



# General Chemistry Laboratory

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## Synthesis of Superconductor



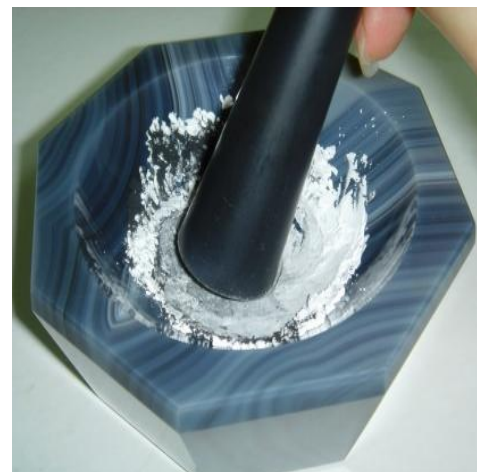
# Preparation

## Collect the following items

- ☐ Agate mortar (clean with sponge after use)
- ☐ Label one zip-lock bag with student ID and name  
(To collect the synthesized superconductor)

## From your personal equipment

- ☐ One plastic spatula
- ☐ Mask (self-prepared)





# Superconductors

- **Metal conductors**

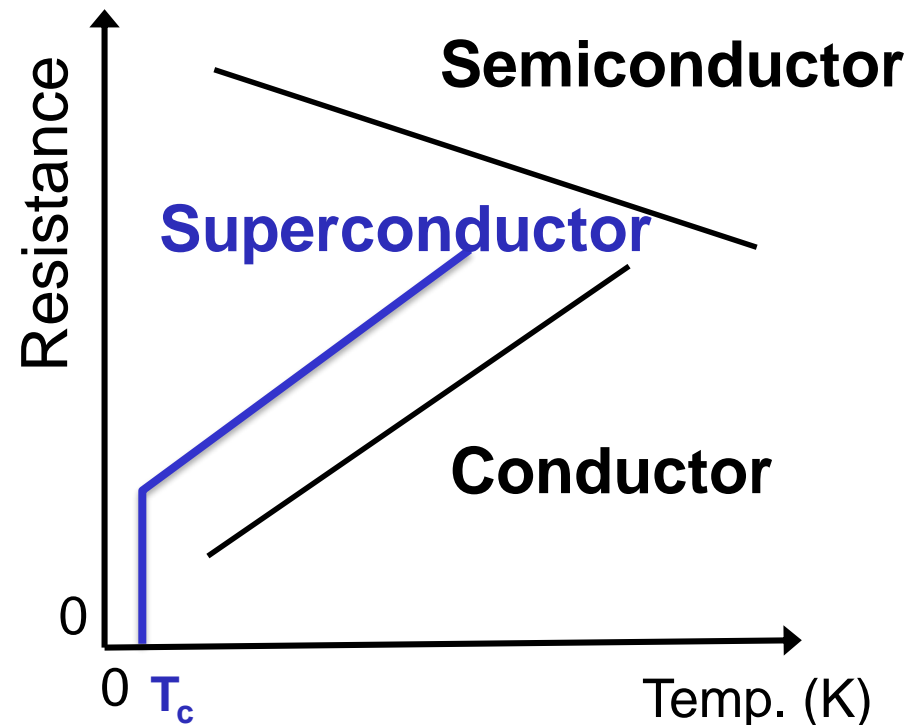
Conductivity decreases with increasing temperature

