ABSTRACT

MAGNETORESISTANCE IN PERMALLOY/GAMNAS CIRCULAR MICROSTRUCTURES

by Justin Guenther

When two ferromagnetic materials are deposited directly on top of one another, their magnetic moments lock together and will no longer switch independently. This effect is known as exchange spring coupling. Reports in literature indicate that a bilayer composed of GaMnAs and permalloy may be a rare exception. Such a bilayer would allow for independent switching and, as a result, giant magnetoresistance. For this thesis, we verified the independent switching of continuous films of GaMnAs and expanded on existing literature. We also investigated GMR in bilayers. Samples were fabricated and measured using novel techniques and software developed specifically for this project. Transport measurements of GaMnAs/Py bilayers revealed a minimal to non-existent GMR effect; instead, the main discernible effect was due to AMR of the bulk substrate of the samples. This thesis also details the construction process of an inexpensive, temporary cleanroom environment.
MAGNETORESISTANCE IN PERMALLOY/GAMNAS CIRCULAR MICROSTRUCTURES

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Above all, my gratitude is to God, my source of strength, light, hope, and meaning. I am blessed to have the privilege to unfold the wonders of His creation.
Chapter 1: Introduction and Theoretical Background

The vast majority of modern electronic devices function by taking advantage of the charge property of electrons. Spintronics has emerged in recent years as a different approach, wherein the spin property of electrons is utilized, in addition to charge. Magnetoresistance has received a great deal of attention and played a significant role in industry and research. Magnetoresistance refers to the change in electrical resistance of a material when it is placed in a magnetic field. It is a broad effect which can result from a variety of different mechanisms, all of which involve the interaction between the spin of electrons in a current and the magnetization of the material or materials the current passes through.  

1.1 Ferromagnetism

A material exhibits magnetic properties when the electrons within that material have ordered spins. In a ferromagnet, the electron spins within an atom are ordered so as to produce a net spin—and therefore, a magnetic moment—for that atom. Moreover, the energy of a ferromagnetic material is minimized when the magnetic moments of the constituent atoms are also fully aligned. This means that a ferromagnetic material will fully and spontaneously become magnetized to saturation. In order to minimize the magnetostatic energy of a ferromagnet, micro-scale domains may form. Within each domain, the magnetization of neighboring atoms will be aligned; however, neighboring domains will be magnetized in different directions, resulting in no overall magnetization on the macroscopic level.

With the exception of amorphous materials, which are beyond the scope of this thesis, most ferromagnetic materials are crystalline, with a periodic molecular structure. The structure of a crystal arises from the interaction between the electron orbitals of the constituent atoms. Different crystalline axes have different orbitals; these orbitals interact with the spins of electrons, resulting in axis-dependent magnetization behavior. This means that certain crystalline axes are easy to magnetize; a relatively small external magnetic field along those axes will result in alignment of magnetic moments in the crystal. Hard axes of magnetization require a much larger external magnetic field to force the magnetic moments to align along those axes.
Due to the spin-orbit interaction described above, the energy of conduction bands within a ferromagnet has a spin dependency. This spin-based difference in energy results in a different density at the Fermi energy for electrons with spin parallel or opposite to the magnetization of the material. This split can lead to dramatic effects on the transport properties of a ferromagnetic material. The resulting interaction between the spin of an electron moving through the material and the magnetization of the material itself gives rise to a phenomenon known as magnetoresistance.4,5

1.2 Magnetoresistance overview

Magnetoresistance is a family of solid-state phenomenon describing the change in resistivity of a conductive structure (such as a multilayer) or material due to the change in magnetization of either part or all of the structure or material. Members of the magnetoresistance family include anisotropic magnetoresistance,1,6 tunneling magnetoresistance,1,6 and giant magnetoresistance.1,6 There are additional types, but they are beyond the scope of this thesis.

Anisotropic magnetoresistance (AMR) describes the change in a material’s resistivity due to the angle between its magnetization and an applied current. AMR is a result of magnetocrystalline anisotropy, which occurs due to interactions between electron spins and orbital angular momenta, known as spin-orbit coupling.1,3

Within a material, the electron orbitals are deformed anisotropically by the magnetization of the material. Electrons moving through the material are scattered, with higher scattering resulting in higher resistivity. It is the differential deformation of the electron orbitals that causes this variation in scattering.6 For most materials, this deformation causes a reduction in resistivity for electrons moving perpendicular to the direction of magnetization, while electrons moving parallel to the magnetization will scatter more, encountering greater resistivity (Figure 1.1).6,7
While AMR occurs within a single crystalline material, giant magnetoresistance (GMR) requires a multilayer structure in order to be observed. GMR describes a change in resistivity due to the relative magnetization direction of ferromagnetic layers within the multilayer structure.\textsuperscript{1,2,6} The simplest multilayer structure consists of two ferromagnets separated by a non-ferromagnetic conductor. One ferromagnet changes magnetization under a relatively small applied field—a soft magnet—while the other requires a larger applied field to change magnetization—a hard magnet. This allows for the magnetization of the soft material to be varied independently from that of the hard magnet. Current is typically directed perpendicularly through the multilayer, as it was in the experiments for this thesis, though studies exist which have analyzed the effect when current is directed parallel to the multilayer.\textsuperscript{1,2,6}

1.3 The two-current series resistor model

The physics behind the GMR effect can be explained using the two-current series resistor model (2CSRM). In this model, each ferromagnet is evaluated as a separate resistor. As the name implies, the single electrical current is considered instead as two distinct currents: spin up and spin down. As the two currents travel through a layer, they encounter different values of
resistance. This resistance is based on the relative direction of the magnetization of the material with respect to the spin direction of the electrons within that current.\textsuperscript{4,9}

This effect arises from differential, spin-based scattering of electrons moving through the material. When the spin direction of the current opposes the direction of magnetization in a layer—the anti-parallel state—that current encounters a high resistivity. When the spin direction is aligned with the magnetization direction in a layer, then the current encounters a low resistivity in that layer (Figure 1.2).\textsuperscript{4,9} This difference in resistivity is caused by the spin-dependency of the density of states at the Fermi level, which is described in greater detail below.

