Ferromagnetic granular exchange interactions of nickel and iron

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Micromagnetic grains of nickel and iron were blended to investigate whether the magnetic exchange interaction was significant to produce a bulk result in a measured deflection by a fixed applied magnetic field. The results follow the same trend as Slater-Pauling's magnetization density calculations for nickel iron films, most notably a stronger following with a finer grain. Furthermore, by adding chromium into nickel and iron blends, the Invar minimum shifts toward less iron in nickel. Hysteresis was determined for the same samples through a homemade vibrating sample magnetometer (VSM). Areas of each loop are determined that show a minimum around the Invar transition, followed by sharp increases based on concentration of iron in nickel. These results help further understand the extent of magnetic exchange interactions in granular materials.

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1. Introduction

Granular ferromagnetic materials have significant impact in fields ranging from granular powders [1] to paleomagnetism studies [2]. Nano-sized granular materials are of interest due to applications in magnetic recording and solar cells [3,4]. The size of the grains can play a significant role. The surface area and volume fraction of atoms that occupy grain boundaries increases greatly as the grain size gets smaller [5–7]. Only 1–3% of the atoms are at the grain boundaries of average grain sizes of 100 nm and considerably less when the grain size is larger [4].

Ferromagnetic atoms are not only bound to their individual grain, but may also contain cohesive bonds between the grains. These bonds can vary in reason, including chemical and environmental [8]. The strength of the bond is highly dependent on morphology (size and shape), although the usual models for studying these grains is random sphere packing [9]. If the grain size is smaller than the exchange length, the alloy can be considered as to have a random anisotropy due to the random distribution of nanoscale grains [10]. Exchange lengths are significant and can be observed in the exchange bias effect which is seen as a shift in the hysteresis curve typically from the interactions of a ferromagnet with an antiferromagnet [11].

Understanding the interaction amongst the individual grains in an alloy can improve magnetic storage devices. These devices commonly use ferromagnetic materials, such as iron. However transferring power using iron can result in losses from eddy current. In order to increase the magnetic permeability and lower the losses, one could alloy with another magnetic material [12]. Iron nickel alloys have the highest permeability among soft magnetic alloys, making this an ideal material to study [13].

Slater-Pauling categorized multiple ferromagnetic alloy combinations via neutron scattering [14]. The iron–nickel curve in Slater-Pauling illustrates that by alloying iron into nickel there is a collapse in the magnetization until ~65% iron in nickel, named invar. A Martensetic phase transition occurs at this point from FCC to BCC. By adding more than 65% iron in nickel, the magnetization recovers and increases until the curve matches bulk iron. We set out to test if granular nickel iron alloys have significant cohesive bonds between the grains, relating closely to the bulk's magnetization.

A homemade vibrating sample magnetometer (VSM) was constructed with an electromagnet and pickup coils. VSM is a common analysis tool for magnetic characterization first proposed by S. Foner in 1955 [15]. This instrument operates by Faraday's law. A sample was placed in a long straw attached to a mechanical vibrator at a set frequency. Stationary pickup coils were placed inside a steady magnetic field produced by an electromagnet. The induced voltage in the pickup coils is proportional to the magnetic moment of the sample. Recording the induced voltage versus the applied magnetic field is called a hysteresis loop where the pickup voltage at a particular field is different depending on whether the applied field is increasing or decreasing. The area of such a loop was the energy required by the remagnetization processes. Labview was used to record both the applied voltage to the electromagnet and the measured voltage from the pickup coil. A similar homemade VSM was reported previously [16].

2. Materials and methods

Magnetic powders were purchased from Alpha Aesar varying in size. Table 1 shows the average grain size of each sample as retrieved from the manufacturer. Furthermore, the percentage of atoms at the grain

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boundary for each grain is included in Table 1. Each grain is assumed to be spherical in determining percentage of grains at the boundary. These trends are comparable to Gratzel’s work [4].

Blends of nickel and iron were weighed out to the nearest hundredth of a gram. The total weight of the mixture was consistently 0.20 g. Each sample was placed in a glass tube that can freely pivot at the top. The powder sample was located at the bottom of the glass tube where a uniformly applied external magnetic field was set by a Veriflux Magnet. The rigid glass rod acts as magnetic cantilever that has a fixed end leaving the other free to rotate based on stress applied to it. In our case the magnetic cantilever pivots due to the external magnetic field acting on the nickel–iron sample located at the free end of the cantilever. The Veriflux Magnet has a range of 21.1 G to 313.9 G that can be set by freely rotating disks on either side of the magnet. The uniformity of the field was checked with a Gaussmeter, resulting in 0.2 G difference in uniformity across the sample location. The resulting magnetic deflection of the powder in the tube was measured by digital calipers from the resulting endpoint of the deflection to the original location of the tube which was marked. These results are shown in the next section.