- **Semiconductors**

Conductivity increases with increasing temperature

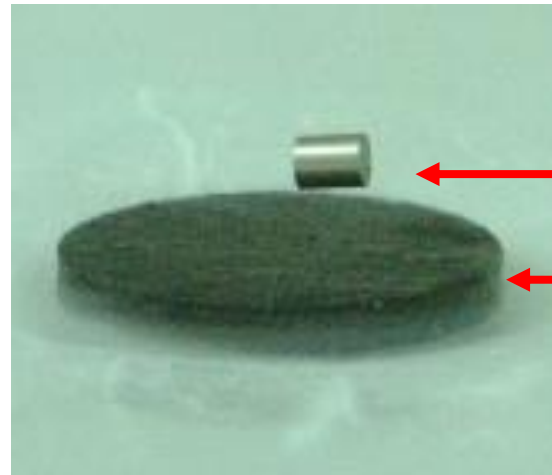
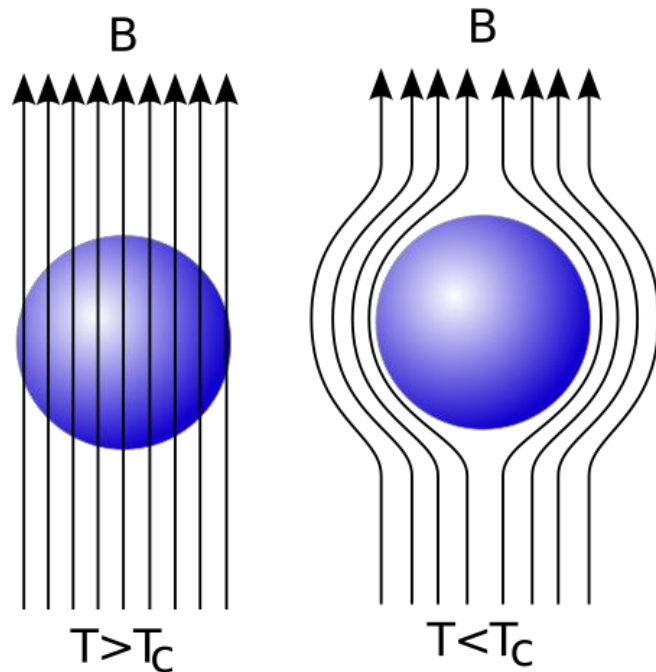
- **Superconductors**

Zero resistance at transition temperature,  $T_c$





# Properties of Superconductor



Magnet

Superconductor

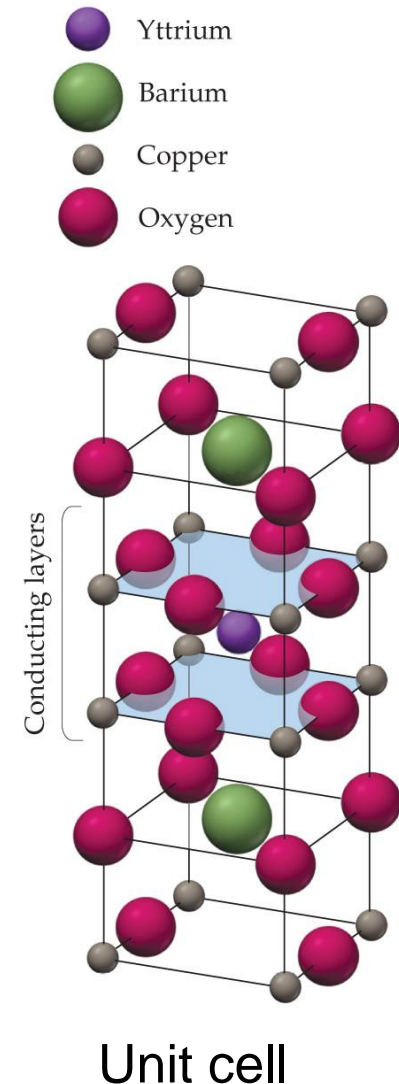
The Meissner effect

- At superconducting transition temperature,  $T_c$ 
  - Zero resistance
  - Meissner effect



# YBCO Superconductor

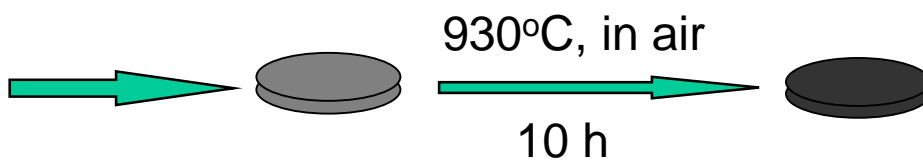
- Yttrium Barium Copper Oxide (YBCO) superconductor
- Chemical formula:  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$
- Crystalline structure
- $T_c$ : **95 K**  
(Boiling point of liquid nitrogen: 77 K)
- Synthetic methods
  - **Solid-state reaction**
  - Sol-gel method
  - Co-precipitation





# Experiment Tasks

Yttrium oxide,  $\text{Y}_2\text{O}_3$   
Barium carbonate,  $\text{BaCO}_3$   
Copper(II) oxide,  $\text{CuO}$



