Midterm Report

Genaro[1]

[1]Department of Physics, University of Kentucky, Lexington

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1 Getting Started

I arrived at UK a week after the program started so I planned on working overtime in order to get caught up. However, when I arrived I learned that the main objective for our research group currently was to move from the basement to the newly renovated third floor. This meant I didn't really start my research until the third week of the program.

Moving the lab took patience and perseverance. A lot of the specialized equipment in Seo's lab is very delicate, like the vacuum chambers and the laser. With the help of the campus moving group and the other three REU students, MacKenzie, Alkhatab, and Tina, we got the lab moved and we were able to begin research.

2 Preliminary Research

I began studying and preliminary research for this program in May before my University left for summer break. I was told I would be researching $SrCoO_{2.5}$ so I began looking into finding its balanced stoichiometric equation from the two reactants $SrCO_3$ and Co_3O_4. However, when I presented my work to Professor Seo's graduate student Sujan I learned that my compound was switched for another, Sr_2RhO_4 so I again found the balanced stoichiometric equation with the two reactants $SrCO_3$ and Rh_2O_3. The balanced equation is as follows:

$$4SrCO_3 + 1Rh_2O_3 => 2Sr_2RhO_4 + 3CO_2 + 1CO \tag{1}$$

I found the molar mass of each of the atoms in the compounds and used them to determine how many grams of the reactants would be required for one gram of the finished product. The results are in tables 1 and 2.

Table 1: Molar Masses of Elements in Reaction

Element	molar mass (gram/mol)
Sr	87.62
C	12.011
O	15.999
Rh	102.9055

Table 2: Required Grams of Reactants

Compound	Required Amount (grams)
$SrCO_3$	0.863
Rh_2O_3	0.3709

Getting the necessary amounts of each reactant is important, but we also needed a recipe for making Sr_2RhO_4. Just putting the reactants next to each other does not induce a chemical reaction. After looking through many papers, the preferred way of forming Sr_2RhO_4 was established. We would grind the two reactants together in a mortar and pestle, then press the combined mixture into a small pellet, see figure 1. This pellet would make it so the powder was tightly bound when it went into the furnace. The furnace is what will actually induce the solid state reaction between the reactants and allow them to change into the product.

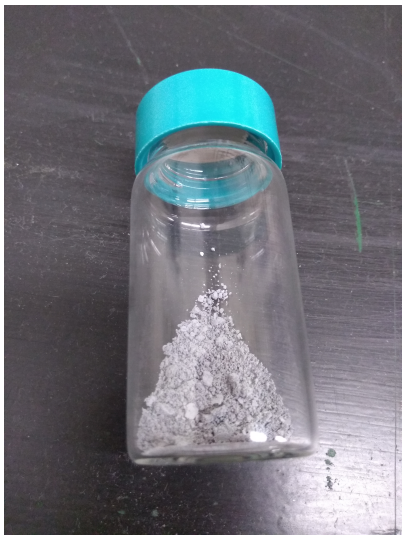

Figure 1: Mixed Powder of $SrCO_3$ + Rh_2O_3

3 Experimental

When I started grinding the powders together I was only allowed to do so under supervision of another student or grad student. This meant I could not grind the powders together as long as recommended. Typically, the longer the powders are ground together, the better the resulting reaction will be in the furnace. Professor Seo recommended a grinding time of three hours, but I only had time to grind for 30 minutes. This may explain why my resulting product wasn't as pure as hoped. After grinding the pellet was placed into a dry hydraulic press for one hour at 19 tons of pressure. After the hour was up the press read only 17 tons of pressure. See figure 2 for a picture of the pellet press and figure 3 of the completed pellet. The pellet was placed into an aluminum crucible and then into a furnace at room temperature, around $\sim 20^oC$ and the furnace rose in temperature at a rate of 1 degree Celsius per minute until at 900 degrees Celsius. Then the pellet stayed in the furnace at constant

