

Exchange Bias in [Co/Pd]/IrMn Thin Films

Young Byun

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Bachelor of Science

Karine Chesnel, Advisor

Department of Physics and Astronomy

Brigham Young University

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ABSTRACT

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We are studying ferromagnetic materials made of Co/Pd multilayers. Vibrating Sample Magnetometry (VSM) is one of the magnetometry instruments for determining magnetic properties in high magnetic fields and in a temperature range of 10 - 400 K. My research project is to measure exchange bias effects in Co/Pd ferromagnetic multilayers interfaced with IrMn anti-ferromagnetic layers. This system, made at Hitachi, presents magnetic domains in the range of 200 nm in width. The magnetization in the domains is alternatively pointing up and down, perpendicular to the layer. The question is to understand the behavior of the net magnetization with applied field and study bias effects in different field cooling conditions. We have studied the film in various states such as field-cooling states and zero-field-cooled states, and we have also studied the remanent coercive point.

Keywords: Ferromagnetic films, anti-ferromagnetic films, Exchange bias, Vibrating Sample Magnetometry

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Chapter 1

Introduction

The system I have been studying is an exchange bias thin film composed of ferromagnetic and anti-ferromagnetic layers. I will describe these magnetic phases in the following paragraphs. I will then describe the composition of our sample and our plan of study.

1.1 Magnetic phase and Exchange coupling

The atoms in magnetic materials carry intrinsic magnetic spins, but the nature of the coupling between these spins give rise to different behaviors. When different states are put together, they can interact and induce exchange couplings. There are different types of magnetism in nature: diamagnetism, paramagnetism, and ferromagnetism are the most common phases. For diamagnetism, a magnetic moment is induced in the direction opposite to the field. In case of paramagnetism, spins are aligned along the applied field. In a ferromagnetic film, there are intrinsic spins that tend to align even in the absent of field, and they form domains. Also, hysteresis effects can be observed in a ferromagnetic film. Hysteresis indicates that reversal processes of the magnetic domains are not reversible. The net magnetization is not following the scale behavior when the magnetic field is ramped in the ascending or descending way.

1.1.1 Ferromagnetism

Ferromagnetism (F) is one type of magnetism where the magnet spins in the domain are aligned in the same direction in Figure 1.1.A, so H_C material has a net magnetization. Materials containing atoms of Co, Iron, and Fe are usually ferromagnetic. One key feature of ferromagnetic materials is that there is still a magnetic moment after the external field is turned off, unlike paramagnetic and diamagnetic materials. When we apply a strong magnetic field to a ferromagnetic material, the magnetic spins tend to align with field. When field is reversed, domains start to nucleate and reverse in the direction opposite to the field. The spins of ferromagnetic atoms are all aligned and form domains. The size of a single domain is typically between 100 nm and 1 μm . In our sample, it is about 200 nm. In ferromagnetic material, the magnetic coupling between the spins is so strong that they tend to align, creating a powerful net moment at the macroscopic scale. When spins align in a thin film, they form domains at the microscopic scale as domain naturally appear when symmetry is broken by the shape of the material. Ferromagnetic materials are best known for their nonlinear responses to an applied magnetic field, which allow them to form the permanent magnets [1]. In ferromagnetic materials, there can be a net magnetization even when external magnetic field is zero; this is called remanent magnetization. We study the behavior of magnetization in ferromagnetic thin films, typically about 10 nm to 50 nm in thickness.

1.1.2 Anti-ferromagnetism

In anti-ferromagnetism (AF) materials, the magnetic spins in the domain are alternately up and down like in Figure 1.1.B, so the net magnetization is generally zero in the bulk of the material. AF is hard to measure, but we can observe coupling effects between F and AF materials that are called exchange couplings. At the interface between the F and AF layers, there are uncompensated spins that form domains. Exchange coupling can then occur between the AF domains and the F domains at the interface.

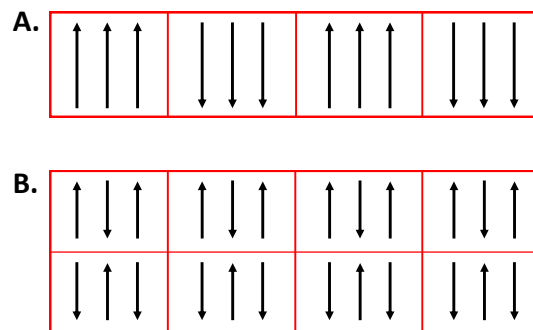


Figure 1.1 A: Sketch showing the alignment of spins in a ferromagnetic film, and formation of ferromagnetic domains. B: Sketch showing the alignment of spin in a anti-ferromagnetic materials.

1.1.3 Exchange Bias

Exchange bias occurs when a Ferromagnetic (F) layer is placed at the vicinity of an anti-ferromagnetic (AF) layer. The bias effect is induced in the F layer by uncompensated spins at the interface with the AF layer, and results in a shift in the magnetization loop, meaning that ascending and descending branches are not symmetric about the origin but shifted in field value. Exchange bias (EB) arises from the anisotropy found in a (F) layer exchanged-coupled to an (AF). It was first observed about 50 years ago, and is widely exploited in magnetic sensor technologies [2]. A full theoretical understanding of the phenomenon remains elusive. When a F thin film in contact with an AF thin film is cooled through the AF layer under an applied magnetic field, the hysteresis loop of the F film develops a loop shift and an enhanced coercivity. These exchange-biasing effects arise as the spin order of the AF film is established in the presence of the F film through the interfacial F exchange interaction [3]. The exchange coupling between the interfacial planes of spins are acting on either side of the F/AF interface [4]. My IrMn exchange-biased films are promising building blocks for the fabrication of magnetic spin valves and tunnel junctions with out of plane magnetization [4].

1.2 Description of our EB film

In my research, the samples I have been studying are multilayered thin films consisting of Co/Pd multilayers interspaced with IrMn layers. My ferromagnetic film is made of Cobalt and Palladium multi-layers, and our AF layers is made of IrMn. The structure of the film is $[[\text{Co}(4\text{\AA})\text{Pd}(7\text{\AA})]_{\times 12} / \text{IrMn}(24\text{\AA})]_{\times 4}$. The bracket is used indicate a multilayer and a number behind bracket means the number of repetition in the multilayer. The thickness of the singer layers are 4\AA of Cobalt(Co) and 7\AA of Pd(Palladium) repeated 12 times then interlayed with IrMn alloy. The total thickness of the film is therefore 228\AA or 22.8 nm . Co/Pt and Co/Pd multilayers have been intensively investigated as the next generation of magneto-optical recording materials [5]. This film is made at Hitachi, San

Jose, CA [6].