![Figure 1.2: Two current series resistor model. When the magnetization of the outer layers is antiparallel (left image), each current path will see a similar resistance. When the magnetization of the outer layers is parallel (right image), one current path will see a high resistance, while the other will see a low resistance.](image)

Typically, when the layers have the same magnetization direction, the overall resistivity is lower, while opposing magnetization directions result in a higher resistivity. The magnitude of this change for common GMR multilayers is on the order of 10-20\%.\textsuperscript{1,2,6} However, larger differences have been observed.

The net change in resistivity for a simple ferromagnet-spacer-ferromagnet structure is determined by the spin polarization of the ferromagnetic materials involved. Spin polarization is a property of the material itself. Non-magnetic materials have no polarization, while half-metals are completely polarized in one direction. Ferromagnetic materials fall in-between non-
magnetics and half-metals. The degree to which a material is polarized determines the magnitude of the difference in resistivity encountered by the spin up and spin down currents.$^{4,5}$

Spin polarization is caused by a difference in energy level between the spin-up and spin-down conduction bands. For a ferromagnet to have a net magnetization there must be more electron spins in one direction than in the other. This means that one conduction band is more populated than the other; essentially, there is more room for electrons in the direction opposing the magnetization. This difference in spin population is known as static spin polarization. The static spin polarization of a material is quantified by Equation 1.1.$^{4,5}$

$$P = \frac{D^\uparrow - D^\downarrow}{D^\uparrow + D^\downarrow}$$

*Equation 1.1*

Hybridization of the conduction bands with the non-polarized s-orbitals allows for multiple paths for an electron to follow. The difference in conduction bands gives rise to a different density of states at the Fermi level for each spin conduction band. If the density is small, the electron is more likely to conduct via the s-orbital, while a greater density means a lower chance of the electron conducting via the s-orbital. Because the s-orbital path has a lower resistivity than the d-orbital conduction band, electrons travelling via the s-orbital encounter a lower overall resistivity as they travel through the material, as illustrated in Figure 1.3.$^{5,7}$ The difference in scattering for spin-up and spin-down electrons traveling through the material is known as transport spin polarization.

![Figure 1.3. Conduction bands in a ferromagnetic material. As shown here, spin-up electrons would encounter lower resistivity, since they are more likely to be present in the s-orbital. Spin-down electrons have a greater probability of conducting via the d-orbital, and as such, these electrons would encounter a higher resistivity.](image)
While a relatively newly understood phenomenon, GMR has been implemented on a massive and crucial scale in the field of magnetic storage. Information on a disc can be encoded as magnetic domains of opposing magnetization. When a GMR multilayer structure moves over the surface of the disc, the magnetic domains cause the soft layer to switch magnetization while the hard layer is unaffected. The result is a measurable change in resistance corresponding to the magnetization of the domain on the disc.\textsuperscript{1,6}

### 1.4 Coupling in ferromagnetic bilayers

The spacer between the ferromagnetic layers serves a crucial purpose in current implementations of GMR. Ferromagnetic materials will couple in the absence of a non-magnetic spacer. This coupling may be ferromagnetic, wherein the magnetization directions of the two layers spontaneously align. Antiferromagnetic coupling is similar, as the magnetization directions will spontaneously change; however, they will preferentially anti-align, pointing opposite directions. The net result of this coupling is that the layers cannot be switched independently. Instead, the layers will switch together to achieve the lowest energy relative alignment.\textsuperscript{10,11}

If the magnitude of the applied field necessary to cause switching is different enough for the two materials, it is possible to break the coupling and switch one layer independently. However, a new effect arises, known as exchange spring coupling. Opposing the “desired” ferromagnetic or antiferromagnetic coupling results in a volatile, high-energy boundary. In order to resolve this discontinuity and achieve the lowest energy state, the magnetization near the boundary will rotate, producing a continuous boundary.\textsuperscript{10,11,12,13}

Exchange spring coupling eliminates ferromagnetic bilayers as an option for GMR structures. Not only would it require a much high applied field to overcome the natural ferromagnetic or antiferromagnetic coupling, but the continuous boundary destroys the GMR effect. In order to work, GMR requires that the spin of an electron remains unaltered as it travels through the multilayer. The continuous boundary causes the spin to gradually change as it passes from one layer to the next. This spin flipping all but eliminates GMR.\textsuperscript{14}

Whereas GMR structures eliminate coupling through introduction of a non-magnetic conductor, tunneling magnetoresistance (TMR) devices achieve the same result by introducing
an insulating layer between the ferromagnets. This insulator is only a few nanometers thick, allowing electrons to tunnel from one ferromagnet to the other. The probability of tunneling is greater in the event that the magnetization directions of the ferromagnets match. TMR is most effective at lower temperatures; as the temperature increases, thermionic emission becomes a greater factor, with the higher thermal energy providing the necessary kick to transport electrons from one layer to the other, regardless of the spin or relative magnetization.\textsuperscript{1,15}

