Growth (Both High and Low Temperature Growth):

Low temperatures yield amorphous cuprite (Cu2O), while High temperatures yield microcrystalline tenorite (CuO)

1. Wafer Preparation
    I. Clean silicon wafer with an acetone and IPA rinse, by squirting directly with solvent bottles or soaking in glassware; remove and dry using nitrogen gun
    II. (Optional) If desire to work with wafers with ~150nm thermal SiO_2, Si oxidation is carried out in the Thermco 4
        A. Enable Thermco 4 furnace
        B. Load wet oxidation recipe
        C. Set time for 30 min, temperature of 950°C
        D. Load clean wafers and wait for processing
        E. Remove wafers, disable tool
2. Metal stack deposition
    I. Enable the Lesker sputter
    II. Deposit adhesion layer
        A. Either: ~5nm Ti (200W DC, 5mTorr, 60sec)
        B. Or: ~5nm Ta (200W DC, 3mTorr, 42sec)
        C. (Note: these values and recipes were adopted from entries in the log book, namely entry by ‘bfrend’ on 3/30/21 for Ti, and entry by ‘Nikita’ on 10/22/20 for Ta)
    III. Deposit Copper (either ~200nm or ~700nm)
        A. If single sputter gun: 200W DC, 3mTorr, 4nm/sec deposition rate
        B. If co-sputter: 200W DC, 3mTorr, 8nm/sec deposition rate (doubled)
        C. (Note: these values and recipes were adopted from logbook entries, namely entry by ‘ajs021’ on 02/23/21)
3. Thermal Oxidation
    I. Cleave 1 in by 1 in chips from deposited wafers to serve as samples (a single wafer can be used for multiple oxidation attempts)
    II. Load samples into furnace (either the Thermolyne or Tylan9)
    III. Oxidation Anneal
        A. Thermolyne
            i. Place samples in chamber
            ii. Turn on furnace using power switch and arrow keys to select temperature (300°C or 800°C)
            iii. Start separate timer for 3hr
            iv. At the end of the anneal, turn off the furnace, prop open the chamber, and let cool for around 1hr
            v. Remove Samples
        B. Tylan9
            i. Enable furnace
            ii. Load recipe (AIR300 or AIR800)
            iii. Enter time for 3hr

iv. Load samples, wait for processing
v. After samples are automatically unloaded, cool for 10 min, remove

Sample Storage

1. Use a 4” wafer container with spring and place sputtered and oxidized samples inside.
2. If desired, place in a vacuum glovebox in SNF or private facilities
    I. Isolate the loading chuck from the glovebox and vent
    II. Place sample inside 4” wafer container inside loading chuck
    III. Load the chuck and isolate from outside and inside glovebox
    IV. Purge with N_2 and pump to vacuum at least 3 times
    V. After final purge, put hands into glovebox
    VI. Fill loading chuck with gas and open loading chuck from inside the glovebox
    VII. Take samples out of loading chuck and place somewhere in the glovebox
    VIII. Close loading chuck, then purge and pump again at least 3 times
    IX. Isolate the loading chuck from glovebox and outside

Characterizations

1. SEM
    I. Cleave samples into quarters to expose film cross section
    II. Mount sample using black sticky tape on 45 degree angled holder, with angled side facing towards the edge of the shuttle for ease of imaging orientation, and face the newly cleaved edge up for imaging
    III. Enable Tool
    IV. Load sample into the SEM, using the quickloader if samples sufficiently small
    V. Image both the sample surface as well as the cleaved cross section, especially checking for evidence of complete oxidation and microcrystallinity
    VI. When finished, unload sample and disable tool

2. XPS
    I. Enable tool and mount/ load samples
    II. After tool initialization steps, take initial survey with 224eV pass energy and 2-3 cycles to identify main elemental signatures
    III. Select high resolution regions, including Cu2p, Cu LMM, O1s, and, depending on the survey information, regions for Ti, Si, Ta (usually surface contaminants)
    IV. Adjust pass energy to 55eV for 2-3 cycles (cycle count can be adjusted depending on signal strength for the day and for the region)
    V. Use multipak software to adjust for any drift due to charging, account for background, fit peaks, and determine chemical phase ratios
    VI. (Optional) If sputtering, use the 5kV 1x1 sputter gun file and sputter for 5 min to remove surface layers and probe the bulk
    VII. (Optional) If conducting a depth profile, use 5kv 1x1 sputter gun file and adjust sputter table to take measurements between every 1-3 minutes of sputtering
    VIII. Close down tool and unload samples
    IX. Disable tool

3. XRD
    I. Enable the tool, open the data collector software and perform x-ray alignment without the sample mounted
    II. Once the x-ray is aligned, mount the sample, with the long side of the sample chip parallel to the beam direction
    III. Find the z-location of the sample either manually or by script. For 700nm Cu depositions, this is usually around z=9.0mm The location is determined by sharpest decrease in x-ray signal as a function of the stage's z location
    IV. For optimal signal, mount a ½" slit on the incident beam lens and remove the PPC collimator slit from the collection optics
    V. Begin a grazing incidence measurement with GIX_01 script (located in Users/Yakub in the XPert2 user computer). Depending on the longitudinal length of the sample, an omega of about 5^{o} to 10^{o} will yield optimal signal counts
    VI. After data is acquired, open the XRD spectrum in data viewer and convert the files to proper format (.csv) for processing
    VII. Close x-ray shutter, unload sample and load a new sample if continuing measurements
        1. If continuing measurements, start from step 3, but use the 1/32" slit on incident beam and PPC collimating slit on the collection optics
        2. If finished with all samples, set the stage positions back to default values and place the 1/32" slit on incident beam and PPC collimating slit on the collection optics
    VIII. Disable tool

4. Optical Characterization
    I. Mount sample on stage
    II. Turn on the 532nm laser and focus onto the sample surface. This is indicated by brightest reflection of the laser onto the narrowest spot
    III. Connect collection fiber or optics to spectrometer receiving port
        1. Use a 550nm longpass filter on collection path
    IV. Set grating of spectrometer to 150 g/mm for broad scans and 1714 g/mm for narrow scans
    V. Set the central wavelength for the spectrum
        1. For room temperature experiments, center the spectrometer grating at 620 nm for optimal signal in region of interest
        2. For cryogenic temperature experiments, set the center wavelength at 600 nm for orthoexciton emission and at 570 nm for Rydberg series emission
    VI. Collect emission for 10s, 30s, or for 60s
    VII. Save and export data
    VIII. Turn off laser
    IX. Remove sample from sample stage