1.3 Plan of study

I have been studying how bias changes with temperature and how bias changes with different cooling field. Moreover, I have been studying how the shape of the loop changes with different field cooling. To measure the amount of bias, we have been using Vibrating Sample Magnetometry (VSM), which is one of the magnetometry instruments used to determine magnetic properties. I will describe the VSM technique in the next chapter.

Chapter 2

Experimental Techniques

2.1 Vibrating Sample Magnetometry

To measure the exchange bias, we use VSM. Many methods are available to measure magnetization among which the most common ones are the force method and the induction method. The former involves the measurement of the force experienced by a magnetic dipole moment in a known uniform field gradient as in a Faraday balance, and the latter involves the measurement of magnetic induction due to the relative motion between the sample and the detection coil system as vibrating sample magnetometer (VSM) [7].

A Vibrating Sample Magnetometer (VSM) is one of the magnetometer instruments used to determine magnetic properties. This experimental technique was introduced in 1956 at MIT by Simon Foner who started the commercialization of these magnetometry in the sixties as reported in [7]. Many papers on VSMs have been published; commercial VSMs with various configurations are also available. A review of the VSM was published by Foner. [8] The principle of VSM is to measure the magnetization of a sample by detecting the electromagnetic force induced in a coil

when magnetic flux is changing in time (Faraday's law):

$$\frac{\partial\Phi}{\partial t} = -V_{em}, \quad (2.1)$$

where Φ is the magnetic flux and V_{em} is the voltage.

Any change in the magnetic flux of a coil of wire will induce a voltage. No matter how the change is produced, the voltage will be generated. We use this effect to quantify the magnetic moment M in the sample as a function of applied magnetic field $H(\text{Oe})$, or temperature $T(\text{K})$. Our VSM instrument consists of the following subsystems: superconductor and power supply, sample vibrator with associated electronic current, detection coils, signal recovery using lock-in-amplifier, cryostat and sample holder [9]. The linear motor is moving up and down at 40 HZ.

I have used BYU's recently-acquired Vibrating Sample Magnetometer(VSM) for all of our magnetization measurements. Our instrument can operate in high magnetic fields up to 9 T and in the temperature range of 10K - 400K. Fig. 2.1 is the VSM instrument we use, and Fig. 2.2 shows a sketch of the sample and detection coils.

2.2 Hysteresis Loop

Ferromagnetic materials are interesting because they exhibit hysteresis. A great amount of information can be learned about the magnetic properties of a material by studying the hysteresis loop.

Fig. 2.3 shows an example of a hysteresis loop from our [Co/Pd]/IrMn film. In this graph, there are four remarkable points we can observe, labeled A to D in the figure. If we start at positive saturation and as the applied field is decreased, magnetization in the sample follows the descending branch. Point A corresponds to nucleation, where domains antiparallel begin to form. At point B, the external field is zero, but there is still some magnetization; this is called remnance, and is what makes permanent magnetization possible. As the field continues to decrease (now getting



Figure 2.1 Picture of our VSM instrument: A black cylinder contains a superconducting magnet, and the attachment top of it is the linear motor for vertical motion.

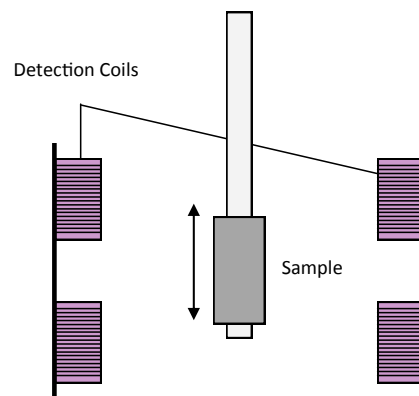


Figure 2.2 Sketch of sample and detection coils in VSM

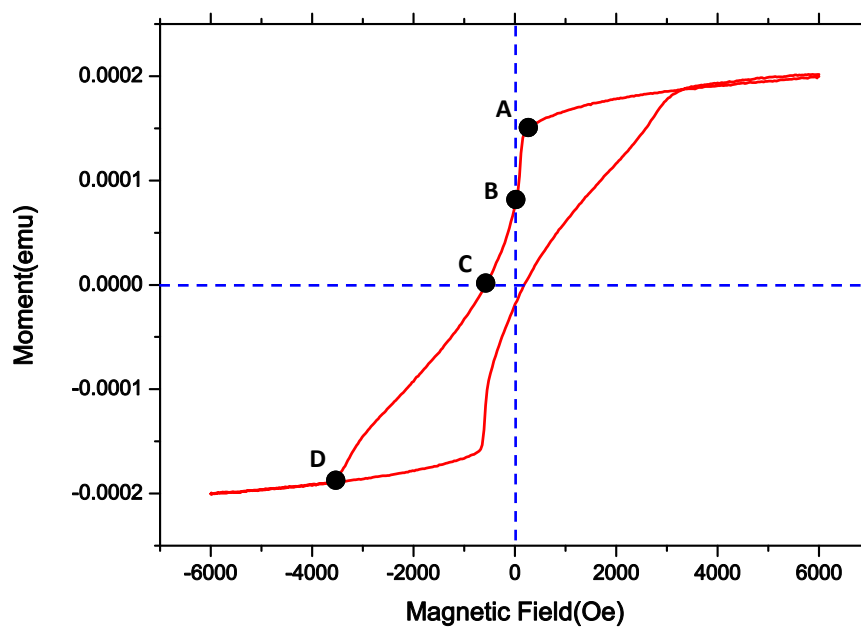


Figure 2.3 [Co/Pd]/IrMn sample of Ferromagnetic hysteresis loop at room temperature measured using VSM. A, nucleation; B, remnance; C, coercive point; and D, saturation. The ascending branch (not labeled), is the mirror image of the descending branch.

