

Manufacturing and Characterization of Paper-Based Magnetic Coatings

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Abstract

Please note the results of this experiment are being published. Therefore, no data can be released at this time.

Employing paper as an engineering material is emerging in industry today. Paper-based technology has grown in the last several years. Current technology includes, but is not limited to, foldable paper microscopes, a disk jockey board made from paper, and interactive wallpapers and newspapers^[1]. In terms of magnetic paper-based devices, these systems are limited to the use of hard magnetic materials for bitwise writing and reading, and actuating. At the Institute of Micro-Production Technology, embedding soft magnetic materials into the paper itself is being researched to enable a novel paper-based technology such as spintronic devices^[2]. This report will show the details of the ongoing research of the characteristics and properties of soft magnetic coatings on paper-based substrates.

The paper industry roots itself deep into history. Today, the industry is well established and produces papers with various properties^[3]. A method of selecting papers to best represent all of the paper types must be employed. Not only will these selected papers represent different aspects of all papers, they will reduce the amount of testing and analyses needed, saving time and resources. Once the papers are selected to test, methods of these tests will have to be altered to accompany the different properties of paper.

Several tests in use today characterize soft magnetic materials deposited on conventional substrates such as silicon. These tests can also be used for soft magnetic materials on paper, but the same methods of preparing and conducting these tests will have to be altered to account for the many different properties of paper compared with silicon. Before using these tests, literature searches were conducted to understand the underlying physics. This knowledge, with the results from the tests, proved helpful when determining the combination of optimal thickness and best paper substrate.

Making structures of permalloy on paper requires a sputtering deposition process with the use of a shadow mask. The problem with conventional shadow masks is the non-uniformity of the pressure applied on the substrate. When used to mask a paper substrate, the paper does not lay flat, which causes an inhomogeneous distribution of the permalloy. Therefore, a magnetic shadow mask is pulled down with uniform pressure by a magnet underneath the paper substrate allowing a minimal gap between paper and shadow mask.

Since the magnet will be used, the magnet's effects on the magnetic response of the permalloy are studied. The magnet increases the sputtering rate and betters the magnetic properties of the permalloy. For the future production of AMR sensors, a recommended thickness of permalloy and a paper with optimal characteristics results in the best possible coercivity and retentivity.

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Nomenclature

- **AMR Sensor**
 - Anisotropic Magneto-Resistive Sensor
 - Detects changes in magnetic fields
 - Linear or rotational positional sensors
- **CLSM**
 - Confocal Laser Scanning Microscopy
 - Laser scans distances from top to bottom of the material
 - Used to obtain surface detail
 - **Surface roughness** [μm]
 - Average (R_A), Root-mean-square (R_Q), and Peak to Peak (R_Z)
- **Coercivity**
 - Force required, in the opposite direction, to demagnetize a magnetic material
- **EDS**
 - Energy Dispersive X-ray Spectroscopy
 - Shows the elemental composition of a material
- **Hysteresis curve**
 - Also known as BH loop
 - Magnetic material's result of a changing external magnetic field
 - The output is the magnetic flux density
- **Permalloy**
 - Py: Ni₈₁Fe₁₉
 - 81% Nickel and 19% Iron
 - Soft magnetic material
 - Magnetizes and demagnetizes very quickly
 - Used in highly sensitive input devices and positioning sensors
- **Retentivity (Remanence)**
 - Magnetization of the material with zero external magnetic field strength
- **SEM**
 - Scanning Electron Microscopy
 - Shows micrographs of a surface at high resolution
- **Shadow Effect**
 - During the sputtering process, if material passes under the shadow mask, the result will be a structure without a clearly defined boundary
 - Results in inhomogeneous depositions and electrical shorts
- **Shadow Mask**
 - A metallic plate with a machined pattern to allow structures to be sputtered onto substrates
- **Sputter Deposition**
 - Also known as "sputtering" or "depositing"
 - Used to place a thin film of material onto a substrate

- Substrate is placed in a vacuum chamber. An inert gas flows into a chamber and is converted to gaseous plasma, such as positively charged Argon atoms. An electrode behind the magnetic material attracts the ions. The Argon atoms accelerate towards the electrode, and hit a “chunk” of the material, which consequently lands on the substrate.
- **VSM**
 - Vibrating Sample Magnetometer
 - Used to obtain hysteresis of magnetic material
 - Measures magnetic flux density due to varied magnetic field strength
 - **Magnetic Flux Density** [Tesla] or [N/A/m]
 - The amount of magnetic field passing through an area
 - **Magnetization** [A/m]
 - The magnetic field applied to the vibrating sample

Introduction

Paper is a versatile material established in a manufacturing industry dating back for centuries. Recently, paper has been employed to construct cost effective and inspiring technology. However, the use of paper in magnetics and magneto-electronics is constrained to hard magnetic materials for wiring, reading and writing of bits, amongst others^[1]. As shown in this study, paper and magneto-electronics can become one system with the use of soft magnetic materials. The purpose of this research is to study the properties of soft magnetic material, in this case permalloy (Py: Ni81Fe19), deposited in thin films on various paper substrates. This study will determine the optimal thickness of permalloy and the best paper substrate to construct a paper-based AMR sensor.