There are a few exceptions which exist, a very small number of materials that can be deposited on one another and still switch independently. Recent research has indicated that GaMnAs—a ferromagnetic semiconductor—and permalloy—a nickel-iron ferromagnetic alloy—are two such materials.\textsuperscript{16}

\textbf{1.5 Choice of materials}

Studies of GMR and TMR have used a variety of different ferromagnetic materials. Derivatives of gallium arsenide (GaAs), particularly manganese-doped gallium arsenide (GaMnAs), are especially common. GaMnAs is a ferromagnetic semiconductor with a Curie temperature on the order of 50K (though more highly doped GaAs has a Curie temperature above 150K).
Figure 1.4: GaMnAs unit cell. This geometry is known as a Zincblende structure, a variation of face-centered cubic geometry. Manganese atoms will bond in substituent positions, taking the place of some gallium atoms. Some manganese atoms will rest in interstitial positions, sitting in the empty space within the crystal.\textsuperscript{17}

The manganese substitutes for gallium atoms, serving as an electron acceptor. However, doping processes will also produce interstitial manganese, atoms which do not replace gallium but instead sit within gaps in the crystal. An interstitial manganese atom serves as an electron donor rather than acceptor, meaning it not only fails to contribute to the magnetic properties, but will even reduce the effectiveness of the substituent manganese atoms (See Figure 1.5).\textsuperscript{16}

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{figure1_5.png}
\caption{Sample crystal lattice, consisting mainly of atoms (a) and (b). Atom (c) is in a substituent position for atom (a). Atom (d) is in an interstitial position, while location (e) represents a lattice defect, where a (b) atom has been dislocated.}
\end{figure}

Fortunately, interstitial manganese occupies a shallow potential well, and as such, can be dislocated through annealing. Upon exiting the crystal, the free manganese will bond with oxygen in the environment, producing a layer of manganese oxide.\textsuperscript{18} Etching with HCl or H\textsubscript{2}SO\textsubscript{4} is effective in the removal of the oxide layer (See Figure 1.6).
Figure 1.6: Effect of annealing GaMnAs substrate. Following epitaxial growth, the substrate contains a large quantity of interstitial manganese atoms (a). Annealing causes these interstitial atoms to migrate to the surface of the sample, where they form an oxide layer (b). Acid etching removes the oxide layer, resulting in a clean substrate with a reduced concentration of interstitial manganese (c).

As a material in GMR and TMR studies, GaMnAs has a complex and varied range of behaviors when placed in contact with other ferromagnetic materials. For example, a GaMnAs and iron bilayer can couple either ferromagnetically or antiferromagnetically, depending on the thickness of the GaMnAs layer. When placed in contact with permalloy (Py), a nickel-iron alloy, GaMnAs appears to not couple with the Py layer, according to recent research. In a 2013 study by Mark, S. et al., no change was observed in the hysteresis loop of a Py/MgO bilayer deposited directly on a GaMnAs substrate. This independence suggests an absence of coupling in GaMnAs/Py bilayers. 18

1.6 Hypothesis

Given that there should be no coupling between Py/GaMnAs, and whereas the non-magnetic conductive spacer serves the sole purpose of preventing coupling, we hypothesize that giant magnetoresistance should be observed in a bilayer consisting of Py and GaMnAs, even without any spacer. Successful demonstration of this hypothesis would allow us to determine the spin polarization of GaMnAs, which has yet to be consistently achieved and will vary from sample to sample.
Chapter 2: Methods and Apparatus

2.1 The circular transfer line method

The circular transfer line method (CTLM) is a sample geometry which suppresses the interference of AMR when making GMR and TMR measurements. The CTLM geometry consists of a uniform, ferromagnetic substrate. The spacer and second ferromagnet are deposited on top of the substrate. Circular portions of the deposited layers are then removed, resulting in islands which are isolated from the background by an empty gap. Overall diameter of each feature is on the order of 500 µm, while the gap between the island and the background ranges from about 5 to 50 µm. This is illustrated in Figure 2.1.

![Figure 2.1](image)

*Figure 2.1. The circular transfer line method (CTLM) basic geometry. Current spreads anisotropically through the top layer (dark gray), crosses the circular gap through the lower layer (black), and returns immediately to the top layer. A four-probe measurement technique eliminates external resistances.*

Electrical contacts are placed at the center of a circular island, as well as on the sample background. Current flows into the island, spreads isotropically, then crosses the gap between the feature and the background via the substrate. After crossing the gap, the current returns to the top layer and exits the sample through the electrical contact on the background (Figure 2.2).
Figure 2.2: Cross-section of CTLM bilayer. The top layer is permalloy, while the bottom layer is GaMnAs. The rough gray line traces the current, which spreads through the permalloy before entering the GaMnAs to cross the gap. After crossing the gap, the current reenters the permalloy.

Because the current is spreading isotropically as it travels through the sample, the directional effects associated with AMR are cancelled out. CTLM geometry can be further improved through use of a four-probe measurement, with the probe contacts applied to the center of each circular feature and the sample background, at a position far from the background contact for the current. Use of this technique isolates the resistance of the sample, eliminating external sources of resistance.\(^5,19\)

Measurement of a single feature with this geometry provides minimal insight. However, measuring multiple features on a single sample, with varying feature diameters and gap widths, will provide enough data to determine the contribution of the contact resistance and the substrate bulk. For a feature with a given diameter and gap width, the total resistance follows equation 2.1.\(^19,20\)

\[
R_T = \frac{\rho}{2\pi \ast t \ast r} \left( w + 2L_T \right) C
\]

Equation 2.1

where

\[
C = \frac{r}{w} \ln\left(1 + \frac{r}{w}\right)
\]

Equation 2.2

In the above equations, \(\rho\) and \(t\) are the resistivity and thickness of the GaMnAs substrate, \(r\) is the radius of the feature “island,” and
is the transfer length, the total length at the perimeter of the contact where the current goes from the permalloy layer into the GaMnAs. In the transfer length equation, $A$ is the total contact area of the feature and $R_C$ is the contact resistance, which arises from a depletion zone that forms when electrons from the permalloy occupy electron holes in the GaMnAs at or near the surface.