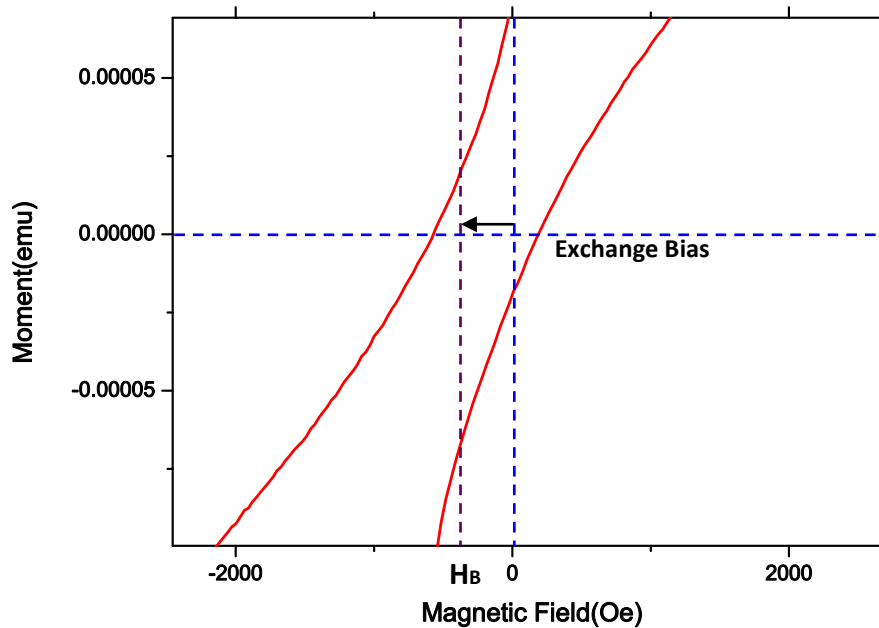


Figure 2.4 Observing bias in the hysteresis loop, here $H_B = -482$ Oe

negative), the sample reaches the coercive point (point C), at which there are equal domains parallel and antiparallel, and thus net magnetization is zero. Finally, the film saturates in the negative direction at point D, so that the entire sample is magnetized parallel to the reversed field. From this point, if we increase the field back to its original value at the positive saturation point, the sample would follow the ascending branch of the hysteresis loop. However, in presence of exchange coupling, a horizontal bias appears in the loop, which is shifting the center to the left as shown in Fig.2.4.

2.3 Field-Cooling Procedure

In a field-cooling (FC) experiment, the sample is cooled down to 20 K in the presence of magnetic field at various field values. As a result, we observe a bias in the magnetization loop. We studied the bias and the width of the loop as a function of the cooling field, and a function of temperature for a given cooling field. All the results are presented in Chapter 3.

2.4 Zero-Field Cooling Procedure

In a zero-field cooling (ZFC) experiment, the sample is cooled down to 20 K with no external magnetic field. As a result, we do not observe any net bias in the magnetization loop, but there is still a change in the magnetization loop, as well as a phase transition. We have studied the effect of temperature on ZFC state, as well as the remanent coercive point, H_{RC} , which is the value of the field that we should apply, so that the system goes back to magnetization zero when the field is zero.

Chapter 3

Results/Discussion

We have measured hysteresis loops on [Co/Pd]/IrMn film using VSM at room temperature in FC and ZFC states.

3.1 Hysteresis Loop at Room Temperature

Fig. 3.1 shows a measurement of magnetic loop at 400 K. As mentioned in Chapter 2, we can observe the main features unique to ferromagnetic materials, namely: saturation, nucleation, remanence, and coercive points. Starting with positive saturation, ($H_S > 3100$ Oe, $M = 0.00016$ emu), the graph goes down to nucleation point, which is labeled as A, ($H_N = 752$ Oe, $M = 0.00015$ emu) and keeps decreasing and hits remanence point, which is labeled as B, where external field is zero ($H_R = 752$ Oe, $M_R = 3.2 \times 10^{-4}$ emu). As the field continues to decrease, the sample reaches the coercive point, which is labeled C, where there is no net magnetization ($-H_C = -740$ Oe). Finally, it arrives negative saturation point, which is labeled as D at ($-H_S = -3600$ Oe). From this point, if we increased the field back to its original value at the positive saturation point about ($H_S = 3100$ Oe), the sample would follow the ascending branch of the hysteresis loop. These two branches are symmetrical at room temperature in the absence of exchange couplings.

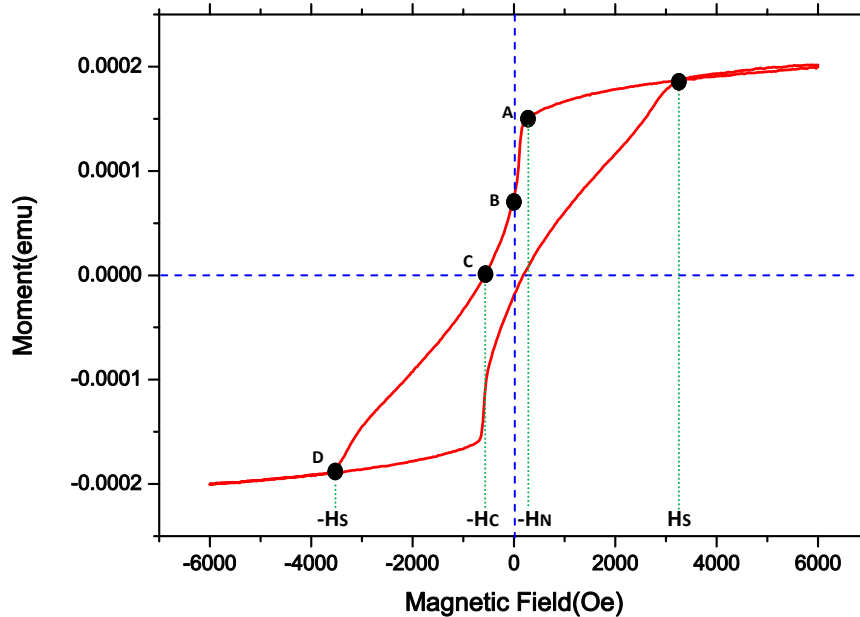


Figure 3.1 Hysteresis loop at room temperature.

3.2 Field Cooled State

In field cooling (FC), we cool down our sample to low temperatures in the presence of a magnetic field. In our measurements, we cooled from 400 K down to 20 K under application of a field with varying magnitudes. As a result, a bias appears in the magnetization loop, which a shift in the magnetization loop. Then, we measure a full loop at each temperature as we heat the sample back up. We have been studying and analyzing the primary factors inducing a bias. Also, we have been studying how they affect the shape of the loop by studying the bias and the width of the loop as a function of the cooling field, and temperatures for a given cooling field. We performed this measurement four times in May, June, November 2010, and March 2011 as shown in Figs. 3.2 to look at reproducibility and possible aging effects.

3.2.1 Study of Bias vs Temperature

Figs. 3.2 shows the results of measurements of magnetization loops recorded at different temperatures after FC. All measurements A, B, C, and D in Fig. 3.2 show the loops measured at increasing temperatures after field cooling from 20 K up to 400 K. We observe some bias which arises from the field cooling. In Fig. 3.3, the amount of bias is plotted as a function of temperature on one graph for all four measurements. The first measurement in May showed expected behavior. Bias is exponentially increasing when temperature decreases. However, after May 2010, the amount of bias was diminished at lower temperature below 150 K. We do not know the reason why bias is diminished, but we suspect that there are some aging effects in the sample. However, we do have consistent results at high temperature between 150 K to 400 K. All four measurements show same the behavior in that region.

Fig. 3.4 shows the amount of coercivity (H_C) as a function of temperature. H_C is where the magnetic moment is zero. Because of the hysteresis, there are two coercive points in a hysteresis loop, one on the ascending H_C and descending branch $-H_C$. To evaluate H_C , I measured the difference between the two points and divided by two. We observe that the behavior of H_C mimics the behavior of H_B (Bias): it increases when temperature decreases. The effect was stronger in the first measurement May 2011, but all measurements exponentially decrease at high temperature.

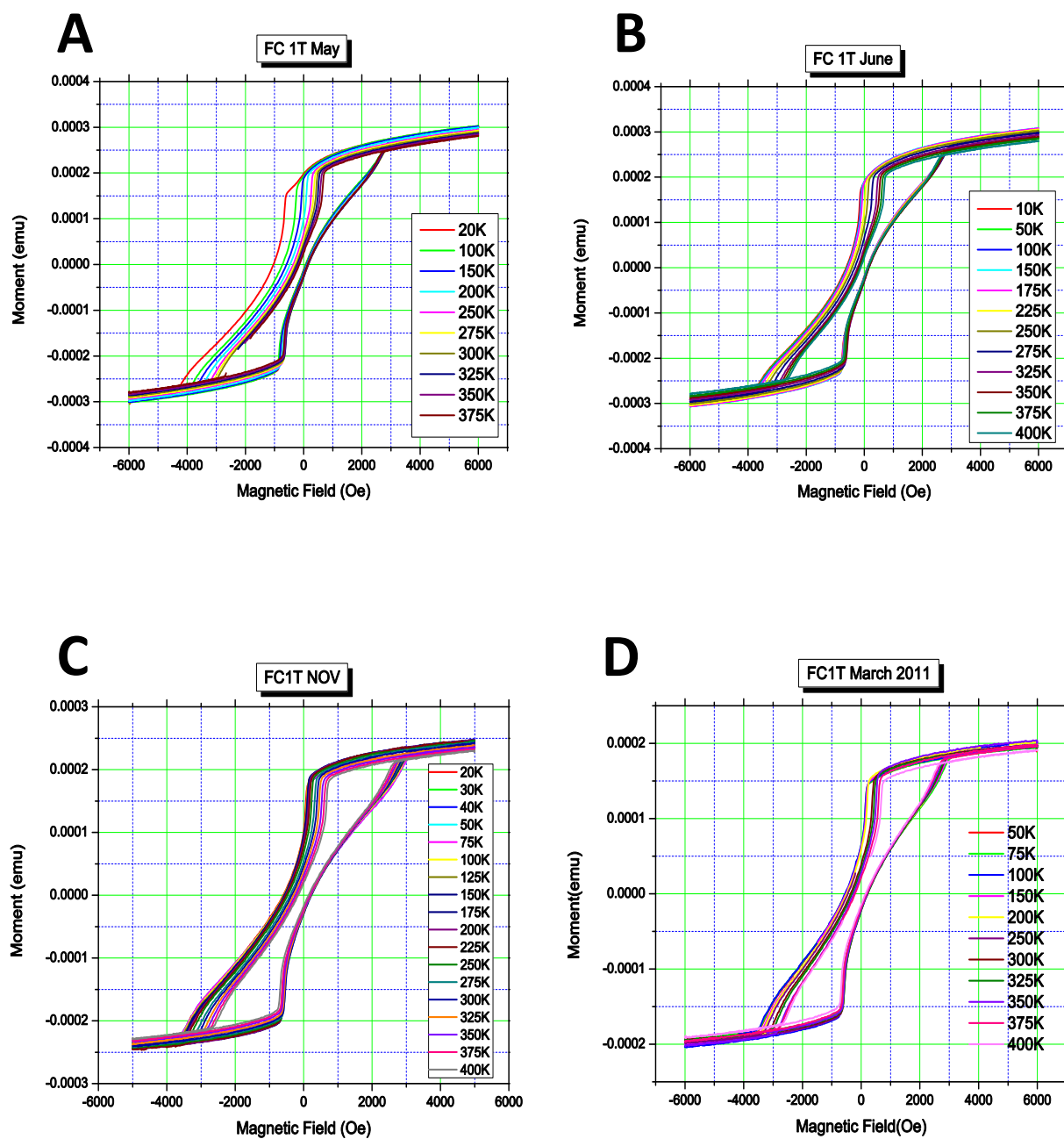


Figure 3.2 Hysteresis loops measured in May, June, November 2010, and March 2011.

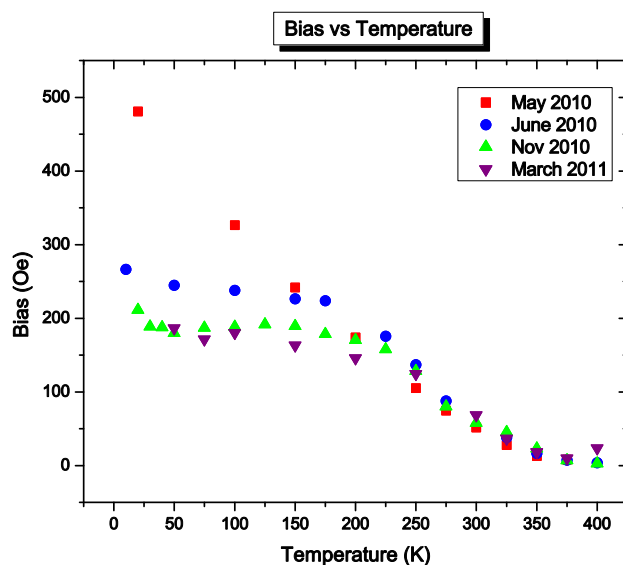


Figure 3.3 Bias versus temperature in FC state under 1T for the four measurements in May, June, November 2010, and March 2011.

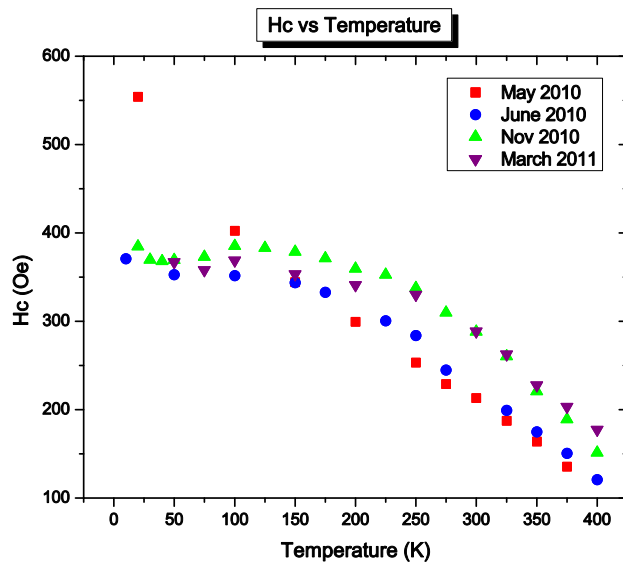


Figure 3.4 Coercivity versus temperatures in FC state for the four measurements in May, June, November 2010, and March 2011.