The optimal combination of permalloy thickness and paper substrate will yield magnetic properties such as a low coercivity and high retentivity. A magnetic material exerts a common response to a change in magnetic field. This response is a B-H loop, or a hysteresis curve shown in Figure 1^[4].

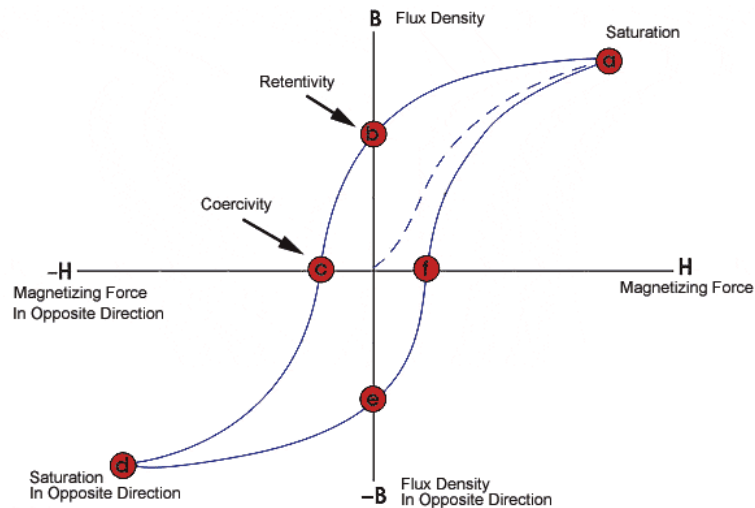


Figure 1. Hysteresis curve of a magnetic material shows the coercivity and retentivity of the magnetic material^[4]

The lower coercivity and higher retentivity correspond to a thinner and taller loop, respectively. The goal of manufacturing a paper-based magnetic sensor is to achieve these properties with the optimal permalloy thickness and paper substrate combination. These properties are critical to insure a large change in resistance, and therefore a high signal to noise ratio in the AMR sensors^[5].

Methods and Material

Sputter Deposition is the method of choice to apply thin films of permalloy on various paper substrates, which will be characterized. The VSM gives the Hysteresis Loop of the material and is the main test used to characterize the permalloy on paper. More tests will be conducted to supplement the characterization. EDS shows the elemental composition of the paper and permalloy materials at a certain scanning depth. CLSM scans the sample and measures the surface roughness of the material. SEM shows surface micrographics of the deposited permalloy on the paper substrates. In combination, these tests characterize and help show how the properties of the paper substrates differ between various paper substrates.

Method of Coating

First, the soft magnetic material must be deposited onto the paper substrate. Shadow masks will be used to construct AMR sensors on paper. However, knowing the mechanical flexibility of paper^[3], the paper must lay flat on the pallet during sputtering. Using the conventional rigid, steel mask and ring clamping system does not apply a unified force over the paper, which allows the paper to flex. This will result in an inhomogeneous deposition of the permalloy. The shadow effect could also occur, where the material is deposited under the mask, patterning of the deposited coating not successful. A new method must be devised to keep the paper flat around the entire 4π in² (diameter 4 in) area of the pallet so the magnetic material will have the optimal properties.

A magnet applies a unified force in a given area. A shadow mask composed of a magnetic material, if forced down on a paper, will provide the necessary unified pressure on the paper substrate. This, in theory, will help flatten the paper, allowing for a unified sputtering process to occur and minimizing the shadow effect.

With a mechanically fixed shadow mask, the substrate will rest on the pallet and the mask will be applied on top. With the magnetic shadow mask, the magnet rests on the 4-inch diameter pallet, in which the magnetic shadow mask presses on the paper substrate. Before using this magnetic fixation in the manufacturing of AMR sensors, it is important to understand the physics behind the manufacturing process, considering the properties of the paper and permalloy.

Sputter deposition is widely used in industry and research. At the Institute of Micro-Production Technology, a magnetron sputter deposition is used. The magnetron behind the target material (permalloy) attracts free electrons from the flowing Argon to increase the sputtering rate^[6]. At the beginning of the project, it was not known how the introduction of a magnet on the bottom of the substrate would influence the magnetron sputter deposition rate or process, or even more importantly, the magnetic properties of the permalloy on paper. Therefore, isolating the effect of the magnet below the paper substrate is critical.

