# Interplay Between Magnetic and Superconducting Layers in Thin Film Heterostructures

By

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Master Thesis in Solid-state Physics

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#### **Dedication**

I would like to dedicate this thesis to my lovely family for their boundless love and my beautiful native land, Georgia. Furthermore, I lovingly dedicate this thesis to the most people with valuable professions, my teachers - Teachers sometimes childless, but thousands of the child's parent.

**Declaration** 

I, Grigol Abuladze, declare that this thesis "Interplay between magnetic and

superconducting layers in thin film heterostructures" and present work - with the

exception of guidance from the supervisors, have prepared independently without

assistance and have been generated by me as the result of my own original research

and that it has not been submitted earlier elsewhere and that I have all relevant

information to the knowledge and belief have made that they are true and I have not

nothing concealed.

I confirm that this work was done under the supervision of Professor Tamaz

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#### 1. Motivation

**Ferromagnetism** and conventional **superconductivity** have long been considered as antagonist phenomena. As a matter of fact, the quantum mechanical exchange interaction leads to the tendency to parallel alignment of the electron spins in neighbouring atoms, whereas electron-phonon interaction in conventional superconductors leads to the tendency of antiparallel alignment of electron spins in Cooper pairs.

Master Thesis and the experiments have been carried out in Germany, Jülich Center for Neutron Science with a joint project of the Georgian-German scientific bridge. The research has been conducted under the solid state physics such important issues, such as Magnetism and Superconductivity. For the implementation of the project, we produced and developed ferromagnetic and superconducting thin films by using of molecular beam epitaxial machine. The films are periodically measured by X-ray refflectivity and diffraction machines, magnetic property measurement system and physical property measurement system in order to reach the ultimate goal of sample preparation. It should be noted that the sample was made in good order and successfully implemented all the planned experiments. The analysis of the data obtained and the results of the project to the beginning of the questions. This study is an important role in the development of such a big event like Superconductivity.

# 2. Theory

#### 2.1 Magnetism

# 2.1.1 Basics to Magnetism

Magnetic fields are either caused by currents, or by the intrinsic magnetic moment of elementary particles. The simplest case of a charge distribution is a circular closed current loop. If there is a current I around an elementary oriented loop of area |dS| then magnetic moment  $d\mu$  is given by:

$$d\mu = I \cdot dS \tag{1}$$

Where I is the electrical current and dS is equal to the area of the loop and it is oriented area enclosed by the current. The unit of magnetic moment is  $Am^2$ . The direction of the vector is normal to the loop and in a sense determined by the direction of the current around the elementary loop. The dipolar field it causes is described by the following equation:

$$\boldsymbol{B}(r) = \frac{\mu_0}{4\pi} \left( \frac{3(r \cdot m)r}{r^5} - \frac{m}{r^3} \right) \tag{2}$$

Where, r is the position vector in relation to the dipole. The energy of a magnetic dipole in a magnetic field  $\mathbf{B}$  is:

$$E = -m \cdot \mathbf{B}$$

This shows, that a dipole will always align parallel to an external field **B**.

Today, it is known that every fundamental particle has an intrinsic magnetic moment, which is tightly connected to its intrinsic angular momentum called spin. Therefore, all matter is interacting with magnetic fields in some way.

In atoms the magnetic moment associated with an orbiting electron lies along the same direction as the angular momentum of that electron and is proportional to it. Respectively, if there is a case then a single point charge is moving on a circular orbit, equation 1 can be reformulated by:

$$m = \frac{q}{2m} \cdot L \tag{3}$$

Where q is the charge and m is the mass of the particle and L is the angular momentum of the particle. All the charges which we will considering are associated with particles

that have mass. As it turns out, equation 3 does not hold in quantum mechanics and has to be generalized. Since both the electrical charge and the angular momentum are quantized, for an electron the formula becomes:

$$m = -g \cdot \mu_{\rm B} \frac{L}{\hbar} \tag{4}$$

Where  $\mu_{\rm B}$  is Bohr magneton, defined by  $\mu_{\rm B}=\frac{e\hbar}{2m_e}$ 

Which combines the particle properties from the equation 3, as they are fixed for an electron. This is a convenient unit for describing the size of atomic magnetic moments and takes the value  $9.274 \times 10^{-24}$  Am². Note that sign of the magnetic moment in equation 4 is negative. Because of the negative charge of the electron, its magnetic moment is antiparallel to its angular momentum. The new parameter g is dimensionless and simply called g-factor. For the orbital movement it is equal to 1, but for the spin it roughly equal 2. For other kinds of particles, e.g. nucleons,  $\mu_{\rm B}$  has to be replaced with their respective magnetons. Consequently it only plays a small role in the magnetism of solid objects and the magnetic moment of electron is mostly responsible for the magnetic moment of an atom.

#### 2.1.2 Magnetization and Field

A magnetic solid consists of a large number of atoms with magnetic moments. The magnetization M is defined as the magnetic moment per unit volume. Usually this vector quantity is considered in the "continuum approximation", i.e. on a length-scale large enough so that one does not see the graininess due to the individual atomic magnetic moments. Hence M can be considered to be a smooth vector field, continuous everywhere except at the edges of the magnetic solid.

