CRYSTAL SYNTHESIS OF SELECTED QUANTUM MATERIALS

by

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ABSTRACT

CRYSTAL SYNTHESIS OF SELECTED QUANTUM MATERIALS

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Cutting edge technology requires breakthrough materials, driving the exploration of condensed matter physics to quantum materials - materials with properties that are only explained through quantum mechanics. This thesis features three quantum materials, which are superconductors, topological insulators, and geometrically frustrated anti-ferromagnetic (AFM) materials. Using three different growth methods, I grow crystals that are suspected to be in one or more of these classes of quantum materials. Niobium has been shown to be an effective superconducting dopant and has a high superconducting critical temperature, similar to Magnesium Boride ($\text{MgB}_2$), in addition to potentially being a topological insulator due to the strong spin-orbit coupling of Niobium [1–5]. A dip probe, physical property measurement system, and X-ray diffraction measure the properties of these materials and finds the critical temperature of Yttrium Barium Copper Oxide to be 92.3 K and the X-ray
diffraction patterns of Niobium Boride, Tantalum Boride, and the Kagome Lattice AFM materials are identified [6]. This work deepens the understanding of these materials for future use in designing quantum technology.
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Chapter 1

Introduction

1.1 Motivation

The research in this thesis is to develop the understanding of material properties to guide future work in solid state devices. The study of condensed matter physics and solid state materials has revolutionized the electronics industry, laser technology, biophysics, quantum computing, and nanotechnology. As the understanding of material properties grows, each of these fields can implement improved technologies. Of particular interest are materials that exhibit ideal material properties.

Computers used to be the size of entire rooms and could not store information. Now, computers fit in your pocket and can store over a terabyte of data. Lasers are now far more precise than their beginning as the Microwave Amplification by Stimulated Emission of Radiation (MASER) and can produce short broadband coherent light burst of down to a femtosecond. As technology develops, engineers encounter obstacles in design that required materials with different electrical, magnetic, and heat conduction properties than those which currently exist. With a better understanding of material science, researchers can better design custom materials with desired
properties. The next generation of solid state devices will rely on quantum materials which are compounds that have properties that can be explained with quantum mechanics.

Quantum phenomenon have been studied since the early 20th century, but its application at the microscale has only been explored over the past 20 years. This research has led to the discovery of materials that have altered the way we understand quantum mechanics. This has opened up an entirely new field of physics and mathematics since the behavior of matter at the microscale differs from that of classical matter at the macroscale and quantum matter at the nanoscale. Because of the lack of knowledge and the potential economic benefits in this area, condensed quantum matter physics has become the most widely research and heavily funded area in physics.

Three types of solid state materials that this thesis discusses are superconductors, topological insulators, and anti-ferromagnetic materials. This research is pioneering the next generation of solid state devices.

1.2 Background

1.2.1 Superconductors

Superconductors are a class of material which have three key characteristics: conduction of electricity with zero resistivity which allows large currents to run through the material with zero power loss, the Meissner effect which expels external magnetic fields making superconductors ideal diamagnets, and a specific heat discontinuity which sharply increases the specific heat at the critical temperature [7]. The temperature at which a material undergoes a second order phase change from its normal state to its superconducting state is called the critical temperature and is denoted $T_c$. 


1.2 Background

In the 20th century, scientists attempted to measure the resistivity of superconductors by running then measuring currents overtime in superconducting loops. They observed that the current did not decrease over time indicating zero resistivity. This method is still used today [8]. This result fundamentally changed the way electricity understood. Such a discovery has led to new theories on how superconductors work microscopically and has further developed quantum mechanical theory.

Superconductors have two types. Type I superconductors, when $T < T_c$, remain superconducting in external magnetic field ($B_{ext}$) and create currents inside themselves to perfectly oppose the $b_{ext}$, thus exhibiting ideal diamagnetism. This superconducting state exists in the material until the $b_{ext}$ exceeds a temperature dependent critical field ($B_c$), then the material transitions back to its normal state. Type II superconductors, when $T < T_c$, remain superconducting until $B_{ext} > B_{c1}$, its first or lower critical field, at which point the external field begins to penetrate the material in quantized flux tunnels called Abrikosov Vortices. These tunnels form a 2 dimensional triangular lattice and each tunnel increases in radius as does the tunnels’ penetration depth when the $B_{ext}$ increases. Once the $B_{ext}$ reaches $B_{c2}$, the material’s second or higher critical field, the material returns to its normal state, and this $B_{c2}$ is usually significantly higher than the $B_c$ for type I superconductors.

