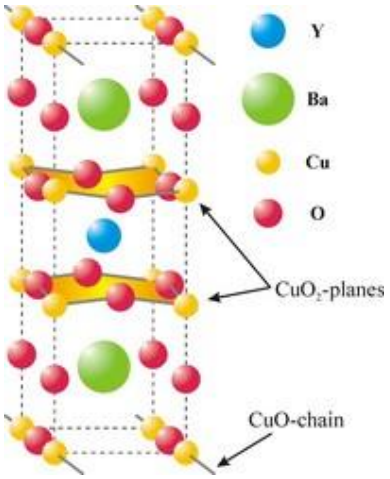
Making a sample $TmBa_2Cu_3O_7$, 2 grams



Start with $BaCO_3$ + CuO , in this case Tm_4O_7

> Before starting dry the starting materials in :
> ❖ $BaCO_3$ in 125^0C , CuO in 450^0C , Tm_4O_7 in 900^0C for 1 day

Find the relevant molecular weights:

- Mass of the sample : $\mathbf{m_s}$=$TmBa_2Cu_3O_7$ = (168.9342)+2(137.33)+3(63.546)+7(15.9994) = 746.23 g/mol
- m ($BaCO_3$)= (137.33) + (12.011) + 3 (15.9994) = 197.34 g/mol
- m (CuO)= 63.146 + 15.9994 = 79.5454 g/mol
- m (Tm_4O_7) = 4 (168.9342) + 7 (15.9994)= 787.7326 g/mol

also $\frac{\text{m (BaCO3)}}{\text{m (Ba)}} = \frac{197.34}{137.33} = 1.43698$, $\frac{\text{m (CuO)}}{\text{m (Cu)}} = 1.25178$, $\frac{\text{m (Tm4O7)}}{\text{m (Tm)}} = 1.16574$

We want 2 grams of $TmBa_2Cu_3O_7$, thus

g of Tm : $\frac{1 \text{ mole (Tm)}}{\text{mole (sample)}} \left(\frac{m(Tm)}{m_s}\right) \times 2.0\ g\ sample = 0.4528\ g\ of\ Tm \rightarrow\rightarrow$

✓ # g of Tm_4O_7 : $0.4528\ Tm \times \frac{\text{m (Tm4O7)}}{\text{m (Tm)}} = 0.5278\ gram$

g of Ba : $\frac{2 \text{ mole (Ba)}}{\text{mole (sample)}} \left(\frac{m(Ba)}{m_s}\right) \times 2.0\ g\ sample = 0.7361\ g\ of\ Ba \rightarrow\rightarrow$

✓ # g of $BaCO_3$: $0.7361\ Ba \times \frac{\text{m (BaCO3)}}{\text{m (Ba)}} = 1.0578\ gram$

g of Cu : $\frac{3 \text{ mole (Cu)}}{\text{mole (sample)}} \left(\frac{m(Cu)}{m_s}\right) \times 2.0\ g\ sample = 0.5109\ g\ of\ Cu \rightarrow\rightarrow$

✓ # g of CuO : $0.5109\ Cu \times \frac{\text{m (CuO)}}{\text{m (CuO)}} = 0.6396\ gram$

Check: # gram of Tm + # gram of Ba + # gram of Cu= 2 g

Consider adding 15% to the #g of each compound, and then dry each compound at the specific oven"

❖ $BaCO_3$ in 125^0C , CuO in 450^0C , Tm_4O_7 in 900^0C for 1 day

Then weigh #g of each compound very very exactly.

Mix all the powders in a mortar (when adding powders, add the heaviest one first) and grind for at least 30 minutes in acetone, put the mixture in an Alumina crucible and place it in 900^0C oven for > 1 day.

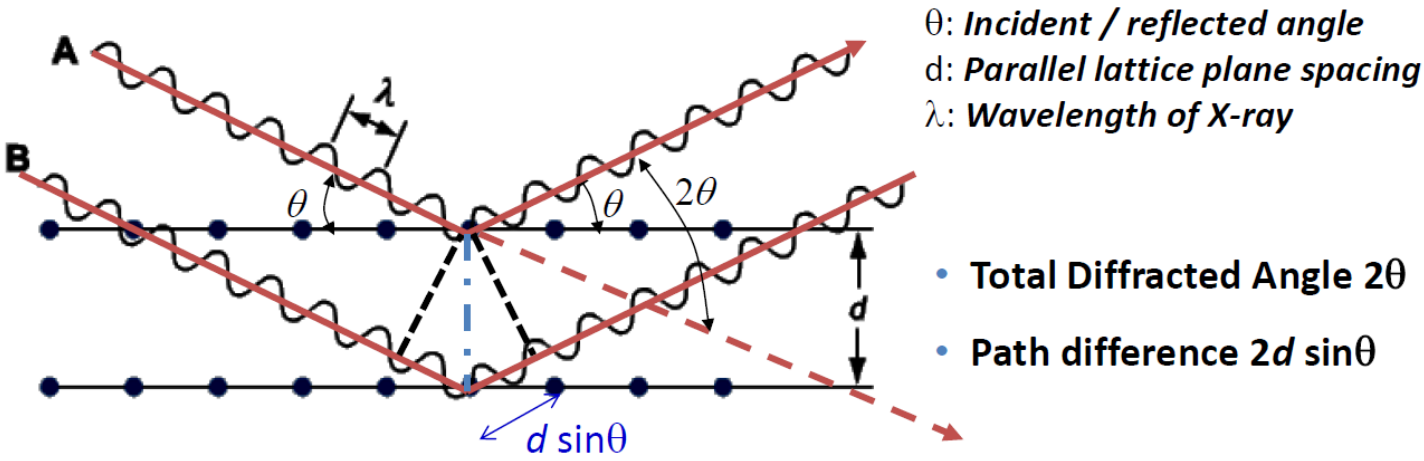
Repeat grinding the mixture 3 times

Finally, press the powers into pellets and anneal the pellets in oxygen (tube oven)----ask for the detail of this step