In vacuum there is now magnetization. The magnetic field can be described by the vector fields **B** and **H** which are linearly related by

$$\mathbf{B} = \mu_0 \mathbf{H} \tag{5}$$

Where  $\mu_0 = 4\pi \times 10^{-7}~{\rm Hm^{-1}}$  is permeability of free space. The two magnetic fields  $\textbf{\textit{B}}$  and  $\textbf{\textit{M}}$  are just scaled versions of each other, the former measured in Tesla and the latter measured in A m<sup>-1</sup>.

In a magnetic solid a relation between B and H is more complicated and the two vector fields may be very different in magnitude and direction. The general vector relationship is:

$$\mathbf{B} = \mu_0(\mathbf{H} + \mathbf{M}) \tag{6}$$

In the special case that the magnetization M is linearly related to the magnetic field H, the solid is called a linear material, it is in case isotropic and we write

$$\mathbf{M} = \mathbf{\chi} \, \mathbf{H} \tag{7}$$

Where  $\chi$  is a dimensionless quantity and called the magnetic susceptibility. In this special case there is still a linear relationship between B and H, this simplifies the above equation to:

$$\mathbf{B} = \mu_0 (1 + \chi) \mathbf{H} = \mu_0 \mu_r \mathbf{H} \tag{8}$$

Where  $\mu_r = 1 + \chi$  is relative permeability of the material.

The magnetic susceptibility is a material property and characterizes its magnetic behavior.

#### 2.1.3 Diamagnetism

Diamagnetism is a fundamental property of all matter, although it is usually very weak. It is due to non-cooperative behavior of orbiting electrons when exposed to an applied magnetic field. Diamagnetic substances are composed of atoms which have no net magnetic moments. However, when exposed to a field, a negative magnetization is produced and thus the susceptibility is smaller than zero. That means, an external

magnetic field induces a negative magnetic moment. It is a purely quantum mechanical phenomenon.

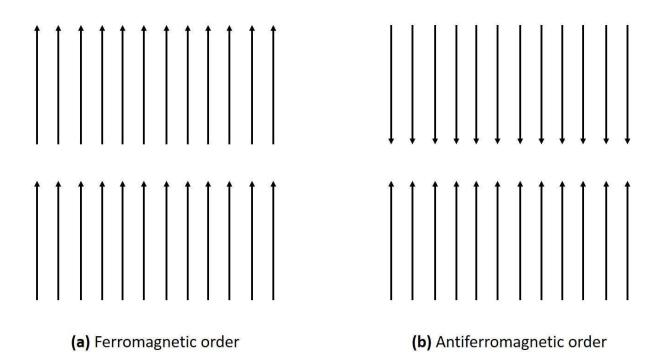
#### 2.1.4 Paramagnetism

This class of materials, some of the atoms or irons in the material have a net magnetic moment due to unpaired electrons in partially filled orbitals. One of the most important atoms with unpaired electrons is irons. However, the individual magnetic moments do not interact magnetically, and like diamagnetism, the magnetization is zero when the field is removed. In the presence of a field, there is now a partial alignment of the atomic magnetic moments in the direction of the field, resulting in a net positive magnetization and positive susceptibility. For a paramagnet  $0 < \chi < 1$ , meaning that an external field gets slightly amplified. Paramagnet materials contain magnetic moments that can be caused by the spin of the electrons or their orbital moment. They can be modeled as a thermodynamic ensemble of non-interacting magnetic moments of equal strength. Their direction is determined by the Zeeman energy, the thermal energy and magnetocrystalline anisotropy. Without an external field the moments are randomly aligned and the net magnetization is zero. If an external field is applied, the moments gradually align more and more as the field strength is increased. As the system is in thermal equilibrium, removing the external field will cause the moments to disorganize again.

# 2.1.5 Ferromagnetism

In a Ferromagnet, the exchange interaction causes the magnetic moments to be aligned parallel to each other, which leads to a spontaneous local magnetization even without an external field. Ferromagnetism is a kind of magnetism that is associated with iron, cobalt, nickel, and some alloys or compounds containing one or more of these elements. Unlike paramagnetic materials, the atomic moments in these materials exhibit very strong interactions. The thermal excitations are working against the ordering of the moments and for high temperatures they overcome the magnetic order. Therefore, the average magnetization becomes zero and the material becomes paramagnetism. These interactions are produced by electronic exchange forces and

result in a parallel or antiparallel alignment of atomic moments. Whereas paramagnetism and diamagnetism are properties of individual atoms or molecules, ferromagnetism is a property of a group of atoms or molecules in a solid crystal or lattice.



All ferromagnetic substances have unpaired electron spins that are strongly entwined by a quantum mechanical force, exchange interaction. Large groups of atoms in a ferromagnetic substance form magnetic domains in which arrays of electron spins become locked together in alignment. When heated to a certain temperature called the Curie point, which is different for each substances, ferromagnetic materials lose their characteristic properties and cease to be magnetic. However, they become ferromagnetic again on cooling.

# 2.1.6 Antiferromagnetism

If a material has two antiparallel magnetic sub-lattices with magnetic moments of the same magnitude, Therefore the material exhibits non spontaneous magnetization. This materials are called antiferromagnees. In this type of materials the magnetic

moments of atoms, usually related to the spins of electrons, align in a regular pattern with neighboring spins pointing in opposite direction.

#### 2.1.7 Ferrimagnetism

Ferrimagnetism is only observed in compounds, which have more complex crystal structures than pure elements. Ferrimagnetic materials have two sub-lattices of antiparallel magnetic moments that do not cancel each other out and a net magnetization can be expected in this case. Unlike ferromagnetic materials, which are typically metals, ferrimagnetic materials are ceramics, in particular, ceramic oxides. The most widely used ferrimagnets in technological devices are materials known as ferrites.

Ferrimagnetic materials contains magnetic moments aligned antiparallel to one other, similar to the antiferromagnetic materials. However, instead of having a zero net magnetic moment, different numbers of unpaired electrons in the component transition metals result do not cancel one other out, resulting in a spontaneous magnetization.

#### 2.1.8 Magnetic Domains

Magnetic domains are regions with the same magnetic ordering in a magnetic material. They are separated by domain walls. Magnetic moments within one domain align themselves along the same direction, and produce a net magnetization. The main implication of the domains is that there is already a high degree of magnetization in ferromagnetic materials within individual domains, but that in the absence of external magnetic fields those domains are randomly oriented. In different magnetic domains, net magnetizations can point into different directions. If an external field is applied to a ferromagnetic material, different processes can occur. This depending on the field strength. All these effects together produce a dependence of the imminent behavior on the history of the material. This is called hysteresis and plot of the variation of magnetization with magnetic field is called a hysteresis loop. Figures below shows different ferromagnetic materials and hysteresis loops, respectively on the figure 1 and figure 2.

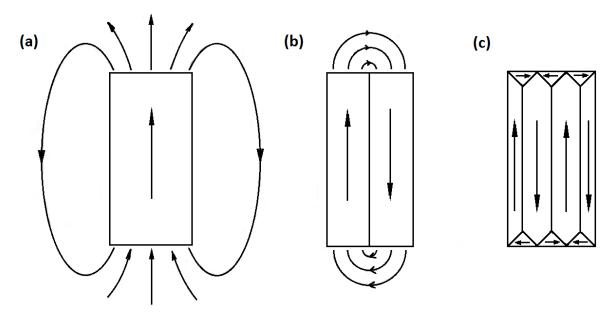


Figure 1: Different domain structures for ferromagnetic material: (a) single domain state, (b) two domains, (c) closure domains state.

On the Figure 2, there is shown hysteresis loop for ferromagnetic material.

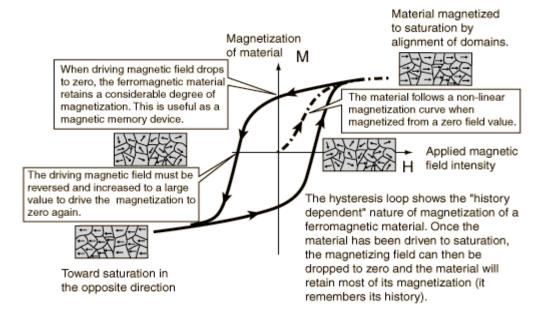


Figure 2: Hysteresis loop. Ref: [http://hyperphysics.phy-astr.gsu.edu/hbase/solids/imgsol/hyloop.gif]

#### 2.1.9 Magnetic Anisotropy

Ferromagnetic materials exhibit intrinsic easy and hard directions of the magnetization and it shows anisotropic behavior which is expressed in terms of a magnetocrystalline anisotropy energy. It means that the properties of a material are

direction dependent. These directions are called easy axis. The spontaneous magnetizations are not equivalent in all directions and this effect are called magnetocrystalline anisotropy. An example of magnetocrystalline anisotropy is the uniaxial anisotropy, which has one single easy axis. The alignment of the magnetization along the two opposite directions of the easy axis cost the same amount of energy, which is the minimum of anisotropy energy. As the magnetic moment turns perpendicular to the easy axis, it costs most energy.

Here, the energy density can be calculated by following equation as:

$$\frac{E}{V} = K_1 sin^2(\theta) + K_2 sin^4(\theta)$$

Where  $\theta$  is the angle, between magnetization and easy axis.  $K_1$ ,  $K_2$  are called anisotropy constants.

## 2.2 Superconductivity

#### 2.2.1 Occurrence of Superconductivity

When a sample is cooled to a sufficiently low temperature, the electrical resistivity of many metals drops suddenly to zero. It means that resistance of certain material completely vanishes at low temperatures, which is called as critical temperature. It is also occurred when we have alloys of metals, ferromagnetic-superconductor materials and so on. This phenomenon is called superconductivity.

In the early 1900s, Kamerlingh Onnes had begun an investigation concerning the electrical resistance of very pure metals at low temperatures. At that time, the purest metal mercury was available. In 1911, he was measuring electrical resistance of pure mercury as a function of temperature when he discovered that the mercury's resistance suddenly dropped to zero below critical temperature 4 K. It is very important to know that the critical temperature for superconductors is the temperature at which the electrical resistivity of a metal drops to zero. Some of superconductors are: Aluminum, niobium, mercury, gallium and alloys of some materials. Figure 3 shows a measurement results of Onnes experiment.

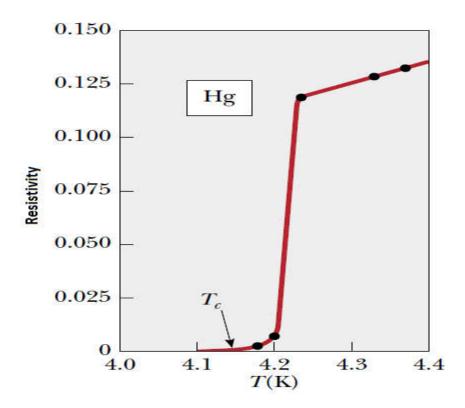


Figure 3: Plot of resistance versus temperature for a mercury. Note that the resistance of mercury follows the path of a normal metal above the critical temperature, *Tc*, and then suddenly drops to zero at the critical temperature, which is 4.15 K for mercury. Ref: Raymond Serway modern physics chapter 12\_page486

That means, the sample undergoes a phase transition from a state normal electrical resistivity to a superconducting state.

#### 2.2.2 Meissner Effect

In 1933, Walter Meissner and Robert Ochsenfeld discovered a magnetic phenomenon that showed that the superconductors are not just a perfect conductors. When a sample is cooled below critical temperatures in the presence of a magnetic field, these materials lost their superconducting behavior above a certain critical temperature-dependent critical magnetic field. This magnetic phenomenon means that, the magnetic flux is expelled from the interior of the superconductor. Figure 4 illustrates the difference between ideal conductor and superconductor material in the case than B field is applied. Idea is that, both are above their critical temperature  $T_c$ . This means that, both are in normal conducting state and have electrical resistance. After, both materials are cooled so that the ideal conductor now has zero electrical

resistance. It is found that the superconductor expels the magnetic field from inside it, while the ideal conductor maintains its interior field.

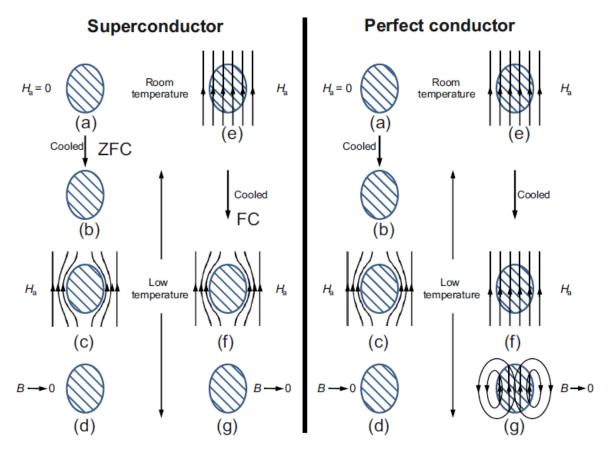


Figure 4: Schematic behavior of applied field Ha and magnetic flux density B in field-cooled and zero-field-cooled experiments on a type I superconductor and non-superconducting material with perfect conductivity.

Adapted from Rose-Innes and Rhoderick (1978, pp. 18 and 20).

# 2.2.3 Two Types of Superconductors

There are two types of superconductors, as shown Figure 5. Type I superconductors display the normal phase and the Meissner phase. Type II superconductors also display the normal and Meissner phase. However, at intermediate temperatures and magnetic fields, they are in a mixed state. In this case, the magnetic field penetrates in a material in the form of vortices. This is called Abrikosov vortex. The graph shows internal magnetic field strength behavior in type I and type II superconductors.

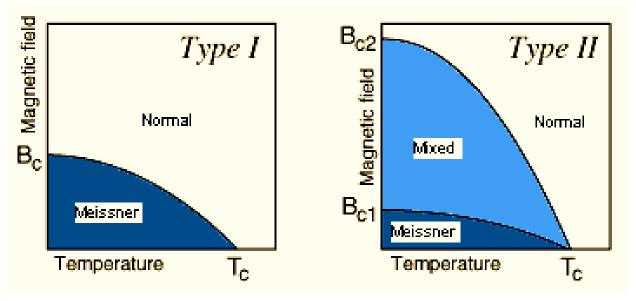


Figure 5: Phase diagram of type I and type II superconductors. Ref: Hays, Aryn M.; Bechtel, Lindsay; Turbeville, Colton; Johnsen, Anthony; and Tanner, Chris A. (2013) "Electrical and Magnetic Properties of High Temperature Superconductors Using Varying forms of Data Acquisition," *Journal of the Advanced Undergraduate Physics Laboratory Investigation*: Vol. 1: Iss. 1, Article 3.

For Type I superconductor, at temperatures above  $T_c$  and magnetic fields above  $B_c$  the material is normal the normal state, while at temperatures below  $T_c$  and magnetic fields below  $B_c$  the material is normal the Meissner phase. For Type II, at temperatures above  $T_c$  and magnetic fields above  $B_{c2}$  the material is in the normal state. At temperatures below the  $T_c$  and magnetic fields between  $B_{c1}$  and  $B_{c2}$  the material is in the Mixed state, while at temperature below  $T_c$  and magnetic fields below  $T_c$ .

High magnetic fields destroy superconductivity and restore that normal conducting state. Depending on the character of this transition, we may distinguish between type I and II superconductors. The graph shown in Figure 6 illustrates the internal magnetic field strength  $B_i$  with increasing applied magnetic field for the both cases.

Type II superconductors are characterized by two critical magnetic fields. When the external magnetic field is less than the lower critical field  $B_{c1}$ , the material is fully superconducting. It means that, there is now flux penetration. When the external field exceeds the upper critical field  $B_{c2}$ , the flux penetrates completely and the superconducting state is destroyed. As on the picture is shown, there is also mixed state in the case when fileds lying  $B_{c1}$  and  $B_{c2}$ .

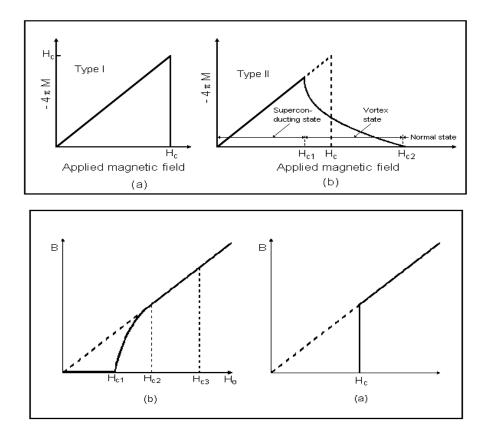


Figure 6: Two type of superconducting materials, according to the behavior in the presence of a mangnetic field. Ref: http://www.materia.coppe.ufrj.br/sarra/artigos/artigo10114/

# 2.3. Scattering Theory

#### 2.3.1 Basic of Scattering

Scattering methods are widely used as a non-destructive method to study materials in condensed matter physics.

Magnetic nanoparticles and assemblies thereof exhibit correlations in a wide size range that can be investigated by various scattering techniques. The nanoparticle morphology can be investigated in the lower nm size range by small-angle scattering methods. Conventional powder diffraction gives insight into a possible long-range order of the atomic structure. Whereas Bragg diffraction gives information on the

averaged long range atomic order, the atomic Pair Distribution Function (PDF) provides insight into the local structure in real space and is complementary to X-ray absorption techniques, which probe mainly the first coordination sphere of a chosen element. Figure 7 shows schematic condition of Bragg scattering.

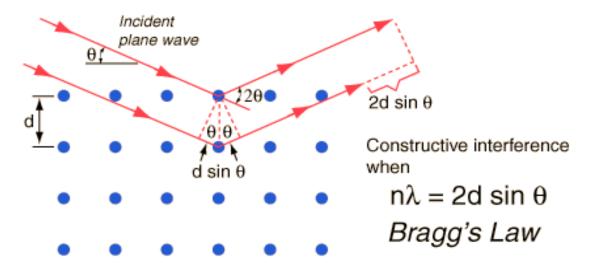


Figure 7: Schematic of Bragg scattering condition. Picture taken from <a href="http://hyperphysics.phy-astr.gsu.edu/hbase/quantum/bragg.html">http://hyperphysics.phy-astr.gsu.edu/hbase/quantum/bragg.html</a>

Bragg scattering occurs when a crystalline arrangement of atoms is irradiated with an X-ray or Neutron beam of wavelengths comparable to the atomic distances. When the incident beam is scattered from a set of lattice planes with a distance  $d_{hkl}$ , the scatter beams interfere constructively for a difference in path lengths of  $n\lambda$ . Figure 8 shows that Bragg reflections are observed for constructive interference of the scattered beams at scattering angles  $2\theta$  according to Bragg's law.

$2dsin\theta = n\lambda$	

For elastic scattering the energy of incident and scattering waves is identical to this formula:

$$|\vec{k}_i| = |\vec{k}_f| = \frac{2\pi}{\lambda}$$

The scattering vector  $\vec{Q}$  is defined as the difference vector between wave vectors of the incident and scattered beams:

$$\vec{Q} = \vec{k_f} - \vec{k_l}$$

With a magnitude of

$$|\vec{Q}| = Q = \frac{4\pi}{\lambda} \sin\theta$$

From this equation we can calculate Q

$$Q = \frac{2\pi}{d}$$

As we explained d is a distance between the lattice planes.

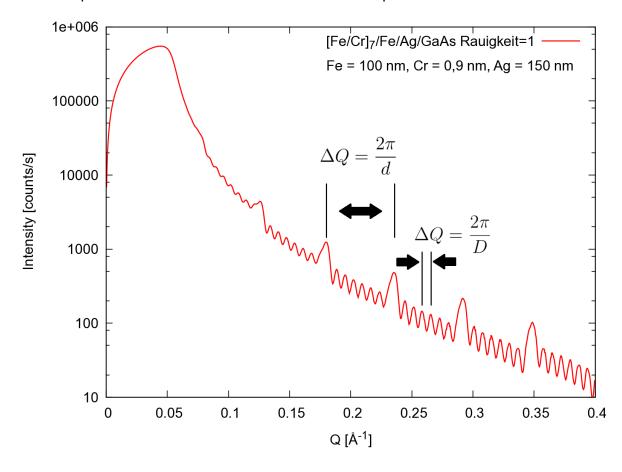


Figure 8: Simulation of a reflectivity measurement of a multilayer Fe/Cr-system. Picture taken from [2]

# 2.3.2. X-ray Reflectometry

In order to characterize the sample there is possibility to use X-ray reflectometry. It is very suitable for the investigation of thin films like grown with the MBE. It has X-ray source and wavelength is 1.54 Angstrom. With the X-ray reflectometer you have access to depth resolved information on layer thickness, roughness and the periodicity of your layer system. The following steps show you how to use the reflectometer to determine the layer thickness.



Figure 9. X-ray Diffractometer in Juelich Research Centre

#### 2.3.2 X-ray Diffraction

X-ray diffraction technique is one of the best tool to characterize the solid state materials. Its analysis is a powerful tool for measuring crystallinity and orientation of thin film samples.