Notably, Yttrium Barium Copper Oxide - YBCO for short - a ceramic material, has been found to be a type II superconductor and is the first superconductor that has $T_c$ higher than liquid Nitrogen. The experimental methods section discusses the synthesis of YBCO.

1.2.2 Topological Insulators

Until the early 1970’s, solid state scientists only thought of material bulk properties, but this changed when J. Michael Kosterlitz from Brown University and David J.
Thouless from the University of Washington used topological principles to explain why some materials exhibit superconducting properties at low temperatures, but not at high temperatures [9]. This application of topology to condensed matter physics opened the doors to topological materials - materials which have a surface state different than the bulk or interior state. This research features 2-dimensional topological insulators whose bulk is an insulator, but whose surface conducts electricity at zero resistance [10]. Topological insulators’ zero resistance surface state closely relates them to superconductors, but the lack of the other two characteristics distinguishes them as not superconducting. However, this state of zero resistance happens at temperatures far higher than the critical temperature of any known superconductor, and since their chemical compositions are more simple than superconductors’, their production is easier than superconductors.

Classical insulators have an energy gap larger than 4.2 eV between their valence band and their conduction band with the valence band being completely filled with valence electrons and the conduction band having nearly zero electron occupancy; in the surface state, it is slightly easier to excite electrons to the conduction band as the band gap is smaller than in the bulk, but still significant. In topological insulators, the bulk has strong electron spin-orbit coupling (SOC) that causes an electron band inversion, meaning the high energy valence electrons and low energy conduction electrons switch energy levels so that conduction electrons are at energy levels just below the gap and valence electrons are at energy levels just above the gap, as shown in Figure 1.1. Strong SOC also creates an effect in an external magnetic field in which the electrons spins align causing the internal current to be net zero, but the spins create a spin carried current along the edge of each layer [11–13]. This effect is called the spin quantum Hall effect because of its similarity to the integer quantum Hall effect, and was first observed in graphene [14–17]. The transition metals have
strong SOC, so they and materials containing them are likely to have novel quantum
topological states [18]. Since topological properties occur only at the surface of a
grain in a crystal, useful topological materials are grown as single crystals.

![Figure 1.1 Topological Insulator Band Structure [13]](image)

1.2.3 Geometrically Frustrated Anti-Ferromagnetic Materials

Anti-ferromagnetism is a phenomenon in which the magnetic moments in a crystal
align antiparallel to their nearest neighbors. This arrangement is straightforward
in rectangular and cubic lattices, but some lattices make this antiparallel orientation
impossible - geometrically frustrated lattices, i.e. triangular or tetrahedral lattices.
In a triangular lattice, for example, if one lattice site is spin up then another will be
spin down, but what will the third be? Regardless of which direction it spins one of
its nearest neighbors will be the same spin, defying the antiparallelism of the lattice.
Three different arrangements may occur: 1) the triangular lattice with have 6-fold
degeneracy because the each lattice site can be spin up or down with the other two
being the opposite spin; 2) one site will be spin up, another spin down, and the third
at some angle; 3) the lattice sites will all point toward or away from the center of the triangle. This class of materials has interesting magnetic properties that are relevant to the field of spintronics.

1.2.4 Growth Methods

Most elements and chemical compounds in their solid phase of matter do not spontaneously react with other solid elements and chemical compounds at room temperature, but they become more reactive at higher temperatures. To grow crystalline solids, furnaces heat up the solid chemicals which allows them to react and form new compounds. Chemicals that are stoichiometrically measured and combined together to produce an anticipated produce for a single experiment are called a sample. There are 3 different crystal growth methods that this thesis will discuss: solid state, metal flux, and arc melting. The first two methods mentioned use a temperature profile that is determined before every experiment which entails what temperatures to heat the sample to, how long it will take to reach that temperature, and how long that temperature will be maintained.

The method used for an experiment depends on the reactants individual properties, the way they react with one another, how they can react with the container in which they are placed during the reaction, and the container’s melting temperature [19]. The containers used in this thesis are Alumina (Al$_2$O$_3$) crucibles and quartz (SiO$_2$ glass single crystal) tubes.

Solid State Reaction

The solid state reaction is the most simple crystal growth method discussed in this thesis. This method heats the sample and the reactants to form a product through a chemical reaction at high temperatures. The sample reacts more completely when the
reactants have more contact with each other so the reactants should be thoroughly
ground into powder and mixed together. The reactants are then placed in their
container and into the furnace set to the determined temperature profile.

**Metal Flux Method**

The metal flux method places the reactants in a metal that has a low melting point to
act as a catalyst for the reaction. This makes reactions between two or more elements
with high melting temperatures more reasonable as the flux-reactant mixture will
have a lower melting temperature than the reactants alone, and the greater amount
of flux the closer the melting temperature of the mixture will be to that of the flux.
Phase diagrams depict this phenomenon; notice in Figure 1.2 that the liquid phase
of a 96%-4% Aluminum-Niobium mixture occurs at 1400°C rather than the melting
temperature of Niobium at 2469°C.

![Figure 1.2 Aluminum-Niobium Phase Diagram](image)

**Figure 1.2** Aluminum-Niobium Phase Diagram [20]
1.2 Background

Having the reactants in their liquid phase enables them to nucleate as they cool, which means as the temperature lowers, a small solid crystal starts to form in the liquid and acts as a nucleus on which all the other atoms solidify and align in the crystal lattice. This process of nucleation usually results in many small single crystals and the removal of the metal flux once it hardens can be difficult. Figure 1.3 details the flux method and that nucleation occurs in the time period that temperature is decreasing, which is the period of crystal growth in every method.

![Diagram of Flux Method](image)

**Figure 1.3** Diagram of Flux Method [21]

**Arc Melting Method**

The arc melting method uses an arc of ionized gas to rapidly melt a sample. In this thesis, the arc melting reactions occur in a vacuum chamber. The reactants are placed in a Copper crucible along with Zirconium and the crucible placed in the vacuum chamber. The vacuum evacuates the chamber three times and Argon is pumped in after the first and second time, leaving the chamber a vacuum. Then a current places charges on the Tungsten electrode over the Zirconium, and the electric field between the electrode and the Copper crucible ionizes the dilute gas in the chamber causing a 3000°C arc to run through the Zirconium in the crucible, which in turn
melts the Zirconium, as depicted in Figure 1.4a. The Zirconium readily reacts with any remaining Oxygen in the chamber, and thus precedes any reaction that produces a sample. Then, the experiment proceeds in the same manner, completely melting the sample 3 times to ensure the reaction is complete. During the experiment, the dilute gas thermodynamically expands causing a rise in pressure, at which point the chamber should be evacuated, again.

![Arc Melting Apparatus](image1)

![Arc Melting Chamber in use](image2)

(a) Arc Melting Apparatus [19] (b) Arc Melting Chamber in use

**Figure 1.4** Diagram of Arc Melting Chamber

Since the arc melting method happens in such a short amount of time, the crystal purity and quality of the samples are low, so arc melted samples are often annealed. The process of annealing purifies the sample and improves crystal structure by heating the crystal to 750°C in 5 hours, and maintaining that temperature for 100 hours.

### 1.3 Research Objective

\(\text{MgB}_2\) was shown to be a high critical temperature (39 K) superconductor [1]. We proposed altering the compound to contain a transition metal in place of Magnesium
1.3 Research Objective

could make a material that was both a high temperature superconductor [4] and
topological insulator, due to the strong SOC in transition metals. The transition
metals selected were Niobium and Tantalum, because they are in the same period
as Magnesium so have similar electric properties. This thesis discusses the syntheses
and outcomes of the Niobium Boride and Tantalum Boride crystals. Niobium Boride
grew based on the methods discussed in [22], and Tantalum Boride grew according
to [23], for a baseline experiment.

The Kagome lattice - shown in Figure 1.5 - is a known naturally occurring crystal
structure that causes frustration in AFM materials. The compound TbMn$_6$Sn$_6$ is a
material that forms this structure with AFM properties [24], so to find more mate-
rials with this structure and AFM property we alter the composition to ZrCo$_6$Ge$_6$
and AFe$_6$Ge$_6$ (A=Zr, Nb, Hf). These are logical candidates for replacements since
Manganese (Mn), Iron (Fe), and Cobalt (Co) are all ferromagnetic, therefore having
similar magnetic properties.

![Figure 1.5](image.png)

**Figure 1.5** Structure of TbMn$_6$Sn$_6$ [24]
Chapter 2

Experimental Methods

2.1 YBa$_2$Cu$_3$O$_7$

The Yttrium Barium Copper Oxide synthesized by combining three compounds Y$_2$O$_3$, BaCO$_3$, and CuO in stoichiometric proportion according to the chemical equation

$$Y_2O_3 + 4BaCO_3 + 6CuO \rightarrow 2YBa_2Cu_3O_7-x (+O_2).$$ (2.1)

Note the $+O_2$ indicates the byproduct of oxygen gas evaporated during the reaction.

We weighed these powders using a scale accurate within ±0.5mg, metal spatulas, and aluminum weighing dishes. The powders were then ground together by an agate mortar and pestle for thirty minutes and placed in an alumina (Al$_2$O$_3$) crucible.

A box furnace heated the sample to 920°C in 5 hours, then kept at that temperature for 10 hours, then returned to room temperature over about 10 hours, thus reacted by solid state reaction. We ground this sample again and pressed it into a pellet using a hydraulic press at 1.5 tons and a dye. The sample underwent another stint in the box furnace at the same temperature profile as before.

Once the crystal synthesis finished, small pieces of the crystal, broken off with
a hammer, went onto a dip probe for testing. First, fine platinum wires adhered to
the surface, using silver paste as a conducting adhesive, in a pattern that allowed a
voltage reading in a circuit built upon the crystal shard, as shown in figure 2.1. Then,
the probe descended into a container of liquid nitrogen to measure the resistance as
the temperature decreased from 300\(K\) to below 80\(K\).

After grinding some of the sample by hand with an agate, mortar, and pestle, we
placed the powders on glass sample slides and then into the x-ray diffraction apparatus
(XRD), as described in [20]. This same process occurs for all of the samples in this
thesis.

2.2 \( XB_{1.6} (X=\text{Nb, Ta}) \)

Niobium Boride and Tantalum Boride grew close to the same way. Each began by
tearing pieces of 99.9999\% pure Niobium and Tantalum tubes and measuring an
amount of each that was within 0.1 g of the desired weight. Then, a stoichiometric
amount of Boron was measured and added according to the measured weight of Nio-
bium and Tantalum. Each sample went into a deep alumina crucible with 99.9999\%
pure Aluminum shots added to each sample to act as a metal flux. The aluminum flux
additions were 35g \(\pm 1g\) to the Niobium-Boron sample and 50g \(\pm 1g\) to the Tantalum-
Boron sample.
The Niobium Boride sample went into the center of a tube furnace along with a strip of Zirconium, which more readily reacts with any remaining Oxygen at lower temperatures than elements in the sample. We sealed it then evacuated the tube and filled it with Argon gas 3 times to remove atmospheric contamination. As the pressure increased inside the tube during heating, the Argon gas escaped through a small tube at one end of the furnace tube. The samples’ heating profile was 900°C in 2 hours, to 1560°C in 2 hours, kept at 1560°C for 10 hours, to 700°C over the course of 100 hours. The Tantalum Boride synthesis followed the same procedures as Niobium Boride.

Once the samples finished with their growth, they went in a Hydrochloric Acid (HCl) bath for a week to dissolve the then hardened Aluminum flux in which they were sitting.

Small pieces of each sample went onto a probe in a circuit similar to YBCO and then the Physical Property Measurement System (PPMS) measured their resistivity from 300 K down to 2.2 K.

2.3 \(Zr\text{Co}_6\text{Ge}_6\) and \(A\text{Fe}_6\text{Ge}_6\) (\(A=Zr, Nb, Hf\))

Each of these samples grew by combining stoichiometric quantities of the individual elements, which were in pieces much larger than powder with the exception of Cobalt which was in powder form and pressed into a pellet with a hydraulic press.

The samples reacted via arc melting method in Argon gas. To increase crystal quality and purity, the samples were annealed by heating to 750° in 5 hours and keeping it there for 100 hours.
Chapter 3

Results

3.1 $\text{YBa}_2\text{Cu}_3\text{O}_7$

The $\text{YBa}_2\text{Cu}_3\text{O}_7$ polycrystal was 1 cm long and .5 cm in diameter, as seen in Figure 3.1. The black smudges in the alumina crucible are $\text{YBa}_2\text{Cu}_3\text{O}_7$ powder that remained after when the powder was pressed into a pellet.
The analysis of Figure 3.2 using the cross method found the critical temperature of the YBa$_2$Cu$_3$O$_{7}$ sample at 92.3 K ± 0.1 K, which the intersection of the two lines indicates. The sharp resistance drop is characteristic of a superconductor. This experimental critical temperature is close to the optimized critical temperature of 93 K, suggesting a small amount of chemical impurity or a high yet imperfect crystal quality, which means a flaw in the crystal structure. The x in the oxygen subscript of YBa$_2$Cu$_3$O$_{7}$ in the figure appears because different levels of oxygen doping influences this material’s properties, but this was not the purpose of the experiment so x was not calculated.
The YBa$_2$Cu$_3$O$_7$ sample’s XRD plot, shown in Figure 3.6, has the 2 times the angle of incidence in degrees along the x-axis and the normalized intensity along the y-axis. The intensity is normalized with the maximum intensity equal to 1, since the $2\theta$ position and relative intensity of each peak are what is most important to compare XRD signatures of samples to XRD signatures of recorded materials. The sample data (blue line) is 1 normalized intensity unit higher than the known YBa$_2$Cu$_3$O$_7$ XRD signature so the x-axis values align and comparison is easy and its sample label, JH01, are my initials and which sample number it is - the rest of the XRD plots are labeled similarly. The sample data shows a hill from 10 to 40 on the $2\theta$ axis, which is common among all XRD sample data in this thesis. This hill occurs because the samples have a small amount of amorphous SiO$_2$ from their sample tubes. Each peak in Figure 3.6 matches the expected XRD signature of YBa$_2$Cu$_3$O$_7$ indicating that the sample was pure.
The NbB$_2$ crystals measured 2 to 3 millimeters long and 1 millimeter across with a high quality cubic crystal structure. Figure 3.4 shows the NbB$_2$ crystals on a 1 mm grid paper for scale. The crystals had partially dissolved in the hydrochloric acid, during its week long acid bath. The crystals were reflective, hard, and metallic.
This crystal’s resistivity versus temperature graph in Figure 3.5 has an x-axis that is raw temperature data in Kelvin from the PPMS and the y-axis has resistivity ($\rho$) in $\mu\Omega$cm, which is a calculated value from the equation

$$Resistance = \rho \frac{\text{Length}}{\text{Area}}.$$  

(3.1)

The PPMS returned the resistance as raw data output along with temperature. The length and area corresponded to the physical dimensions of the NbB$_2$ sample on the PPMS probe. The graph not showing a resistivity drop indicated that our sample was not a superconductor with critical temperature above 2.2 K. It could potentially have a critical temperature below 2.2 K, but the resistivity flattens out at around 2.7 $\mu\Omega$cm like a classical conductor does. This suggests that the cubic state of NbB$_2$ is not superconducting.
This XRD signature matches that of Nb$_{0.943}$B$_2$ which means our crystal was the correct compound, but not at the doping level we hoped to achieve.

The crystals grew in two different phases. The one dimensional phase was just over 1 mm long and the other was around .4 mm by .3 mm. These dimensions are
smaller than what would be useful in most industrial uses, likely because there was too much flux. Greater the amount of flux, the longer the sample needs to dissolve and the more the crystal will dissolve resulting in smaller crystals.

![Figure 3.8 TaB₂ XRD](image)

This figure shows that there were two phases in the Tantalum Boride sample, and yet neither were the anticipated composition. Measurements for resistivity verses temperature could not be obtained due to a faulty circuit on the sample in the PPMS.
3.3 ZrCo$_6$Ge$_6$ and AFe$_6$Ge$_6$ (A=Zr, Nb, Hf)

These XRD data plots show peaks that do not appear on the predicted XRD signature of the expected material, yet there enough matching peaks to conclude that they are the same materials. These additional peaks indicate impurities in our samples and the blue asterisks mark them for convenience. The arc melting method commonly produces impurity peaks, but the crystal can be annealed to decrease or eliminate impurities.
Chapter 4

Conclusion

The YBa$_2$Cu$_3$O$_7$ sample having critical temperature to be 92.3 K showed that this material’s synthesis and high temperature superconducting property is reproducible.

The NbB$_2$ growth and analysis showed that this material can grow using an Aluminum flux. Future work involves decreasing Boron contents to form the compound NbB$_{1-x}$ with $0 \leq x \leq .4$ and testing for superconductivity to make a well established doping range that yields superconductors. The superconducting samples will then be tested for topological properties.

The attempted TaB$_2$ growth indicated that the flux method with 50 g of Aluminum flux does not produce TaB$_2$. Future work involves decreasing the amount of flux, decreasing the temperature gradient in the nucleation stage of crystal synthesis, and changing input chemical composition to obtain TaB$_{1-x}$ with $0 \leq x \leq .4$. Testing will be carried out as described in the previous paragraph.

Producing the precursors to the Kagome lattice materials shows arc melting is effective for precursors, but needs annealing to improve crystal quality and purity.
Bibliography