The key strength of using the CTLM geometry is the ability to eliminate the bulk resistance. The total resistance of the current path through the sample includes contributions from the deposited ferromagnet, the resistance of the substrate, and the contact resistance due to interfacial effects. By measuring the total resistance as a function of gap width and fitting a line to the data, it is possible to determine what the resistance would be with a gap width of zero. Conceptually, this eliminates all contribution from the substrate, leaving only the resistance of the deposited ferromagnet and the contact resistance.

2.2 Sample fabrication

In order to make magnetotransport measurements, we fabricated samples using a bilayer based on GaMnAs and permalloy. These materials were chosen based on reports in literature that demonstrate that they do not couple, even in the absence of a non-magnetic spacer. The process began with GaAs substrate, which was then coated with GaMnAs using molecular beam epitaxy to produce a layer between 30 and 45 nm thick. We received these blank samples from the group of Prof. Jacek Furdyna at the University of Notre Dame. For as-grown samples, we cleaned the blank substrate and were ready to begin photolithography. For some samples, we annealed at 180°C for 24 hours or more in air, removed the resulting manganese oxide layer with hydrochloric acid, and then cleaned the sample to prepare for photolithography. We used a typical photolithography process, after which samples were placed in the vacuum chamber for permalloy deposition. Ion milling was then used to clean the surface of the GaMnAs substrate further. Sputtering was used to deposit a layer of between 60 and 90 nm of Py on the substrate. Afterwards, samples were bathed in acetone to remove the unwanted metal. Following analysis to determine feature quality, the samples were wired for four-probe measurement.
Measurements were performed in liquid helium. Samples were situated within an electromagnet, with the length of the sample being coaxial with the magnet coil. The current in the coil was ranged incrementally to produce fields up to 2000 Gauss. The voltage drop across the samples was measured and used to calculate resistance. Following initial measurements, samples were typically annealed again for 30 minutes and then measured both immediately and 24 hours after annealing to discern time-related effects. Some samples were also measured under slight rotation (between 15 and 20 degrees) to discern the type of angle-related effects that would be expected from AMR.
Chapter 3: Results and Discussion

3.1 Verification of independent switching

The samples were fabricated with features that were either 600 or 400 microns in diameter, with gap widths of 80, 40, 20, 15, 10, 8, 6, or 4 microns. In order to verify that our GaMnAs and permalloy layers would switch independently, we made samples without features, then measured the major and minor magnetization loops to confirm the absence of coupling effects. Sample 1 consisted of a GaMnAs substrate, a copper spacer, and a permalloy layer capped with copper. Sample 2 omitted the spacer, instead featuring only the copper cap (Figure 3.1).

![Sample 1 and Sample 2](image)

*Figure 3.1. Geometries for featureless samples used in magnetization measurements. Sample 1 (GaMnAs/Cu/Pt/Cu) matches typical GMR geometries, with a spacer to prevent coupling. Sample 2 (GaMnAs/Pt/Cu) matches our experimental geometry, omitting the usual spacer.*

The magnetization of the samples as a result of applied field was measured for both samples (Figure 3.2). A field was applied that was strong enough to set the magnetization of both the permalloy and the GaMnAs to the same orientation. Then, the field was gradually decreased to zero before changing the field direction. Shortly after changing the direction, the magnetization of the permalloy switched, resulting in the large change in the magnetic moment of the sample. At a larger negative field, the GaMnAs changed magnetization direction as well. The field was then brought slowly to zero before changing the field direction again. As the
magnitude again increased, the permalloy switched first, followed eventually by the GaMnAs. The result was a complete hysteresis loop, known as a major loop.

We next confirmed the absence of coupling in our samples. This was done by applying a high field to set the orientation of the GaMnAs, then varying the field to cause switching in only the permalloy layer. The resulting hysteresis behavior is called a minor loop. After switching just the permalloy with the GaMnAs fixed in one magnetization direction (the forward loop), the applied field was strengthened until the GaMnAs layer switched magnetizations. Then, the permalloy layer was again switched independently (reverse loop). The result is shown in Figures 3.3 and 3.4.
Figure 3.3: Permalloy switching behavior at constant GaMnAs magnetization with a copper spacer between the substrate and the permalloy.

In Figures 3.3 and 3.4, permalloy changed magnetization at the same applied field for both the forward and reverse loops. This clearly demonstrates that the magnetization of the
GaMnAs did not affect the switching behavior of the permalloy. Therefore, we know that the two layers are switching completely independently.

