Growth and Characterization of Magnetoresisitive Semiconductors

Honors Thesis

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Abstract

We have grown iron-doped indium antimonide (nominally 45% In, 45% Sb, 10% Fe) via RF magnetron sputtering. Analysis of the resulting samples by means of the van der Pauw method failed to detect any measurable magnetoresistance. However, detection of the Kerr effect, using a novel approach, indicates the presence of paramagnetic behavior in some films. This approach, if it can be refined, could prove to be a valuable and convenient tool for measuring magnetization.

Introduction

Objectives and Challenges¹

Magnetic semiconductors are a topic of particular interest, since such materials – if they can be made integrable with current semiconductor technology – could allow for significant advancements in the computing industry. The combination of semiconductors (currently used for information processing and communications) with magnetic materials (currently used for information recording) offers the possibility of storing and processing information simultaneously. Controlling the spin of charge carriers might also be a step on the way to quantum computing. Magnetic field-dependant resistance could be used in magnetic recording heads or field-actuator devices.

Although conventional semiconductors (those, such as Si and GaAs, that are used in everyday electronics) are nonmagnetic, there are types of magnetic semiconductors – europium chalcogenides and semiconducting spinels – that have been around for decades. These compounds, whose structures incorporate a periodic magnetic element, exhibit ferromagnetic behavior. The drawbacks, however, are that they are very difficult to grow and cannot be easily combined with conventional semiconductors, due to a very different crystal structure.

Rather than pure magnetic semiconductors, the answer may lie in diluted magnetic semiconductors (DMSs), which are actually regular, nonmagnetic semiconductors that have been doped with magnetic materials. Manganese doping of Group III-V semiconductors has been successful in producing compounds that exhibit ferromagnetic behavior at low temperatures; (Ga,Mn)As can have a magnetic transition temperature as high as 110 K. Obviously, much higher transition temperatures will have to be achieved before these substances can be used in everyday electronics. Alternatively, iron doping of GaAs has been successful in producing materials that exhibit superparamagnetic behavior even at room temperatures², a phenomenon that could be especially useful if it can be used to generate giant magnetoresistance (GMR). Large magnetoresistive effects have already been observed at low temperatures (-26% at 20 K) in $Zn_{0.80}Cr_{0.20}Te$, a ferromagnetic Cr-doped II-VI semiconductor.³

Iron-Doped Gallium Arsenide

GMR, a change in resistivity of 50 percent or more in magnetic fields, has been observed in magnetic superlattices and magnetic sandwiches (types of thin films with alternating layers of magnetic and nonmagnetic materials), as well as in granular metallic systems (Co-Cu, Fe-Cu, Co-Ag)⁴. Although the exact physics of the phenomenon is still being explored, the basic mechanism is that of spin-dependant scattering: charge carriers (electrons or holes) with spin parallel to the material's magnetization pass through virtually unaffected and therefore encounter a decreased resistance. (Charge carriers that are not aligned initially will become aligned after scattering, and thereafter also experience the lower

resistivity.) Competing with the magnetization, however, are thermal effects that will unalign the charge carriers' spins; for magnetization to effectively decrease resistance, the distance between magnetic elements (layers or grains, depending on the material) must be much smaller than the mean free path (Λ) between spin flip scattering events. If GMR can be achieved in Group III-V semiconductors at room temperature, not only will that allow direct integration of magnetoresistive elements into electronics, but it will also allow for greatly increased design variation, since the properties of semiconductors (such as charge carrier density and type) can be manipulated by changes in doping.

Iron-doped GaAs semiconductors have successfully been fabricated that exhibit paramagnetic behavior, although not GMR². Iron (1%) was implanted into commercial semi-insulating GaAs wafers (using an energy of 170 keV), and samples were then subjected to rapid thermal annealing. This induced the iron to migrate, forming ferromagnetic Fe₃GaAs clusters within the GaAs, the size of which were shown to increase with annealing temperature and duration (an example of Ostwald ripening). Larger cluster sizes produced correspondingly larger effective magnetic moments, until limited by the introduction of multiple domains per cluster. Cluster sizes of 1.4; 4.4; and 28 nm demonstrated effective magnetic moments of 240; 6000; and 10,000 Bohr magnetons, respectively.