Y : Ba : Cu = 1 : 2 : 3

Grind, press, and sinter

Property test

Synthesize YBCO superconductor by solid-state reaction

1. Weigh starting materials according to atomic ratio
2. Grind the chemicals to homogeneous
3. Press the mixture into pellet
4. Sinter at 930°C for 10 h
5. Test the Meissner effect



# Step 1: Calculation of Formula

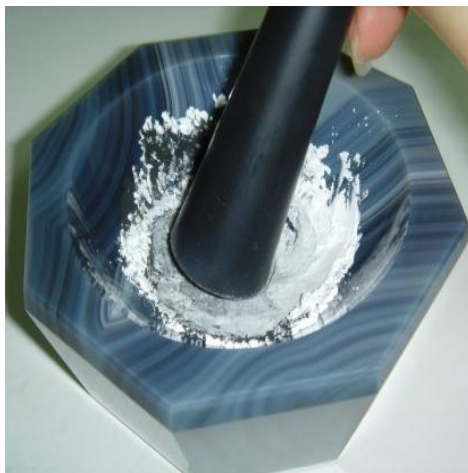
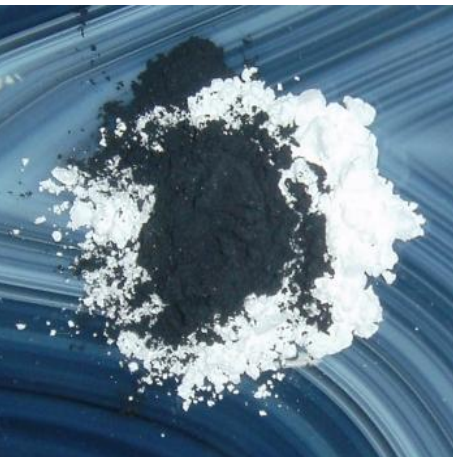
- To synthesize **0.004 mol**  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$   
(Y : Ba : Cu = 1 : 2 : 3 )

- $\text{Y}_2\text{O}_3$        $1/2 \times 0.004 \times 225.82 = 0.4516 \Rightarrow 0.45 \text{ g}$
- $\text{BaCO}_3$        $2 \times 0.004 \times 197.31 = 1.5785 \Rightarrow 1.58 \text{ g}$
- $\text{CuO}$            $3 \times 0.004 \times 79.55 = 0.9546 \Rightarrow 0.95 \text{ g}$

✓ The stoichiometric amount of starting materials is critical to the success of the experiment!



## Step 2: Grind and Mix the Reactants



↑  
Still contains white powder

- Use agate mortar to contain the chemicals
- Mix chemicals with plastic spatula first, then grind
- Grind until color appears gray and homogeneous, that may take about 10~15 min.
- Collect the ground powder in a piece of weighing paper

✓ Agate mortar is expensive (NTD 9,000~15,000) and easily broken, handle with cares