3.2 Magnetotransport samples

![Figure 3.5: 80 micron gap width feature.](image)

![Figure 3.6: 40 micron gap feature.](image)
Figures 3.5 through 3.8 show the change in resistance of CTLM samples consisting of a permalloy layer deposited directly on a GaMnAs substrate. Samples were mounted according to the procedure described earlier, then measured in liquid helium. A magnetic field was applied
using a superconducting electromagnet. The field ranged from -1500 Gauss to +1500 Gauss, by increments of ~6 Gauss. The peaks appearing at the low fields (<150 G) correspond to the expected switching of the magnetization for the permalloy and GaMnAs. As the applied field increases or decreases from 0 G, the permalloy switches first. This happens at extremely low fields, on the order of 20 G. This is seen in the graphs as a jump in resistance immediately to the left or right of zero applied field. As the magnitude of the field continues to increase, the GaMnAs switches magnetization next. This occurs at an applied field around 100 G. This switching event is seen in the graphs as a drop in resistance. In all of the samples where this effect was present, the magnitude of the change in resistance varied with the overall resistance of the sample, with the jump remaining around .3% of the overall sample resistance. This constant percent change indicates that the effect is not a result of the interface, meaning it must be a result of the sample bulk.

Measuring these samples over a wider range (up to +/-1600 G) revealed some unexpected switching behavior, occurring in the range of 800 to 1200 G and -800 to -1200 G. Unlike the low-field switching events, which all occurred at the same field regardless of gap width, these higher-field dips changed shape and location depending on the gap width of the sample. This switching behavior also exhibited an angular dependence; rotation of the sample by approximately 15° resulted in a significant change in switching field (Figures X, X, and X). This was likely due to the magnetization snapping to easy axes, and then slowly shifting to align with the applied magnetic field, as depicted in Figure 3.9.

![Image of magnetization diagrams](attachment:image.png)

**Figure 3.9:** Sample magnetization (in black) under applied field (dark grey) with respect to crystallographic axes (light grey). Under smaller applied fields (a, b), the magnetization remains along the easiest axis. As the field increases (c), the magnetization snaps to a harder axis, bringing
magnetization closer to the applied field. When the applied field is strong enough (d), the magnetization of the sample will move away from the axes and align fully with the applied field.

As a means of establishing an experimental control, the same measurements were performed on a sample with identical features, but with the permalloy layer replaced by niobium (Figure 3.10). While permalloy is a typical ferromagnetic conductor, niobium is a non-magnetic superconductor at liquid helium temperatures. Because the effect we are looking for requires two ferromagnets in a bilayer, using a non-magnet would allow us to assess how much of what we observed was due to the permalloy/GaMnAs structure, as opposed to simply being due to the bulk substrate.

![Graph showing normalized resistance vs. field (G) for different feature gap widths: 600-80, 400-80, 600-40.](image)

**Figure 3.10**: Niobium control sample. Note that low-field switching events, similar to those observed in the GaMnAs/Py bilayer, are present here, as well.

Though there are some differences between the Nb control samples and Py experimental samples, the similarities show that the major effects occur independently of Py. We still encounter a resistance change at very low fields, with a corresponding change back around +/- 100 G. This strongly suggests that the effect seen in our magnetoresistance measurements was due to the bulk substrate, with permalloy making little or no contribution.

This conclusion is further supported by measuring the resistance at zero applied field as a function of feature gap width (Figure 3.11).
The intercept of 0.477 Ω accounts for the contact resistance and the resistance of the permalloy layer. Since the sample was measured in liquid helium, the resistance of permalloy is negligible. What this graph indicates is that sample resistance is a function almost exclusively of gap width. This relationship is mediated by the relationship between gap width and the amount of GaMnAs the current must travel through. From this data, we can conclude that the behavior of these samples was due almost entirely to the substrate bulk.

Because of the high resistance seen in the sample and the relatively low magnetoresistance effects observed, new samples were fabricated using different parameters. The samples were annealed for 20 hours, the surface was etched with dilute HCl to remove manganese oxide at the surface, and then annealing was continued for another 4 hours. Lithography was done as normal, and the sample was etched with oxygen plasma to clean the surface and re-etched with dilute HCl to remove organic impurities and manganese oxide. Ion milling was conducted prior to metal deposition.

Ion milling is a process where a surface is bombarded with high velocity ions to etch away the surface. Because of the repeated annealing and etching, the GaMnAs surface may have been depleted of substituent Mn. Since the ferromagnetism of GaMnAs relies on the presence of the Mn substituting for some of the Ga, depletion of these crucial atoms could create a depleted
zone, resulting in a high resistance and dampening the desired effect. Ion milling would be an effective solution for removing the depleted zone without creating an oxide at the surface.

Measurement of an older sample that was not annealed prior to deposition showed some potentially promising results (Figure 3.12). Unfortunately, due to the age of the sample, it was unknown what was responsible for the effects seen. It is possible that natural migration of interstitial manganese resulted in a barrier at the surface large enough to produce a TMR effect. This is, however, speculative.

![Figure 3.12: Old as-grown sample. The sharp drop seen asymmetrically is similar in shape to the TMR effect seen in other samples, though the relative size of the drop is much smaller than would be expected. The asymmetry may be due to impurities in the sample affecting propagation of domain walls.](image)

### 3.3 Annealed magnetotransport samples
Figure 3.13: 80 micron gap width annealed sample.

Figure 3.14: 40 micron gap width annealed sample.
Figure 3.15: 20 micron gap width annealed sample.

Figure 3.16: 15 micron gap width annealed sample.

The symmetric, low-field switching events observed in these features (which were characteristic of the other features) were consistently low in magnitude, on the order of .05 to .1% of the total resistance. This was consistent with our sample geometry, designed to suppress AMR effects.
Measurements of the resistance as a function of gap width yielded a result similar to the findings for the earlier samples (Figure 3.17). The intercept was still low, with the resistance depending almost solely on the gap width. This suggests that the modified sample fabrication procedure was not effective. Fabrication and testing of diagnostic samples revealed that one likely issue was the effectiveness of our ion mill. Even when samples were placed in close proximity to the ion mill and the mill was run for an extended length of time—as long as possible without risking damage to the mill—virtually nothing was removed from the diagnostic samples. If a depleted zone was present and responsible for the lack of visible GMR, running the ion mill would not have had any effect.