Isolating Effect of Magnet

Comparison tests between a regular pallet, a magnetic pallet, and a raised pallet exemplifies the effect of the magnet. The raised pallet uses a stainless steel round stock with the same height increase as the magnet. The three pallets, as shown in Figure 2, will be compared to isolate the magnet's effect.



Figure 2. From left to right, the “regular pallet”, “magnetic pallet”, and “raised pallet” will be used to isolate the effect of the magnet.

The first pallet comparison test compares the sputtering rate between the three pallets. In this experiment, as in most performed throughout this research, the sputter duration is conducted in five time increments at low power. Paper as a substrate is entirely different than the conventional silicon substrates; therefore, conventions to sputtering must be revisited. While silicon can withstand high temperatures, paper cannot^[3]. The plasma generator power in the sputtering process must therefore be lowered to reduce the heat generated. This parameter was chosen to insure low temperature to accommodate the papers during sputtering.

In order to determine deposition thickness, permalloy is deposited on two 10mm x 10mm silicon chips in time increments, for a total of 10 chips for each pallet. Before inserting each pallet into the chamber, the samples are cleaned with acetone and isopropyl alcohol. A slim piece of tape is then applied to the middle of each chip, blocking the incoming material from being deposited on the chip. Figure 3 shows the sample after sputtering with tape applied.

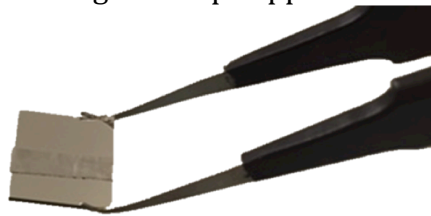


Figure 3. Tape is applied to the silicon sample to block deposited Permalloy.

The tape is then removed to reveal a step in the material, or thickness of permalloy. Each sample's step is measured at three locations to obtain a range of data. With two samples for each hour sputtered per pallet, thirty samples are required. The data, once compiled, averaged, and graphed, shows the differences in deposition rates between the three pallets.

To further the isolation of the magnet, the same amount of permalloy is deposited on samples for each pallet, and the samples are then measured in the VSM. Using the results of the previous thickness experiment, sputter deposition durations for each pallet are calculated to achieve equally deposited material. Once this data is obtained, samples are prepared. To ensure accurate results, 3 samples

for each thickness are measured on the VSM. Also, for reference, and further understanding of the relationship between permalloy and paper, 3 glass chips are sputtered and measured.

Insuring the same amount of permalloy is critical not only in the duration of sputtering, but also in the structure of the sample. Using the VSM restricts the sample size to a maximum width of 5 mm and a maximum length of 10 mm. Magnetic material has an easy axis of magnetization^[7]. If the sample is not symmetrical about both axes, different results could occur. Therefore a sample size of 5mm x 5mm is required. A magnetic shadow mask with 5mm x 5mm cutouts was machined and used for these experiments, as seen in figure A1 in the appendix.

Equal amounts of permalloy are sputtered on the samples. Since each pallet's sputtering rate differs, different sputter durations are required. Once permalloy is sputtered on paper and glass, each sample's magnetic response is measured in the VSM, recorded, and compared.

Paper Selection Process

The magnetic pallet's effect is isolated and understood. Now the properties of paper and the thicknesses of permalloy are compared. Before any experiments are designed, certain papers must be selected to best represent the population of all paper types. Paper samples from various vendors in Germany were gathered. Amongst the many papers gathered, 40 were chosen in previous work to be analyzed. Experimenting on all 40 papers would take too much time and resources; therefore, the selected papers must be further reduced.

Using the properties of paper, density, surface roughness, and thickness, the number of papers used in experiments is reduced. In addition, each paper is briefly sputtered to give a light coating of permalloy on the paper. The CLSM is then used to scan the paper, and an image processing software is used to measure the fiber size. These properties are organized to select a group of paper to best represent the population.

A process was developed to reduce the number of papers to best represent the entire population. First, the densities of the papers are ordered from least to greatest. Next, the papers are divided into groups by density. Within each of these density groups, the papers are reordered from smoothest to roughest, based off of Ra. Within each subgroup, the papers are ordered from thinnest to thickest. Once organized, papers were selected while keeping in mind the fiber size and texture of the paper. The papers are now selected, and multiple experiments are constructed to characterize the interplay between paper properties and permalloy thickness.

Characterizing Paper and Permalloy

The shadow mask, developed knowing the constraints of the VSM, is used for uniformity between all tests. There are 13 groups of three openings where a slip of paper can be placed underneath the mask. There is room for a slip of paper 11mm x 33 mm in each group. As such, 1 slip of each of the 13 papers is cut from the bulk material. Again, as in the pallet comparison, these paper samples will be measured

with a thickness of permalloy sputtered in time increments. Also, glass samples are placed on top of the mask, saving room and resources.

To measure the magnetic properties of permalloy on paper, the hysteresis curve of the magnetic material must be obtained. Therefore, the first test uses the VSM. Three samples of each paper per hour are required to insure a wide data set, minimizing error. After a batch of samples is sputtered, they are either measured immediately or stored in a desiccator for preservation.

Supplementing the magnetic properties of the various permalloy thickness and paper samples with other characterizing tests will result in a better understanding of the physics of the interplay between the two. The first of such tests is the CLSM. For each time increment, a paper sample with permalloy is measured for surface roughness. In addition, SEM and EDS experiments are performed on the paper and reference samples. Together, these tests will help characterize the paper samples and the magnetic material.

Results

Pallet Comparison

The first experiment compared the sputter deposition rate between the three pallets. After sputtering on silicon a chip, the thicknesses of the permalloy was measured. There is a differing sputtering rate between all three pallets, and the magnetic pallet has the highest rate, as shown in figure 4.

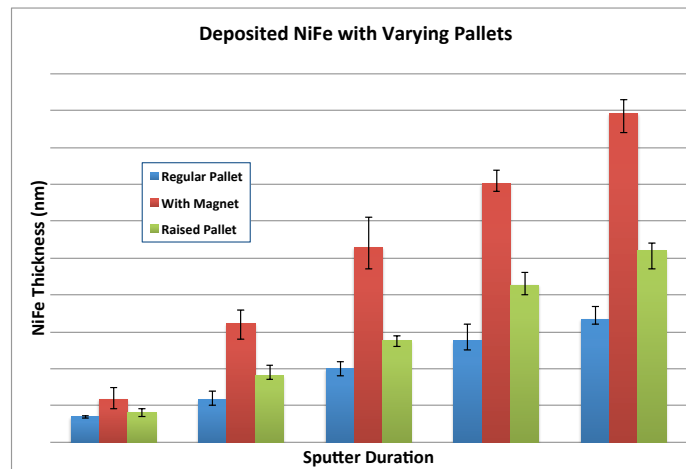


Figure 4. Thickness of permalloy on the three pallets

The difference in sputtering rates is interesting and is noted for discussion, but further testing must be conducted to fully isolate the effect of the magnet on the magnetic properties of permalloy on paper. To obtain an equal amount of permalloy, each pallet required different sputter durations. Therefore, the sputter duration was modeled as a function of permalloy thickness based on the aforementioned sputtering experiments.

The thicknesses are chosen to sputter on paper and glass samples for VSM measurements. The equations generated from the model were used to interpolate the times required for equal thicknesses for each pallet. Once the samples were sputtered on each pallet, the next comparison experiment compared the magnetic response of permalloy on the paper and glass samples. The VSM results show there is in fact a difference in the magnetic response of permalloy on paper and glass samples compared between pallets, even with equal thicknesses of permalloy. Figure 5 shows the coercivities of the paper and glass compared between Paper pallets.

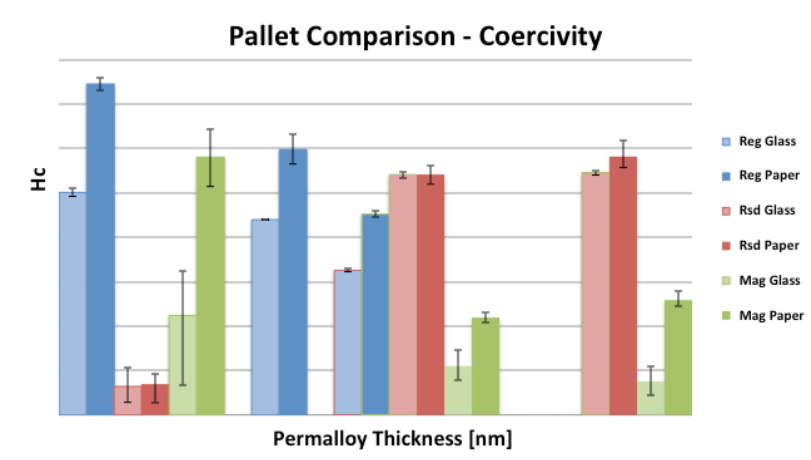


Figure 5. Coercivities of the three pallets of equal amounts of permalloy

The graph shows the trend of decreased coercivity as permalloy thickness increases for the regular pallet and the magnetic pallet. However, the coercivity of the raised pallet increases because the pallet is outside the optimal range for the grains of the permalloy to form correctly. Nonetheless, the magnetic pallet showed the best magnetic response between the three pallets. Further data are compared in the magnetic responses of permalloy on paper and glass on the regular, raised, and magnetic pallets, shown in figure A2. Clearly, the magnetic response of the permalloy sputtered on the magnetic pallet best represents the properties desired: low coercivity and high retentivity.

