XRD of iron nanopowders synthesized using a microwave plasma

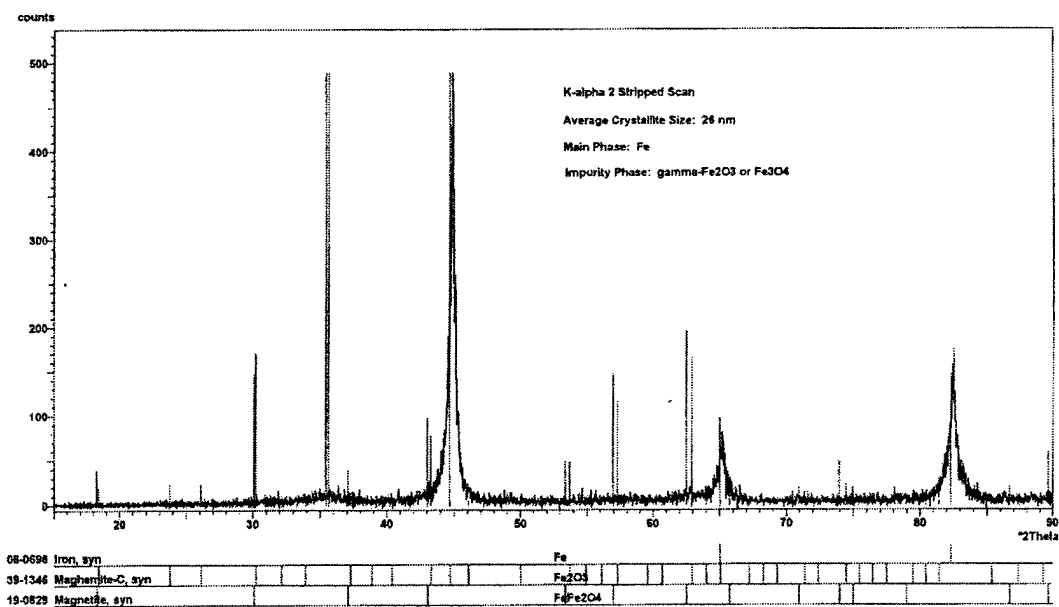


Figure 3b. X-ray scan of iron nanopowders at slow scan rates (0.8 degrees/minute)

Powder particle size was measured using transmission electron microscopy (TEM). Figure 4 shows a micrograph of the synthesized nanocrystalline iron powders. From the TEM analysis, the particle size ranged between 15-20 nm.

BET analysis was conducted and the mean particle surface area was calculated to be $42 \text{ m}^2/\text{g}$. This corresponds to a particle size of 26 nm, which is close to the results obtained from TEM.

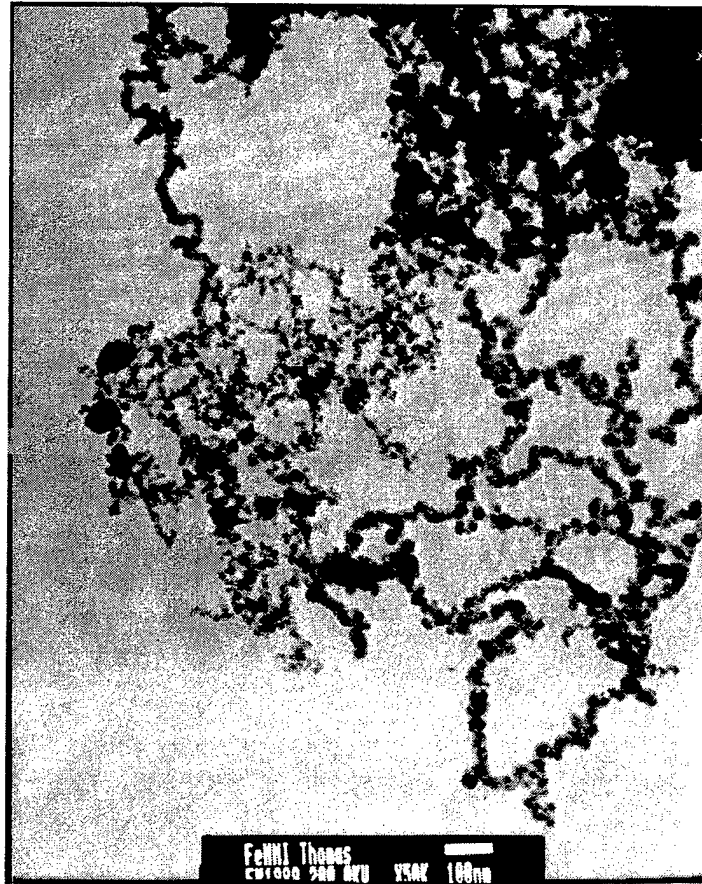


Figure 4: TEM of iron nanopowders

4.3 Preparation of the MR Fluid Filled Strut

The preliminary design of the strut was a thin-walled tubular container filled with magnetic particles and fluids in order to obtain tunable stiffness and damping characteristics. The composition of the tube can play a significant role in the fabrication of the strut so that (1) the tube is flexible and is not brittle to induce a stress failure upon application of a magnetic field and (2) the diameter is large enough for insertion of powders and fluids.

Two materials were initially used as tubing materials: (1) a composite material such as a graphite epoxy and (2) Polymer composite such as polyethylene. The former was too brittle while the latter was too small and the surface forces did not allow for powder and fluids to enter the interior of the tube. After careful investigation, a vinyl tubing, with a diameter of $7/16''$ x $1/16''$ thickness was used as the container for the MR fluid filled strut.

Two techniques were used in preparing the MR fluid filled strut. The *first technique* involved filling the container with a mixture of micron-sized and nano-sized iron powder, so that a bimodal powder size distribution was obtained. The container was then vibrated to reduce the porosity of the powder compact to be as small as possible and then infiltrated with hydraulic oil to fill up the existing pores.