![Figure 3.17: Resistance as a function of gap width for annealed samples. Intercept error is 0.447Ω](image)

Having analyzed the sample bulk, it was also necessary to characterize switching behavior in the permalloy alone. In order to characterize the magnitude of this AMR and the external field at which it would occur, we measured the magnetoresistance through only the permalloy layer (Figure 3.18). The switching behavior seen in the permalloy alone was also consistent with the overall switching observed in the sample.
Figure 3.18: AMR of permalloy background. The AMR of permalloy is extremely small (~0.3%) and occurs at a small switching field (+/- 20 Gauss).
Chapter 4: Conclusions and Outlook

Because of the small extent of the AMR in permalloy, its effect can be considered negligible. Based on the behavior of the background and the sample as a whole, the effect can be conclusively attributed to AMR in the sample bulk, with TMR either not occurring under these conditions, or occurring in such a small proportion that it is lost in noise. Our hypothesis was that a Py/GaMnAs bilayer should be capable of producing a GMR effect. Because the bulk AMR was so dominant and we were unable to observe GMR or TMR, we cannot support our hypothesis at this time; however, we cannot reject it either, and will thus continue to research the possibility.

The next step would be to further investigate the effect rotation has on the sample; there may be an ideal angle which minimizes the bulk effect and better allows for seeing TMR in our samples. Because AMR is dependent upon the angle between the current and the applied magnetic field, observing a range of values for sample rotation would allow us to quantify the magnitude of the AMR effect. Given that our sample geometry should be suppressing AMR, analyzing different sample rotations could also reveal an alternate explanation. However, at this time, AMR of the bulk is still the most logical explanation for the effects observed.

Based on the literature, the observed results run contrary to our expectations. Assuming GaMnAs and permalloy do not couple, we should expect to be able to achieve GMR/TMR. Even in the presence of a strong AMR effect, at the low temperatures used for measurement, we should be able to observe large GMR switching events. In order to diagnose the cause of our unexpected results, we need to confirm the lack of coupling between the materials. This can be addressed through two channels. The magnetization data we currently have lacks precision; our measurements have shown that Py will switch magnetization under a field of just 20 Gauss. Additional magnetization data, generated under much higher control and precision, would be invaluable.

In addition to the magnetization measurements done using a SQUID, it would also be extremely useful to analyze the samples using neutron diffraction. Because neutrons have a non-zero spin and no charge, they can be used to probe magnetization. They interact only with the magnetic fields. This technique could be used to investigate the magnetization layer by layer to see if we coupling, exchange spring coupling, or spin-flipping occurring at the interface. Spin-
flipping is a distinct possibility, as the processes of ion milling and acid etching both roughen the surface of the substrate. This low-quality surface may cause electrons to change spin when they reach the irregular interface.
Chapter 5: Special Projects and Contributions

There is, of course, much more to research than being in the lab and reading or writing papers. A good experimentalist must be creative and be able to identify and solve problems as they arise, even when those problems are outside of the usual range of experience. I encountered several such opportunities over the course of my Master’s work.

5.1 Novel wiring technique

In order to wire a sample for measurement, each feature being measured requires a connection to two separate pins. These pins are simply the ends of the copper wires in two CAT-5 cables; the cables run through the length of a tube that is inserted into a liquid helium Dewar when measurements are performed. Ultra-fine gold wires are used to connect the copper pins to the centers of features on the sample. The connections are held in place using a cold-press technique with indium solder.

The original method of wiring the sample was to coat both ends of the gold wire with indium, then attach one end of the wire to a pin and the other end to the center of the sample. This procedure would be performed twice for each feature being measured. There was one key issue, however: the act of attaching the second wire to a feature would almost invariably dislodge the wire already attached. Moreover, having to attach the free end of the wire to the sample was often very difficult, as the wire would spring and vibrate. Additionally, the indium loaded on the end of the wire would often be as large as the feature, making shorts and solder bridges common, especially since two wire ends would be attached to the sample. Connecting four features would take around an hour and a half, with poor connections being a typical issue.
The solution I devised to expedite this inefficient process was simple but effective. Rather than connecting two wires to each feature, one longer wire would be used to join two pins. The center of the wire would then be doped with indium solder, and brought into contact with the sample. This process cuts the number of connections made on the sample surface in half, reducing the likelihood of dislodging connections already made. Since the wire is secured at both ends, it doesn’t bounce and spring to the same extent. Since only one connection per feature is necessary, shorts and bridges are a rare occurrence. Implementation of this new procedure has allowed for six features to be connected on virtually every sample, with wiring taking about an hour.

5.2 Development of Instrumentation Software

Once samples are wired, they are ready to be measured. This became a major issue when transferring between computers and researchers. The original software used for measurement of samples was written using LabView, which presented a number of issues. First was migration of virtual instruments (VIs) and sub-VIs. In order for a LabView VI to function properly, every VI referenced within the program must be in the correct file location. When migrating to new users
on a new computer, it was virtually impossible to collect all the VIs needed to make the
programs work properly. This was compounded by the quality of the programs themselves; there
was an abject lack of documentation or comments, and the complexity far exceeded the needs of
the experiments. Development of new programs in LabView was stymied by the lack of a large
user support base online and the high cost of the software preventing development outside of the
laboratory.