GMR was not observed, however, because the samples were nonconducting. This effect was caused by the formation of Schottky barriers around the metallic Fe_3GaAs clusters, depleting the surrounding GaAs of its charge carriers.

To reduce this problem, subsequent work was done with In_{0.53}Ga_{0.47}As⁵. (Schottky barriers are due to band offsets, and In_{0.53}Ga_{0.47}As has a smaller band gap than GaAs - 0.74 eV versus 1.42 eV, at 300 K therefore reducing the amount of depletion.) The same technique (1% iron implantation at 170 keV, then thermal annealing) was used to embed 6.2 nm superparamagnetic clusters with an effective moment of 7000 bohr magnetons. Samples were successfully fabricated demonstrated that large negative magnetoresistance at low temperatures (3.2% at 5 K in .5 T). Small magnetoresistive effects (-0.1% in 1.5 T) were also present at temperatures as high as 175 K, but could only be discerned by comparisons with iron-free In_{0.53}Ga_{0.47}As (Figure 1).



Fig. 1: Subtracting the magnetoresistance of ironfree $In_{0.53}Ga_{0.47}As$ revealed the influence of paramagnetic clusters at 175 K⁵.

Indium Antimonide

InSb, like GaAs, is a Group III-V semiconductor (thus named because they contain one element from each of those two columns of the periodic table). It has an even smaller band gap than $In_{0.53}Ga_{0.47}As$, only .17 eV at 300K, which should result in even smaller Schottky barriers around metallic precipitates. This will hopefully allow for the formation of larger clusters while maintaining a satisfactory carrier density.

There is some question, however, as to what kind of metallic precipitates, if any, might be formed in InSb. Low-temperature molecular beam epitaxy



(MBE) has been used to grow $(In_{1-x_3}Mn_x)As$, in which the ferromagnetic impurity merely replaces the indium, up to x nearing 20%. As mentioned earlier, iron implanted in GaAs migrated to clusters of Fe₃GaAs, a hexagonal compound with lattice parameters such that it can fit well into the GaAs crystal structure⁶. There has been no such synthesis of a corresponding Fe₃InSb, however.

Indium antimonide, like all Group III-V semiconductors, has a zincblende structure (Figure $2a^7$); it has a lattice constant of 6.48 angstroms at 300K (compare to 5.65 for GaAs). If doped with iron, one possible candidate for cluster formation (given the elements present) is FeSb₂, a semiconductor with an extremely narrow bandgap, resulting in very high conductivity due to thermal excitation⁸. This compound has a Marcasite structure (Figure $2b^9$) with spacing in the 6 angstrom range, and is paramagnetic at room temperatures.

Whether iron impurities might migrate to form FeSb₂, or any other type of cluster, is yet to be determined. If so, the cluster size is likely to be tunable (as with Fe₃GaAs in GaAs). Should that be the case, the goal would then be to form clusters small enough to be single-domain, large enough to be easily magnetized by reasonable magnetic fields at room temperature, and close enough to one another to minimize spin flip scattering.

Growth

Each film was grown using RF magnetron sputtering, in which the target (a pressed pellet of powdered elements to be incorporated into the semiconductor, mixed at the appropriate stoichometry) is bombarded by an argon plasma. Atoms are knocked from the target and migrate to the substrate

positioned above it – here, sapphire (0001) – which encourages them to arrange in the appropriate fashion.

Sapphire (0001) has been shown to be an effective substrate material, on account of its mechanical strength and ability to produce epitaxial InSb (111) films. Although there is a four percent lattice mismatch, pure InSb (111) films grown on sapphire (0001) using molecular beam epitaxy have shown mobilities nearing that of the bulk material $(1x10^4 \text{ cm}^2/\text{Vs})$, as compared to the bulk value of $6x10^4 \text{ cm}^2/\text{Vs}$).¹⁰ InSb films doped with small percentages of Fe have also been grown epitaxially on sapphire (0001) via RF magnetron sputtering, yielding mobilities of 400-800 cm²/Vs.¹¹



Fig. 3: Sapphire has a hexagonal crystal structure. Its (0001) face has been shown to be compatible with the (111) face of InSb.