3.2.2 Study of Bias vs. Cooling Field

We have also measured how the bias and coercivity depend on the magnitude of the cooling field.

Fig. 3.5 shows loop measurement after cooling under different fields. We cooled the sample down to 25 K and measured the hysteresis loop at 25 K. We then heated the sample back up and cooled it again under different applied field. It was a very long experiment to complete, taking about 50 hours. We applied fields from 100 Oe up to 4000 Oe.

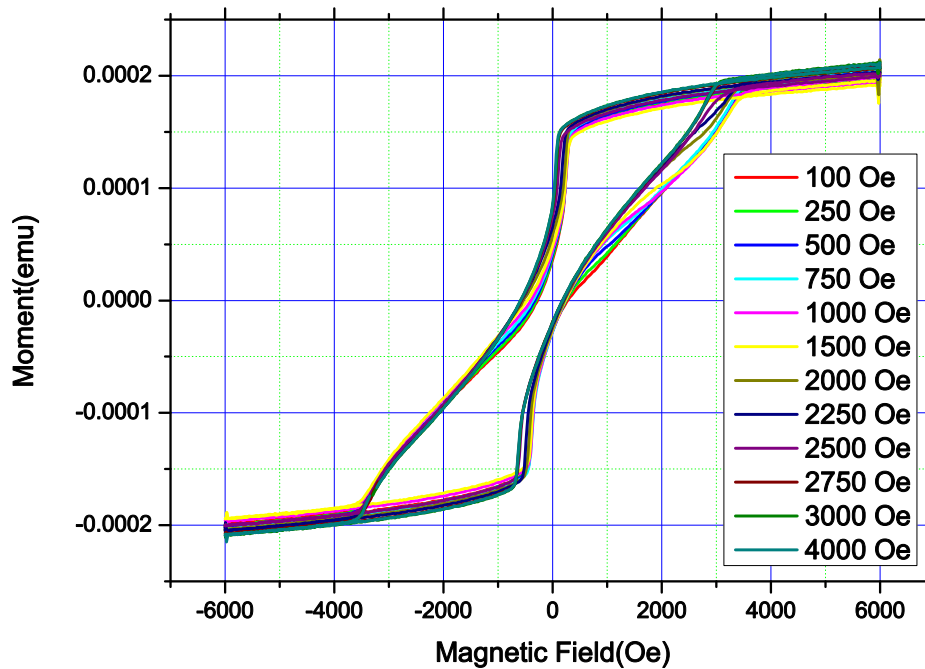


Figure 3.5 Hysteresis loops measured at 25K after field cooling under different fixed magnetic field values.

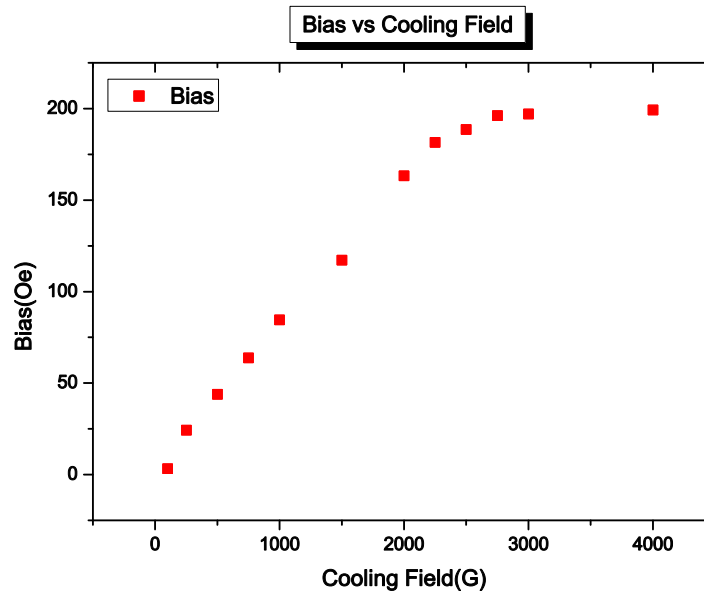


Figure 3.6 Bias versus magnitude of cooling field.

Fig. 3.6 shows the amount of bias at 25 K as function of the magnitude of cooling field. We observe that the bias increases as the cooling field increases and saturates at some point when the cooling field is around $H_S = 2600$ Oe. We observed linear behavior in the bias with a cooling field, until the cooling field is about 2500 Oe. There is obviously no bias for zero cooling field. Linear behavior appears between 0 and 2500 Oe and saturation above 2500 Oe.

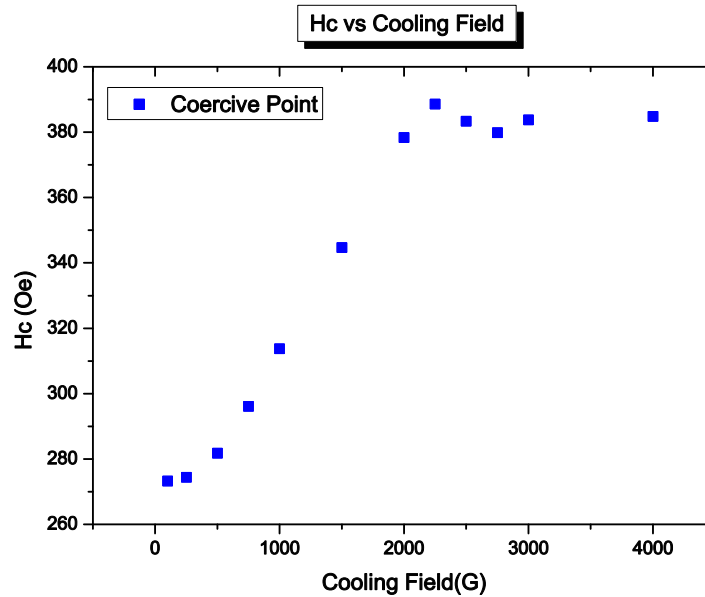


Figure 3.7 Coercivity versus magnitude of the cooling field at 25 K.

In Fig. 3.7, the amount of coercivity is plotted as a function of the cooling fields. There is still some coercivity where the cooling field equals zero, as we do observe coercivity at 400K. However, while the coercivity increases with cooling field values, it also shows a saturating behavior for $H > 3000$ Oe. This point matches the saturation point of the sample at 400K.

3.2.3 Study of Shape of Loop vs. Field cooling

We observed some change in the shape of loop for different cooling fields in Fig. 3.5. The shape at the end of descending branch and the shape at the end of ascending branch are not symmetrical. Also, the width at top and at bottom are not the same. The width at the bottom is thinner than the width at top. This change with the magnitude of cooling field is more drastic than change in temperature seen in Fig. 3.2.