The scheme of a typical scattering experiment can be described by following way: A monochromatic beam, defined by a plane wave with wavelength  $\lambda$ , momentum  $\hbar k$ , and wave number k which equals to  $\frac{2\pi}{\lambda}$  hits the sample. The term diffraction distinguishes those scattering experiments, in which the scattered waves are detected without any analysis of the energy. In general, the information of static structure is given by elastic scattering. For Bragg scattering of an ideal crystal, constructive interference only occurs under the Bragg condition.

There are many possibility to use X-ray diffraction technic for another type of investigation also. Many of them are: Thin film diffraction and grazing incidence X-ray diffraction may be used to characterize the crystallographic structure and preferred orientation of substrate-anchored thin films. High-resolution X-ray diffraction is used to characterize thickness, crystallographic structure, and strain in thin epitaxial films. It employs parallel-beam optics. X-ray pole figure analysis enables one to analyze and determine the distribution of crystalline orientations within a crystalline thin-film sample. X-ray rocking curve analysis is used to quantify grain size and mosaic spread in crystalline materials. Thin film diffraction and grazing incidence X-ray diffraction may be used to characterize the crystallographic structure and preferred orientation of substrate-anchored thin films.

The schematic illustration of X-ray diffraction is shown on the Figure 10.

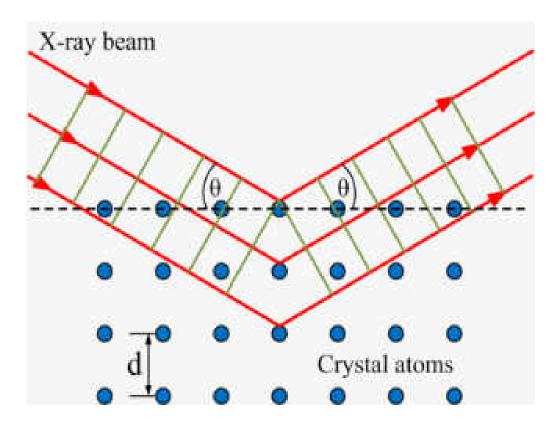


Figure 10: X-ray diffraction scheme. This shows that the difference in path length between X-ray beams incident on a stack of atomic planes is 2d sinθ. The condition for constructive interference of two waves is that they must be shifted by an integral number of wavelengths. Picture taken from <a href="http://ssp.physics.upatras.gr/x-ray">http://ssp.physics.upatras.gr/x-ray</a> diffraction.

#### 2.3.3 Grazing Incidence Small Angle X-ray Scattering

By grazing incidence small-angle scattering (GISAXS), a third dimension of the scattering vector is investigated as for each defined incident angle, a full 2D GISAS pattern is measured with  $\alpha_i \neq \alpha_f$  and  $2\theta_f \neq 0$ . In order to achieve a good resolution in all directions, an incident beam collimated in two dimensions is required. For measurement of a full 2D pattern, also a position sensitive 2D detector is useful. For these reasons GISAS experiments are usually performed on small-angle scattering instruments. The 2D GISAS pattern can be described by the  $2\theta_f$  and  $\alpha_f$  contributions measured in direction of the x and y axis of the detector image, respectively. The specular reflection is detected at  $\alpha_i = \alpha_f$  and  $2\theta_f = 0$ .

In case of neutron scattering, the transmission is often large enough to allow for detection of the transmitted beam, which is located below the sample horizon. Both reflections are located on the off-specular scattering line at  $2\theta_f=0$  and can thus also determined by a single off-specular scan. This is useful because in many GISAXS measurements a beam stop is located at this high intensity line, in order to allow for longer exposure times and thus better statistics in the lower intensity regions. The GISAXS structure is shown on the Figure 11.

GISAXS does not require any specific sample preparation other than thin film deposition techniques. Film thicknesses may range from a few nm to several 100 nm, and such thin films are still fully penetrated by the x-ray beam. The film surface, the film interior, as well as the substrate-film interface are all accessible. By varying the incidence angle the various contributions can be identified.

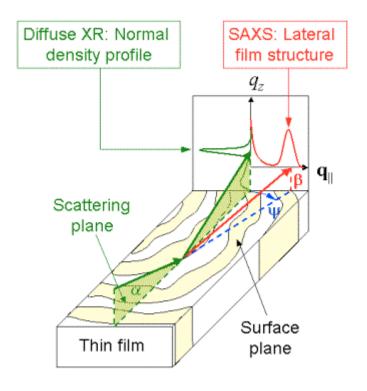


Figure 11: Structure of GISAXS. Picture taken from []

# 3. Experimental Methods

#### 3.1 Sample Preparation

In this chapter, a review of the various experimental techniques used in the work is made. The first section deals with sample preparation methods, whereas the second one introduces the utilized characterization techniques.

The sample under investigation is a typical hybrid of Superconductor-Ferromagnetic (S/F) layer and basically it is alloy of thin film compounds. In order to carry out research according to the project, we started to growth Iron and Palladium (FePd) thin film layers as a compound of ferromagnetic alloy. As for superconductor thin film, we chose to produce material Niobium (Nb), which is type II superconductor material and we had started to prepare Nb thin film. The basic idea is that a superconducting Nb film is placed on top of the ferromagnetic (F) substrate with the easy axis of magnetization in the Z direction.

For the implementation of this research, as I noted, main was to prepare thin film multilayers and we used Molecular Beam Epitaxy machine (MBE). It is process, in which one or more materials are heated up and hence are evaporated and finally recondensed on a substrate. Depending on the material substrate combination the crystal grows layer by layer. In general this is very slow process but it has many advantages. For instance you can develop multilayers of different materials which change from layer to layer. On the picture 12 is shown Molecular Beam Epitaxy machine, which is located in Juelich center neutron science, it is which I used during the working period.

#### 3.2 Thin Film Techniques

Producing of thin films is very importance and necessary for research in Solid-state Physics. It is highly important if techniques have to be fast in order to keep cost law. MBE machine is very slow technique to work in research but it has variety of methods to use. This including sputtering, spin coating, pulsed laser deposition and so on. Here I tried to introduce shortly most useful method sputtering.



Figure 12: Molecular Beam Epitaxy Machine for Sample Preparation in Juelich Researh Centre

The term sputtering refers to a variety of methods. The most common technique is magnetron sputtering. A strong magnetic field is placed around a target made of the material you want to evaporate. Applying of high voltage, depending on the conductivity of the target will ionize the gas and accelerate it towards the target. After this process, the collisions of the gas with the target will break out atoms, molecules or clusters which can then move to the substrate.

A special technique used for oxides is high pressure sputtering. Here the sputter gas is Oxygen at pressures around one milibar. The substrate is placed in close proximity to the target. The high oxygen pressure minimizes re-sputtering on the substrate as the mean free path is very short. Additionally the high oxygen pressure minimizes oxygen deficiency.

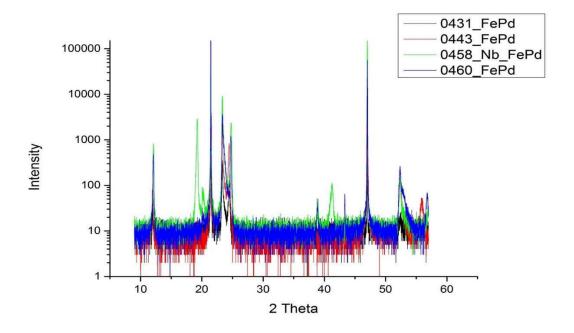
Good vacuum is the prerequisite for thin film growth with MBE and most of the other techniques. You cannot grow a clean thin film if your surface is contaminated with "dirt" from the air.

Magnesium oxide is used in the preparation of the surface layer of sandwich whose thickness is 0.5 mm and the surface layer was prepared at 550 °C. After the buffer layer, we prepared layer of palladium and iron compound for this buffer layer is chromium and palladium, and appropriate thickness of 2 nm and 60 nm, while the temperatures have trained at room temperature. Then we prepared a sample of ferromagnetic compound as iron and palladium thin films consisting of the compound. With a thickness of 50 nm is prepared and 430 °C. As for the second type of superconducting layer we put niobium, thickness of 50 nm which is composed of niobium and 50 °C. Thus the end we got the alloy under study. Which is pretty good and the status of invisible information is shown below the images. Sample size was 10 mm x 10 mm and a thickness of 0.2 cm. It is shown on the picture below.

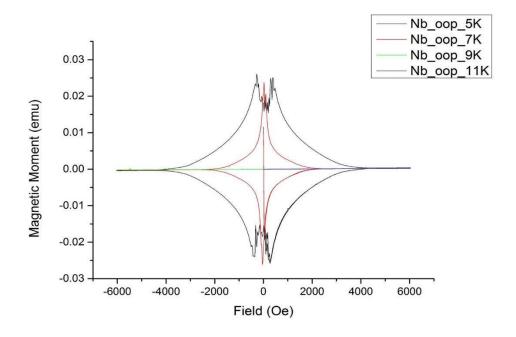


#### 3.3 Measurements

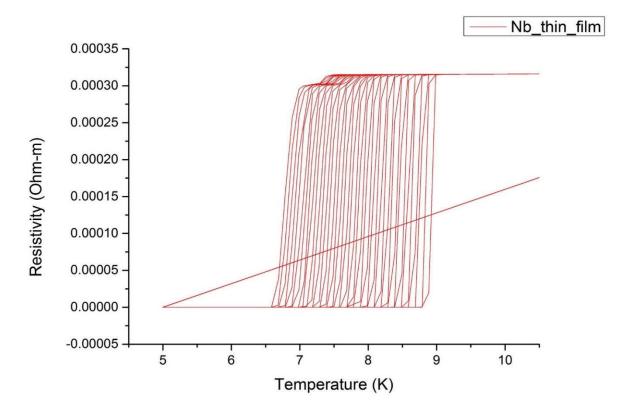
We used X-Ray diffraction measurement method to determine the planes conducted which clearly showed that the intensity of both the plane in case of an equal. The experiment is shown in the picture below. Total we were prepared 4 different layer. A Photo shows that the temperature increase of more and better structures have received the order. The intensity was not good, however, several layers of production test, we have achieved the desired result.



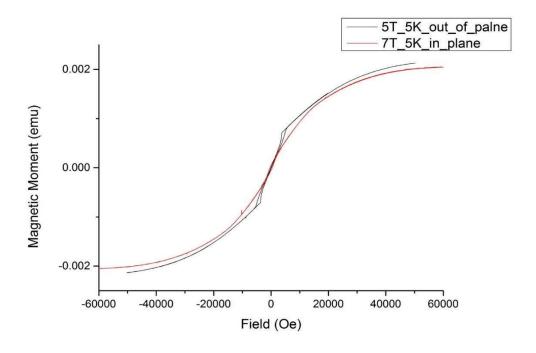
We also conduct measurement for Superconducting layer, dependent of the magnetization on the magnetic field. Whereby the magnetic field is perpendicular to the sample's own magnetic moment. The measurement was performed in 5, 7, 9 and 11 Kelvin temperatures. Estimated that 9 and 11 Kelvin temperature superconducting niobium became paramagnetic. And 5 Kelvin temperature during its magnetization was bigger than 7 Kelvin temperature. The measurements are shown in the picture below. Magnetic measurement systems used in the data set.



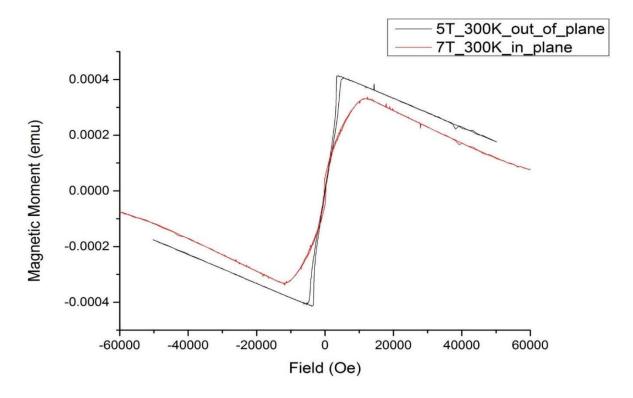
At the same time, we have met resistance temperature dependence for the superconducting layer, at the physical property measurement systems. The measurement was quite determined and fruitful. Chart is shown in the picture below.



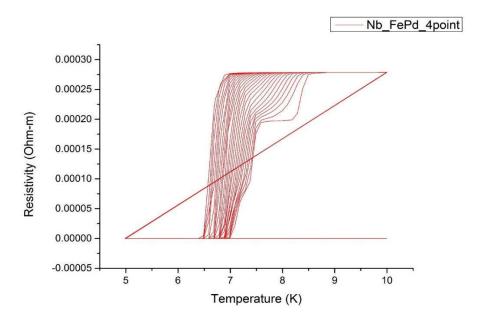
Also, we also performed the magnetization measurements in ferromagnetic compounds in two different cases, depending on the direction of the magnetic field. In the first case, the field is perpendicular to the magnetic moment of a sample of its own, and in the second case it was a concurrent design their own magnetic moment. The first case the measurement was performed at a temperature of 5 K, 300 K and the other in case of temperature. Chart is shown in the picture below.



This time showing the same kind of measurement, but as mentioned above are compatible with the magnetic field and the magnetic moment of the sample for the measurement was performed at a temperature of 300 Kelvin. Bear in mind that in both cases the magnetic field of 7 tesla and 5 respectively.



Last, the measurement was performed in which the project period, the temperature dependence of the resistivity measurement in the study design and the temperature varied from 10 K in the range of 5 Kelvin. The measurement results are shown in the picture.



#### 4. Results

Such measurements and analysis of the data, given the different types of results based on which it has been determined that the magnetic moment is greater than the superconducting layer of ferromagnetic materials, iron and palladium compounds. Impedance critical temperatures obtained niobium layer lower than the massive niobium samples. As for the particular sample, which is a pretty good line of prepared for it, we got a much wider transitions than only in a thin layer of niobium than specifically. It is caused by iron and palladium ferromagnetic properties.

#### 5. Literature

- 1. Magnetism in Condensed Matter Stephen Blundell
- 2. Introduction to Solid state Physics Charles Kittel
- 3. Preparation of Functional Oxide Multilayers Part 1 Physical and technical background Alexander Weber February 25, 2015
- 4. Preparation of Functional Oxide Multilayers Part 2 Experimental Tasks Alexander Weber February 26, 2015
- 5. Domain-wall superconductivity in superconductor–ferromagnet hybrids. An article by ZHAORONG YANG, MARTIN LANGE, ALEXANDER VOLODIN, RITTA SZYMCZAK AND VICTOR V. MOSHCHALKOV. Published online 3 October 2004.
- 6. Magnetic domains in epitaxial ordered FePd(001) thin films with perpendicular magnetic anisotropy. V. Gehanno and A. Marty *Service de Physique des Mate riaux et Microstructures/De partement de Recherche Fondamentale sur la Matie re Condense e/CEA.*

38 054 Grenoble, France

- 7. Electrical and Magnetic Properties of High Temperature Superconductors Using Varying forms of Data Acquisition. Hays, Aryn M.; Bechtel, Lindsay; Turbeville, Colton; Johnsen, Anthony; and Tanner, Chris A. (2013) "Electrical and Magnetic Properties of High Temperature Superconductors Using Varying forms of Data Acquisition," *Journal of the Advanced Undergraduate Physics Laboratory Investigation*: Vol. 1: Iss. 1, Article 3.
- 8. Fundamentals of Superconductivity C.M. Rey, A.P. Malozemoff 1Energy to Power Solutions (E2P), Knoxville, TN, USA; 2AMSC, Devens, MA, USA.

9. Modern Physics by Serway, Moses and Moyer.