The sputtering target was prepared by mixing 6 g of powder with a $In_{.45}Sb_{.45}Fe_{.10}$ ratio (9.4561 g, 9.6224 g, and 6.9906 g, respectively), and pressing this on top of an older target. It is important to note that the films grown are unlikely to have the same stoichiometry as the target. The older target, which had had a $In_{.475}Sb_{.475}Fe_{.5}$ ratio, had been used to grow a film at 360 C that electron microprobe data found to have concentrations of 55% Sb, 44% In and 1.4% Fe.¹¹ One possible explanation for the discrepancy is the difference in melting points of the elements (156.61 C for In, 630.74 C for Sb, and 1535 C for Fe), causing the In to vaporize from the sample more readily. The iron, not being part of the regular InSb lattice, may not have been incorporated easily into the film, or may have precipitated out.

Before each sample was grown, the sputtering system was pumped down overnight (by means of the diffusion pump) with the substrate at a temperature around 125 °C to promote outgassing. The pressure typically decreased to values on the order of 10⁻⁶ torr. During sputtering, liquid nitrogen cooling above the diffusion pump was maintained, in order to decrease the partial pressure of any contaminating vapors (water, air, etc.). For one hour prior to growth, the substrate was additionally heated at least 500 °C, before being decreased to the growth temperature. At that point, argon flow was begun, and was regulated throughout at pressures of 5-10 mtorr. A power of 200-300 W was used to initiate the plasma, from which it was decreased to 50 W for the duration of film growth. For the first 15 minutes of growth, the substrate was protected by a shutter, to decrease incorporation into the film of any impurities that may have been absorbed onto the outermost layer of the target. After this, the shutter was moved aside and the target moved nearer to the substrate. Typical samples were grown for two hours, producing thicknesses of approximately .40 microns (measured by a crystal thickness monitor). Films were grown at a range of temperatures, from 196-306 °C.

Of the 10 films made with this target, seven seemed to have grown epitaxially, as they were characterized by mirror-like reflectivity, though three of these appeared slightly cloudy/smoky. The other three samples were light to medium gray and completely unreflective.

Resistivity Measurements

Because our samples are round, they do not lend themselves to the traditional six-lead Hall measurements. Instead, the ac van der Pauw technique (Figure 4) was used, in which four leads are attached to the sample arbitrarily (each approximately a quarter of the way around). By measuring two "resistances," rotated 90 degrees from one another, one can calculate the resistivity, if the sample thickness is known.¹² By applying a variable magnetic field perpendicular to the sample, the resistivity can be graphed with respect to this field. The mobility and carrier density are calculated from the resistivity, and from the Hall voltage of the van der Pauw measurement.

The results for the seven epitaxial films are summarized in Table 1. As is readily evident, a progressive decrease in the substrate temperature on subsequent films produced a steady increase in films' resistivity (and the corresponding trends in carrier density and mobility). Sample 24, though it appeared epitaxial, was completely nonconducting. We suspect this was caused by an air or water leak, since the pressure in the chamber would not decrease to its usual levels after this run; regreasing of all suspect areas brought pressures back to normal operating levels.

The graph of resistivity with respect to magnetic field exhibited a small slope (ranging from



Fig. 4: The van der Pauw technique involves three measurements (shown above), from which resistivity, carrier density and mobility can be calculated if the sample thickness is known.

| Sample | T (°C) | ρ (x10 ⁻³) | c.d. | μ |
|--------|---------|------------------------|----------------------|-----|
| 21 | 283-298 | 5.7 | 5x10 ¹⁸ | 200 |
| 24 | 280-281 | infinite | N/A | N/A |
| 25 | 295 | 8.9 | 5x10 ¹⁸ | 140 |
| 26 | 251-257 | 8.4 | $7x10^{18}$ | 106 |
| 27 | 235-237 | 10.5 | $7x10^{18}$ | 80 |
| 28 | 215-212 | 14.1 | 6.5×10^{18} | 70 |
| 29 | 196 | 36.1 | $7x10^{18}$ | 25 |

Table 1: Resistivity, carrier density and mobility were calculated using the ac van der Pauw method, with d = .40 microns.

0.1 to 0.5 % of the total resistivity for various samples, over the entire range of fields measured: from -.7 T to +.7 T). This slope continued linearly on either side of zero field, implying some kind of dependence

on the direction of the magnetic field applied. It was positive for some samples and negative for others. It was not always proportional for R_A and R_B of the same sample. For sample 26, it was even positive for R_A and negative for R_B (Figure 5).

This abnormal behavior turned out to be dependent on the placement of the leads, and as such, is probably a very small Hall effect due to the connections not being exactly opposite one another. Rotating the connections (without disturbing the attachment of the wires to the sample) produced a corresponding change in the graphs. RA and R_B switched upon the first 90 degree rotation, and were restored but with the opposite slope upon the second The change in slope (Figure 7). attributable steepness is to the downward drift the measurements experience over time (Figure 6).











Fig. 7: The strong slope (found in V_{12}/I_{34} and its analogues) is a fractional Hall effect due to misalignment, producing a 0.5% change in resistivity. The 0.1% change in other "resistance" of each pair is consistent with drift (see Figure 6).

This -0.1% drift over the standard measurement time is most likely caused by a slight increase in temperature of the sample, simply from the current being used to take the measurements. This is significant not only because it can produce the illusion of a magnetoresistive effect (particularly if one is not careful to check for correspondence between R_A and R_B before calculating ρ), but also because it means that a small real effect (such as the 0.1% change observed in $In_{0.53}Ga_{0.47}As$ at 175 K) would be masked entirely. Thus, if there are any magnetoresistive effects in these samples at room temperature, they are beneath the resolution of our instrumentation.

Magnetization Measurements

In order to measure the magnetization of our samples in the absence of a SQUID magnetometer, we have attempted to develop a technique based on the magneto-optical Kerr effect: the phenomenon in which polarized light, reflected off a magnetized surface, experiences a rotation of its polarization and a change in its ellipticity. There are three variations of the Kerr effect, one for each possible configuration of the incident light with the direction of magnetization of the material (Figure 8¹³).



Fig. 8: The polar Kerr effect (left) occurs when the magnetization is perpendicular to the surface of the material. The longitudinal Kerr effect (center) occurs when the magnetization is parallel to the surface of the material and to the plane of the incident light. The transverse Kerr effect (right) occurs when the magnetization is parallel to the surface of the material and perpendicular to the plane of incident light.

Both the part of the electric field polarized parallel to the angle of incidence and the part of the electric field polarized perpendicular to the angle of incidence will experience a rotation; however, these rotations are not necessarily equal (thereby resulting in the change in ellipticity). For example, in the study of a Cu/Co multilayer, they were found to be different everywhere except at normal and parallel incidence¹⁴. Thus, precisely what the Kerr Effect will produce in any given situation is dependent on the angle and polarization of the incident light, as well as the direction and degree of the magnetization. By controlling the first three variables, however, we seek to standardize a system for measuring the last.

Set-up

Rather than measuring the change in polarization of a single beam, we have measured instead the change in phase difference between a right circularly polarized and a left circularly polarized beam, relative to a reference detector. This particular set-up (Figure 9) has been used very successfully for detecting the denaturation of collagen, a chiral molecule the exhibits different indices of refraction for left- and right-handed circularly polarized light (until it denatures).¹⁵



Fig. 9: Few modifications were required to adapt the apparatus's function from detection of collagen denaturation (its original purpose) to measurement of semiconductor magnetization. Essentially, the film was put into the path of the beam in place of the collagen cuvette, and the measurement detector was realigned appropriately.

The angle of the incident beam is easily adjustable (within the limitation imposed by the structure of the magnet in which it is positioned). Since the magnitude and ellipticity of the Kerr Effect are angledependent, this enabled us to investigated whether alternate arrangements would produce a stronger signal.

Data acquisition was run through Lab View.