# Step 3.1: Pressing Apparatus



Hydraulic press



Parts of dies



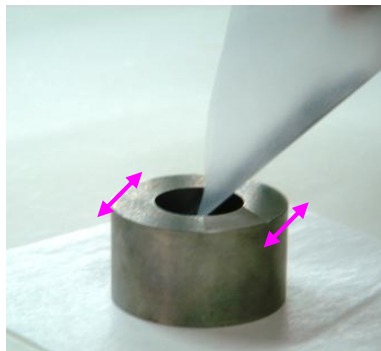
Hollow ring



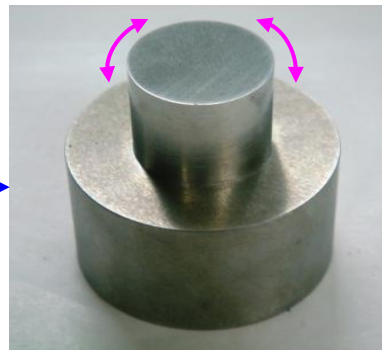
## Step 3.2: Press into Pellet



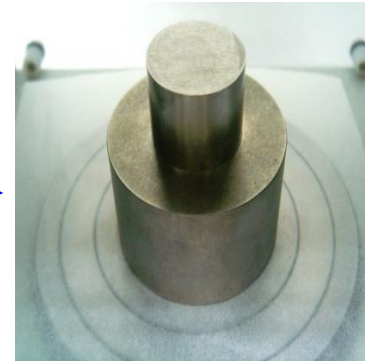
Place die on a weighing paper



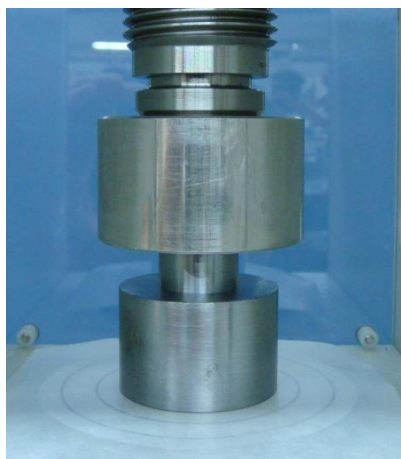
Transfer powder into die evenly



Rotate the die to even up powder



Place the dies on **center of hydraulic platform**



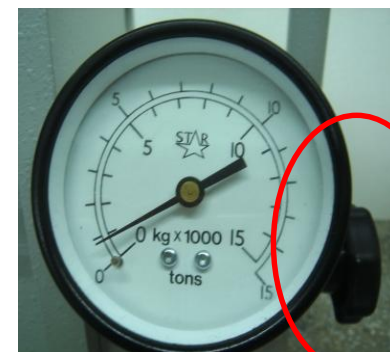
Fix the dies in position



Close the valve clockwise



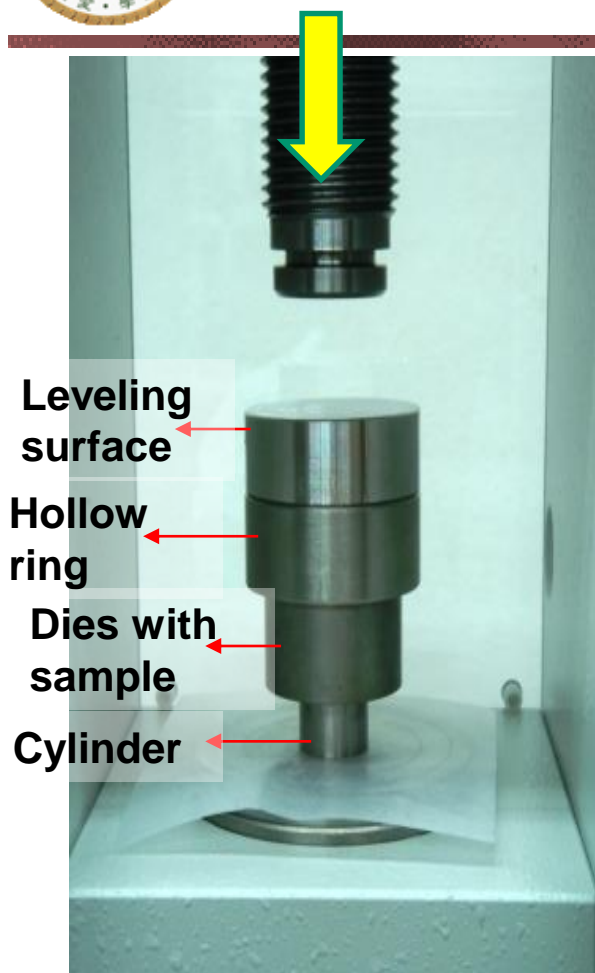
Apply the **pressure to 1 ton/cm<sup>2</sup> for 1 min.**



Release the valve **counter-clockwise**



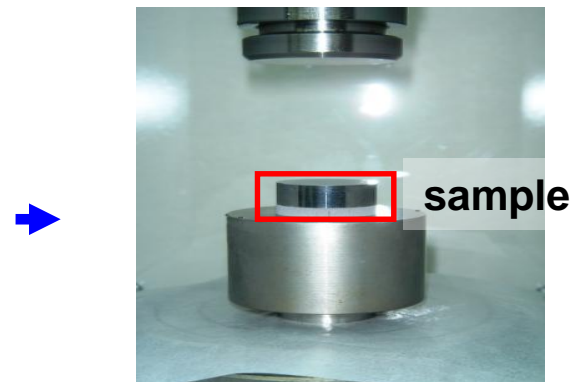
# Step 3.3: Obtain Pellet



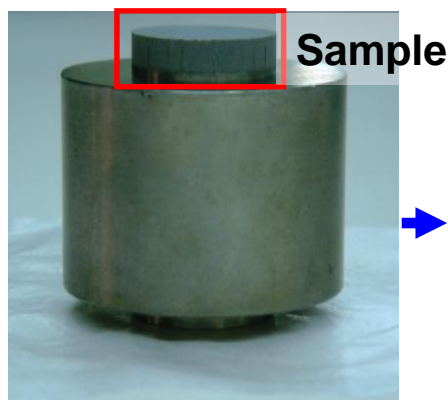
- Place the dies **in center of platform upside down**
- Setup the hollow ring and leveling surface



- Close the valve clockwise
- Pull leverage to surface the superconductor pellet



- Release the valve
- Take out the hollow ring



Resulting sample pellet



Place pellet onto an alumina plate with plastic tweezers carefully





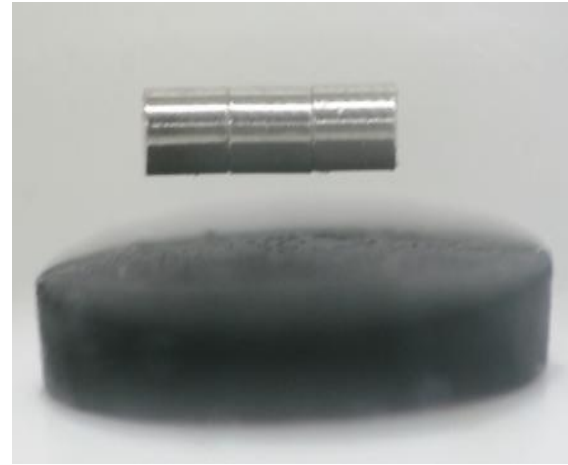
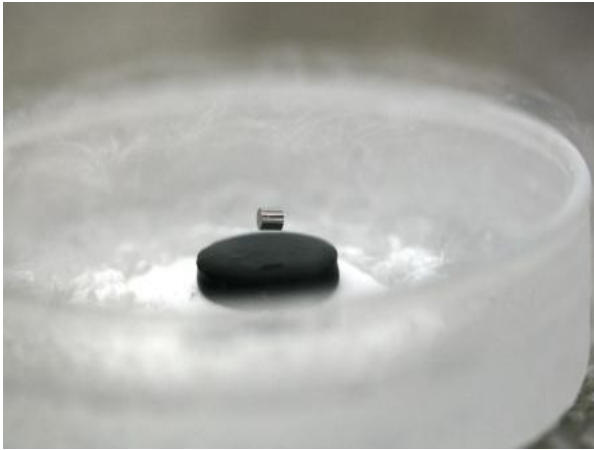
## Step 4: Sinter at 930°C



- Place the sample pellet into box furnace
  - Sinter the samples at 930°C for 10 h. by raising and lowering the temperature at a rate of 5 °C/min.
- ✓ The box furnace can hold  $4 \times 4 = 16$  samples
  - ✓ Alumina plate cannot be labeled, record the position in furnace instead



## Step 5: Property Test



- Examine the Meissner Effect by observing the levitation of magnets
  - Place superconductor in petri dish
  - Place magnetic bars on superconductor pellet with plastic tweezers, compare levitation height and amount of magnetic bars
  - Add liquid nitrogen and immerse the superconductor
  - Dry and warm the superconductor with a hair dryer to room temp.

✓ Notice that the magnetic bars are small and easily lost





# Additional Notes

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- The dies should be placed in center of platform to prevent uneven application of pressure when pressing
- Pressure should be kept at 1 ton/cm<sup>2</sup> for 1 min.
- Clean up the dies thoroughly after each use to avoid the powders pile up in dies that cause it to get stuck
- Avoid direct contact with liquid nitrogen which may cause frostbite



# Clean-Up and Check-Out

- Use sponge to clean agate mortar and pressing dies
- Tuck the lab stools underneath the lab bench
- Clean up the lab bench and check personal equipment inventory (have an associate TA signed the check list)
- This is a **Brief Report** experiment:
  - Hand in prelab/lab note/report together to the TA
- Groups on duty shall stay and help clean up the lab