The *second technique* for preparing the MR fluid filled strut involved preparing a MR fluid, then pouring it into the vinyl tube. MR fluids were prepared from both micron-sized iron powders and nanocrystalline iron powders. In addition, a fluid composed of a hybrid mixture of micron-sized and nano-sized iron powders was also prepared. Hydraulic oil was treated with a surfactant, Lecithin, and the iron powder mixture was added to the hydraulic oil using shear mixing. The total solids loading in all the fluids was 60 wt% except the fluid prepared from nanopowders where the solids loading was 43 wt%. The following hybrid compositions were used in the trials.

- 70 % micron-sized iron powder, 30 % nano-sized iron powder
- 70 % nano-sized iron powder, 30 % micron-sized iron powder

Approximately five and one half inches of the vinyl tube was used for each experiment. The tube was sealed by singeing the end and clamping it shut manually.

The initial experiments were conducted solely with micron-sized (45 μm – 200 μm) iron powders. The strut could not be made using the first technique as the surface tension disallowed the infiltration of oil through the pores between the powders. The second technique was however successful but eventually lead to the settling of the powders and the oil due to the large density gradient. Modifying the surface of the powders with a coating of lecithin improved the dispersion of particles but a chemical modification of the surface would be required to prevent settling.

In an attempt to use a lesser volume of iron powders, a tube composed of nano-sized iron powders was attempted. The procedure for preparing the tube was similar to that used for micron-sized iron powders. However, the nano-sized iron powders were passivated with nitrogen before exposing it to air because the powders are highly volatile. After passivation, the iron powders were packed in the tube, and hydraulic oil was added to the powders. The tube was partially burn sealed, and then the seal was completed using plumber's goop. It was difficult to singe the tube containing nano-sized iron powders because of their pyrophoricity. The powders burn along with the tube, thus making a seal very difficult. In addition, the iron powders are very dark, thus making it very difficult to observe the flow of oil between the powders. Although it is hard to see the mixture within the tube, it is noted that the tubes made with nano-sized powders are stiffer than those made with micron-sized iron powders. The nano-sized powders and the hydraulic oil solution become very viscous. The highest concentration that can be made is one of 33-wt% of nano-iron powders. The MR struts made with nano-sized iron particles display lesser magnetic properties than those made with micron-sized iron powders, however they are also stiffer. A proper combination of both micron-and nano-sized iron

particles will most likely create the right volumetric ratio of particles to develop a working MR strut.

4.4 Investigation of the effects of changing the volumetric ratio of nano- to micron-sized particles

The effects of changing volumetric ratio of nano- to micron-sized particles were investigated using hybrids of 70-wt% micron-sized iron to 30-wt% nano-sized solutions and vice versa. The 70-wt% micron- to 30-wt% nano- hybrid iron powder mixture displayed similar behavior to the strut prepared from 100-wt% micron-sized iron powders. The hydraulic oil continually settled atop the solution. This is most likely due to the weight of the micron-sized powders. The 70-wt% nano-to 30-wt% micron- hybrid iron powders displayed properties very similar to the strut containing fluid prepared from nano-iron powders. Although the solution was viscous, it was not too thick to be added to the vinyl tube container. The hydraulic oil also settles atop the iron nanopowders, however the extent of separation between the oil and the powders was drastically reduced. In addition to micron-sized and nano-sized iron powders, nano-sized iron oxide powders were also investigated for the preparation of the MR strut. The density of iron oxide is less than that of iron and thereby reduces the separation between the powders and the oil. Iron oxide nanopowders prepared using microwave plasma synthesis were milled for one hour to remove agglomeration. A MR fluid solution containing 60 wt% nano iron oxide powders was prepared in hydraulic oil. The solution was then added to a vinyl tube and sealed. No separation was noticed, however it appeared as if the hydraulic oil was trapped in the agglomerates. As a result, the magnetic properties of the strut were reduced.

The MR struts prepared using micron-sized iron powders displayed the best magnetic properties. However, they also exhibited the most separation between the iron powders and the hydraulic oil. The struts containing MR fluids prepared from nano iron powders exhibited a slower response to the magnetic field than the strut containing MR fluids prepared from micron-sized iron powders. This difference in magnetic response is possibly due to the difference in the iron powder content between the fluid containing nanopowders and micron sized powder. Although there was a separation between the iron nanopowders and the hydraulic oil, it was significantly less in comparison to the separation between the hydraulic oil and micron-sized iron powders.

5. Conclusions

The following conclusions can be drawn from the experiments and the observed results.

- (i) The preparation of a MR fluid and its subsequent addition to the tube was easier when compared to the addition of oil to a strut containing the powder.
- (ii) A narrow particle size distribution does not allow the infiltration of oil due to surface tension.
- (iii) Surface coating/modification of the iron particles is necessary to avoid/minimize surface tension.
- (iv) The response of the MR fluid filled struts to magnetic fields depends on the concentration of magnetic particles and their particle size.

- (v) The rheological behavior of the hybrid fluids were strongly influenced by the particles with the higher weight fraction.

5. Future Work

The experiments performed indicate that a wide particle size distribution is necessary to minimize the voids between the particles. A narrow particle size distribution does not lock the MR fluid in between the voids and results in separation of the hydraulic oil and the powders. In order to make a strut with the required tunability in stiffness and damping properties, the following would be required

1. A strut with a wider diameter for ease of insertion of MR fluid.
2. Coating of the strut material or modification of the fluid properties to minimize surface forces upon mixing.
3. Vacuum driven infiltration of the fluids
4. Preparation of MR fluids in low viscous carrier medium.