How to check the sample ? take X-ray diffraction (XRD) in powder form

Study the crystal structure through diffraction of X-ray photons:

Crystals are regular arrays of atoms, and X-rays can be considered waves of electromagnetic radiation. Atoms scatter X-ray waves, primarily through the atoms' electrons. Just as an ocean wave striking a lighthouse produces secondary circular waves emanating from the lighthouse, so an X-ray striking an electron produces secondary spherical waves emanating from the electron. This phenomenon is known as elastic scattering, and the electron (or lighthouse) is known as the scatterer. A regular array of scatterers produces a regular array of spherical waves. Although these waves cancel one another out in most directions through destructive interference, they add constructively in a few specific directions, determined by Bragg's law: 2d sin θ= n λ



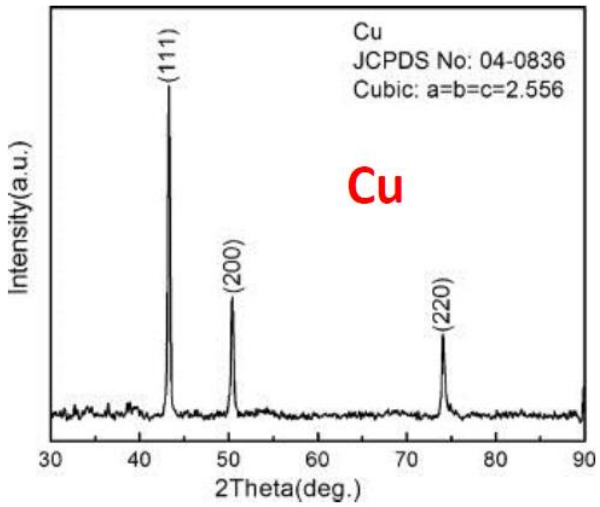
Constructive interference occurs at

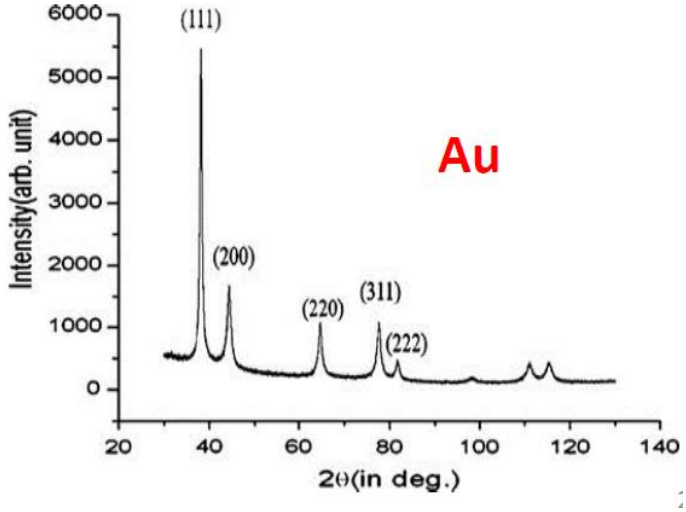
$$2d \sin\theta = n\lambda$$

Bragg's Law

To see a diffraction patter, the size of d would have to be approximately the same as λ of x-rays , which indeed is.

Sample XRD patterns





Crystal Planes

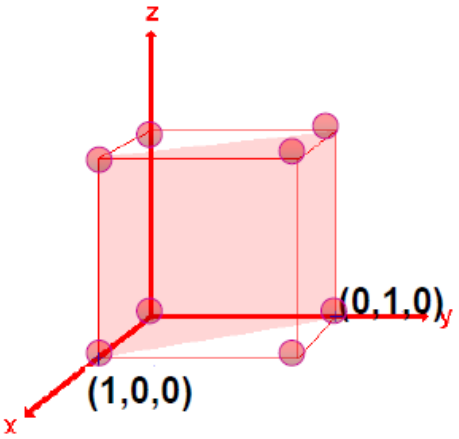
Sets of equally spaced planes within a crystal lattice

1.Express intercepts of the plane with the crystal axes $u\mathbf{a}1, v\mathbf{a}2, w\mathbf{a}3$

2.Take reciprocals of these numbers $1u$,$1v$,$1w$

3.Take the integer $h{:}k{:}l$=$1/u{:}1/v{:}1/w$

4.Use parentheses (hkl) – Miller Indices



Axis	X	Y	Z
Intercept points	1	1	∞
Reciprocals	1/1	1/ 1	1/ ∞
Smallest Ratio	1	1	0
Miller Index (110)			

more examples: