FERROMAGNETIC AND EXCHANGE BIAS STUDIES IN THE DILUTED MAGNETIC SEMICONDUCTOR (GA,MN)AS

A Thesis in Materials
by
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ABSTRACT

In this dissertation, the ferromagnetic properties of the diluted magnetic semiconductor (Ga,Mn)As will be explored. The aim of this research is to characterize these properties using a wide variety of methods, then to explore their utility in the effect of exchange bias.

We begin this dissertation by discussing the theoretical explanations as to why (Ga,Mn)As becomes ferromagnetic after the inclusion of Mn atoms into the GaAs lattice structure. Previous experiments detailing the work done in annealing will be explained, as well as the material properties of the dilute magnetic semiconductor. The dissertation will then deal with the magneto-optical Kerr effect (MOKE), an important tool in characterizing ferromagnetic materials. MOKE can be used to determine the Curie temperature of (Ga,Mn)As, and the experimental set-up for this will be shown. Once the case of generalized MOKE is explained, the dissertation will delve into a specific type of MOKE experiment, that of component-resolved MOKE. Component-resolved MOKE is an elegant experiment that can be particularly useful for materials undergoing a phase transition in magnetic anisotropy. It will be shown that tensile-strained (Ga,Mn)As undergoes such a transition, and the effect that annealing has on this transition will be demonstrated. The dissertation will then discuss the origin of the exchange bias effect, and how it relates to (Ga,Mn)As. Data will then be shown demonstrating that (Ga,Mn)As has been successfully exchange-biased, the first semiconductor to be done so. The various experiments exploring the chemical and structural properties of the MnO layer
that exchange biases (Ga,Mn)As will be shown. Finally, the dissertation will finish with possible directions in which the MOKE and exchange bias experiments can be pursued.
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List of Abbreviations

AFM: Anti-ferromagnet

DMS: Diluted magnetic semiconductor

EDS: Energy dispersion spectrometry

EPR: Electron paramagnetic resonance

FM: Ferromagnet

FR: Ferromagnetic resonance

GMR: Giant magnetoresistance

H_C: Coercive field

H_E: Exchange field

HH: Heavy hole

IB: Impurity band

LH: Light hole

MBE: Molecular beam epitaxy

MCD: Magnetic circular dichroism

MIR: Midinfrared spectroscopy

MOCVD: Metal organic chemical vapor deposition

MOKE: Magneto-optical Kerr effect

PEM: Photoelastic modulator
PHE: Planar Hall effect
RBS: Rutherford back scattering
RHEED: Reflection high energy electron diffraction
SQUID: Superconducting quantum interference device
T_B: Blocking temperature
T_C: Curie temperature
T_N: Neel temperature
TEM: Transmission electron microscopy
VB: Valence band
XPS: X-ray photoelectron spectroscopy
XRD: X-ray diffraction
XRR: X-ray reflectometry
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Chapter 1

Introduction to (Ga,Mn)As and Exchange Bias

This chapter serves as an introduction to the diluted magnetic semiconductor (DMS), (Ga,Mn)As, as well as providing the layout of the thesis. The material structure of (Ga,Mn)As is given, the history surrounding its study, and the current theory of the origin of the ferromagnetism in the material is discussed. Furthermore, the concept of exchange bias will be introduced, and the evidence of the exchange biasing of (Ga,Mn)As will be shown. Finally, the goals of our research will be explained.

1.1 Review of (Ga,Mn)As

Diluted magnetic semiconductors (DMS’s), of which (Ga,Mn)As is an example, provide a potentially new avenue for electronics. The interest in DMS’s comes from the fact that they are both semiconductors and ferromagnetic. Traditional electronics uses the flow of charge carriers in the device to carry information. By incorporating magnetic elements into the semiconductor, however, the quantum nature of the spin of these charges (up or down) can be manipulated can be manipulated as well. Since the field incorporates the spin of the charges into electronics, it has been given the name of spintronics [1]. At its heart, spintronics relies on spin-polarized current flowing through
a semiconductor, which is then either detected or manipulated to perform an operation.

Towards the first goal of spintronics research, several methods have already been shown to be successful in generating a spin-polarized current. Among them are using quantum mechanical selection rules to transfer the photon’s angular momentum to the electron spin angular momentum [2]. Also shown to be successful in generating spin-polarized currents are spin injection from ferromagnetic contacts [3]. Finally, in order to generate spin-based phenomenon we can incorporate magnetic atoms into the lattice of a semiconductor, making the system into a DMS [4].

Several devices have already been constructed, in a proof-of-concept manner: magnetic tunnel junctions, spin-dependent resonant tunneling diodes and spin-polarized light-emitting diodes [5-11]. Another device of interest is that of a magnetic bipolar transistor, allowing the transfer of information from magnetic media to conventional semiconductor devices [12].

Since (Ga,Mn)As is based upon GaAs, it has the advantages that GaAs possesses: compared to silicon, GaAs has a higher electron mobility and breakdown voltage, is a direct bandgap material (silicon is an indirect bandgap material), and generates less noise when operated at higher frequencies. Due to these advantages, it has been extensively studied, and has already found use in technological applications such as mobile phones and satellite systems. Besides the advantages that GaAs has, the inclusion of Mn into the lattice structure makes the system ferromagnetic, hence making it attractive for potential new devices. However, the current Curie temperature, or $T_c$, of ~170 K limits the practical uses any devices constructed with (Ga,Mn)As in the heterostructures [13].
While there are hopes that the $T_c$ can be raised up to a suitable level for practical use, it remains to be seen whether this will come to pass.

Since the physics of III-V materials is already well-studied and incorporated into modern electronics applications, when the first successful growth of ferromagnetic (In,Mn)As was shown in 1992, it created a great deal of interest [14]. The first initial challenge was strictly that of growth. It is necessary to grow (III,Mn)As via molecular beam epitaxy (MBE), as opposed to metal organic chemical vapor deposition (MOCVD), because the former allows for low-temperature growth. Since under equilibrium conditions, Mn will only incorporate itself into III-As to $\sim 1\%$ without surface segregation and phase separation occurring, it is necessary to use a non-equilibrium technique in conjunction with the low-temperature growth. The implementation of this technique solved the first problem in creating ferromagnetic (In,Mn)As, that of increasing the Mn content to the point at which ferromagnetism occurs (beyond $\sim 1\%$). The reason that low-temperature growth is necessary is that the (III,Mn)As will phase segregate into sections of III-As mixed with small clusters of MnAs precipitates. As MnAs is also ferromagnetic, this has the result of affecting the overall magnetism of the structure.

After the successful growth of ferromagnetic (In,Mn)As, this was followed up in 1997 by the growth of (Ga,Mn)As with a Curie temperature of $\sim 50$ K [15]. This was followed shortly by an increase in $T_c$ to $\sim 110$ K in the following year [16]. Following the discovery of the effect of low-temperature annealing (to be discussed further on), this has reached as high as $\sim 170$ K [13]. Recent works performed on GaAs heterostructures with Mn $\delta$-doping, in particular the work done by Tanaka et al., claim to demonstrate a Curie temperature as high as 250 K [17 – 20]. Mn $\delta$-doping refers to inserting approximately a
monolayer (ML) of Mn into the heterostructure, thus achieving a $\delta$-like profile. In the work performed by Tanaka et al., two samples were grown: in the first, a GaAs layer with a Mn $\delta$-doping content of 0.5 ML was grown on top of a $p$-type Be-doped Al$_{0.5}$Ga$_{0.5}$As layer. In the second, a GaAs layer with a Mn $\delta$-doping content of 0.6 ML was grown below a $p$-type Be-doped Al$_{0.3}$Ga$_{0.7}$As layer. Since the magnetic content of the samples too small for a bulk magnetization method such as a superconducting quantum interference device (SQUID), the anomalous Hall effect was used to study the two samples. The authors claim a $T_c$ of 190 K for the first sample, and a $T_c$ of 250 K for the second sample, both well above the previous high of $\sim$170 K.

1.1.1 Material Characteristics

We now focus on the material characteristics of (Ga,Mn)As. The material is formed by the inclusion of Mn into the GaAs lattice structure, and thus shares most of the same material characteristics as GaAs. Both are direct band gap materials, i.e, there is no phonon needed for emission of light, an advantage that GaAs has over Si-based structures. (Ga,Mn)As retains the zinc-blende structure of GaAs, as shown in Figure 1.1. It should be noted that the strain in the film is determined by the substrate used: to achieve compressive strain, the film is grown on a GaAs substrate, whereas for tensile strain the film is grown on (In,Ga)As that has been allowed to relax to its own lattice constant.
Figure 1.1: Representation of the lattice structure of (Ga,Mn)As, where a Mn atom has substituted into a Ga site.

Ga has an atomic structure of \([\text{Ar}]3d^{10}4s^2p^1\), Mn is \([\text{Ar}]3d^54s^2\), and As has a structure of \([\text{Ar}]\ 3d^{10}4s^2p^3\), which leads to the conclusion that the natural substitution of Mn into the GaAs lattice structure would be to substitute into a Ga site. When the Mn substitutes in this manner, it acts as an acceptor, as well as providing a localized spin of \(S = 5/2\). The hole will be weakly bound, and since the Mn will then be in the \(d^5\) state it will possess zero angular momentum. These assumptions have been confirmed through the use of electron paramagnetic resonance (EPR) and ferromagnetic resonance (FR) experiments \([21, 22]\). Direct evidence of the substitutional Mn providing a hole was given by a scanning tunneling microscopy experiment performed by Yakunin, et al. \([23 - 25]\). In the experiment, a single impurity Mn atom was switched from the ionized and
neutral states (neutral being that of Mn in the d$^5$ configuration with a weakly localized hole) by applying a voltage with the STM tip. This voltage corresponds to a binding energy of the hole of ~0.1 meV. This binding energy, $E_b$, is also inferred through the use of infrared spectroscopy and photoluminescence experiments, which placed the energy at $E_b = .1124$ eV [26 - 29]. The value of the binding energy plays an important role in determining the strength of the $p$-$d$ exchange, which as we will see, plays an essential role in the ferromagnetism of the system.

As previously mentioned, it is necessary to grow (Ga,Mn)As via low-temperature MBE. Low temperature MBE growth, while necessary, results in a high density of point defects. These point defects are the following:

A) The first type of defect is an As antisite defect. In this defect, As occupies Ga sites in the lattice, becoming $\text{As}_{Ga}$. These act as deep-level donors, compensating the holes provided by the substitutional Mn. $\text{As}_{Ga}$ defects form because at the low growth temperatures of (Ga,Mn), As has a higher sticking coefficient than for the usual GaAs growth temperature of approximately 600 $^\circ$C [30 – 32].

B) The second type of defect is that of interstitial Mn, $\text{Mn}_I$. In this case, Mn does not substitute into the Ga site, and instead occupies an interstitial position in the lattice. Here, it also acts as a double donor, further compensating the holes provided for by the $\text{Mn}_{Ga}$ [33]. $\text{Mn}_I$ acts as a double donor, because it is a divalent atom occupying an interstitial position in the lattice.
Earlier studies have shown that the hole concentration plays a very important role in determining the Curie temperature of the film (see e.g., figure 1.2). Therefore, the control of both types of compensating defects is essential in creating robust ferromagnetism in (Ga,Mn)As [14].

Figure 1.2: Reprint from reference 34. Curie temperatures for a series of (Ga,Mn)As samples with a range of Mn concentration from 2% to 8.5%, and thicknesses ranging from 300 to 1200 nm. The data shows an empirical relationship of $T_c \approx \rho^{1/3}$, note however that mean-field theory predicts a relationship of $T_c \approx \chi \rho^{1/3}$. 
For the first type of defect, a heat treatment solution is unavailable, as the As antisites are chemically stable until ~500 °C [35]. Any heat treatment that would displace the As from the Ga sites would also form clusters of MnAs, something that is to be avoided. It has been shown that these defects can reach a density of up to $10^{20}$ cm$^{-3}$, which corresponds to ~1% of the Ga sites [36]. It has been suggested by Campion et al., that by using As$_2$ dimers instead of As$_4$ trimers and maintaining a strictly stoichiometric growth mode, these defects can be reduced [37]. A study of the effect of As$_{Ga}$ on (Ga,Mn)As was performed by Myers, et al. [38]. In the study, the As:Ga flux ratio during MBE growth is smoothly varied within individual samples through growth without substrate rotation. The hole densities and Curie temperatures of the samples were then measured, and it was shown that the hole concentrations can vary by up to two orders of magnitude, depending on the As:Ga flux ratio. In the As-rich films, the excess As produced samples with hole concentrations as low as ~$10^{17}$ cm$^{-3}$, whereas in the Ga-rich samples, the excess Ga suppressed the substitution of Mn into the lattice, also resulting in low hole concentrations. Since the As:Ga flux ratio is a controllable variable during growth, the results are very useful in increasing the $T_c$ of (Ga,Mn)As.

After the Curie temperature of (Ga,Mn)As was increased from ~50 to ~110 K, it became apparent that there existed metastable defects in the system that could be manipulated, and it was theorized that the defects were Mn occupying interstitial sites in the lattice. Direct evidence of this came from an experiment that combined Rutherford back-scattering and particle-induced x-ray emission measurements [39]. The experiment was able to distinguish between interstitial and substitutional Mn, and showed that for as-
grown samples exhibiting robust ferromagnetism up to 20% of the Mn existed in an interstitial state.

It is now known that Mn\textsubscript{i} defects can be reduced by post-growth annealing, at temperatures ranging from 190 to 250 °C (see for instance figure 1.3) [18 - 20, 40 - 42].

It has been shown that post-growth annealing the sample will increase the Curie temperature, up to as high as 173 K [13]. Ion channeling experiments have shown that annealing causes a decrease in the concentration of the interstitial Mn. The model proposed by Edmonds \textit{et al.} [18], is that interstitial Mn out-diffuses towards a free surface during the annealing process. Once at the free surface, they are passivated by oxygen. Since there are fewer interstitial Mn compensating the donors provided by the substitutional Mn, this has the effect of enhancing the ferromagnetism.
Experiments performed on (Ga,Mn)As layers capped with undoped GaAs add evidence to the free surface model proposed [42]. The (Ga,Mn)As layers were capped with a layer of GaAs of varying thickness. For all of the layers capped in this manner the beneficial effect of annealing on the samples was suppressed. However, when additional free surface was made available by lateral patterning, the annealing effect was restored.

It is currently hypothesized that capping the (Ga,Mn)As layer with GaAs prevents an increase in annealing because Mn interstitials diffusing into the GaAs layer act as donors, creating a p-n junction. The p-n junction creates a repulsive Coulomb barrier, and it is this barrier that limits the diffusion of Mn into the capping layer [18].
1.1.2 Ferromagnetism in (Ga,Mn)As

There are several competing theories regarding the origin of the ferromagnetism in (Ga,Mn)As. In this section, we will attempt a general overview of these, and discuss the controversies involved.

Before delving into the specifics of (Ga,Mn)As, we need to discuss the mechanics of exchange interactions. Zener’s kinetic-exchange or the indirect-exchange interaction was proposed in 1951 [43]. In this model, there will exist a local moment, typically in the \( f \)-shell or \( d \)-shell (for the case of (Ga,Mn)As, it is in the \( d \)-shell). The coupling between these moments is mediated by \( s \) or \( p \)-band itinerant carriers. The moments can have either a ferromagnetic direct exchange between the band electrons on the same site, or an antiferromagnetic exchange due to hybridization between the local moment and the band electrons on the neighboring site [4, 44]. The polarization of the magnetic electrons is then propagated to neighboring sites.

Zener also proposed another model known as “double-exchange” [45]. In this theory, the interaction occurs between two isolated magnetic atoms with different number of electrons in the magnetic shell, and hopping occurs between them through an intermediate, non-magnetic atom. Due to Hund’s rule, this couples the two magnetic moments ferromagnetically.

At very low concentrations of Mn, the average distance between the Mn moments is much larger than the radius of the bound hole. It has been shown experimentally that
the onset of ferromagnetism in (Ga,Mn)As occurs when the Mn concentration reaches
~1% [46 – 48], with the system near the Mott insulator-to-metal transition point (which is
when the average distance between the Mn impurities is approximately equal to the
effectivework Bohr radius of the impurity). At this point, the localization length of the
impurity band is extended such that it allows them to mediate the ferromagnetic exchange
between the local moments. Therefore, the exchange interaction in question would be
that of double-exchange.

The controversy over the ferromagnetism arises when the Mn concentrations
reach a higher level. There are two competing views for what occurs. The first such
view is that of the mean-field theory [49]. Since it is a mean-field theory (and thus deals
with an average), the theory can not account for any sort of positional distribution in the
(Ga,Mn)As layer. Furthermore, the theory’s limits break down under samples with high
carrier concentrations, where the mean distance between the carriers is approximately
equal to the mean distance between the spins. That being said, the model has correctly
predicted trends in $T_C$ as well as strain-dependent magnetic characteristics (discussed
further in Chapter 3).

Before going into the theory, it is necessary to briefly explain $p$-$d$ exchange, as it
plays an extremely important part in the ferromagnetism. $P$-$d$ exchange arises from the
fact that the occupied d-band in the Mn atom overlaps with the valence band of GaAs. It
is very similar to the $p$-$d$ exchange in II-VI semiconductors, except here the Mn is acting
as both a dopant and a local moment [49]. This $p$-$d$ exchange interaction results in a
spin-dependent coupling between the holes and the $\text{Mn}_{\text{Ga}}$, and is given by:
\[ H_{pd} = -\beta N_o s \cdot S, \]  

where \( \beta \) is the \( p-d \) exchange integral and \( N_o \) is the concentration of the cation sites. This exchange interaction is anti-ferromagnetic in nature. Besides the antiferromagnetic interaction between the carriers and the localized spin, the \( p-d \) exchange also results in a short-range antiferromagnetic coupling between the Mn spins known as superexchange. While this dominates in II-VI undoped semiconductors, it is effectively neglected in uncompensated (Ga,Mn)As [4, 49].

A more heuristic picture of the mean-field theory is as follows: what the model suggests is that the holes mediate the ferromagnetic interaction between the localized spins. As mentioned before, the holes and the localized spins are antiferromagnetically coupled through the \( p-d \) exchange. As shown in Figure 1.4, one Mn spin will antiferromagnetically couple to a weakly localized hole. Meanwhile, another Mn spin will also be coupled to the hole. Since both Mn spins are antiferromagnetically coupled to the hole, this produces a ferromagnetic coupling between them, and is the origin of the ferromagnetism in (Ga,Mn)As, according to the theory.
Figure 1.4: Coupling between the localized Mn spin of 5/2 and the hole produces a ferromagnetic coupling between the two Mn spins.

The model calculates the free energy contribution from the holes by diagonalizing the 6x6 Kohn-Luttinger k·p while including the contribution from the p-d exchange. The Hamiltonian for the mean-field model takes the following form:

\[ H = H_{\text{holes}} + J_{pd} \sum_{i,j} S_i \cdot s_j \delta(r_i - R_j), \]  

where the first term includes the k·p Kohn-Luttinger Hamiltonian and the interaction of holes with other holes and random disorder potentials [50]. The second term in the equation is for the p-d exchange interaction between the localized spins (S_i) and holes (s_j). J_{pd} is an exchange constant, and from photoemission data is inferred to be 54±9 meV nm^3 [51]. Since the mean-field model uses the experimental value for J_{pd}, it correctly
identifies the strength of the magnetic interaction. Since it includes the spin-orbit interaction, it also takes into account the strong spin-orbit interaction in the valence band.

The model is a mean-field model, and as such, each local moment is described by a Hamiltonian of $H = S_I \cdot H_{MF}$. Here, $S_I$ is the local Mn spin operator, and $H_{MF}$ is the effective field experienced by the local moments from the spin polarization of the band holes, and can also be expressed as $H_{MF} = J_{pd} \langle s \rangle$, where $\langle s \rangle$ is the mean spin density of the valence band holes [50]. This leads to the following temperature and field dependence:

$$\langle S \rangle = \frac{H_{MF}}{|H_{MF}|} S B_s \left( \frac{S |H_{MF}|}{k_B T} \right),$$  

where $B_s$ is given by the following upon linearizing about $\langle S \rangle = 0$:

$$B_s = \frac{S + 1}{3} \frac{S |H_{MF}|}{k_B T_c}.$$  

Upon rearranging this and putting it in terms of the itinerant-hole spin susceptibility, $\chi_f$ = $\frac{d \langle s \rangle}{dh_{MF}}$, where $h_{MF}$ is the local field experienced by the valence band holes, we then reach the predicted formula for the Curie temperature:

$$T_c = \frac{N_{Mn} S (S + 1)}{3} J_{pd}^2 \chi_f,$$
where $N_{\text{Mn}}$ is the density of local Mn moments [50].

The mean-field theory treats the holes as itinerant, and the exchange, as noted above, as mediated by the $p$-$d$ exchange, and is therefore based upon Zener’s indirect-exchange interaction. However, there is evidence that this may not be the correct picture.

Recent experiments show evidence that the impurity band does not merge into the valence band with increasing Mn concentration, but instead persists throughout [52]. An impurity band is formed in a semiconductor through doping. As the semiconductor is doped with acceptors, as for (Ga,Mn)As, energy states close to the valence band are formed. As the concentration of dopants is increased, the individual donor states merge into an energy band, thus forming the impurity band. The experiment measured the band structure of (Ga,Mn)As across the phase diagram using infrared and optical spectroscopy. The result of the experiment was strong evidence that the Fermi energy resides in the Mn-induced impurity band. Furthermore, they find the effective mass to be on the order of $10m_e$, whereas for $p$-type GaAs with the Fermi energy lying in the conduction band, the effective mass is $0.38m_e$ [53]. This leads to an optical mass, $m_{\text{opt}}$, between 0.7 and 1.4 $m_e$. This is much larger than the value of 0.24 to 0.27 $m_e$ expected assuming that the Fermi energy lies in the valence band [54]. Finally, the experiment provides a reason for the low mobility seen in samples with robust ferromagnetism (as low as $1 – 5 \text{ cm}^2/\text{V s}$), since the effective mass is much higher than seen in GaAs.

There exists a competing theory that suggests that Zener’s indirect-exchange interaction is not the correct picture, but a modified form of Zener’s double-exchange interaction. In this modified form, the Mn impurities form an impurity band with mixed
character, and the electrical conduction and Mn-Mn coupling are mediated by hole hopping through the impurity band [55 – 62]. In this viewpoint, the Hamiltonian takes the following general form, instead of the one previously discussed:

\[ H = -t \sum_{\langle ij \rangle, \sigma} \hat{c}_i^{\dagger} \hat{c}_j + J \sum_I S_I \cdot \sigma_I, \]

where \( \hat{c}_i^{\dagger} \) creates a hole at site \( i \) with spin \( \sigma \), \( \sigma_I \) is a hole spin operator, \( \sigma_I = \hat{c}_I^{\dagger} \sigma_{a\beta} \hat{c}_I \).

Shown in Figure 1.5, we see a band energy schematic detailing the difference between the two scenarios of impurity band versus mean-field theory. In the experiment, the resonance that is observed by the midinfrared spectroscopy (MIR) can occur from either transitions from the light hole (LH) to heavy hole (HH) bands, which is consistent with the mean-field picture of the IB having been absorbed, or from transitions between the MnGa impurity band (IB) and valence band (VB), consistent with the impurity band view. However, as the hole concentrations increase, the two viewpoints differ, as shown in figures 1.5(b) and 1.5(d). With increasing hole concentration, the Fermi energy, \( E_F \), which resides in the VB will shift further into the VB. Therefore, the optical transition observed by MIR will occur at higher energies as the hole concentration increases. In the competing viewpoint, the \( E_F \) will reside in the IB. As the hole concentration increases, the Coulomb attraction between the holes and the impurities, which determines the separation of the IB and VB, will be screened. Therefore, with increasing hole concentration, the optical transition observed by MIR will occur at lower energies. Since
the experiment reveals that with increasing hole concentration, the optical transition red-shifts this provides evidence of the IB picture being correct.

Figure 1.5: Reprinted from reference 52. Band energy schematic for the two competing viewpoints of the origin of the ferromagnetism in (Ga,Mn)As. In a) and b) the transition occurs between the LH and HH bands, whereas for c) and d) the transition occurs between the IB and VB.

This experiment strongly suggests that the mean-field model’s interpretation of the origin of the ferromagnetism is incorrect. However, it has been argued that the MIR transitions that occur are the result of intra-band transitions within the valence band [41]. If the results hold up to further study, the view of the origins of the ferromagnetism must
be changed. At the very least, the idea that the impurity band disappears into the valence band with increasing Mn concentration must be changed. However, that is not to say that the mean-field theory needs to be discarded entirely. It has provided accurate predictions for the Curie temperature in a range of materials, and has successfully predicted the magnetic anisotropy behavior as a function of temperature and carrier concentration. Furthermore, the incorporation of Mn into GaAs enhances the magnetic circular dichroism (MCD) near the band-gap frequencies in such a way that the sign of the enhancement is consistent with the theory that assumes antiferromagnetic $p$-$d$ exchange coupling [64 - 65]. Closer study of this issue is clearly warranted.

1.2 Exchange Bias

Having discussed the current understanding of the ferromagnetism in (Ga,Mn)As, we now switch our focus to the exchange biasing of (Ga,Mn)As. In this section, we discuss the basic theory behind the exchange bias phenomenon and the successful exchange biasing of the semiconductor (Ga,Mn)As (the first demonstration of exchange bias in an antiferromagnet/semiconductor system) by growing an antiferromagnetic oxide layer of $\text{Mn}_x\text{O}_y$. These experiments were carried out by Eid et al. [66, 67]. In this dissertation, we discuss the material and chemical characteristics of these exchange-bias structures. As will be shown in chapter 4, the oxide will be found to be of the species MnO. Finally, we provide an overview of attempts to standardize and improve the
strength of the exchange bias in the system by systemically annealing the samples in an oxygen environment.

We note that there were earlier (unsuccessful) attempts to exchange bias (Ga,Mn)As using a MnTe or a (Zn,Mn)Se overlayer, performed by Furdyna, et al. [68]. In these experiments, four types of samples were grown and measured via SQUID: a regular (Ga,Mn)As film with no capping layer, a (Ga,Mn)As/MnTe film, and a (Ga,Mn)As/GaAs/MnTe film (where the GaAs buffer layer varied in thickness), and a (Ga,Mn)As/(Zn,Mn)Se film. While the samples do show an increase in coercive field strength upon the addition of the overlayer, which has been shown to occur in exchange-biased systems, there was never any evidence of an exchange field shown. Although the paper concludes that this is likely due to the ferromagnetic layer being too thick (as the exchange field is inversely proportional to the thickness of the ferromagnetic layer), there was no further study demonstrated where the ferromagnetic layer was of an order in which the exchange field would be present [69, 70]. Furthermore, the larger increase in coercivity occurs in the (Ga,Mn)As/GaAs/MnTe system. The contention is that this is due to Mn diffusion into the GaAs buffer layer, blurring the MnTe system into the GaAs buffer layer. However, while this would explain why an increase in coercivity would occur, this does not explain why the increase in coercive field is so much stronger compared to the (Ga,Mn)As/MnTe system. As such, the evidence that an exchange bias effect has occurred in the system is weak.
1.2.1 Exchange Bias Background

Exchange bias was discovered in 1950's, with the famous experiment by Meiklejohn and Bean [71]. Since then, it has become very important in device applications [72, 73]. In particular, it is used in spin valve devices for computer storage as well as in arrays of magnetic sensor devices based on the giant magnetoresistance effect (GMR) [74]. However, the theory behind the actual phenomenon itself is still not completely understood, and there are many aspects that still need to be studied.

Since ferromagnetic semiconductors are compatible with existing semiconductor electronics and photonics devices, this provides a strong reason to pursue potential spintronic devices [5, 75 - 77]. Therefore, since exchange bias is an important device application, it is necessary to be able to exchange bias such a material. (Ga,Mn)As has been extensively studied, both experimentally and theoretically, and is thus a good candidate for such a study.

Exchange bias is observed when there is exchange coupling at the interface between an anti-ferromagnetic layer (AFM) and a ferromagnetic layer (FM), after the sample is cooled in the presence of a magnetic field from a temperature above the Neel temperature, \(T_N\), but below the Curie temperature of the FM. Once cooled below the Neel temperature in a field, the hysteresis loop of the system is shifted along the field axis, generally in the opposite direction of the cooling field [78]. Thus, the coercive fields of the hysteresis have different values. This shift in the loop is known as the exchange bias or field, \(H_E\). This effect will disappear close to or above the Neel temperature of the AFM, providing proof that it is the AFM that causes the exchange bias.
The temperature at which the exchange bias disappears is known as the blocking temperature, $T_B$. Also, it is important to note that the effect will not occur if the sample is cooled from above $T_N$ in zero field if the sample is in a demagnetized state. However, if the sample is zero field cooled from a remnant state, there will be an exchange bias observed [82].

![Figure 1.6: A graphical interpretation of the exchange bias phenomenon. The arrows represent the spins of the AFM and FM layer as the system undergoes a hysteresis loop.](image-url)
Figure 1.6 shows a schematic depiction of the exchange bias phenomenon. First, as shown in 1.6(a), the temperature of the sample is greater than the blocking temperature of the AFM, but below the Curie temperature of the FM ($T_B < T < T_C$). When a magnetic field is applied, the FM spins will align along the field, while the AFM spins will remain randomly arranged.

When the temperature of the sample is cooled below the blocking temperature, the AFM spins next to the AFM-FM interface will align themselves ferromagnetically along the direction of the FM spins, shown in 1.6(b). This is due to a ferromagnetic interaction between the AFM spins at the interface and the FM spins. In this case, a ferromagnetic interaction between the AFM spins and the FM spins is assumed. The spins away from the AFM-FM interface will align themselves in such a way that the net magnetization of the AFM film is zero.

When the magnetic field is reversed, the FM spins begin to rotate, as shown in figure 1.6(c). However, the AFM spins will remain in the original configuration. This produces an interfacial interaction between the FM and AFM spins, in which the AFM spins produce a torque on the FM spins. This torque serves to keep the FM spins aligned in their original configuration, past the point at which they would normally be reversed. Therefore, the field necessary to align the FM spins along the opposite direction of the original cooling field is greater due to the effective internal biasing field from the AFM spins at the interface [83].

Eventually, the magnetic field will reach a point at which the FM spins will align themselves in the opposite direction, as shown in figure 1.6(d). When the magnetic field is put back into the original configuration, there is still the interaction, or torque, from the
AFM spins. This has the effect of decreasing the necessary field needed to put the FM
spins back into their original configuration, as shown in figure 1.6(e) as the spins begin to
rotate back to their original configuration. Thus, assuming a square hysteresis loop, the
loop is no longer symmetric about zero magnetic field. The amount that the hysteresis
loop is shifted towards the positive or negative magnetic field is the exchange field.

The (Ga,Mn)As/MnO system provides unique opportunities for studying a rarely
studied limit of exchange biasing. The Neel temperature of MnO is 118 K, while the
attainable Curie temperature of (Ga,Mn)As has been reported to be higher than 170 K
[13]. There have not been many studies of systems where $T_B > T_C$, as in most AFM/FM
systems the Curie temperature is much higher than the blocking temperature of the AFM.
Therefore, in theory the system can be used to study the cases of when $T_C < T_B$, $T_C \sim T_B$, and $T_C < T_B$.

1.2.2 MBE Growth of MnO/(Ga,Mn)As

We now discuss the experiments by Eid et al. on exchange-biasing of (Ga,Mn)As. Low
temperature molecular beam epitaxy (MBE) growth was performed in an Applied
EPI 930 system with Ga, Mn, and As effusion cells. The substrates used were epi-ready
semi-insulating GaAs (100), and were deoxidized using the standard method of heating to
$\sim 580 ^\circ C$ with an As flux impinging on the surface. A buffer layer of GaAs with a
thickness of 100 nm was then grown. The samples are then cooled to $\sim 250 ^\circ C$ for the
growth of a 5 nm thick low temperature GaAs layer, after which a 10 nm thick 
$\text{Ga}_{0.94}\text{Mn}_{0.06}\text{As}$ is grown. The growth is performed under group V rich conditions with an 
As:Ga beam equivalent pressure ratio of $\sim$12:1. The samples are then transferred in situ 
to an adjoining ultra high vacuum buffer chamber and the As effusion cell is cooled to a 
temperature of 110 °C. This is done to prevent any formation of MnAs clusters during 
the subsequent Mn growth. The As pressure in the growth chamber is monitored, and 
when it reaches an acceptable level, the sample is reintroduced into the growth chamber. 
A Mn capping layer with a thickness of $\sim$4 nm or $\sim$8 nm is then deposited. In order to 
prevent any interdiffusion and chemical reaction between the Mn and (Ga,Mn)As layers, 
the Mn growth is performed at room temperature [84]. While the Mn capping layer is 
expected to be pure Mn, due to the 99.999% source purity, the layer does oxidize rapidly 
once the samples are removed from the ultra high vacuum chamber.

In order to in situ monitor the growth mode and surface reconstruction, reflection 
high-energy electron diffraction (RHEED) is performed at 12 keV. The (Ga,Mn)As 
thickness is then calculated from the RHEED oscillations. The thickness of the Mn layer 
is estimated by using the RHEED oscillations of MnAs, because the growth rate of MnAs 
is largely determined by the sticking coefficient of Mn (and later verified by using 
transmission electron microscopy, Rutherford back scattering, and x-ray reflectometry, 
all of which will be discussed in the following chapter). The Mn concentration of $\sim$0.06 
was estimated from electron probe microanalysis performed on earlier calibration 
samples grown using fluxes similar to the Ga and Mn fluxes used here. The RHEED 
pattern of the (Ga,Mn)As layer growth had a streaky 1x2 reconstruction pattern, which
suggests a good crystalline quality. During the Mn growth, the RHEED pattern consisted of sharp, elongated streaks, with a symmetry suggestive of a cubic phase of Mn [85, 86].

In order to study the affect that *ex situ* annealing has on the Mn layer, two growth protocols were observed when mounting the samples onto the MBE sample holders. For the first protocol, indium covers the entire bottom surface of the wafer. In order to remove the sample from the sample block, the samples had to be annealed at ~220 °C for a few minutes in order to melt the indium and remove the sample. For the second protocol, only the two edges of the sample are attached to the block with indium, and the center portion of the sample is suspended, indium-free. In this type of sample, the center portion can be removed from the sample holder by cleaving without any heating. The indium-bonded edges still require a short thermal annealing in order to remove them. Therefore, a systemic study can be performed on the effect of the short annealing done when the samples are removed, as well as that of *ex situ* annealing of the indium-free portion of the identical sample.

We must stress, for clarification, that when we refer to indium-free or indium-backed samples, that the indium itself plays no role in the actual magnetization of the sample. Rather it only refers to the procedure used in the sample growth.

1.2.3 Magnetization Measurements

Now that we have described the growth of the exchange bias structures, we turn our attention to the magnetization measurements. The magnetization measurements were
performed using a commercial superconducting quantum interference device (SQUID) and the exchange bias effect was later confirmed using a magneto-optical Kerr effect (MOKE) experiment. The samples were measured with the magnetic field in the plane of the sample along the [110] direction, and were measured as a function of temperature and applied magnetic field.

In Figure 1.7, we have the SQUID data demonstrating that the (Ga,Mn)As/MnO system was successfully exchange-biased. In Figure 1.8(a), the Ga$_{0.92}$Mn$_{0.08}$As (10 nm)/MnO (4nm) sample was field-cooled in a field of 2500 Oe, while in Figure 1.8(b) the sample was field-cooled in a field of -2500 Oe. The hysteresis loop is clearly shifted to the left in the first case, and to the right in the second. The loops are shifted in the opposite direction of the applied cooling field. As mentioned earlier, this is common in exchange-biased systems. In Figure 1.8(c), the sample was not field cooled, but instead cooled in a zero field. Although it is expected to be perfectly symmetric about zero field, the hysteresis loop is very slightly shifted. This is because we cannot eliminate the field coming from the magnetization of the (Ga,Mn)As ferromagnetic layer. As a control study, a sample of (Ga,Mn)As of the same composition was grown without a Mn cap (with field-cooling of 1000 Oe), with the SQUID data for this shown in Figure 1.8(d). There is no shift in the hysteresis loop, and thus no exchange bias. This is a clear demonstration that the exchange bias is originating from the Mn capping layer. Also, the hysteresis loops of the capped sample are larger than that of the uncapped sample. This is typically seen in exchange-biased systems, as the AFM-FM system enhances the coercive field of the ferromagnetic layer [87].
Figure 1.7: Reprinted from reference 67. SQUID measurements of a (Ga,Mn)As/MnO. In a) and b) the sample is field-cooled in a field of 2500 and -2500 Oe with the loop shifted to the left and right, respectively. In c) the sample is zero-field cooled with little shift. In d) a control sample is measured, with no shift visible.

Temperature dependent data for a Ga$_{0.92}$Mn$_{0.08}$As/MnO sample is shown in Figure 1.8(a). The thickness of the (Ga,Mn)As layer was 10 nm, while the Mn layer was ~4 nm, and the Curie temperature, as measured by low field measurements of M(T), was ~55 K. The exchange field, $H_E$, decreases monotonically with increasing temperature until it reaches $T_B$ at 48 K. The coercive field decreases, plateaus around the blocking temperature, and then decreases monotonically to zero at $T_C$. Figure 1.8(b) is of a different sample. This sample was of Ga$_{0.94}$Mn$_{0.06}$As/MnO, with the (Ga,Mn)As layer being 10 nm in thickness, while the Mn layer was ~8 nm in thickness. The $T_C$ of the
sample was ~90 K. The blocking temperature of the two samples is the same, which is to be expected because it only depends on the anti-ferromagnetic layer. Similar behavior to the first sample is observed as well.

Figure 1.8: Reprinted from reference 67. Temperature dependent magnetization measurements of the coercive field, $H_C$, and the exchange field, $H_E$ for a) a $\text{Ga}_{0.92}\text{Mn}_{0.08}\text{As}$ (10 nm)/MnO sample (4 nm) and b) a $\text{Ga}_{0.94}\text{Mn}_{0.06}\text{As}$ (10 nm)/MnO (8 nm) sample.
Finally, in Figure 1.9 the dependence of $H_C$ and $H_E$ is plotted as a function of the magnitude of the cooling field for the two samples. A cooling field of only a few Oe is needed in order to produce the exchange bias effect, and there is very little dependence on the cooling field for $H_E$, even up to cooling fields a few orders of magnitude larger than that needed to create the exchange bias. This is important to note, because for some systems, large cooling fields result in the loops shifting towards a positive direction for a positive cooling field, a phenomenon known as positive exchange bias [88]. When the cooling field is sufficiently small (under 7 Oe), there is almost no exchange bias observed.
Figure 1.9: Reprinted from reference 67. $H_E$ and $H_C$ as a function of magnetic field for a) a sample of the first mounting procedure and b) a sample of the second procedure.

1.2.4 Annealing Studies

The second growth protocol allows us to study the effect that post-growth annealing has upon the exchange field. In Figure 1.10, there is the SQUID data from just
such a sample. The data is from two portions of the same sample, both with a $T_c$ of 90 K, one with an indium backing and the other indium-free. The magnetization measurements indicates that there are no large Mn$_2$As, GaMn, or MnAs clusters. In 1.10(c), there is clearly a shift when the indium backed sample is cooled in an applied field of 1 kOe. However, this exchange field is absent when the indium-free portion of the sample is measured, shown in 1.10(b). The indium-free sample was then annealed in atmosphere at 200 °C for one minute. Upon annealing, the indium-free sample demonstrates an exchange bias field, as the hysteresis loop is shifted upon cooling the sample in a field. Therefore, for indium-free samples, a certain amount of post-growth annealing is necessary in order for there to be exchange bias in the system. This emphasizes the importance that oxidation of the Mn layer has on the effect.
Figure 1.10: Reprinted from reference 67. Magnetization measurements of a Ga$_{0.94}$Mn$_{0.06}$As (10 nm)/MnO (8 nm) grown using the second protocol. a) Shows the M vs T measurements of the indium-free and indium-backed portions. In b) the as-grown In-free piece is measured and no exchange bias is observed. In c) the In-mounted sample is measured, and an exchange bias is seen. Finally, in d) after the as-grown In-free piece was annealed in atmosphere for one minute at 200 °C, an exchange bias effect was observed.

However, in none of the samples grown using the second protocol, was the exchange field of the post-annealed sample greater than that of the indium backed portion of the same sample. This was attributed to the annealing conditions being less than optimal, and it was hoped that with the proper annealing conditions, the exchange field
could be controlled and increased. To this end, an annealing study of the
(Ga,Mn)As/MnO system was proposed, and will be discussed further in Chapter 4.

1.2.5 Research Performed by Other Groups

Now we turn our attention to work performed since the work done by Eid et al. There have been two experiments performed by Furdyna et al. since the work discussed above [89, 90]. In the first, ferromagnetic resonance experiments were performed on exchange-biased samples of (Ga,Mn)As/MnO. These experiments suggest that the samples show a unidirectional anisotropy about the $[1\bar{1}0]$ direction, as opposed to the more typical $[100]$ [91]. While this may be a property due to the antiferromagnetic layer, as suggested, this transition also occurs at higher temperatures. The reason that this transition occurs in (Ga,Mn)As (please see Chapter 3 for a fuller discussion of this) has to do with the change in the spin-splitting parameter and the hole concentration. For samples with low hole concentrations, easy axis transitions can occur at lower temperatures. Since the samples have not been annealed, and have been capped during growth, it is possible that their hole concentrations are lower than usual, and this may be affecting the easy axis transition temperature of (Ga,Mn)As.

In the second experiment, the asymmetric planar hall effect (PHE) was measured in order to investigate the exchange field. They concluded that the AF spins remain frozen during the magnetization reversal in (Ga,Mn)As, and they proposed a single ferromagnetic domain model to explain the behavior in the PHE data.
1.3 Research Goals & Layout of Thesis

Having established the background information for our experiments, we now discuss the goals and achievements of this dissertation. These are two-fold:

A) The development of a magneto-optical Kerr effect, or MOKE, experiment to study the ferromagnetism in (Ga,Mn)As. A MOKE experiment was set-up to provide an in-house aid in quickly providing a rough estimate of the Curie temperature. We will also propose a novel way to measure the anisotropy in tensile-strained (Ga,Mn)As with MOKE, through resolving the components of the magnetization. We will show that this system is able to successfully measure anisotropy changes in tensile-strained (Ga,Mn)As.

B) We have previously mentioned the work done on exchange-biasing (Ga,Mn)As using a Mn capping layer. However, the structural and chemical composition of the Mn capping layer was not known at the time of the exchange biasing study – only that (Ga,Mn)As had been capped with a layer of metallic Mn. In this dissertation we analyze the chemical and structural nature of the exchange-biased (Ga,Mn)As system.

In chapter 2, we discuss the theory behind the MOKE experiment, the current experimental set-up, and we demonstrate the experimental results showing we have achieved our goal. This is followed by a discussion of component-resolved MOKE, in
order to measure the anisotropy changes in tensile-strained (Ga,Mn)As in Chapter 3.

Finally, in chapter 4, we turn our attention to the exchange-biased system and analysis of the chemical and structural nature of the system.
Bibliography


Chapter 2
Magneto-optical Kerr Effect

This chapter provides the theoretical background for the magneto-optical Kerr (MOKE) experiment. In addition, we discuss the three different setups (and their advantages and disadvantages) used for measuring MOKE, as the system has evolved over the time of this project.

2.1 Theoretical Background

When a material acquires a net magnetic moment, and linearly polarized light is reflected from it, the off-diagonal components of the Fresnel reflection matrix (to be defined shortly) are non-vanishing, and the diagonal components are modulated, due to the presence of a magnetic field. When the light enters the magnetized material, the light will decompose into beams containing the left and right-circularly polarized modes. Due to the non-vanishing off-diagonal components, this results in a different response to left and right circularly polarized light, as the light will generally propagate with different refractive indices or phase velocities [1]. Upon exiting the material, the two modes will re-combine, however there will now be a rotation in the polarization as well as an ellipticity. The Kerr rotation causes the rotation in the polarization plane of light. The
Kerr ellipticity arises from the fact that the absorption coefficients for right and left circularly polarized light will be different as well, thus resulting in an elliptical polarization. When the geometry of the experiment is in reflection, the effect is known as the Kerr effect, while for transmission it is known as the Faraday effect. However, both arise from the same physics.

MOKE is related to the polarized electronic band structure. For different materials the effect will be stronger or weaker, as it will depend on the net electron spin polarization, the spin-orbit coupling strength, and the joint density of states of the coupling states. It will also depend on the wavelength of light used. Also note that the Kerr ellipticity and the Kerr rotation can have different strength functions for the spectrum of light, related to one another through the Kramer-Kronig relation.

In order to define the coordinate system, please refer to figure 2.1. Note that this will be for the case in which the sample is optically thick, i.e, only the magnetic film needs to be considered.
Figure 2.1: The coordinate system for the MOKE experiment.

The Fresnel reflection matrix is derived from the dielectric tensor, $\varepsilon$:

$$
\varepsilon = \varepsilon_{xx} \begin{bmatrix}
1 & -iQm_z & iQm_y \\
-iQm_z & 1 & -iQm_x \\
-iQm_y & iQm_x & 1
\end{bmatrix}
$$

(1)

where $m_x$, $m_y$, and $m_z$ are the directional cosines of the magnetization vector $M$ [2]. $\varepsilon_{xx}$ is assumed to equal $\varepsilon_{zz}$ for simplicity. $Q$ is a magneto-optical constant equal to:
By solving Maxwell’s equations for the dielectric tensor, we obtain the Fresnel magneto-optical reflection matrix, which is the ratio between the incident polarized electric field and the reflected polarized electric field:

\[
\begin{bmatrix}
E_r^r \\
E_p^r
\end{bmatrix} = \begin{bmatrix}
r_{pp} & r_{ps} \\
r_{sp} & r_{ss}
\end{bmatrix}\begin{bmatrix}
E_{p}' \\
E_{p}'
\end{bmatrix}
\]

The elements of the Fresnel magneto-optical reflection matrix are defined as follows [2]:

\[
r_{pp} = \frac{n_0 \cos \theta_o - n_1 \cos \theta_i}{n_1 \cos \theta_o + n_0 \cos \theta_i} - \frac{i2n_o n_1 \cos \theta_0 \sin \theta_i m_z Q}{n_1 \cos \theta_o + n_0 \cos \theta_i}
\]

\[
r_{sp} = \frac{in_o n_1 \cos \theta_o (m_z \cos \theta_i + m_y \sin \theta_i) Q}{(n_1 \cos \theta_o + n_0 \cos \theta_i)(n_0 \cos \theta_o + n_1 \cos \theta_i) \cos \theta_i}
\]

\[
r_{ss} = \frac{n_0 \cos \theta_o - n_1 \cos \theta_i}{n_o \cos \theta_o + n_1 \cos \theta_i}
\]

\[
r_{ps} = \frac{in_o n_1 \cos \theta_o (m_z \cos \theta_i - m_y \sin \theta_i) Q}{(n_1 \cos \theta_o + n_0 \cos \theta_i)(n_o \cos \theta_o + n_1 \cos \theta_i) \cos \theta_i}
\]
In turn, this leads to the following expressions for the Kerr effect, depending upon whether the incident light is s or p-polarized:

\[ \theta_k^p = \frac{r_{sp}}{r_{pp}} \quad (8) \]

and \[ \theta_k^c = \frac{r_{pl}}{r_{ss}} \quad (9) \]

These expressions will greatly simplify once the three different orientations for the MOKE are explained. The three types of MOKE are the polar, longitudinal, and transverse, and are determined by the direction of magnetization. When the magnetization is along the Z-axis, it is called the polar Kerr effect. When it is along the Y-axis, it is known as the longitudinal Kerr effect. Finally, when the magnetization lies along the X-axis, it is the transverse Kerr effect. This is illustrated in Figure 2.2.

We can immediately see the effect this has on the Fresnel reflection coefficients. For longitudinal MOKE, \( m_y = 1 \), while \( m_x = m_z = 0 \). For transverse MOKE, \( m_x = 1 \), while \( m_y = m_z = 0 \). Finally, for polar MOKE, \( m_z = 1 \), while \( m_x = m_y = 0 \).
2.2 Experimental Background

This section will briefly deal with the past versions of the experiments, as well as their advantages and disadvantages, then discuss the current version of the experimental setup. There are some constants in each of the experiments, which I will note. A laser diode of wavelength 676 nm was used for all three experimental setups. This wavelength was chosen because (Ga,Mn)As exhibits a stronger MOKE at this wavelength, due to the wavelength being close to the band-gap of (Ga,Mn)As. The sample sits inside a cryostat capable of reaching 4.2 K. Around the cryostat is an electromagnet capable of fields up
to 1800 Oe. Finally, a lock-in amplifier tied to the frequency of a chopper (or the photo-elastic modulator) is used to boost the signal and to help with noise reduction.

2.2.1 Cross Polarizers

![Diagram of cross polarizers MOKE version]

Figure 2.3: Experimental setup for the cross-polarizers MOKE version.
Originally, the experimental setup for the MOKE was set up as in Figure 2.3. The light passes through the first linear polarizer, which polarizes it into s-polarized light, after which it reflects off of the sample. The reflected light then passes through a second linear polarizer which is set to $\delta = 1 - 2$ degrees from p-polarized light (and thus extinction). The advantage of this system is that it can be relatively straightforward to derive the values of the Kerr rotation in degrees, as opposed to arbitrary units [3]. The formula for determining the Kerr ellipticity when using such a system is:

$$\phi = \frac{\Delta I}{I_o} \frac{\delta}{4},$$

(10)

where $\Delta I$ is the difference in intensities when the magnetization is saturated in opposite directions and $I_o$ is the Kerr intensity at zero magnetization. However, because the linear polarizers are almost completely crossed, this cuts down on the signal strength greatly. We found that this made the system far too sensitive to any noise or vibrations when measuring (Ga,Mn)As, though for measuring MnAs it served quite well. Particularly when any temperature dependent measurements were made, the signal to noise ratio became a serious issue.
2.2.2 Photo-elastic Modulator

Figure 2.4: Experimental setup using a photo-elastic modulator (PEM).

Figure 2.4: Experimental setup using a photo-elastic modulator (PEM).
In an effort to improve the signal to noise ratio, the above setup was used before switching over to the current set-up. After reflecting off of the sample, the beam passes through a HINDS photoelastic modulator (PEM), which acts as an oscillating quarter wave plate essentially. The PEM will oscillate at either 50k Hz or 100k Hz. The light passes through a linear polarizer set to 45 degrees from the s-polarized direction, and then is detected.

Upon passing through the system, the intensity of the light can be written as [4]:

\[ I(t) = I_o[1 + 2\theta_k \cos(A_o\omega t) - 2\epsilon_k \sin(A_o\omega t)], \tag{11} \]

where \( I_o \) is the DC intensity, \( \theta_k \) and \( \epsilon_k \) are the Kerr rotation and ellipticity (respectively), \( \omega \) is the angular frequency of the PEM, and \( A_o \) is the retardation amplitude of the PEM.

Expanding this equation through a Fourier series, we obtain:

\[ I(t) \equiv I_o[1 + 2\theta_k J_o(A_o) - 4\epsilon_k J_1(A_o)\sin(\omega t) + 4\theta_k J_2(A_o)\cos(2\omega t)], \tag{12} \]

however, the first term may be neglected because \( \theta_k \) is a small number. This in turn yields the equations for the Kerr rotation and ellipticity as:

\[ \theta_k = \frac{\sqrt{5}}{4J_2} \frac{V_{2f}}{V_{DC}}, \quad \text{and} \quad \epsilon_k = \frac{\sqrt{5}}{4J_1} \frac{V_{1f}}{V_{DC}}, \tag{13, 14} \]
where $V_{1f}$ and $V_{2f}$ are the voltages measured by the lock-in amplifier with the PEM operating at the fundamental and two times the fundamental frequency.

This setup greatly improved the signal to noise ratio compared to the original, and it is the set-up used for the experiments in Chapter 3. Furthermore, it has the advantage in that depending upon whether it is oscillating at 50k or 100k Hz, it will independently measure the Kerr rotation and the Kerr ellipticity. However, for (Ga,Mn)As grown on GaAs substrates, the system was not quite able to take effective measurements close to the Curie temperature. For tensile-strained samples, due to their higher Kerr signal, measurements up to $T_c$ were reliably made.
2.2.3 Diode Bridge

The current experimental setup is shown above, in Figure 2.5. Here, after the beam is reflected off of the sample, it passes through a polarizing beam splitter. The polarizing beam splitter will reflect s-polarized light 90 degrees to the original path, while allowing through p-polarized light. These two components are then focused on the detector’s two photodiodes. The photodiode bridge detector is a Nirvana detector from

Figure 2.5: Current experimental setup using a diode bridge detector.
New Focus. The difference between the two signals is then amplified and when balanced, the signal is proportional to \( \sin(2\delta \theta_k) \), where \( \delta \theta_k \) is the change in the rotation due to the magnetic field [5]. The laser intensity and polarization noise appear equally in both photodiodes and are canceled out to leading order, thus greatly reducing the overall noise in the measurement.

With this setup, it was found that the Curie temperature could be measured reliably. The only remaining issue with the system is that when a (Ga,Mn)As layer is capped with another layer, occasionally the measurements were not able to reach \( T_c \) due to the fact that MOKE is most sensitive to the topmost layers.

2.3 MOKE Measurements of \( T_c \)

Now that we have discussed the current experimental setup, we turn our attention to the measurement of the Curie temperature in (Ga,Mn)As. One of the principal reasons for the creation of this experiment was to find a way to quickly and reliably measure the Curie temperature of (Ga,Mn)As, as well as other ferromagnetic samples. While SQUID is always an option, occasionally measurements need to be made quickly, in order to get information about the samples in order to facilitate further growth.

The principle behind measuring the Curie temperature with MOKE and with SQUID is very similar. Both measure the strength of the magnetic signal, and compare the data as temperature is increased. For measuring \( T_c \) with MOKE the following procedure was used. The sample was measured at varying temperatures, taking care to wait enough time so that the temperature had stabilized between measurements. Then,
the data was off-set such that the Kerr signal was about zero (initially, the data is off-set from zero by some amount depending on the signal strength obtained). The size of the hysteresis loop was measured at saturation for each temperature. This data was then graphed versus temperature to obtain $T_c$.