Paper Properties and Permalloy Thickness

The 40 preselected papers' densities, thicknesses, and surface roughness were previously obtained and fiber size was added in during the selection process. A matrix aided in visualizing the spread of the data. The matrix shows an even spread of the data. Twelve papers were chosen to best represent the population of papers gathered, thus optimizing experimental time and resources. In addition, a glass sample was chosen as a reference. Also, a "Nano paper" is currently in research and was added to the paper selection to further develop the theory and characterization of the interplay between paper properties and permalloy thicknesses. All in total, 14 samples were used in the experiments.

The first and main experiment is an investigation of the magnetic responses of permalloy on the paper substrates using the VSM. As expected, as more permalloy accumulates on the paper substrates the coercivities of the papers decrease. Graphing the coercivity of permalloy on glass as a function of thickness of permalloy, figure 8 below, reveals a trend of the decreasing coercivities.

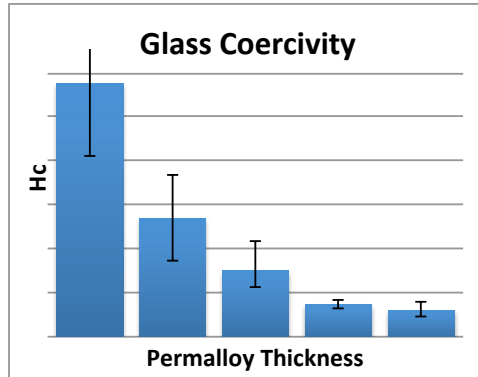


Figure 8. Coercivity of permalloy asymptotically approaches a value

The curve reveals the best coercivity of permalloy deposited on glass obtained from an optimal thickness of permalloy. For the coercivities of the papers, shown in figure A3, there is also a value for which the coercivity asymptotically approaches. The retentivity for the papers also increases as expected and is shown in figure A4. There is no value approached in this graph. Perhaps larger quantities of permalloy would result in such a value, but there was no indication of said value.

The surface roughness of the papers changed during sputtering. The reference glass samples showed a decreasing roughness as sputtering time increases, as shown in figure 9 below.

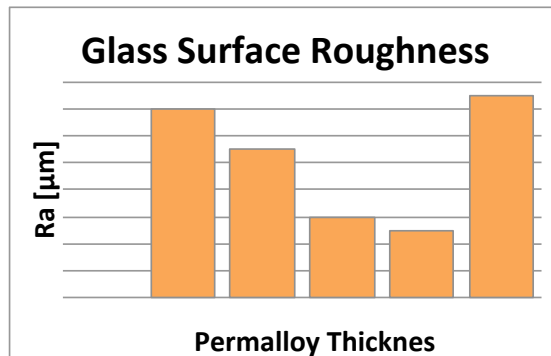


Figure 9. Average surface roughness for permalloy sputtered on reference glass

The average surface roughnesses of the papers are shown in figure A5. From the graph, two trends are prevalent. Rough papers of a certain average roughness value show no change in roughness as permalloy is added. The papers with a roughness under a certain range of value tend to increase in roughness.

Other characterization tests were performed on the paper-permalloy samples. The results of the EDS analysis are shown in figure A6. The weight

composition of Nickel to Iron stays constant throughout the sputtering process. The SEM tests produced micrographs of the paper surfaces in various zooms to aid in the understanding of the permalloy deposited on various paper substrates.

Discussion

Pallet Comparison

The thickness experiment was conducted to study the sputtering rates, or the amounts of permalloy deposited on the substrate, per time increase. As time increases the pallets all showed a linear increase of permalloy thickness, which is expected, as no other factors contributed to the sputtering process. The magnet does in fact pull the magnetic material towards the pallet at a faster and constant rate than without a magnet. The magnet increased the sputtering rate because the material sputtered is magnetic and is attracted to the magnet.

The effect of the magnet on the permalloy was proven during initial testing before the shadow mask was introduced. Paper samples were simply cut into square pieces and placed on top of the magnet. Once the magnetic pallet and paper were sputtered a certain time, the discovery took place. The paper square samples “stood” on one end and were attracted to the outer edge of the pallet as the pallet was taken out of the chamber, shown in figure A7. Once the samples stood on end, it was impossible to tell whether the samples were sputtered while still flat, or while one end. It was noticed once the pallet was moved or shaken by the robot arm, the tendency for the samples to stand on one end increased. Nonetheless, whether the samples were on one end during or after sputtering, they were not used. Therefore, the shadow mask was introduced to keep the samples lying flat and to achieve a perfect 5mm x 5mm area amongst all samples.