In light of the myriad of complications, development of new software for controlling
measurement apparatus was done using Python. The online community of developers is large,
free libraries are available for interfacing over GPIB, the Interactive Development Environment
(IDE) is free as well, and Python is taught in the Miami University computational physics
curriculum. Finally, Python development can be done outside of the laboratory, without the need
for expensive, proprietary software.

The main program written for magnetotransport measurements was successfully used in
dozens of data runs. Additional programs were also developed for measurements of single
electron transistors, Schottky barrier analysis, and n-type/p-type characterization of microwires.
An in-development version of the program is provided in full in Appendix A.

5.3 Construction of Temporary Fume Hood and Cleanroom

Due to the physics department moving to a new building, it was necessary to dismantle
the professional class 100 clean room in late spring. In order to minimize downtime, we built a
temporary, low-cost cleanroom for continued production of samples. The walls were built using
hangers designed for drop ceilings to support doubled-up 15 mil heavy-duty plastic sheeting. The
floor plan was redesigned to minimize the footprint of the cleanroom and maximize the
efficiency of a HEPA blower fan purchased specifically for this project.

One of the main concerns in building the temporary cleanroom was containing the
hazardous fumes emitted by the photolithography chemicals. We addressed this concern through
construction of a basic fume hood. Ventilation was handled using muffin fans and a dryer hose
connected to an exhaust line still in place from the professional cleanroom. The fume hood itself
consisted of one table secured to the top of another, surrounded by cardboard and 15 mil plastic
sheeting to keep it contained. The space under the top table provided a sufficiently large,
protected space for the fume hood equipment.
No official rating was obtained for the temporary cleanroom, although anecdotal evidence suggested that a class 100 environment has been achieved with similar setups, in the region immediately beneath the HEPA blower. The quality of the samples produced in the temporary clean room was sufficient to allow us to proceed with research with only a moderate delay.
Works Cited

8. Ye, Jun et al. (2013) Determination of magnetic anisotropy constants in Fe ultrathin film on vicinal Si(111) by anisotropic magnetoresistance. Scientific Reports, 3(2148), Figure 4.


Appendix A: Python Measurement Script

from __future__ import division
import numpy as np
from matplotlib import pyplot as plt
from matplotlib import animation as anim
import time
import visa
import csv
import datetime
import string
docname = raw_input("Filename: ")
date_time = str(datetime.datetime.now())
datenow,timenow = string.split(date_time," ")
hr,mnt,sc = string.split(timenow,":")
filename = ["C:/Documents and Settings/Labuser/",docname,"-",datenow,"-",hr,"- ",mnt,".csv"]
filename = ".join(filename)

#Prompt user for constants
v1ref=input("Enter reference voltage for lock-in 1 (Volts): ")
v1freq=input("Enter frequency for lock-in 1 (Hertz): ")
if li2==1:
   v2ref=input("Enter reference voltage for lock-in 2 (Volts): ")
v2freq=input("Enter frequency for lock-in 2 (Hertz): ")
else:
   v2ref="Lock-in 2 off"
v2freq="Lock-in 2 off"
imin=input("Enter minimum current value (Amperes): ")
imax=input("Enter maximum current value (Amperes): ")
istep=input("Enter current step value (Amperes): ")
timedelay=input("Enter time delay between steps (milliseconds): ")
exret=",enter value of external resistor (Ohms): ")

#Initialize PS and amps
try:
   amrel=visa.instrument("GPIB::05") #PS gpib address
   print "Amrel power supply ready"
except:
   sys.exit("Power supply error. Program terminated.")

try:
   lockin1=visa.instrument("GPIB::08") #Top lockin address
   print "Lock-In 1 ready"
except:
   sys.exit("Lock-In 1 error. Program terminated.")
try:
    lockin2 = visa.instrument("GPIB::09")  # Bottom lockin address
    li2 = 1
    print "Lock-In 2 ready"
except:
    print "Lock-In 2 error. Resuming without lock-in 2."
    li2 = 0

header_timestamp = [str(datetime.datetime.now())]
header_author = [str(raw_input("Researcher name: "))]
header_const_labels = ["LI 1 Ref", "LI 1 Freq", "LI 2 Ref", "LI 2 Freq", "Time Delay", "External Resistance"]
header_constants = [v1ref, v1freq, v2ref, v2freq, timedelay, rext]
if li2 == 1:
    header_data_labels = ["ISet(A)", "IMeas(A)", "Field(G)", "X1(V)", "Y1(V)", "R1X(Ohms)", "R1Y(Ohms)", "X2(V)", "Y2(V)", "R2X(Ohms)", "R2Y(Ohms)"]
else:
    header_data_labels = ["ISet(A)", "IMeas(A)", "Field(G)", "X1(V)", "Y1(V)", "R1X(Ohms)", "R1Y(Ohms)"]

with open(filename, "wb") as f:
    writer = csv.writer(f, dialect = "excel-tab")
    writer.writerow(header_timestamp)
    writer.writerow(header_author)
    writer.writerow(header_const_labels)
    writer.writerow(header_constants)
    writer.writerow(header_data_labels)

amrel.write("OUTP 1 ON")  # Turns on PS output
amrel.write("".join(["ISET 1 ", str(ilim)]))  # Applies current limit
amrel.write("VSET 1 5.00")  # Applies PS voltage limit
amrel.write("".join(["CURR 1 ", str(imin)]))  # Sets PS current to In
amrel.write("OUTP:REL:POL 1 REV")  # Sets PS output to negative
lockin1.write("".join(["FREQ ", str(v1freq)]))  # Sets LI1 frequency
lockin1.write("".join(["SLVL ", str(v1ref)]))  # Sets LI1 reference voltage
if li2 == 1:
    lockin2.write("".join(["FREQ ", str(v2freq)]))  # Sets LI2 frequency
    lockin2.write("".join(["SLVL ", str(v2ref)]))  # Sets LI2 reference voltage
if abs(imin) < imax:  # This block sets the current limit
    ilim = imax * 1.1  # on the Amrel PS to 110% of whichever
else: ilim = abs(imin) * 1.1  # value is larger, Imin or Imax.