In order to test whether the system was accurate in its measurements, a sample of $\text{Ga}_{0.95}\text{Mn}_{0.05}\text{As}$ 30 nm in thickness was used. The sample had previously been measured using SQUID, and the Curie temperature had been found to be 68 K.
Figure 2.6: MOKE measurements of a 30 nm Ga$_{0.95}$Mn$_{0.05}$As sample with a Curie temperature of 68 K, at a temperature of a) 4.2 K, b) 35 K, c) 45 K, and d) 60 K. The magnetic field was along the [110] direction of the (Ga,Mn)As film.
Figures 2.6 and 2.7 show the data gathered by the MOKE setup. Although it would be necessary to measure more data points in order to obtain a very precise measurement of the Curie temperature, it is plain to see that the $T_c$ measured in this manner agrees well with the one obtained by SQUID measurements. Also, as can be seen in Figure 2.6d, the signal is very clean and free of noise, even close to the Curie temperature. Therefore, it would appear to be safe to say that the experiment can now accurately measure the Curie temperature for compressively strained (Ga,Mn)As samples.
2.4 Conclusion

After several iterations of the magneto-optical Kerr effect experiment, the Curie temperature may now be measured using the MOKE system, accurately and reliably. The largest remaining challenge to the MOKE system comes from ferromagnetic layers capped with another layer. Depending on the reflectivity of the capping layer, MOKE may or may not be able to measure the $T_c$ of the ferromagnetic layer. However, with time, this problem should be solved as well.
Bibliography

Chapter 3

Component-Resolved Magneto-optical Kerr Effect

In the previous chapter, we established the basics of both the theory behind the magneto-optical Kerr effect (MOKE), and the experimental set-ups used to detect the MOKE signal. We now describe an extension of the standard MOKE to study spin reorientation transitions in GaMnAs, a phenomenon of current interest [1, 2]. For certain carrier concentrations in specific amount of strain, both tensile and compressive, it is predicted that the easy axis of magnetization in GaMnAs will undergo a spin reorientation transition from (001) to [001] for tensile-strained films (i.e, from in the plane of the sample to out of the plane of the sample), or from [001] to (001) for compressively strained films, where [001] is the growth direction of the film [3]. While there have been previous studies demonstrating the spin reorientation transition in compressively strained films, there has been little work in the tensile strain regime [1, 2]. Although it is not typically used for such measurements, the MOKE experiment can, with a simple trick in geometry, be used to get a two-dimensional picture of the magnetization, in order to measure such spin reorientation transitions. This chapter will discuss the theory behind the component-resolved MOKE, the experimental set-up, and the study of the spin reorientation transition in tensile-strained GaMnAs.
3.1 Theory

As discussed in the previous chapter, there are three types of MOKE, depending on the direction of the magnetization with respect to the plane of incidence of the light. In materials undergoing a spin reorientation transition, such as in tensile-strained GaMnAs going from a (001) to a [001] easy axis, the signal from the MOKE experiment can be a mixture of all three types of effects: polar, longitudinal, and transverse (please refer to Figure 2.2 in Chapter 2). This is further complicated by the fact that there is a different response factor for each of the types: the same magnitude of magnetization in each of the three directions will not produce the same amount of signal in the experiment. The polar Kerr effect is usually up to an order of magnitude larger than that of the longitudinal Kerr effect, while the longitudinal Kerr effect is typically larger than the transverse Kerr effect. This can lead to complicated hysteresis loops, making analysis of the signal difficult.

As referenced in the previous chapter, there has been previous work done in separating the components of the MOKE signal into its constituent parts through the use of several methods [4, 5]. We focus here on a more recent method proposed by Kirschner, et al. [6]. As shown in the previous chapter, the equation for the polar and longitudinal Kerr effects are related to the Fresnel reflection coefficients, $r_{ij}$. Here
Here, the notation of $s$ or $p$ in the Kerr effect equations refer to whether the incident light is polarized along the $s$ or $p$ direction (for the difference between these two orientations, please refer to figure 3.3). Also, as noted in the previous chapter, for longitudinal MOKE, $m_y = 1$, while both $m_x$ and $m_z$ equal zero. For transverse MOKE, $m_x = 1$, while both $m_y$ and $m_z$ equal zero. Finally, for polar MOKE, $m_z = 1$, while both $m_x$ and $m_y$ equal zero. This has the effect of greatly simplifying the Kerr effect equation, when the signal is composed solely of one type of effect, and leads to the equation for the polar Kerr effect under $s$-polarized light (since this will be the polarization used in the experiment):

\[
\theta_k^s = \frac{r_{ps}}{r_{pp}}, \quad (1)
\]

and \[\theta_k^s = \frac{r_{ps}}{r_{ss}}. \quad (2)\]

\[
(\theta_k^s)^{\text{Polar}} = (\frac{r_{ps}}{r_{ss}})^{\text{Polar}} = -\frac{\cos \theta_o}{\cos(\theta_o - \theta_1)} \ast \frac{in_1n_iQ}{(n_1^2 - n_o^2)}, \quad (3)
\]

For the longitudinal Kerr effect under $s$-polarized light we obtain:

\[
(\theta_k^s)^{\text{Long.}} = (\frac{r_{ps}}{r_{ss}})^{\text{Long.}} = -\frac{\cos \theta_o \tan \theta_1}{\cos(\theta_o - \theta_1)} \ast \frac{in_1n_iQ}{(n_1^2 - n_o^2)}, \quad (4)
\]
where $n_0$ is the refractive index of air, and $n_1$ is the refractive index of (Ga,Mn)As. The angles are defined in the previous chapter in figure 2.1, and $Q$ is a magneto-optical constant related to the off-diagonal components of the dielectric tensor:

$$Q = i \frac{\varepsilon_{xy}}{\varepsilon_{xx}}$$ (5)

![Initial Measurement and Measurement Reversal](image)

Figure 3.1: Experimental set-up for the component-resolved experiment.

The component-resolved Kerr effect method utilizes the fact that the polar Kerr effect is an even function of the incident angle of the light, while the longitudinal Kerr effect is an odd function of the incident angle, i.e:
\[(\theta_k^\prime)^{\text{Polar}} (\pm \theta) = (\theta_k^\prime)^{\text{Polar}}, \text{ while} \]

\[(\theta_k^\prime)^{\text{Long}} (\pm \theta) = \pm (\theta_k^\prime)^{\text{Long}}. \]  

Also, while using s-polarized light, the transverse Kerr effect is zero. This is because of the fact that, classically to a first order, the MOKE can be thought of as a change in the electric-field vector of the light as a response to the Lorentz force from the material’s magnetization. When using s-polarized light, the electric field vector of the light is parallel to the magnetic field for the transverse effect, and thus no MOKE is observed [6]. In a system where we use s-polarized light to measure the Kerr effect, the signal will be composed solely of the polar and longitudinal Kerr effects. Therefore, if we first measure the Kerr effect in a geometry where the incident angle is known, and then reverse the geometry (i.e, the position of the laser and the detector are switched), we obtain:

\[\theta_k^\prime (\theta) = (\theta_k^\prime)^{\text{Polar}} + (\theta_k^\prime)^{\text{Long}}, \text{ and} \]

\[\theta_k^\prime (-\theta) = (\theta_k^\prime)^{\text{Polar}} - (\theta_k^\prime)^{\text{Long}}. \]  

The angle of incidence chosen is 45°. Although the polar and longitudinal Kerr effects have an angular dependence with respect to the magnitude of the effects, they are both stable at this angle, allowing for slight experimental variances in the incident angle
without affecting the overall measurement [7 - 9]. First the measurement is taken, then the incident optics (the laser and its attendant optics) and the detector (and its optics) exchange positions and the measurement is taken anew. Once both sets of data are acquired, we obtain the polar Kerr signal as one half the point-by-point summation of both sets of data, while the longitudinal Kerr is one half the point-by-point difference of both sets of data.

While this analysis deconvolutes the signal into the longitudinal and polar components, there is still one more necessary step before the analysis is finished. We mentioned earlier that the polar and longitudinal signals from the sample do not have the same response factor. In order to resolve the magnetization vector, it is necessary to scale the longitudinal and polar signals relative to their response factor. There are two limits: the ultra-thin limit, defined as:

\[
\frac{2\pi |n_1| d_1}{\lambda} \square 1
\]  

(10)

where \( n_1 \) is the refractive index of (Ga,Mn)As, and \( d_1 \) is the thickness of (Ga,Mn)As. In this case, the scaling factor for comparing the longitudinal and polar Kerr effects is [10]:

\[
\frac{(\theta_k^e)^{\text{Long.}}}{(\theta_k^e)^{\text{Polar}}} = -\frac{\sin^2 \theta_l}{\sin \theta_2 \cos \theta_2},
\]  

(11)

where \( \theta_2 \) will be the angle formed in the layer beneath the ferromagnetic layer (an additional layer will be present in figure 3.1).
The second limit is the bulk limit, when it is safe to consider only the top layer, (Ga,Mn)As. The scaling factor for this limit is given by:

\[
\frac{(\theta_k)^{\text{Long.}}}{(\theta_k)^{\text{Polar}}} = -\tan \theta.
\] (12)

Assuming the index of refraction of GaAs, the scaling factor is 5.07 for bulk GaMnAs and 5.61 for the ultra-thin limit. For all the measurements taken, the bulk scaling factor was used, as all of the samples were beyond the ultra-thin parameters. However, both scaling factors are relatively close to one another. Once the two signals have been scaled, the magnetization vector in the Y-Z plane can be resolved accurately for a given magnetic field, with the caveat that the scaling factor has been determined theoretically.

### 3.2 Experiment

The samples were grown by molecular beam epitaxy (MBE) on epiready semi-insulating GaAs (100) substrates. The samples consist of a low temperature GaAs buffer layer, followed by an (In,Ga)As layer that has been allowed to relax to its lattice constant. On top of the (In,Ga)As layer, a Ga\textsubscript{0.94}Mn\textsubscript{0.06}As layer of varying thickness (15, 30, 60, 120 nm) was grown. The thickness of the (In,Ga)As layer for all the samples, except for the 30 nm sample, was 750 nm, with the 30 nm sample's (In,Ga)As layer having a thickness of 700 nm.
Figure 3.2: Sample structure of the (Ga,Mn)As samples measured. The (In,Ga)As layer for the sample with 30 nm of (Ga,Mn)As was 700 nm in thickness.

<table>
<thead>
<tr>
<th>(Ga,Mn)As Thickness (nm)</th>
<th>$T_c$ As-grown (K)</th>
<th>$T_c$ Annealed (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>70</td>
<td>115</td>
</tr>
<tr>
<td>30</td>
<td>95</td>
<td>130</td>
</tr>
<tr>
<td>60</td>
<td>75</td>
<td>105</td>
</tr>
<tr>
<td>120</td>
<td>60</td>
<td>115</td>
</tr>
</tbody>
</table>

Table 3.1: Curie temperatures of the samples, as-grown and annealed, as measured by SQUID.
The MOKE signal is measured using the Hinds PEM technique described in the previous chapter and illustrated by Figure 2.4 in that chapter. In principle, however, there is nothing to prevent the use of the diode bridge technique from being able to measure the component-resolved MOKE. The samples are placed in a helium-flow cryostat (base temperature of 4.2 K) which lies in between the pole pieces of an electromagnet capable of fields of up to 1800 Oe. A diode laser with a wavelength of 676.4 nm is used as the probe. The optics are placed such that the incident angle is 45°, and care is taken to ensure that the laser spot is on the same portion of the sample each time.

The data from both the 45° and the –45° incident angles is then analyzed, converted into the longitudinal and polar signals, and scaled by the response factor. Once the data is scaled, the hysteresis loops of the polar and longitudinal components are compared, at the magnetic field strength at which the original signal reached saturation, in order to determine the percent character of each, to the overall signal. Once the percent character of the longitudinal or polar components is known, a basic vector analysis is performed to find the direction of the magnetization vector in the X-Z plane at saturation.

Also, in order to confirm that the method was working properly, a control experiment was performed, shown in Figure 3.3. Generally, for compressively strained (Ga,Mn)As, the magnetization of the sample will lie fully in the plane of the sample, i.e., there should be no polar signal observed. Therefore, upon inverting the experimental set-up, the hysteresis loop should simply be inverted, as the only signal coming from the
sample will be the longitudinal MOKE (an odd function of the incident angle). A sample of compressively strained (Ga,Mn)As was measured using this technique. No polar MOKE was observed, and the two hysteresis loops were inverses of one another.

Figure 3.3: MOKE of a compressively strained sample as a control measurement with the magnetic field parallel to [110] at a temperature of 4.2 K: a) Incident angle of $-45^\circ$, b) Incident angle of $45^\circ$, c) Polar MOKE component, d) Longitudinal MOKE component, after scaling.
The samples were measured at different temperatures and crystal orientations with respect to the magnetic field. Finally, they were annealed for two hours at 250°C in air, re-measured, and the results were compared to the as-grown data.

3.3 Theoretical Predictions

Now that we have shown that the method can theoretically resolve the components for (Ga,Mn)As, we discuss our method of analysis and the theoretical predictions. The magnetic field is applied in the plane of the sample, along the longitudinal direction. If the magnetization of the sample lies in the out-of-plane direction, this means that regardless of whether the material is undergoing an easy axis reorientation, there will be some signal from the longitudinal direction as the strength of the magnetic field increases. If, however, the easy axis of the material lies in the plane of the sample and the field is applied in the plane of the sample, there will be no polar MOKE signal observed, as this arises from the out-of-plane magnetization component. Therefore, if a sample initially only shows a longitudinal MOKE component, but a polar MOKE signal forms as the temperature is changed, the sample has undergone a reorientation in the easy axis of magnetization.

Shown in figure 3.4 is a theoretical calculation for the minimum magnetic field necessary to align the magnetization along the hard axis [3]. For low hole concentrations and tensile strain, the minority spin sub-bands become depopulated, and an in-plane easy
axis of magnetization becomes favored. For a given spin-splitting parameter $B_G$, where $B_G$ is a constant proportional to the magnetization of the sample, as the carrier concentration increases, for tensile-strained (Ga,Mn)As the easy axis of the sample will align itself along [001].

From the graph, we can see that the mean-field theory predicts that there are certain inflection points, depending upon the spin-splitting parameter (the points on the graph at which the relative anisotropy field reaches zero). In the tensile-strain regime, any sample whose properties cause it to fall to the left of this inflection point will have an easy axis in the plane of the sample; for any to the right, it will be out-of-plane.