3.3 Zero-Field Cooling

In ZFC, we cool down the sample with no external field. When we cool down with no field, we do not observe any macroscopic bias. However, there are still some changes in the shape of the loop that arise from local exchange coupling.

3.3.1 Shape of Loop vs. Temperature

From the zero-field cooling state, we measure hysteresis loops as function of temperature. Fig. 3.8 shows how the shape of the loop changes with temperature from 20 K to 400 K. The width of loop is getting wider at top and bottom when decreasing temperatures. This effect is most likely due to thermal activation. We can observe the same result on coercivity as well.

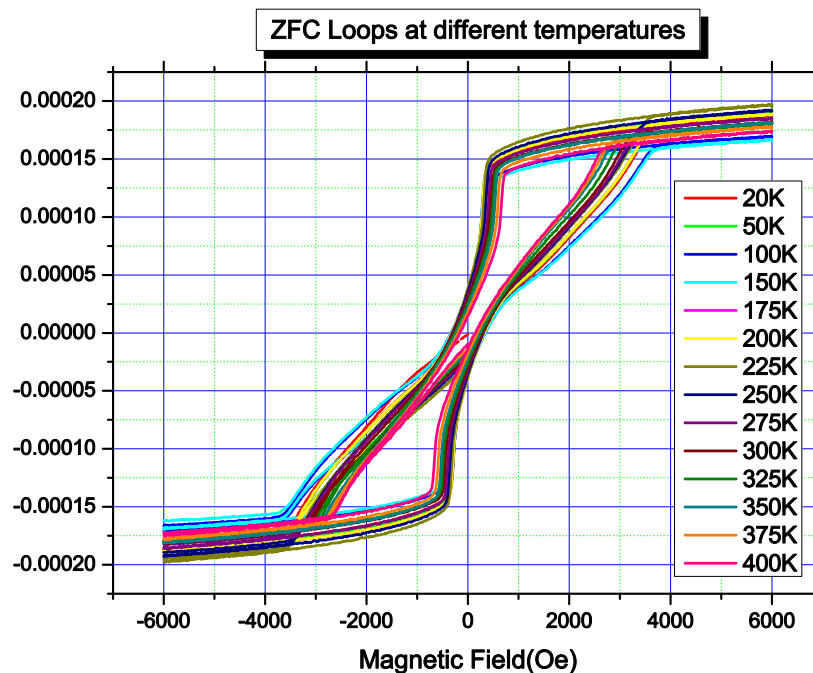


Figure 3.8 Hysteresis loops measured after ZFC at variable temperatures.

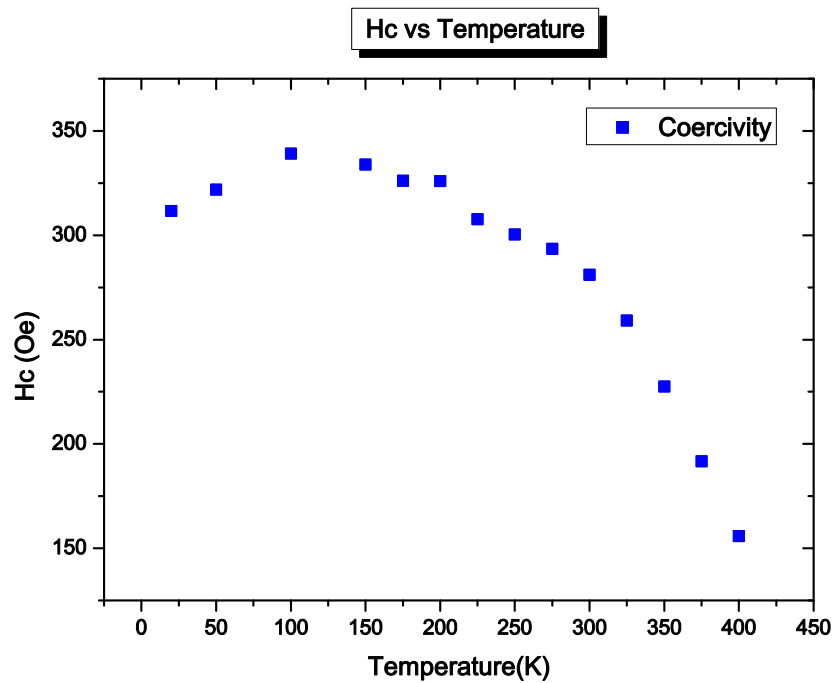


Figure 3.9 Coercivity in zero field cooling state versus temperature.

Fig. 3.9 shows the coercivity in ZFC state as function of temperature. When temperature decreases from 400 K down to 100 K, the coercivity increases. Interestingly, this result is very similar with FC result shown Fig. 3.7. In both FC states and ZFC states, when temperature increases, the coercivity decreases. To measure width of the top of the loop, I measured positive saturation and nucleation points at each temperatures, and then I subtracted H_N from H_S . The result is shown Fig. 3.10.

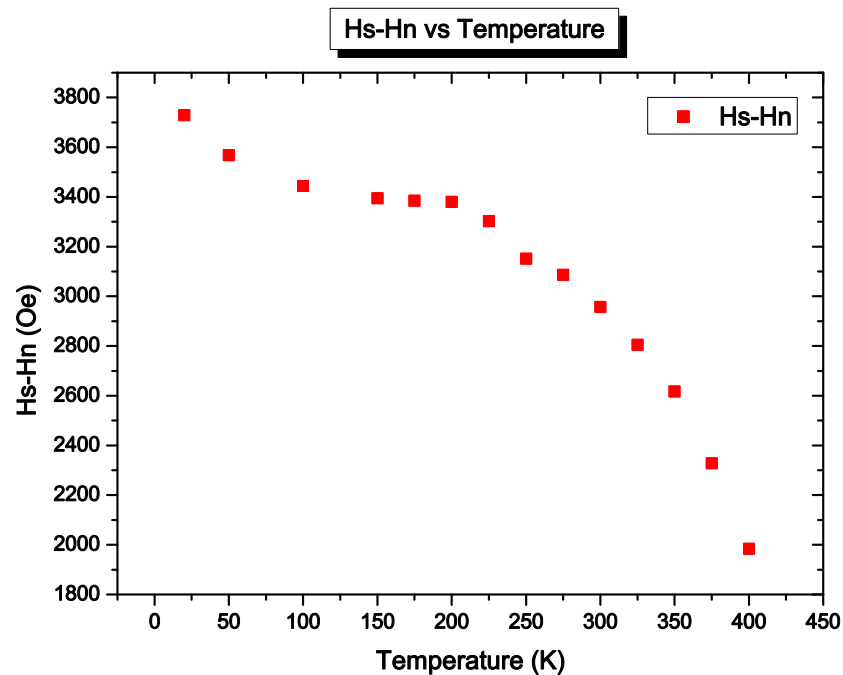


Figure 3.10 Positive saturation point subtract by nucleation point versus temperature.