# Prepare figure for graphing
fig = plt.figure()
ax = fig.add_subplot(111)
line1, = ax.plot([], [], lw=2)
if li2 == 1:
    line2, = ax.plot([], [], lw=2)
ax.set_xlim(imin*290*1.05, imax*290*1.05)
ax.set_ylim(-1, 1)
ax.grid()

def polling_cycle(iset):
    # Define the iterated function
    amrel.write("\n\n\nCURR 1 \n\", str(iset)))  # Change the current for the PS
    iactual = amrel.ask("IOUT? 1")  # Check actual current output
    v1x, delim, v1y = lockin1.ask("SNAP ? 10,11")  # Check lockin1 x,y values
    if li2 == 1:
        v2x, delim, v2y = lockin2.ask("SNAP ? 10,11")  # Check lockin2 x,y values
    else:
        v2x, v2y = 0, 0
    return iset, iactual, v1x, v1y, v2x, v2y

def gendata():
    # Clear all data lists from memory, establish value for iset
    isetlist = []
    iactuallist = []
    fieldlist = []
    v1xlist = []
    v1ylist = []
    r1xlist = []
    r1ylist = []
    v2xlist = []
    v2ylist = []
    r2xlist = []
    r2ylist = []
    iset = imin

    amrel.write("OUTP:REL 1 OFF")  # Front outputs on PS go live, and yes, it should say "OFF"

    while iset > 0:  # Run from minimum current value to 0
        iset, iactual, v1x, v1y, v2x, v2y = polling_cycle(iset)
        yield iset, iactual, v1x, v1y, v2x, v2y
        iset = iset - istep
        time.sleep(timedelay)

    amrel.write("OUTP:REL:POL 1 NORM")  # Return to regular (positive) polarity

    while iset < imax:  # Run from 0 to maximum current value
        iset, iactual, v1x, v1y, v2x, v2y = polling_cycle(iset)
        yield iset, iactual, v1x, v1y, v2x, v2y
        iset = iset + istep
        time.sleep(timedelay)
while iset>0:  # Run from maximum current value to 0
    iset,iactual,v1x,v1y,v2x,v2y = polling_cycle(iset)
    yield iset,iactual,v1x,v1y,v2x,v2y
    iset=iset-istep
    time.sleep(timedelay)

amrel.write("OUTP:REL:POL 1 REV")  # Reverse polarity to negative

while iset<imin:  # Run from 0 to minimum current value
    iset,iactual,v1x,v1y,v2x,v2y = polling_cycle(iset)
    yield iset,iactual,v1x,v1y,v2x,v2y
    iset=iset+istep
    time.sleep(timedelay)

def appender(values):
    iset,iactual,v1x,v1y,v2x,v2y = values
    field = iactual*290
    r1x = rext * v1x / (v1ref - v1x)
    r1y = rext * v1y / (v1ref - v1y)
    r2x = rext * v2x / (v2ref - v2x)
    r2y = rext * v2y / (v2ref - v2y)
    isetlist.append(iset)
    iactuallist.append(iactual)
    fieldlist.append(field)
    v1xlist.append(v1x)
    v1ylist.append(v1y)
    r1xlist.append(r1x)
    r1ylist.append(r1y)
    v2xlist.append(v2x)
    v2ylist.append(v2y)
    r2xlist.append(r2x)
    r2ylist.append(r2y)

    with open(filename,"a+b") as f:
        writer = csv.writer(f,dialect = "excel-tab")
        if li2 == 1:
            writer.writerow([iset,iactual,field,v1x,v1y,r1x,r1y,v2x,v2y,r2x,r2y])
        else:
            writer.writerow([iset,iactual,field,v1x,v1y,r1x,r1y])

    xmin,xmax = ax.get_xlim()
    ymin,ymax = ax.get_ylim()
    if x <= xmin:
        ax.set_xlim(xmin*1.05,xmax)
        ax.figure.canvas.draw()
    if x >= xmax:
        ax.set_xlim(xmin,xmax*1.05)
        ax.figure.canvas.draw()
if y <= ymin:
    ax.set_ylim(ymin*1.05,ymax)
    ax.figure.canvas.draw()
if y >= ymax:
    ax.set_ylim(ymin,ymax*1.05)
    ax.figure.canvas.draw()
line1.set_data(fieldlist,r1x) #whichever lists are being graphed go here
if li2 == 1:
    line2.set_data(fieldlist,r2x)
    try:
        return line1, line2,
    except:
        return line1,

ani = animation.FuncAnimation(fig,appender,gendata,blit = True,interval = 10,repeat = False)
plt.show()

In order to run the program, first make sure that all the equipment is turned on
Next, double-click on the icon on the desktop
The program will prompt the user for values. Make sure you use the appropriate units
(listed) along with the prompt
The program will append to the filename specified by the user in the file location
specified after “filename = “
When a data run is finished, be sure to close the graphs.
Pressing F5 will start the program for another data run.