We have already stated that due to the geometry of the experiment, we will be able to see any changes in the easy axis of magnetization, i.e., if the sample passes through an inflection point. However, the experiment can also judge the predictions of the theory, as the temperature increases. Since the spin-splitting parameter $B_G$ is proportional to the magnetization in the sample, we are able to vary this by varying the temperature (and thus the magnetization). Although we are unable to align the magnetization completely along the hard axis, as in the graph, by plotting

\[
\frac{\theta^P_{k} - \theta^L_{k}}{\theta^P_{k} + \theta^L_{k}}
\]

for a given magnetic field, we can test these predictions qualitatively, and make an estimate based on how much of the signal at this magnetic field comes from $\theta^L_{k}$ as to how close the sample is to the inflection point. The magnetic field chosen is 1080 Oe, and the value of the Kerr effects will be an average of the values taken at $\pm 1080$ Oe.
Figure 3.4: Reprinted from reference 3, the mean-field theoretical predictions for the necessary magnetic field to align the magnetization along the hard axis of magnetization, as a function of hole concentration, spin splitting parameter $B_G$, and strain.
3.4 Results

Shown in the following pages are the temperature dependent component-resolved data taken for the as-grown samples. The magnetic field is aligned along the [110] direction. With the exception of the 120 nm sample (to be discussed later), all samples showed some degree of polar signal at all temperatures.

Figure 3.5: Polar MOKE data for the 15 nm as-grown (Ga,Mn)As sample at a temperature of a) 4.2K, b) 25K, c) 35K, and d) 55K.
Figure 3.6: Longitudinal MOKE data for the 15 nm as-grown (Ga,Mn)As sample at a temperature of a) 4.2K, b) 25K, c) 35K, and d) 55K.
Figure 3.7: Polar MOKE data for the 30 nm as-grown (Ga,Mn)As sample at a temperature of a) 4.2K, b) 25K, c) 45K, and d) 55K.
Figure 3.8: Longitudinal MOKE data for the 30 nm as-grown (Ga,Mn)As sample at a temperature of a) 4.2K, b) 25K, c) 45K, and d) 55K.
Figure 3.9: Polar MOKE data for the 120 nm as-grown (Ga,Mn)As sample at a temperature of a) 4.2K, b) 15K, c) 35K, and d) 50K.
Of particular interest is the as-grown sample with 120 nm of (Ga,Mn)As. At a temperature of 4.2 K, there is no polar MOKE signal observed, as shown in figure 3.9 and 3.10. However, as shown in figure 3.9, as the temperature increases, a polar MOKE signal emerges. This implies that the material has undergone a spin reorientation transition, (the reasons why this is the case are discussed in section 3.3) with the initial
easy axis of magnetization lying in the plane of the sample, contrary to the usual
expectation of a compressively strained (Ga,Mn)As film. Further evidence of this
transition comes from Figure 3.8, which shows temperature dependent SQUID data. The
SQUID data appears to agree with the component-resolved MOKE data, in that there is a
sharp decrease in the in-plane magnetization with a corresponding increase in the out-of-
plane magnetization as temperature increases.

![Graph of SQUID data](image)

Figure 3.11: SQUID data of the 120 nm as-grown (Ga,Mn)As sample, with the
magnetic field in the plane of the sample, along [110] as well as out of the plane of the
sample (along the [001] direction).
Now that we have shown the data, we can compare our results to the mean-field theory. In figure 3.12, we have plotted \( \frac{\theta^\text{Polar} - \theta^\text{Long}}{\theta^\text{Polar} + \theta^\text{Long}} \) for the samples, taken at a field strength of 1800 Oe:

![Graph](image)

Figure 3.12: \( \frac{\theta^\text{Polar} - \theta^\text{Long}}{\theta^\text{Polar} + \theta^\text{Long}} \) as a function of temperature for the as-grown (Ga,Mn)As samples.

In figure 3.12, when the data is close to -1, the sample’s magnetization at 1080 Oe is aligned along the [110] direction (in the plane of the sample). When the data is close to 1, the sample’s magnetization is aligned along [001] (out of the plane of the sample).
When the data is close to 0, the magnetization is evenly split between the two directions, i.e., at 45° to the in-plane direction.

![Graph showing the relationship between hole concentration and relative anisotropy field](image)

Figure 3.13: Reprinted from reference 3, the mean-field theoretical predictions for the necessary magnetic field to align the magnetization along the hard axis of magnetization for tensile-strained (Ga,Mn)As, as a function of hole concentration, spin splitting parameter $B_G$, and strain. The trend line (red) demonstrates the effect that increasing temperature has on the system for a given hole concentration.

We can see from the data, that as the temperature increases, the magnetization of all of the samples increasingly aligns itself along [001]. This means that the anisotropy field necessary to fully align the magnetization along the hard axis of magnetization is increasing. However, for all the samples, this reaches a peak at approximately 45 K,
whereupon the anisotropy field necessary to fully align the magnetization along the hard axis is decreasing. This behavior is in qualitative agreement with the theoretical predictions for tensile-strained (Ga,Mn)As, as shown in figure 3.13. What we observe in the prediction plotted in figure 3.4 is that as the spin-splitting parameter is reduced, the spin reorientation transition for a given hole concentration will occur at lower hole concentrations. This is seen from the inflection point in the curve shifting to lower hole concentrations. As previously mentioned in section 3.3, as we increase the temperature, this has the effect of decreasing the spin-splitting parameter, $B_G$. For a given hole concentration, this has the same effect as if the spin-splitting parameter stayed the same but the hole concentration increased.

Mean-field theory also predicts that (Ga,Mn)As has a contribution to the anisotropy from the $p$-$d$ exchange, beyond the contribution from the strain [3, 11]. Although the electrons in (Ga,Mn)As are in the $d^5$ configuration, and thus have no orbital momentum (and therefore, no anisotropy would be expected) the theory states that the ferromagnetism is mediated by the holes, which have an orbital momentum. In figure 3.14, taken from reference 12, we have the same predictions as in figures 3.4 and 3.13 for an unstrained (Ga,Mn)As film. As can see from the curve, the easy axis is predicted to switch between the [110] and [100] directions.
Figure 3.14: Reprinted from reference 3, the mean-field theoretical predictions for the necessary magnetic field to align the magnetization along the hard axis of magnetization for unstrained (Ga,Mn)As, as a function of hole concentration, spin splitting parameter $B_G$, and strain.

Beyond the data taken with the magnetic field along the [110] direction, similar data was also taken with the magnetic field along the [100] direction. This data for the 120 nm thick sample is plotted in figure 3.15, alongside the data for the [110] direction. As can be seen from the graph, the anisotropic behavior varies between the two directions. Since the contribution to the magnetic anisotropy from the tensile strain is the same for both sets of data, the difference must arise from the contribution from the $p-d$ exchange.
Figure 3.15: \( \frac{(\theta^r_k)^{\text{Polar}} - (\theta^r_k)^{\text{Long.}}}{(\theta^r_k)^{\text{Polar}} + (\theta^r_k)^{\text{Long.}}} \) as a function of temperature for the 120 nm thick (Ga,Mn)As sample, with the magnetic field aligned along the [100] and [110] directions.

Now let us look at what happens to the magnetic anisotropy after we have annealed the samples. In Figure 3.16, we have graphed \( \frac{(\theta^r_k)^{\text{Polar}} - (\theta^r_k)^{\text{Long.}}}{(\theta^r_k)^{\text{Polar}} + (\theta^r_k)^{\text{Long.}}} \) for the 15 nm sample after it has been annealed in air for 2 hours at 250 °C. For comparison, we have also included the data for the as-grown case. In this annealed case, the field chosen for comparison is 1800 Oe, but the comparison field for the as-grown is 1080 Oe.
Figure 3.16: \( \frac{(\theta^p)^{\text{Polar}} - (\theta^l)^{\text{Long.} \}}{(\theta^p)^{\text{Polar}} + (\theta^l)^{\text{Long.} \}} \) as a function of temperature for the 15 nm thick (Ga,Mn)As sample, a) after it has been annealed in air for 2 hours at 250 °C, and b) for the as-grown case.

We know from previous studies that for (Ga,Mn)As, low temperature annealing can change the magnetic behavior of the films [12-14]. As discussed in Chapter 1, Mn interstitials and As anti-sites act as donors, and compensate the carriers from the substitutional Mn. Low temperature annealing has the effect of removing some of the Mn interstitials, most likely to the free surfaces, where they are passivated. This results in a higher carrier concentration in the sample. Thus, though it was not directly measured
in these samples, it is almost certainly true that upon annealing, the carrier concentration of the samples was increased.

The annealed data also agrees well with the theoretical predictions. The increase in carrier concentration has moved the position of the data, when compared to the theory in figure 3.4, to the right. Comparing the two sets of data, we see that upon annealing, the plateau of the data lasts longer than that of the as-grown sample. This is not unexpected though, as low-temperature annealing will also affect the amount of strain in the system, and thus will change the overall shape of the curve [15].

Finally, a third experiment was done with the samples, as shown in Figure 3.17. A second laser, with a wavelength of 488 nm, was placed incident on the same spot on the sample as the probe. As the power of the second laser was increased, the magnetization vector shifts closer to the [001] direction, the same behavior observed as when the temperature of the sample was increased. This is most likely due to a bolometric, or heating, effect since the (In,Ga)As layer has a poor thermal conductivity. Consistent with this explanation, thinner samples required less intensity from the second laser in order to achieve larger shifts of their magnetization towards [001].
Figure 3.17: \( \frac{(\theta_{k}^{s\text{Polar}} - \theta_{k}^{s\text{Long}})}{(\theta_{k}^{s\text{Polar}} + \theta_{k}^{s\text{Long}})} \) as a function of laser pump intensity for the 30 nm thick (Ga,Mn)As sample.
3.5 Conclusion

In summary, we have used component-resolved MOKE to directly probe the magnetization of a set of tensile-strained (Ga,Mn)As samples, both as-grown and annealed. The data suggests that as-grown samples can have a substantial portion of their magnetization in the plane of the sample, even at relatively low fields. Both the effect that annealing has on the samples, and the temperature dependent behavior of the magnetization appear to agree well with mean-field theoretical predictions. Finally, a bolometric effect was observed, most likely due to the poor thermal conductivity of (In,Ga)As.
Bibliography


Chapter 4

Materials Characterization Studies of Antiferromagnetic MnO Layer

In the introduction, we demonstrated that it is possible to exchange bias (Ga,Mn)As by growing a layer of MBE-grown Mn on top of the (Ga,Mn)As. However, initially, there was little information known about the composition of this Mn layer, other than the expectation that the epitaxial layer deposited by MBE would oxidize. There are several species of $\text{Mn}_x\text{O}_y$: MnO, MnO$_2$, and Mn$_2$O$_3$, or the layer could be a combination of multiple species, or have a portion of the Mn remain in its unreacted form of Mn$^0$. Also, the structural composition of the Mn layer was not known. The layer could be amorphous, crystalline, or polycrystalline, or again, a mixture of types. In this chapter, we describe the experiments used to determine the chemical and structural composition of the Mn$_x$O$_y$. Some of these experiments were carried out by collaborators (cross-sectional transmission electron microscopy and Rutherford back-scattering), while others were performed by the author of this thesis (X-ray photoelectron spectroscopy, plan-view transmission electron microscopy and x-ray diffraction).
4.1 Chemical Analysis

4.1.1 X-ray Photoelectron Spectroscopy

X-ray photoelectron spectroscopy (XPS) is an experimental method used to determine the chemical composition of a sample. The underlying principle is simply Einstein's photoelectric effect. X-rays are used to bombard the surface of the sample. When an x-ray photon impacts an atom, an electron from the shell of the atom is ejected, with the kinetic energy given by:

\[ E_k = h\nu - E_b - \phi \]  

(1)

where \( E_k \) is the kinetic energy of the ejected electron, \( h\nu \) is the energy of the x-ray photon, \( E_b \) is the binding energy of the electron, and \( \phi \) is the work function of the spectrometer (specific to each instrument). What makes XPS particularly useful in chemical analysis is that it is very sensitive to the chemical nature of the material, i.e., different chemical binding states will produce slightly different results. Because the electrons can be scattered, etc., the technique is sensitive to approximately the first 5 nm of sample on the surface. However, it is also possible to ion mill the sample in order to achieve depth profiling.

The surface and subsurface of the sample was characterized using a Kratos Analytical Axis Ultra system, with the x-ray source being monochromatic Al K\( \alpha \) x-rays (\( \nu = 1486.6 \text{ eV} \)). Low energy electrons (< 5 eV) were used for charge neutralization.
The charge-induced shifts in the lines were corrected by using the binding energy of the C-C hydrocarbon line (285 eV) as an internal check. For the depth profiling, data was acquired while simultaneously sputtering away the surface of the sample with 4 keV Ar⁺.

As shown in the previous chapter, in order for the exchange bias to exist in the Mn/(Ga,Mn)As structure, a certain amount of ex-situ annealing must be done. Also as discussed previously, there are two growth protocols. In the second growth protocol, the one used in this study, only two edges of the sample are attached to the mount with indium, with the middle section of the sample suspended. After removal from the growth chamber, the center portion is cleaved in order to prevent any heating. Thus, through XPS, we can observe any changes in the chemical structure of the sample before and after annealing.

Depth dependent XPS measurements were performed on a sample grown using the second protocol. Two pieces were then taken from the indium-free portion, and one of the pieces was subsequently annealed in atmosphere at 200 °C for one minute. For such a measurement, the time spent sputtering away the free surface of the sample is proportional to the depth below the surface. Figure 4.1, shown below, depicts the high resolution Mn 2p XPS spectra for the annealed portion of the sample. The Mn 2p 3/2 line from the annealed piece is centered around approximately 641.0 eV, and its position is in agreement with the binding energy of Mn²⁺, indicating the formation of MnO. This line points away from the other possible chemical bondings, because metallic Mn⁰ has a 2p 3/2 line at 639 eV. Also, MnO₂ (Mn⁴⁺) has a binding energy of ~642.5 eV, while Mn₂O₃ (Mn³⁺) has an energy of ~641.7 eV. There also exist two satellite lines spaced 5.5 eV
away from the 2p_{3/2} and 2p_{1/2} lines. These satellite excitations are typical for a MnO structure, and are not present in either Mn_2O_3 or MnO_2 [1 - 3].

Figure 4.1: Mn 2p XPS data for the annealed portion of the sample, before depth profiling was performed.

The shape and position of the lines remain constant with the depth profiled, with only the intensity decreasing due to the decrease in the Mn content, as shown in figure 4.2. This is evidence that the annealed film is nearly uniformly oxidized with MnO being the dominant form of Mn throughout the antiferromagnetic layer.
Figure 4.2: Depth dependent Mn 2p XPS measurements taken of a MnO/(Ga,Mn)As sample after post-growth annealing in atmosphere for 1 minute at 200 °C.

Depth profiling XPS measurements were also performed for the as-grown indium-free sample, as shown in Figure 4.3. In this case, the Mn$_{3/2}$ line has a low binding energy.
shoulder after 60 seconds of Ar⁺ sputtering, and after 90 seconds the line shifts position to 639 eV (which is the expected position for metallic Mn⁰). After 90 seconds of sputtering the satellite lines disappear as well. This is evidence that although the surface layer of the as-grown piece is oxidized into MnO, metallic Mn⁰ is the dominant species towards the lower region of the layer. There are a few options for the composition of the bottom layers: elemental Mn and Mn bonded metallically in a compound such as MnGa or Mn₂As would be consistent with the data. However, previous studies done on Mn grown on GaAs would seem to point towards Mn₂As: Jin et al reported the formation of a Mn₂As-type Mn-Ga-As interfacial layer during Mn growth on GaAs at 400 K [4]. Also, Hilton et al found that an epitaxial Mn₀.₆Ga₀.₂As₀.₂ layer consisting of tetragonal Mn₂As and MnGa formed between Mn and GaAs from solid state interfacial reactions during annealing [5]. Finally, a recent in-situ XPS study showed that Mn growth on GaAs at temperatures as low as 95 °C led to the formation of an eleven monolayer thick Mn₀.₆Ga₀.₂As₀.₂ interfacial reacted layer [6].
Figure 4.3: Depth dependent Mn 2p XPS measurements taken of the same MnO/(Ga,Mn)As sample, as grown.

Further evidence of an oxidation reaction is presented in Figure 4.4. Shown in the figure are the depth dependent oxygen concentrations for the same sample: as-grown and annealed in atmosphere at 200 °C and 250 °C for one minute. Both of the annealed samples take substantially more sputtering time in order for the oxygen concentrations to decrease, with the sample annealed at 250 °C taking only slightly more time. Since the only change in the three samples are whether or not they were annealed, it stands to reason that the increase in the oxygen concentration further into the sample comes from the annealing (oxidation) process.
Figure 4.4: Depth dependent XPS oxygen concentrations for a MnO/(Ga,Mn)As sample, as grown and annealed in atmosphere for one minute at 200 and 250 °C. Sputtering time is proportional to the depth into the sample.
4.1.2 Rutherford Back Scattering

Rutherford back scattering (RBS) experiments were performed by our collaborators at the University of Minnesota in order to characterize the chemical nature of the material as well. RBS uses Rutherford's classic experiment with back-scattering off of atoms in order to determine the atomic number, concentration and thickness of the material. In the experiment, the material is bombarded with ions, the kinetic energy of the back-scattered ions is analyzed, and from this the chemical nature of the material is understood. RBS was performed using 1.4 MeV and 2.3 MeV with 20 µC of integrated charges of He\(^+\) ions in normal (165°) and glancing (108°). Random and <100> channeling measurements were performed to determine the composition and depth profile of the structure. Simulations of the random RBS spectra, as shown in Figure 4.5, agree with the XPS data in that there is formation of MnO with no detectable Ga or As at the surface. The glancing angle <100> channeling RBS data corresponds to more Ga than As (∼5 x 10\(^{15}\) atoms/cm\(^2\)) than the ∼1-2 x 10\(^{15}\) atoms/cm\(^2\) expected for an abrupt interface. Although this increase in the Ga and As would correspond to a ∼2nm Mn\(_{0.6}\)Ga\(_{0.2}\)As\(_{0.2}\) layer with no ion channeling, because the films are grown epitaxially, some channeling may be expected and the reacted layer may be thicker [7].
Figure 4.5: Reprinted from reference 8. RBS channeling spectra of a Mn/(Ga,Mn)As sample before (solid line) and after (dotted line) post-growth annealing.

2.3 MeV He\(^+\) beams were used in a glancing angle geometry.
4.1.3 Transmission Electron Microscopy

Our collaborators at the University of Minnesota also performed cross-sectional transmission electron microscopy (TEM) and energy dispersive spectrometry (EDS) on an as-grown sample grown using the first growth protocol of complete In bonding. For more information regarding EDS and TEM, please refer to the appendix. The samples were prepared by chemical mechanical polishing, dimpling and ion milling with 2.7 keV Ar⁺. The microscopy was performed using a Philips CM30 TEM with an operating voltage of 300 kV. In Figure 4.6(a), there is dark horizontal band, approximately 2.3 nm thick, at the MnO/(Ga,Mn)As interface. The sample does not show any exchange biasing, which suggests that the interfacial reacted layer may consist of Mn₀.₆Ga₀.₂As₀.₂. This is also consistent with the RBS channeling results from the as-grown In-free samples which also did not show exchange bias. EDS done confirms the surface layer as MnOₓ with a thickness of ~9 nm. Upon annealing the In-free samples in air, the samples exhibit exchange bias and the RBS channeling interfacial Ga and As peaks increase slightly (~1 x 10¹⁵ atoms/cm²). Cross-sectional TEM, shown in Figure 4.6(b), of a sample that does demonstrate exchange bias shows a thin bright horizontal line at the interface, by contrast. The change in contrast is consistent with a decrease in density and the increase in the channeled Ga and As interfacial RBS yields as a result of the oxidation of the interfacial Mn-Ga-As.
Figure 4.6: Reprinted from reference 8. Cross-sectional TEM of MnO/(Ga,Mn)As samples with complete In bonding. In a) the sample showed no exchange bias, and in b) the sample showed exchange bias.

4.1.4 X-ray Reflectometry

Finally, we obtained further evidence of the interfacial reaction layer from x-ray reflectometry (XRR) measurements performed on a 10 nm thick (Ga,Mn)As sample that is capped with a Mn layer which is nominally 10-nm in thickness. The second growth
protocol was used, and the indium-free section of the sample was measured. Although the samples were not exchange-biased, the Mn thickness was chosen in order to be able to use XRR. XRR uses glancing angle x-rays in order to probe the thickness, roughness and density of thin films by monitoring the changes in the critical total reflection angle and the oscillations in the reflectometry. Figure 4.7 shows the XRR data from this sample, both as-grown and post-annealed, as well as attempts to fit the XRR data using an oxide-metal-semiconductor tri-layer structure as the theoretical model. After the sample is annealed in atmosphere at 200 °C, the thickness of the MnO layer increases, while the metallic layer decreases, until finally a uniform oxide film is made.
Figure 4.7: Reprinted from reference 8. XRR measurements for an In-free sample and the fits of the data. In a) the sample is as-grown and the fit assumes a oxide-metal-semiconductor structure. In b) the same sample is shown after annealing, and the fit assumes a MnO layer.
4.2 Structural Analysis

4.2.1 X-ray Diffraction

X-ray diffraction (XRD) is one of the standard methods to probe the lattice properties of a material. The technique uses Bragg's equation, given by:

\[ \lambda = 2d \sin(\theta) \]  

where \( d \) is the lattice spacing, \( \theta \) the incident angle of the x-ray, and \( \lambda \) the wavelength of the x-ray.

In order to probe the lattice constant of the MnO layer, a nominally 60 nm thick Mn layer was grown on a GaAs buffer layer with a GaAs substrate using the first, or completely In-bonded, growth protocol. The sample was then measured using a Philips X'Pert Pro MRD XRD machine. The machine was used in the mirror optics set-up (no hybrid monochromator). As a control sample, a piece of GaAs substrate was also measured.

In Figure 4.8, the XRD data is plotted. In particular, Figure 4.8(b) shows the zoomed in section of the data, showing the difference between the two samples. The line at ~59.3° in 2θ corresponds to a known line for the (220) spacing for MnO with a NaCl lattice type and a lattice spacing parameter of 4.43 Å [9]. This means that at least some of the material in the sample is crystalline MnO. The amplitude of the signal from the
(220) line is quite small, and none of the other expected lines for MnO were apparent in the data. However, the small amplitude of the signal from the (220) line may mean that although the other lines are there, they are indistinguishable from the background noise. It is also possible that although part of the sample is in a NaCl crystalline formation, the sample could be polycrystalline or amorphous.
Figure 4.8: XRD data of a MnO/GaAs sample with complete In bonding where the Mn grown was nominally 60 nm. A) the overall spectrum, with b) zoomed-in about the Mn (220) peak.
4.2.2 TEM Diffraction

In order to see whether the material was polycrystalline, single crystal, or partially amorphous, we prepared a plan-view TEM sample. For details of the sample preparation, please refer to the appendix. Using the TEM in diffraction mode is a fairly simple method to determine the crystallinity of the sample. If the sample is polycrystalline, the diffraction pattern will show up as either rings or a circle of spots, as the Bragg conditions are satisfied in multiple orientations all with the same lattice spacing. For a sample that is single crystal, the diffraction pattern will be an array of spots. Finally, for an amorphous sample, there will be a large background in the diffraction pattern.

Figure 4.9 shows the diffraction pattern for the plan-view sample along the [002] zone axis. The position and array of the spots agree with those expected for MnO in a NaCl crystalline formation. The background around the diffraction spots arises from beam saturation. No evidence of polycrystalline or amorphous regions was found. However, it is possible that the material is polycrystalline but highly textured. Therefore, we conclude that the lattice structure of the MnO film is NaCl.

TEM is a site specific measurement technique which measures only a small section of sample, whereas XRD is a bulk measurement. Although the zone axis was along [002], this does not preclude that the overall orientation is preferentially along [022] due to the site-specific nature of the measurement. Furthermore, since the XRD signal from the MnO layer is relatively small compared to the overall signal from the substrate, it is possible that the signal arising from the [002] section is indistinguishable from noise.
4.3 Annealing Study

It was shown in the introduction chapter that some annealing is necessary to achieve exchange bias in the system. However, in none of the samples grown using the second protocol (that of the partially indium-backed growth method described in Chapter 1), was the exchange field of the post-annealed sample greater than that of the indium backed portion of the same sample. This was attributed to the annealing conditions being less than optimal, and it was hoped that with the proper annealing conditions, the
exchange field could be controlled and increased. Towards this end, we carried out an annealing study of the (Ga,Mn)As/MnO system.

A sample of $\text{Ga}_{0.94}\text{Mn}_{0.06}\text{As}/\text{MnO}$ was grown using the second protocol. The (Ga,Mn)As was 10 nm in thickness, while the Mn layer was ~8 nm. The sample was cleaved into many pieces, in order to study the affect that annealing time and annealing temperature has on the exchange bias. The samples were annealed in a rapid thermal annealing furnace, for varying temperatures and times, as indicated in Table 4.1. The annealing was performed in an oxygen overflow environment. Finally, when the samples were not being used, they were kept in a dry box with a nitrogen flow, in order to prevent any annealing in air over time. The exchange field magnitude was measured via MOKE, in the diode bridge configuration.
<table>
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<th>Annealing Time (minutes)</th>
<th>Exchange Field (Oe)</th>
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<td>1.5</td>
<td>0</td>
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<tr>
<td>200</td>
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<td>200</td>
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Table 4.1: A chart detailing the annealing conditions used in the study, with the annealing time, temperature, and the resulting exchange bias field. All measurements were performed at 4 K, with the magnetic field aligned along [100].

Table 4.1 shows the exchange field obtained for different annealing conditions. As can be seen from the chart, we could not determine any systemic effect on the exchange bias magnitude when varying the time or temperature of the annealing process. However, it is possible that the samples themselves were less than optimal, as the exchange field of the indium backed portion also showed little exchange bias. It is
important to note that there is an odd behavior in the hysteresis loops for any sample annealed in a temperature of 280 °C or greater, as shown in Figure 4.10. However, this is most likely due to the fact that it is known that annealing bulk MnO at a temperature of ~300 °C will form other species of manganese oxides, and the behavior of the hysteresis loops can most likely be attributed to this [10].

Figure 4.10: MOKE measurements for the sample annealed at 360 °C for 1.5 minutes in an oxygen environment. The magnetic field is aligned along [100] (black) and [110] (red). All samples annealed at 280 °C or greater show similar behavior to some degree when the magnetic field is aligned along [100].
4.4 Conclusion

XPS, RBS, XRR and cross-sectional TEM were all performed to determine the chemical nature of the MnO layer. The oxide appears to be MnO, with an interfacial layer of Mn$_{0.6}$Ga$_{0.2}$As$_{0.2}$, which reduces in thickness upon annealing. XRD and plan-view TEM diffraction were also performed to determine the structural composition of the samples. The lattice structure of the MnO film was determined to be NaCl.
Bibliography

Chapter 5
Closing Remarks

5.1 Summary

In Chapter 1, it was shown that (Ga,Mn)As becomes ferromagnetic when Mn is substituted into the Ga sites in the GaAs lattice structure. The material characteristics were discussed, with particular attention paid to the role of As anti-site and Mn interstitial defects. Since the ferromagnetism of the Mn spins is mediated by the itinerant holes donated by the substitutional Mn, and both defects compensate these holes, the importance of defects in (Ga,Mn)As can not be overstated.

We also introduced the theory behind exchange-biasing, and also demonstrated the successful exchange-biasing of (Ga,Mn)As, which marks the first successful exchange biasing of a semiconductor. Experiments were performed in order to improve the quality and consistency of the exchange field, via low temperature annealing. However, the results were mixed at best.

In Chapter 2, we introduced the magneto-optical Kerr effect experiment, and the theory behind it. Also introduced were the three iterations of the experiment, as they successively improved the quality of the data, eventually leading to the ability to measure the Curie temperature in (Ga,Mn)As.
Chapter 3 dealt with the component-resolved magneto-optical Kerr effect experiment. This experiment is primarily a way to measure samples that are close to undergoing a phase transition in the easy axis of magnetization. It was shown that the mean-field theory originally introduced in Chapter 1 successfully predicts the behavior of tensile-strained (Ga,Mn)As, which expands the prediction beyond that of compressively-strained samples, which had been previously seen.

Finally, Chapter 4 discussed the efforts to characterize the chemical and structural nature of the MnO layer used to exchange bias (Ga,Mn)As. Upon performing x-ray photoelectron spectroscopy, Rutherford back scattering, transmission electron microscopy, and x-ray reflectometry, it was shown that the species of manganese oxide is MnO. After x-ray diffraction studies and the use of transmission electron microscopy diffraction, it was determined that the MnO was single crystal, with a NaCl-type lattice structure.

5.2 Future Directions

The future direction of these projects is largely divided, like the thesis, into two parts. For the MOKE and component-resolved MOKE experiments, the basis of the experiment is largely finished. Both have shown to be able to provide reliable and accurate data, with the one caveat being ferromagnetic samples capped with a reflective layer. By and large, these two experiments only await samples to measure. For example, one interesting experiment that could be performed with the component-resolved MOKE, would be to test whether the mean-field predictions for high carrier concentration
samples are valid. The mean-field theory predicts that, just as with the low hole (or low spin-splitting parameter) concentration samples, the high concentration samples should also undergo a phase transition.

For the exchange-biasing experiments, quite a bit of work remains to be done. Although the crystalline and chemical structure of the MnO layer was successfully characterized, the exchange bias effect itself remains small, and to some extent dependent upon the person performing the growth in the MBE chamber. It may be worthwhile to explore alternative methods of creating the MnO layer. For example, it may be possible to sputter the MnO onto the (Ga,Mn)As layer after the oxide has been removed *in-situ*. Alternatively, there has been work done in creating MnO by electrochemical deposition. In this case, the MnO tends to form amorphously. However, the role of the interface and crystallinity of the anti-ferromagnet in exchange bias is still under dispute. By creating a single-crystal system (via MBE) and an amorphous/single-crystal system (via electrochemical deposition), it could shed useful light upon this debate.
Appendix A

Transmission Electron Microscopy

In this appendix, the details of the experiments performed with transmission electron microscopy (TEM) are discussed. A brief theoretical background of the experiment is given, as well as the procedure for creating a plan-view and cross-sectional TEM samples, in particular for the MnO exchange bias samples, as there are certain precautions that must be taken when preparing those samples. Finally, some of the images taken are presented.

A.1 TEM Background

Transmission electron microscopy is a very useful technique for analyzing materials. TEM is used over other spectroscopy techniques because it can provide several things. First, because the source of the probe beam is highly charged electrons (a typical operating voltage for the LaB$_6$ TEM at Penn State is 200 keV), the spatial resolution, which is wavelength dependent, is typically on the order of angstroms. Spatially resolved diffraction patterns and chemical information can also be obtained (and this information can be considered from the bulk of the specimen, since the samples are on the order of 100 nm thick), the latter when the TEM is equipped with an energy dispersive spectroscopy (EDS) or electron energy loss spectroscopy (EELS) system.
Finally, the chemical, crystallographic and microstructure information obtained can be correlated, down to atomic length scales.

That is not to say that TEM is not without its drawbacks. The TEM has a very small sampling volume. More importantly, the samples must be prepared in such a way that they are electron transparent. This is time consuming and can be difficult (and extremely frustrating at times!)

This is meant as a very brief background of the theory behind TEM. For a more comprehensive look, I would recommend “Transmission Electron Microscopy” by David B. Williams and C. Barry Carter.

The first kind of contrast in TEM, mass-thickness contrast, arises from the incoherent elastic scatter of electrons – Rutherford scattering. The screened relativistic Rutherford differential cross section is equal to:

$$\frac{d\sigma(\theta)}{d\Omega} = \frac{\lambda_R^2 Z^2}{64\pi^4 (a_o)^2 (\sin^2 (\frac{\theta}{2}) + (\frac{\theta_o}{2})^2)^2},$$

where $\lambda_R$ is the relativistically corrected wavelength, $Z$ is the atomic number, $a_o$ is the Bohr radius of the scattering atom, and $\theta_o$ is the screening parameter [1].

This in turn, is equal to $|f(\theta)|^2$, where $f(\theta)$ is the atomic scattering factor, given by:

$$f(\theta) = \frac{1 + \frac{E_o}{m_o c^2}}{8\pi^2 a_o} \frac{\lambda}{\sin \frac{\theta}{2}} (Z - f_x),$$

where $f_x$ is the exchange term.
where \( E_0 \) is the beam energy, \( m_o \) is the electron mass, and \( f_x \) is the scattering factor for x-rays.

By integrating the differential Rutherford cross section from an angle \( \beta \), the semi-angle of collection of the objective aperture (which is a angle pertaining to the instrument being used), to infinity we can obtain the probability that an electron will be scattered through an angle greater than \( \beta \):

\[
\sigma(\beta) = \frac{[Z^{2/3} \lambda a_o (1 + \frac{E_0}{m_o c^2})]^2}{\pi a_o^2 (1 + (\frac{\beta}{\theta})^2)}
\]

(3)

With all the terms in the equation as previously defined.

As can be seen from the above formula, as the mass of the atoms, \( Z \), increases, this in turn increases the scattering of the electrons. Thicker samples will scatter more as well, as they relate to the above equation through \( \lambda \).

For diffraction and phase contrast imaging, we rely on the fact that the beam undergoes Bragg diffraction as it passes through the material (apart from amorphous materials). There are two types of diffraction contrast imaging: bright field (BF) and dark field (DF). For both of these types, the electron beam must be aligned (ideally) into a two-beam condition. This means that the electron beam will be in two beams: diffracted along a crystalline axis, or not diffracted (as shown in Figure A.1):
Figure A.1: Schematic for a) bright-field imaging and b) dark-field imaging. In a) the diffracted beams are blocked by the objective aperture and only the original beam is transmitted, while in b) the original beam is blocked and only a diffracted beam is allowed to be transmitted to the detector.

Having the beam in a two-beam condition gives the best contrast from any crystalline defects in the sample, as well as being much easier to quantitatively interpret. In phase-contrast imaging, the two beams are allowed to interact, and the resulting phase amplitude difference is periodic with the lattice spacing.
A.2 Sample Preparation

Sample preparation for TEM is an extremely important step. If the preparation is not done correctly, it can introduce new defects into the sample, which can complicate analysis and give false impressions. Using the method about to be described, the samples I have created have shown very little if any damage. For certain steps, a different tactic must be used for the exchange biased samples, and I will note that where relevant. It should also be noted that this procedure is for thin films grown on a GaAs substrate, and for different substrates the conditions may vary. First I will describe the procedure for a plan-view sample and then the procedure for a cross-sectional sample (though the latter shares many of the same steps as the former).

A.2.1 Mechanical Thinning

The first step in the process is mechanically thinning the sample down. A tri-put polisher is used to mechanically thin the sample in a level manner from the substrate side. The goal is to reach ~100 microns. Anything ~80 microns and below will generally not be usable, as it tends to shatter very easily. Typically, the samples are polished down using 15 micron diamond paper until the sample reaches 150 microns in thickness, at which point 1 micron diamond paper is used to polish the remaining 50 microns. A digital micrometer is used to measure the thickness throughout the polishing. Water is used on the diamond paper to help with the process.
Note: For the MnO samples, it is believed that water was contaminating and ruining the exchange bias effect, if the samples came into contact with a large amount of water. Instead of using water with the diamond paper, an oil was used. Also, when attaching the sample to the tri-put polisher, generally Crystal Bond is used. However, for these samples, it is important to minimize the heat exposure. Therefore, the sample holder was only heated to 100 °C using the hot plate – just enough to melt the crystal bond. The time the sample spends on the hot plate should only be a few seconds as well.

A.2.2 Dimpling

Once the appropriate thickness has been reached with mechanical polishing, it is time to dimple the sample. This is accomplished using a Gatan Model 656 Dimple Grinder. Although such a small size is most likely not necessary for the majority of the dimpling, a .01 diamond paste is used throughout the process. Before the sample is bonded to the dimpler mount, it is again measured with the digital micrometer to see the amount to be dimpled. Typically, the sample is dimpled such that the bottom of the dimple is ~15-20 microns in thickness. Upon reaching this point, the sample is very carefully cleaned of any paste, and it is polished using the felt-covered dimpling wheel, which has been soaked in oil.

Once the sample has been dimpled, it is time to remove it from the dimpling mount. The dimpling mount is placed upon a piece of paper and then placed into a Petri dish filled with acetone. The dimpling mount is placed on its side. The acetone in the Petri dish will dissolve the crystal bond, typically in ~5 – 10 minutes. Once the crystal
bond is dissolved, the dimpled sample will float onto the piece of paper (which is used because it makes it very easy to remove the sample from the Petri dish without damaging it).

**A.2.3 Bonding and Milling**

Now it is time to bond the sample onto the sample holder. The sample holders I typically use are molybdenum grids with a 2 x 1 mm oval hole in the center. Molybdenum grids were chosen over copper ones because molybdenum etches much more slowly than copper under ion milling, and copper grids were producing some ion milling damage in the form of sputtered copper on the sample surface. The sample is bonded to the mount with M-bond. The sample is then cured by placing it on a Teflon-tape covered glass side on a hot plate for 20 – 30 minutes at ~150 °C. It is also possible to cure the sample by letting it sit at room temperature overnight. For the MnO samples, this was the way they were cured.

Once the TEM sample has been bonded onto the sample mount, it is time to ion mill. The ion mill used is a Fischione Model 1010 Low Angle Ion Milling & Polishing System. The angle of incidence for the beam is typically 12° (the practical minimum for this machine is said to be 10°), and the sample undergoes continuous 360° rotation. Finally, I found that the samples incur much less damage if the machine is operated at liquid N₂ temperatures. The Ar⁺ beam conditions generally used are 4.0 keV and 3 μA. Using these conditions, the typical ion milling time was roughly 3 – 4 hours. However,
the only way to know for certain if the sample is done is to look at it under the Fischione’s microscope periodically until a hole appears.

A.2.4 Cross-sectional Sample Preparation

By and large, cross-sectional sample preparation is very similar to plan-view sample preparation. Where the two differ is in the initial structure of the sample. All of the cross-sectional samples I have prepared were done in the following manner: A strip of silicon and a strip of sample are cleaved, with the width of the strip being ~2 mm and the length a few cm. Both strips are then cleaved into half, such that the length has been halved. With this done, the sample strips are mounted onto the tri-put polisher, and the indium used to mount the sample for MBE growth is polished off, using the method described earlier. Once this is done, the samples are bonded together in a sandwich-like structure, as illustrated in Figure A.2. The samples are bonded using M-bond. It is important that the surface of each layer is totally covered with the M-bond, however, not excessively. Once this is done, the sample is covered in Teflon-tape, placed inside a clamp-like device, and put on the hot plate for ~30 minutes at ~150 – 200 °C. The size of the sandwich-like structure should not exceed 3 mm in any dimension.

Note: For the MnO sample, it is possible to create the structure using the same method. However, when it comes to the curing process it is necessary to not use the clamp device. It is better to have the hot plate at ~125 °C, and to manually press down on the sandwich structure, which is laying on a Teflon-tape covered glass slide, for 1 – 2
minutes. This minimizes the time spent on the hot plate, but it sometimes has led to the samples not curing strongly enough (such that during the other steps, the Si-sample or sample-sample interface breaks).

Figure A.2: Top-down view of the schematic for the sandwich-like structure used in cross-sectional sample preparation. The polished side of the silicon is bonded to the substrate side of the sample pieces using M-bond, and the thin films are bonded together in the same manner.

The other steps in the preparation of a cross-sectional sample are the same, with the caveat that the sample is polished, dimpled, and milled such that the 4 layers are all visible from a top-down perspective. Also, when the hole appears in the sample, if it does not appear at the sample-sample interface, it must be allowed to grow in size until it reaches the interface.
A.3 TEM Images

The first set of images were taken at the University of Minnesota using a Philips CM30 TEM with an operating voltage of 300 keV. They are of the 30 nm tensile strained sample discussed in Chapter 3. The goal was to see how many dislocations existed in the sample and if they were concentrated in the thicker portion of the sample (implying that they existed more heavily in the (In,Ga)As layer). Figures A.3(a) – A.3(c) are of the thinner portions of the sample, and showed that the dislocations extended into the (Ga,Mn)As layer. However, Figure A.3(d), which is in a thicker section of the sample, shows that although the dislocations are present in the (Ga,Mn)As layer, they are more heavily concentrated in the (In,Ga)As layer. All of the images are in bright-field mode.
Figure A.3: A, b, and c) BF TEM images of dislocations present in the (Ga,Mn)As layer. d) shows the thicker section of the sample, where the dislocations are more concentrated.
The following images were taken, at Penn State using a LaB$_6$ TEM, of two separate quantum well structures used to make micro-disk lasers. They consist of five quantum wells, and the goal of the imaging was to make sure that the layers were of the appropriate thicknesses. Figure 6.4(a) is a bright-field image of a micro-disk laser sample where the middle quantum well is supposed to be roughly one half the thickness of the other quantum wells. Figure 6.4(b) shows a micro-disk sample where the quantum wells are evenly spaced, and is a dark-field image using the (220) spot.

Figure A.4: TEM micrographs of two different quantum well structures used in micro-disk lasers. In a) a BF image, and in b) a DF image using the (220) spot.
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