Results

In response to polar Kerr measurements (those discussed here were taken at an incident angle of 42 degrees), our reflecting, 10% iron samples produced a wide variety of responses, some more easily interpreted than others. Throughout, a record of the magnet current was cross-referenced with scan times, since signals would not be distinguishable from the ambient noise without such a guide (Figure 10).



Fig. 10: Left: Application of a negative current through the magnet (corresponding to an applied field of approximately 6000 G) produced a negative "peak" during scans 17455-17485. *Right*: An applied field of -5600 G corresponds to the positive "peak" located from 6750-6780 scans. Abrupt ons and offs were used, rather than a ramped current (as had been originally envisioned), in order to more clearly discern any polarization changes.

Samples 28 and 29 were generally well-behaved. They responded to decreases in applied field with increases in phase, and vice versa (an example of each is shown in Figure 10). When the applied field was turned off, the phase returned to its original position, and changes seemed to be roughly related to the strength of the field in question. A detailed graph of phase change amplitude vs. applied field amplitude is shown in Figure 11 (positive and negative peaks were combined on the same graph, due to the uniform behavior). Unfortunately, the constant fluctuations make low-field measurements nearly undetectable, and even at high fields it was difficult to assign peak heights precisely. Averaging produces

a very broad range of results (such as the four vastly different peak heights for sample 29 near 6000 G, despite those peaks appearing to be fairly consistent in the raw data), but the unpredictability of the noise leaves few other alternatives. Nevertheless, it is still reasonable to conclude that these two samples exhibited paramagnetic behavior, which could be indicative of magnetic clusters.



Fig. 11: In these data, about 10 scans on either side of the peak were averaged as a baseline, and subtracted from the average peak height.

Sample 27, however, was more difficult to interpret. First, peak heights did not increase noticeably with larger applied fields, though this could merely be attributable to early saturation. More curiously, this film had the habit of reacting with a sharp positive peak every time the applied field was turned off (Fig. 12). Furthermore, unlike samples 28 and 29, it responded identically to applied fields in opposite directions.

Similar behavior was observed in sample 24, though with additional complications.





Fig. 12: Green "down" arrows indicate when the magnetic field was turned on, red "up" arrows when it was turned off.

Although this film consistently responded with that same sharp positive peak when the applied field was turned off, occasionally it would also respond with a sharp negative peak when the applied field was turned on, but then return to its original phase (while the magnetic field was still on), showing no net displacement. Furthermore, this happened repeatedly with the stronger applied fields; some of this sample's larger peaks were produced by fields around 2000 G, while fields of 4000-7000 G produced no reaction at all. Last but not least, although sample 24 initially responded similarly to both positive and negative applied fields (as sample 27 had), it later switched and began responding to large negative fields (around -6000 G) with abnormally large positive phase displacements (.04-.06 degrees). Recall, however, that this sample was nonconducting, making its structural properties questionable.



Fig. 13: A thin film iron sample showed polarization displacements proportional to the applied field for a series of positive field measurements (scans 550-975, graphed in inset). Whenever magnetization was applied in a "new" direction, however, the displacements were abnormally large (A,C) and/or in the "wrong" direction (B,C).

Standardization

As a comparison, we decided to try our method on a sample with more predicable magnetization: a simple iron film. For "thin films" such as this one (technically, anything between 10 monolayers to several thousand angstroms), magnetization at zero field is in-plane, and the polar Kerr effect (which pulls the magnetization out of the field) produces a paramagnetic response up to the saturation field of 2.2 T. We were, indeed, able to detect and measure this behavior, but also encountered some more complicated phenomena (Figure 13).

The first time it was magnetized, the iron film showed an unusually large displacement when the field was turned off (peak A). After this, it responded linearly to applied fields (Figure 13 inset), as expected. Application of negative fields, however, generated two initial responses, in which the displacement was not only still negative, but was also displaced in the same direction both when the field was turned on and when it was turned off (Figure 13, B and C). Subsequently, continued application of negative fields produced positive peaks mirroring the negative peaks produced by the positive fields (the first of these can be seen in Figure 13, at scan 1470). Similarly, switching back to positive fields generated the same uni-directional displacements (in the positive direction) both when the field was turned on and off.