We see an evolution on the width at the top of loop with temperature. The shape of loop is opening or widening up while temperature drops. There is an interesting result below 100 K that we do not expect to measure. The width increases more at these temperatures, which is unpredictable result to be further understood.

3.3.2 Effect of Demagnetization

We have seen that no bias is observed in ZFC, and the loop is symmetrical. However, the shape of the loop is strongly modified depending on the magnetic history of the sample. We see how magnetic history can influence the shape of the loop. Fig. 3.11 shows the hysteresis loop zero field cooling state from 400 K without demagnetization. In particular, demagnetizing the sample before ZFC can have a great effect. Demagnetization corresponds to applying successive magneti-

zation loops with decreasing amplitudes down to zero. In Fig. 3.12, when we heat up to 400 K and apply a demagnetization prior to ZFC, we see an change in the shape of loop in zero field cooling state as illustrated in Fig. 3.11 and Fig. 3.12. We call plateau effect. We can obviously see the difference between Fig. 3.11 and 3.12, which is that the enlargement of the loop in Fig. 3.12 is observed compared with Fig. 3.11.

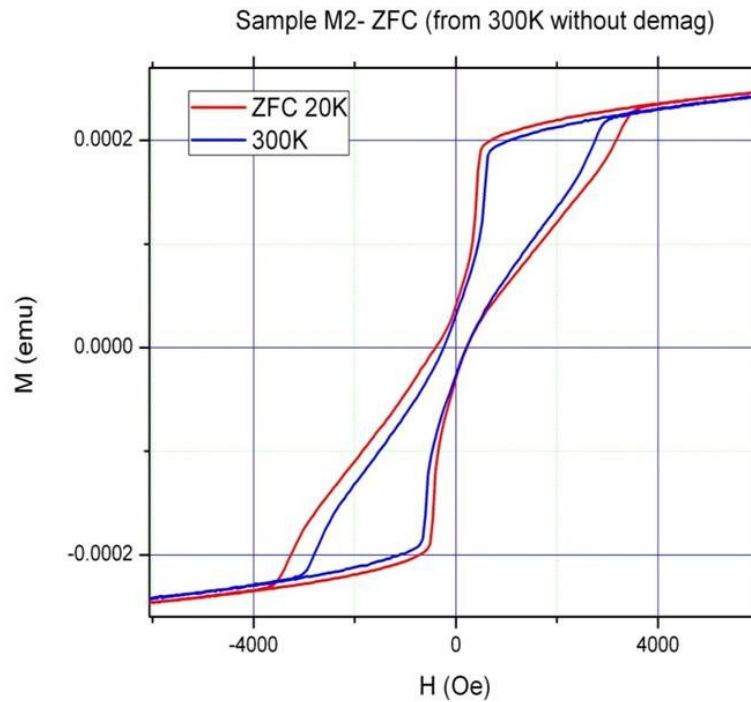


Figure 3.11 Hysteresis loops at 300 K and in zero field cooling state at 20 K after cooling down from 400 K without demagnetization

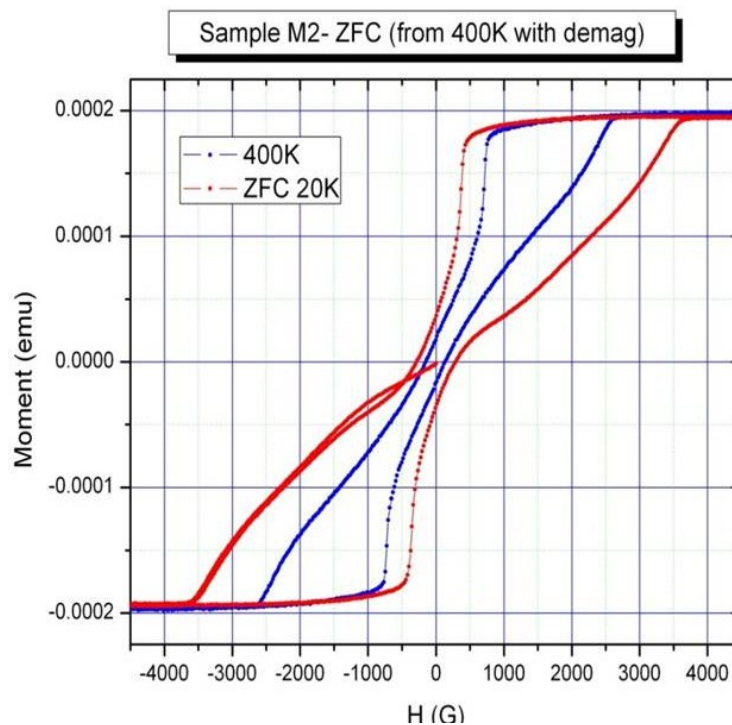


Figure 3.12 Hysteresis loops at 400 K and in zero field cooling state after demagnetization at 400 K

3.3.3 Study of the Remanent Coercive Point

Because of coercivity, net magnetization at $H = 0$ is not zero on the major loop. The coercive point, H_C , is the field we need to apply to get $M = 0$, but when we release the field back to $H = 0$ from H_C , then magnetic moment M is not zero. Remnant coercivity is different from the coercivity mentioned earlier. Remnant coercivity, designated by H_{RC} , is the value of the field that we should apply to the sample, so that the magnetization M goes back to zero when the field is released to zero. In other words, we are looking for the value of field for which the sample has reached a zero net magnetization, in a irreversible way. We have been searching for this point in ZFC state at low temperature, by applying various fields, coming from negative saturation as shown in Fig. 3.13. In Fig. 3.14, we can observe that the value of magnetic field for which the sample reaches a zero net

magnetization is a bit above $H_{RC} = 500$ Oe.

