Thin Film Deposition and X-Ray Spectroscopy

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Magnetron sputtering was employed in order to deposit a couple thin films on to a glass plate. In order to test the structure, X-Ray spectroscopy was employed. Though the results of the spectroscopy did not immediately match any of the values found in databases, there was significant evidence of the correct compounds. Using the aforementioned techniques, it was found that magnetron sputtering can deposit thin films with relatively few imperfections in their structures. These imperfections could likely be further reduced by annealing the sample.

I. INTRODUCTION

In this work, magnetron sputtering deposition was used to deposit a thin film of $Nd_2Fe_{14}B$. Thin films are small layers of a target material that has been depositied onto a substrate using one of a variety of deposition methods. Thin films have seen applications in Optical Coatings,dielectric materials[3], thin-film solar cells [1],Lithium Ion batteries[2].

Magnetron sputtering is a physical vapor deposition method used to deposit a thin layer of a material onto a substrate. A target material and a substrate are placed in a vacuum and the target material is then bombarded with charged molecules of an inert gas. The efficiency of this process is then increased by using a magnetic field to increase the bombardment rate with the target materials.

The crystalline structure of the deposited $Nd_2Fe_{14}B$ can be analyzed using X-Ray Diffraction (XRD).In this case, the material will have relatively low levels of $Nd_2Fe_{14}B$ but will have an abundance of the constituent elements, until the thin film is subjected to annealing.

II. EXPERIMENTS

A. Thin Film Deposition

The magnetron sputtering machine, displayed in figure 1, was generously provided for use to us by Dr. Cheng's lab. After receiving instruction on how to conduct the experiment, we were enabled to conduct the full process of thin film deposition using magnetron sputtering.

Several steps must be conducted in order to achieve the proper conditions for magnetron sputtering. After ensuring that the machine is maintaining a proper vacuum, we then introduce Argon into the chamber at a rate of $10cm^3/s$. Argon is utilized specifically so that no reaction between the sputtering target and the gas occurs on impact of the Argon ion. After verifying that Argon is being introduced to the chamber, we set the chamber pressure to $16millitorr$ to ensure that enough Argon is present in the chamber to generate the plasma field required for sputtering while still minimizing the Argon present in the rest of the chamber, thereby increasing the mean free path.

After we ensure that the pressure in the chamber is at $16millitorr$, we start planning to turn on the sputtering gun. First and foremost is to turn on the water cooling system. Without water cooling, the sputtering gun is likely to be dammaged. After we ensure the water cooling is functioning properly we provide power to the sputtering gun and ensure that we are providing $30W$ of power. At this point, the sputtering gun has begun creating the plasma field and we give the system time to stabilize. It is common that if the equipment is not used for a while, uneven rates of deposition/sputtering will occur initially. After this is done, we open the shutter and wait for 30 minutes - at which time we shall have enough material sputtered onto the target to complete the experiment. The process of sputtering, as well as the Argon plasma, can be seen in figure 2.

After 30 minutes, we first close the shutter to stop all deposition instantly. Only after this do we begin the process of shutting down the system. First we power off the sputtering gun. Next, we stop the flow of Argon into the chamber. After this, we reset the vacuum state of the chamber to previous levels. Finally, we turn the water cooling off.

When all deposition is completed, we are able to remove the thin film samples we generated. First we must idle the vacuum pumps so that the chamber can be brought back to atmospheric pressure. Next, we carefully introduce gas into the chamber in a way that brings the pressure of the chamber back to atmospheric while not damaging the expensive vacuum pumps. Once pressure inside of the chamber matches atmosphere, the system can be opened and the samples can be removed.

B. X-Ray Spectroscopy

Fortunately for the students in this lab, we were able to gain access to a bran new, state of the art, X-Ray spectroscopy machine. The machine is presented in figure 3. We were able to use this machine to study the solid state composition of thin film we had produced previously by using the machine.

The most important part of conducting the X-Ray diffraction experiment is ensuring that the sample is lined up. This ensures that the X-Rays are incident upon the

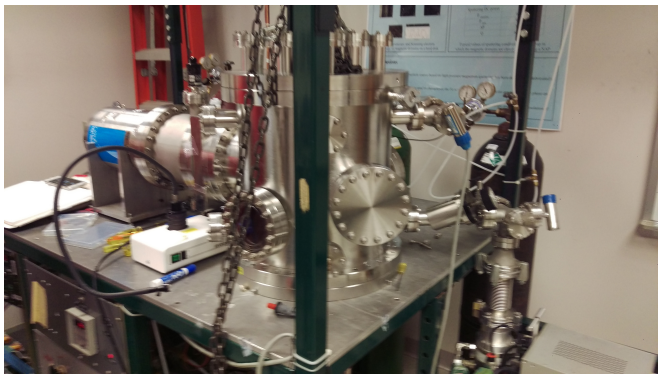


FIG. 1. Magnetron Sputtering Machine

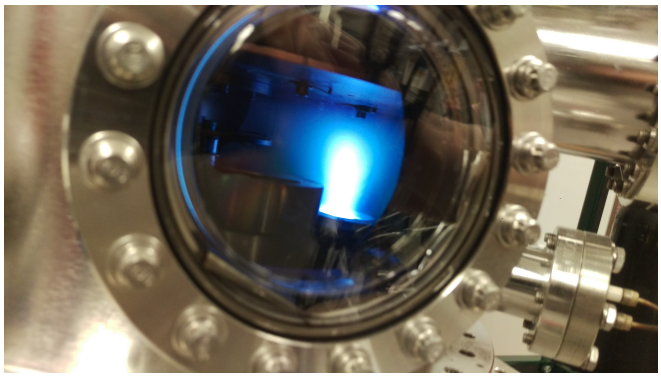


FIG. 2. Sputtering Process and Plasma

sample in such a way that the reflected rays are captured by the machine for analysis.

We set up the machine to sweep through a 2θ range of $30 - 50$ based on domain knowledge of the materials we expected to find before hand. The time per step, $\Delta t/s$ was set to be $0.4s$. A total of 1782 steps were required for the completion of the analysis.

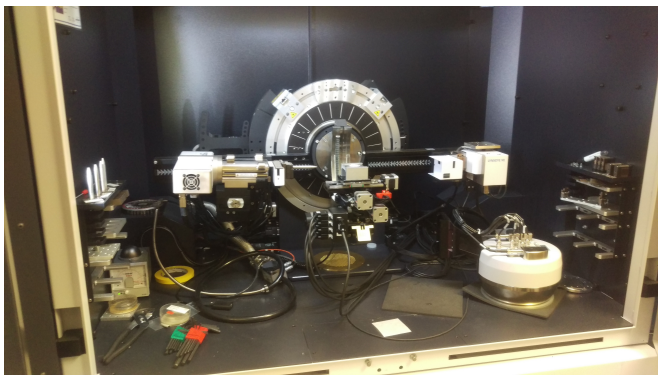


FIG. 3. X-Ray Spectroscopy Machine

III. DISCUSSION

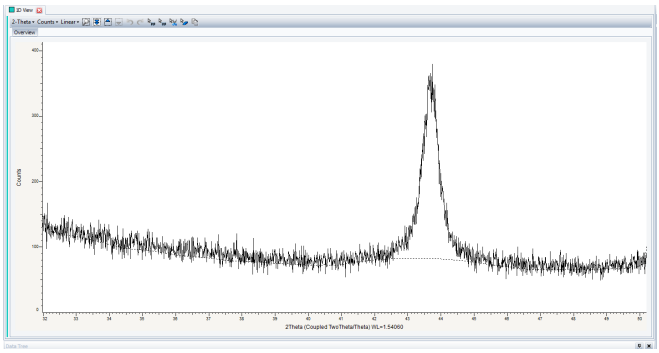
A. Thin Film Deposition

Though the content of the thin films that was of interest was the layer with neodymium, this layer was covered with a thin layer of copper. This was in order to reduce the ability of the layer of interest to oxidize and lose important properties. Though copper would react with the air as well, it would do so much slower and protect the layer underneath.

B. X-Ray Spectroscopy

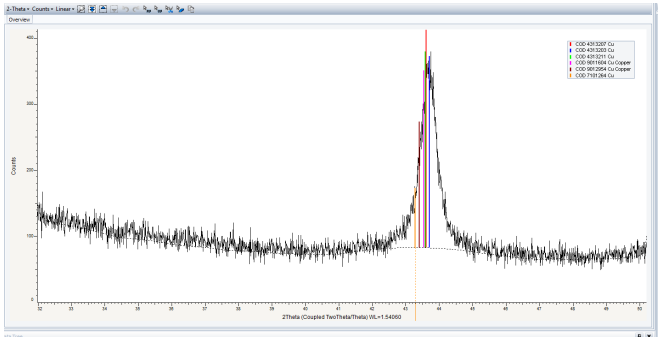
Using the X-Rays, it was possible to determine the structure of the thin film which was deposited with far greater accuracy than what would be capable with almost any other form of technology. The software used compared the structure found to those which have been recorded in established databases. By inspection of the graphs in figures X and Y (I think we had 2 graphs), it is clear that the deposited material does indeed have the properties of copper as expected. There are also traces of many different Boron, Iron, and Neodymium structures, but it is hard to determine accurately which one.

It can be seen that the samples aren't exact matches to the logs from the databases, but that is in part due to the fact that the samples were not yet annealed. Had they been annealed, then the structure would likely have been more organized with fewer impurities. Alas, due to the glass plate on which the films were deposited, it was impossible to anneal them as the glass would melt in due to the high temperatures necessary for annealing.



IV. CONCLUSION

The goal of this research was to create a thin film of $Nd_2Fe_{14}B$ and then analyze its structure via X-Ray spectroscopy. The methods implemented to examine structure provided data which indicated general success of the depositions. Due to the substances not being annealed, the values are not quite as accurate as one could



hope. Thus for future work, it would be greatly beneficial to deposit the thin films on a substance other than glass which can better withstand heat. This way the substances could be annealed and X-Ray spectroscopy could better verify which compounds are present in the thin films.

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[1] S. Grama. A survey of thin-film solar photovoltaic industry technologies. *Massachusetts Institute of Technology*, 2008.

[2] et al Hu, Liangbing. Thin, flexible secondary li-ion paper batteries. *ACS Nano*, 2010.

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