All samples were not sputtered in a perfect 5mm x 5mm area with the shadow mask. There were two factors contributing to the non-uniform sputtering across all samples in the same pallet. First, if the physical properties of the paper, dimensions of shadow mask, and the paths of the magnetic particles are taken into account, a theory can be developed. Paper is very porous with about 70% air⁴. The fiber composition of paper and its porosity make the paper easily penetrable by foreign particles. When the particles are released from the target and accelerate towards the pallet, a majority of the particles land on the substrate within the square area. However, there is the chance the particle’s path is at an angle to the area and travels under the mask and through the porous paper, until it finally hits a fiber and stops below the mask. This is known as the shadow effect. Initially, cutting the samples prior to sputtering without the mask, the shadow effect was avoided. However, the paper samples stood on end and were attracted to the edges of the pallet, so the mask was necessary. Also, to manufacture sensors on paper, the shadow mask must be used to construct the AMR stripe. Using the mask to sputter the square samples is a better representation of the final product’s magnetic response.

The second factor when discussing non-uniform sputtering on the magnetic pallet was initially discovered by visually inspecting the plasma during sputtering

on all pallet types. The distribution of plasma of the magnetic pallet is more intense than the other pallets as the cone is completely focused on the pallet. This intensity contributes to the increased sputtering rate of the pallet, as fewer particles miss the paper samples. Viewing the substrates after sputtering shows the distribution of the intensity of the plasma. The shadow effect was more concentrated in the center than on the outer radii of the pallet, implying more permalloy was deposited in the center. However, the initial testing without the shadow mask proved the permalloy is attracted to the poles of the magnet when the samples were attracted to the outer edge of the magnet. Further testing is required to understand the distribution of permalloy on the magnetic pallet. Nonetheless, in order to minimize the difference in radial thickness of permalloy, the paper substrates are placed randomly on the pallet during testing.

The three pallets compared with each other isolated the effects of the magnet. The sputtering rate is increased and the magnetic response of permalloy is more optimal than sputtering on the regular pallet. More experiments can be devised to further study these effects, as there is an optimal distance from target to pallet. Finding an optimal distance of the magnetic pallet is crucial to the formation of grain boundaries, which affects the magnetic response of the material.

Paper Properties and Permalloy Thickness

The magnetic pallet is used for sputtering permalloy on paper substrates. The shadow mask exerts a uniform pressure on the paper samples, minimizing the shadow effect which results in a more homogenous sputter deposition. With various papers sputtered on the same pallets, the thicknesses of the papers will vary and was taken into consideration when trying to minimize the shadow effect. On the inner radii of the pallet, the shadow effect was more intense than the outer radii. The paper sample placements were thus randomized during all tests to insure more accurate test results.

The paper substrates selected best represented the population of papers gathered. Density, thickness, surface roughness (average, RMS, and P-P) and fiber size were all considered. The paper densities plotted with the thickness reveals a linear relationship. Throughout sputtering, the average surface roughness was consistently linear with the root-mean-squared roughness and the peak-to-peak roughness. As a result, generally the papers were not completely covered by the permalloy and the surface properties of the papers were retained throughout sputtering. The amount of permalloy was not enough to have the same effects of permalloy on glass. However, it was enough to show promising results of the magnetic response of the permalloy on paper.

Measuring the magnetic response of permalloy on paper allowed for a deeper understanding of the influence of the paper properties. As shown in figures 8 and A3, there is an asymptotic coercivity the permalloy reaches when sputtered on glass and paper substrates. The magnetic response of the permalloy from a thin, near 2D structure, changes as it reaches its bulk value. The ratio of grain boundaries to surface area plays a role in the change. As more permalloy is added, the number of grains restricted by the surface of the paper and fibers stay constant. The paper surface's effect gradually decreases as the permalloy fills the gaps in the paper.

Eventually the permalloy structure is more uniform and the coercivity tends to the optimal value.

The retentivity increases as permalloy thickness increases, because the more permalloy material on the substrate, the higher the magnetic flux. Since there is no value the retentivity approaches, the limiting factor for the optimal permalloy thickness is coercivity. For glass and paper samples, an optimal range of permalloy thickness achieves the best coercivity.

Once the optimal thickness is increased, the magnetic response of the permalloy reaches its best performance. The magnetic response of permalloy on paper also never reaches the response of permalloy on glass. Figure A8 shows the magnetic responses of permalloy, with the optimal thickness, on glass substrates and the paper substrates showing optimal coercivity. These selected papers have similar properties. Under a certain value of surface roughness, the papers show a better magnetic response. Then, these papers' densities are compared. Some of these smooth papers are the least dense papers tested, and some are in the middle of the range of densities. As the papers are sputtered on the pallet for the duration of the optimal thickness, the magnet heats up. If the papers are under a certain density, the paper warps under this heat. The wattage used during sputtering should be further tested to minimize this effect. Therefore, papers with a roughness under a certain value and over a certain density are the optimal papers.