The most likely explanation for this behavior would be that an applied field into the plane somehow favors a different in-plane magnetization than does an applied field out of the plane, and we are detecting a longitudinal or transverse Kerr effect at the same time as the polar Kerr effect. (The magnet we are using has a remnant magnetization of under 50 G; this in itself could possibly have accounted for a small displacement at zero field, if it had been in the opposite direction.) However, attempts to measure the longitudinal Kerr effect itself (see Figure 9) were unsuccessful; applied magnetic fields up to 6500 G in either direction produced no detectable change in phase. Our set-up currently does not have the capability to measure the transverse Kerr effect, due to the geometry of the magnet.

Method Feasibility

One significant deterrent to this method, thus far, has been the struggle to detect the signal above the noise of the system. Because we are measuring such small changes in phase (and of light, no less), the signal is noticeably affected even by variations in temperature and air currents through the room; electrical noise (from ambient radiation, and/or disturbing wires attached to the system) is also an issue. A number of modifications have been made to reduce the amount of noise, including the addition of terminators to any empty plugs attached to the system, the proper grounding of the detectors, the enclosure of the system in a foam box, and the taking of data in the middle of the night. Although some of these seem to have improved the signal slightly, it remains to be the case that it is too noisy to collect data at all 70-90 percent of the time (Figure 14). Even during quiet periods, measurements at low field are difficult with more responsive samples and impossible for less responsive ones, as the background noise alone is on the order of the signals for which we are looking (Figure 15).

A smooth surface is also extremely important for a clear signal (Figure 16). Surface imperfections on the order of nanometers are enough to scatter the reflected beam with greater variations in path length than the change in polarization for which we are looking. This was why comprehensive data could not be gathered for samples 25 and 26. A few measurements were managed with sample 25, showing a small response (.01 at 6300 G), similar to that observed for samples 28 and 29. No measurements at all were possible for sample 26.



Fig. 14: Of the 31 minutes of scan time shown, there were three in which it was possible to take usable data. (The scan rate is .667/s.) The oscillatory behavior is most likely due to temperature variations.



Fig. 15: Even when the measurement detector is receiving no signal at all, the electrical noise causes both a drift and a .005-.01 degree vibration in the reported signal.



Fig. 16: A sanded steel surface produced a much "fuzzier" signal than did a sputtered InSb sample (doped with 5% iron) under identical conditions.

Conclusions

The samples we have grown from the 10 percent iron target do not exhibit any magnetoresistive behavior at room temperature, and show decreasing resistivity as a function of growth temperature. Magnetic measurements indicate the presence of paramagnetic behavior in some samples. This response, however, is



Fig. 17: Sample 5 (nominally 5% iron, grown at 315° C, $\rho=9x10^{-3}$) exhibited a near-normal polar Kerr rotation of .025 degrees.

weaker than that seen for older samples with lower iron concentrations (Figure 17). Other samples exhibited consistent magnetic responses that were not paramagnetic; this behavior can not yet be clearly defined, due to its complexity and in the absence of a complete understanding of how magnetization changes will affect the measured signal.

The adoption of the existing laser set-up to detect the Kerr effect has indeed been successful in detecting magnetization of samples. However, much more work is needed before this can be a viable measurement method. Unexpected results from the iron film alone indicate that further investigation must be made into how, exactly, Kerr rotation in different configurations (polar, longitudinal, and transverse) and at different incident angles affects the phase change measured by the system. Perhaps experimental measurements with a material whose magnetization is well documented would be in order, and/or a theoretical analysis of how various changes in rotation and ellipticity might be "perceived" by the phase meter.

The former approach would also be helpful in standardizing the paramagnetic behavior (which we believe to have clearly identified), so that an observed change in phase can be translated into a known magnetization. This, in turn, could then be correlated to cluster size in the proposed model and compared to samples' growth temperature and iron concentration, towards the ultimate goal of fabricating InSb with appropriately sized and spaced clusters to produce giant magnetoresistance.

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