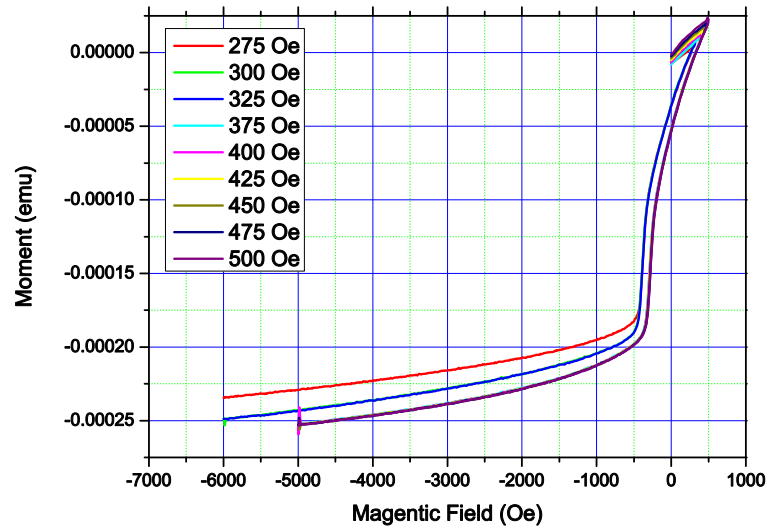


Figure 3.13 Successive partial minor loops coming from negative saturation, in search for the remanent coercive field H_{RC} . Measurement done at 20K in ZFC state after demagnetization at 300 K.

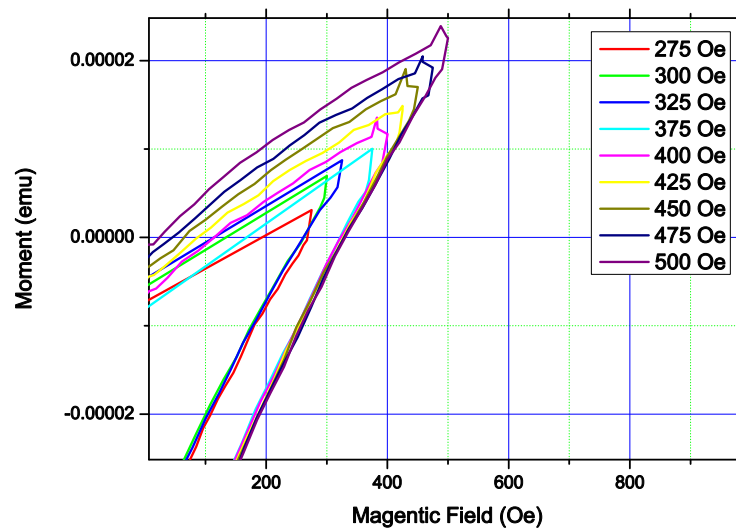


Figure 3.14 Enlargement of Fig. 3.13. It appears that H_{RC} is a bit above 500 Oe.

Other measurements of remanent coercive point are shown in Fig. 3.15 and 3.16. Fig. 3.15 shows remanent coercive point at 400 K with no demagnetization. It appears that $H_{RC} = 475$ Oe. Fig. 3.16 shows remanent coercive point at 400 K with demagnetization. In this plot, it appears that $H_{RC} = 450$ Oe. This first study of remnant coercive point shows that the H_{RC} value varies slightly between 450 Oe and 500 Oe depending on the measurement. It appears that this value does not depend too much on the application of demagnetization prior to zero field cooling. It probably depends mostly on temperature.

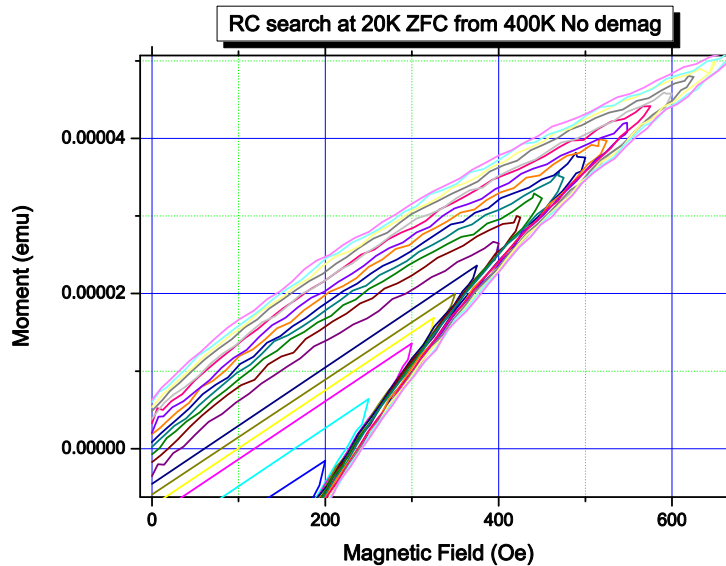


Figure 3.15 Remanent Coercive Point at 20 K after cooling down from 400 K without demagnetization.

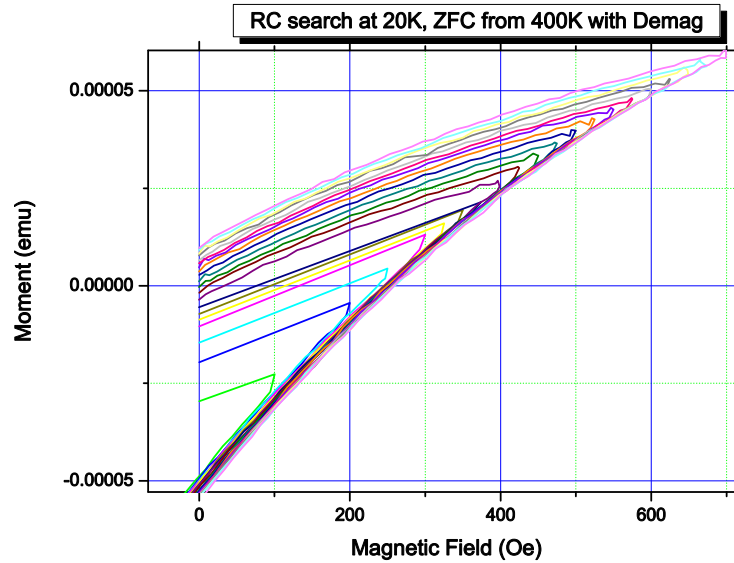


Figure 3.16 Remanent Coercive Point at 20 K after cooling down from 400 K with demagnetization.

3.4 Conclusion

We have studied exchange bias using Vibrating Sample Magnetometry (VSM). When the sample is cooled down in the presence of a magnetic field, through various experiments, in May, June, November 2010, and March 2011, we observed the amount of bias decreases as temperature increases in Field Cooling (FC) states. We also showed a similar phenomenon for coercivity. We studied the saturation, coercive and nucleation points at different temperatures and saw an evolution in the shape. We also measured zero-field-cooling (ZFC) states and effect of demagnetization, and we characterized remnant coercive points. In the future, we plan to study further the occurrence of exchange bias in [Co/Pd]/ IrMn thin films, the variation of the bias with different thicknesses for the Co, Pd, and IrMn layers. We want to further understand the impact of demagnetization on the behavior of the magnetic film in zero-field cooled and the occurrence of memory effects [10].

In particular, we want to understand the correlation between biasing effects in the magnetization and the occurrence of magnetic memory at different fields values and different spatial scales [11].

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