Conclusion

The effects of a magnet introduced to sputter deposition were isolated and studied. The magnet on the pallet increases the sputtering rate and more quickly structures the formation of NiFe grains for a better magnetic response compared with sputtering on a regular pallet. The optimal distance from target to substrate using the magnetic pallet and the power used during sputtering still needs to be studied to obtain an optimal distance and Wattage.

Optimal permalloy thickness and paper properties to achieve the best possible magnetic response were discovered using various characterization experiments. The more permalloy, up to a certain thickness, betters the magnetic response, or the lowest coercivity. Smoother paper results in a better magnetic response, with no limit to how smooth the paper. The paper must be a certain density to withstand the heat from the pallet.

These paper properties contribute to the optimal paper recommended to use for further studies and prototypes of AMR sensors constructed on paper. The next step recommended is to study the papers over the optimal density and under the optimal surface roughness. This way, more of the physics can be studied of the paper properties, such as the role of fiber size and texture of the papers.

Appendix

Figure A1. Magnetic shadow mask with (39) 5mm x 5mm openings

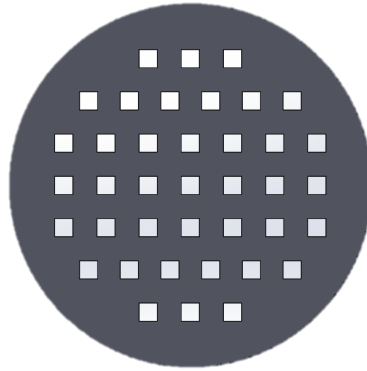


Figure A2. Magnetic responses of permalloy on regular, raised, and magnetic pallets