Figure 2: Pellet Press

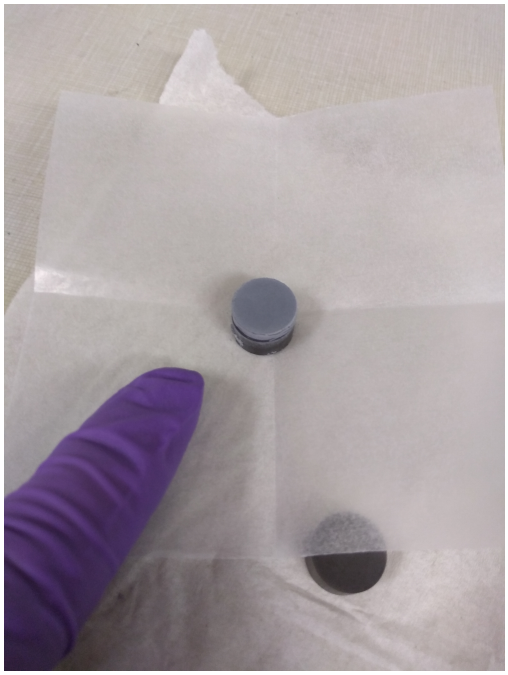

Figure 3: Pellet

temperature for 24 hours before the furnace dropped to room temperature again at a rate of 1 degree per minute. This meant the pellet was in the furnace for a total of 54 hours. The crucible allowed for the pellet to not be placed on the 'floor' of the oven which kept the sample pure.

In order to check if the furnace induced the correct solid state reaction and created the proper product it was necessary to learn about X-Ray diffraction. This X-Ray diffraction (XRD) technique allows us to identify compounds by their crystal lattice. This means that after we take our mixture out of the furnace, we can use the XRD machine to accurately tell if what we have created is the desired product or if it is something else. XRD works on the principle of constructive interference. Because our reactants and eventual product have a crystalline structure meaning the atoms follow a regular repeating pattern, we can shoot the crystal structure with X-rays and track the intensity of the refracted X-rays at different angles. Because we typically shoot these X-rays at a powder of the crystalline substance, the X-rays will diffract in every direction, but will constructively interfere when they travel through a distance that is an integer multiple of the wavelength. See figure 4 courtesy of Anton Paar.com for a pictograph representation. The reason X-rays are used is because the wavelength of X-rays is extremely small, it is so small it is comparable to the spacing in a crystal lattice. This allows for the previously mentioned constructive interference effect. Because all unique compounds will have a unique X-ray diffraction interference graph, we can identify the compound from the furnace by its XRD graph.