For hysteresis measurements, well blended samples of 0.4 g of nickel and iron were placed into a straw. Each end of the straw was then glued so that the grains couldn’t shift during vibration. A vibrating drum was attached to the straw that vibrated at 50.0 Hz for each trial. New samples were created with different mass percentages of nickel and iron. An electromagnet capable of up to 5100 G was used. Areas of each loop were calculated based on the results.

### 3. Results and discussion

#### 3.1. Magnetic deflections

Magnetic deflections were recorded based on the sample studied and compared with the Slater-Pauling magnetization density of thin films (labeled theory). Nickel–iron powders of two grain sizes are tested at 60 G of applied field and the results are plotted in Fig. 1. We note a similar trend in the deflection of both the coarse grain and fine grain compared to the Slater-Pauling magnetization density. Most notably, a decrease in the deflection around 65% iron in nickel was observed, which shows a break from anisotropy and finding a significant exchange length interactions at work. Furthermore, as the grain size became smaller/finer, the results matched closer to the theoretical curve, thereby illustrating a more cohesive exchange of magnetism in the sample.

Furthermore, 5% and 10% by weight of chromium was added to a blend of nickel iron and the same test was performed, Fig. 2. The Invar minimum of only iron and nickel grains is 65% iron in nickel. The minimum in the magnetic deflection was noted at 60% iron in nickel with 10% of additional chromium and at 45% iron in nickel with 5% of additional chromium. Each grain size was tested as performed earlier with no significant correlation found. The shift of the invar transition to lower iron content in nickel was likely due to the presence of antiferromagnetic chromium, forcing the iron to align antiferromagnetically at lower concentrations than without any chromium. Also, the antiferromagnetic exchange of chromium brings about a drop in the magnetization sooner with a smaller amount of iron in nickel.

<table>
<thead>
<tr>
<th>Material Label</th>
<th>Average grain size in (nm)</th>
<th>Percent of atoms at grain boundary</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel Coarse</td>
<td>$5.0 \times 10^3$</td>
<td>0.0015</td>
</tr>
<tr>
<td>Nickel Fine</td>
<td>$1.5 \times 10^2$</td>
<td>0.4952</td>
</tr>
<tr>
<td>Iron Coarse</td>
<td>$2.5 \times 10^5$</td>
<td>0.0003</td>
</tr>
<tr>
<td>Iron Fine</td>
<td>$2.0 \times 10^6$</td>
<td>0.0038</td>
</tr>
</tbody>
</table>

Table 1

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**Fig. 1.** Magnetic deflection of both coarse and fine powders of nickel iron were experimentally determined and plotted along with the magnetization density (theory) by Slater-Pauling. We note a similar trend in these results, including a precipitous drop in deflection and magnetization density at 63% of iron in nickel.

**Fig. 2.** Chromium at 5% and 10% by weight was added to nickel and iron samples. The total mass of the sample is fixed at 0.2 g. Magnetic deflections were measured with an applied magnetic field of ~400 G. The Invar minimum for each sample has a notable shift in location regardless of grain size. This shift is attributed to the antiferromagnetic exchange of chromium added into the nickel iron blend.

**Fig. 3.** The area under each hysteresis curve is plotted (y-axis) versus percent of iron in nickel (by weight). The line is drawn as a guide to the eye. A minimum is noted at 50% followed by sharp increases in the amounts of magnetic work.
3.2. Hysteresis

Samples were created in 10% steps by mass composed of nickel and iron. The total mass of the sample was fixed at 0.4 g. All hysteresis loops were similar in shape however the area under each loop varied widely. The resulting areas of each loop versus concentration are shown in Fig. 3. We note a minimum in the area or magnetic work around 50% iron in nickel. Albeit not at 65% iron in nickel where Invar is located, discovering a minimum in the area suggests that there is a concentration where it is energetically favorable to switch the orientation of the magnetic grains, thereby transitioning from a mostly nickel to mostly iron blend.

4. Conclusions

Magnetic exchange interactions were found to be significant in micromagnetic grains blended together, notably in nickel–iron blends. The significance is even more notable when the grain size decreases in size. By adding chromium, the Invar minimum shifts toward less iron in nickel regardless of grain size. Furthermore, hysteresis measurements via a homemade VSM suggest that there is a transition around 50% iron in nickel that is energetically favorable to switch its magnetic orientation. These results validate a granular magnetic model of a thin film to avoid costly manufacturing of a material that doesn’t have the desired properties.

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