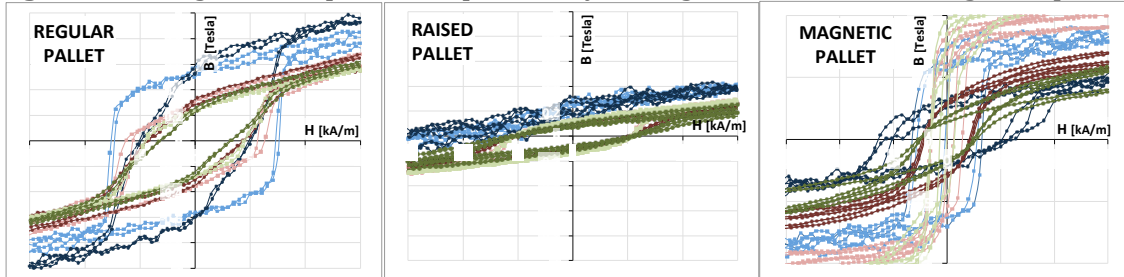


Figure A3. Coercivities of paper samples and glass

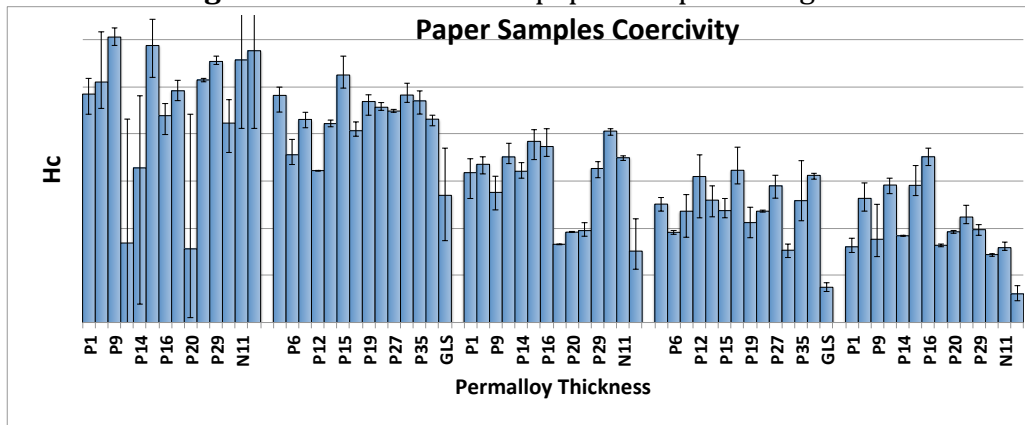


Figure A4. Retentivities of paper samples and glass

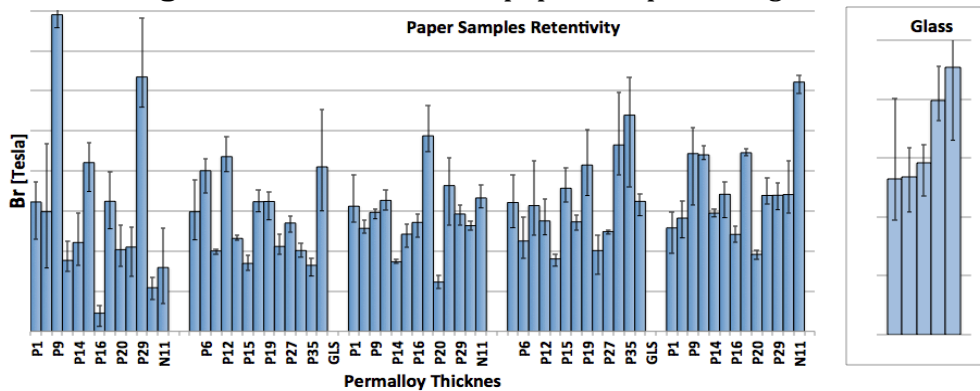


Figure A5. Surface roughness of paper samples

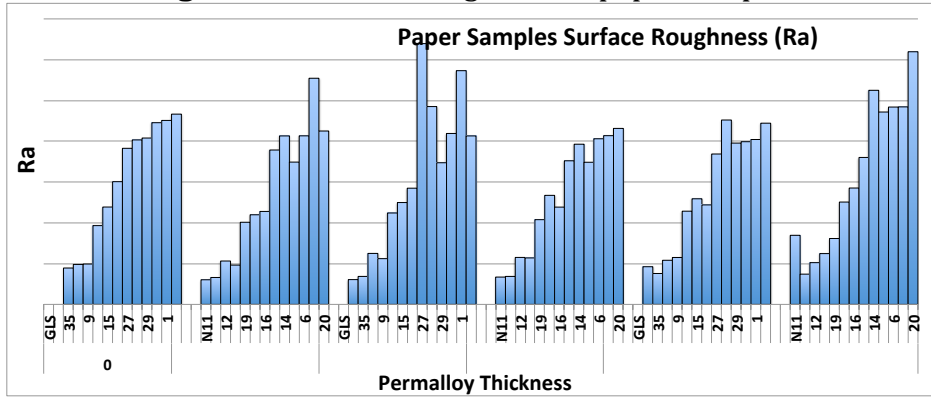


Figure A6. Weight percentages of permalloy

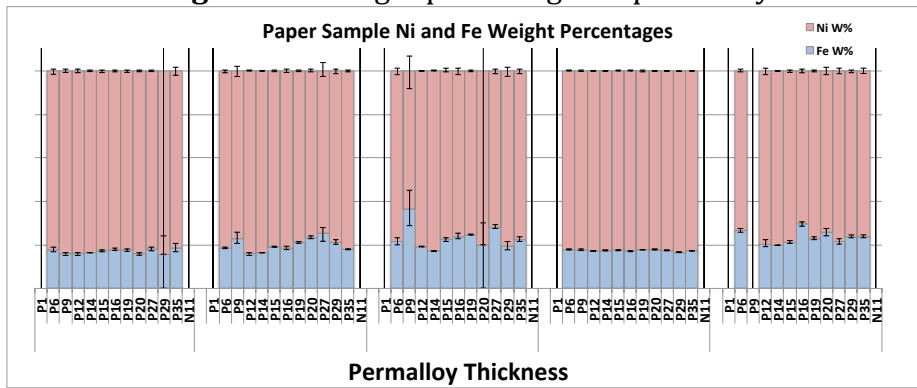


Figure A7. Result of sputtering without shadow mask

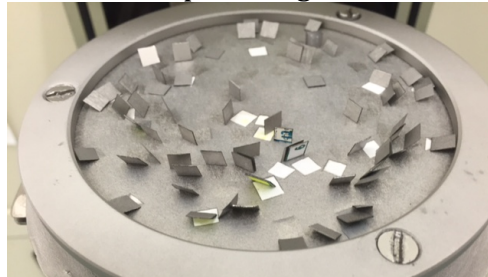
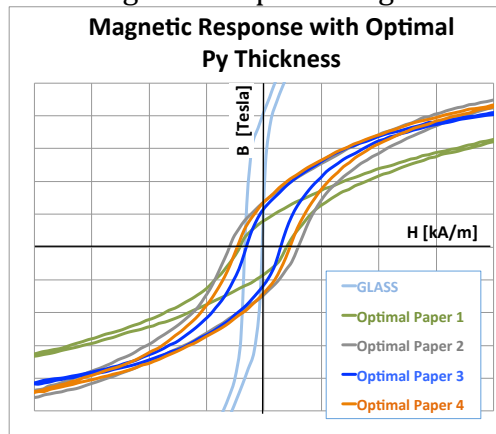


Figure A8. Optimal magnetic response of glass and selected paper



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