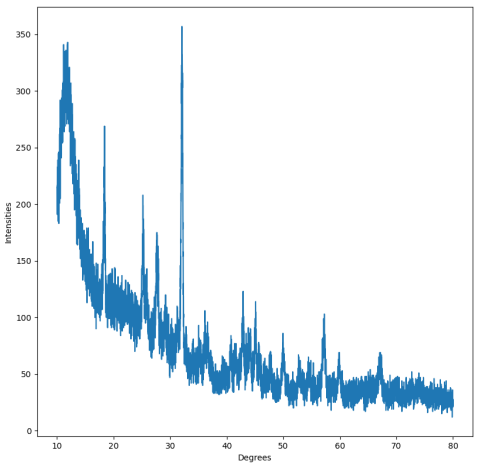

Figure 5: Calcinated XRD Profile

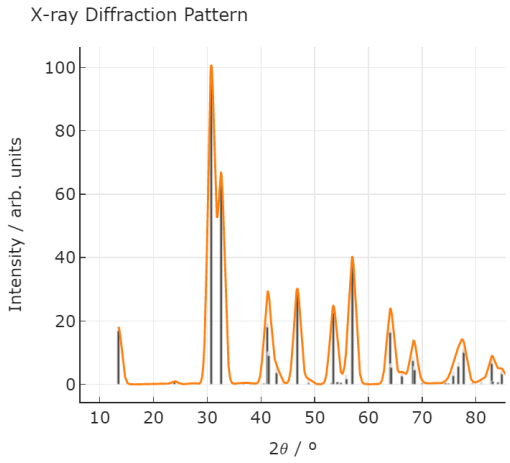

Figure 6: Materials Project XRD for Sr_2RhO_4

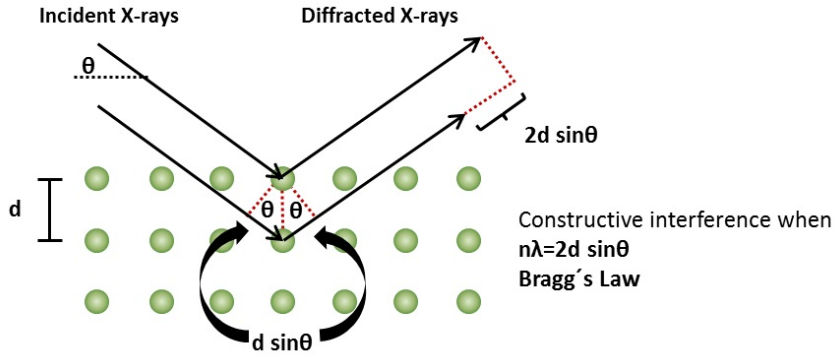

Figure 4: XRD Schematic

After putting the calcined powder through the XRD machine I obtained a diffraction profile. I compared my diffraction profile, figure 5 to a diffraction profile downloaded from The Materials Project, 6. It is clear that my data does not precisely match the data as reported by the materials project. Possible reasons for these discrepancies include: my powder didn't react completely and some the reactants are still present in the mixture, the Materials Project may have used different parameters when generating their diffraction pattern, and the XRD machine we used may be misaligned. Because the latter two reasons are much more unlikely than the first, I will be grinding the calcined powder for two and a half hours, then re-pressing into a pellet, followed by placing the pellet back into the furnace. By re-grinding and re-firing the pellet I hope to remove all contaminants from the mixture and end up with only the desired product.

4 Next Steps

The main goal of my time here is to synthesize this compound, Sr_2RhO_4, and create a thin film of it using pulsed laser deposition. This pulsed laser deposition (PLD) technique involves shooting a laser of varying intensities at a target of Sr_2RhO_4. The laser will take the solid pellet of Sr_2RhO_4 and turn it into a hot plasma which will deposit onto a pre-selected substrate material. This is what we are hoping to study with atomic force microscopy. The deposition of Sr_2RhO_4 will form a thin film which is ideal for atomic force microscopy. However, due to the lab moving and a lack of necessary outlets for the laser, we have not started this part of the program yet. We are estimating that in week 6 we will be creating our first thin films. See figure 7 for an overview of how PLD works provided by Ted Sanders.

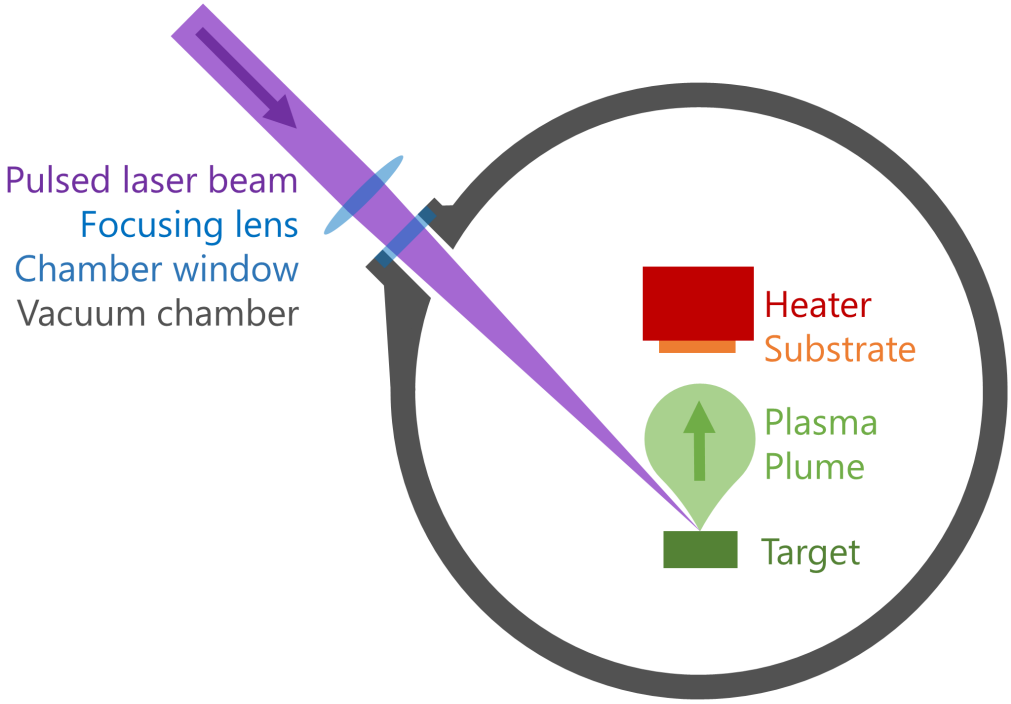

Figure 7: PLD Overview