

This instruction covers the capabilities, operation, and compatibilities of the Nitric Oxide anneal furnace system. This furnace is located in the P bay of the cleanroom and is only to be operated by trained and authorized users.

1 SAFETY REQUIREMENTS

1.1 General Safety Information

- Safety glasses must be worn whenever in the cleanroom, except when using a microscope or when wearing protective goggles.
- Be mindful of cross-contamination of controls, equipment, and tools.
- Furnace surfaces can be hot. Do not load/unload above 140°C and use heat resistant gloves if necessary.
- Nitric Oxide gas is particularly dangerous and this system is monitored by a local alarm instead of the main toxic gas monitoring system. If the alarm sounds, evacuate the area and hit the TGMS alarm button in the hallway. See hazard ID in figure 1 and refer to the MSDS for this material before operating the furnace.

2 PROCESS MATERIALS

2.1 Equipment

Wafer handling tweezers

2.2 Gasses

Ar Argon

NO Nitric Oxide (TOXIC, CORROSIVE, OXIDIZER)



GHS03



GHS04



GHS05



GHS06

Danger

H270 - MAY CAUSE OR INTENSIFY FIRE; OXIDIZER

H280 - CONTAINS GAS UNDER PRESSURE; MAY EXPLODE IF HEATED

H314 - CAUSES SEVERE SKIN BURNS AND EYE DAMAGE

H330 - FATAL IF INHALED

CGA-HG22 - CORROSIVE TO THE RESPIRATORY TRACT

CGA-HG11 - SYMPTOMS MAY BE DELAYED

P202 - Do not handle until all safety precautions have been read and understood.

P220 - Keep/Store away from clothing, combustible materials

Figure 1 - Nitric Oxide Gas Hazard Identification

3 System Overview

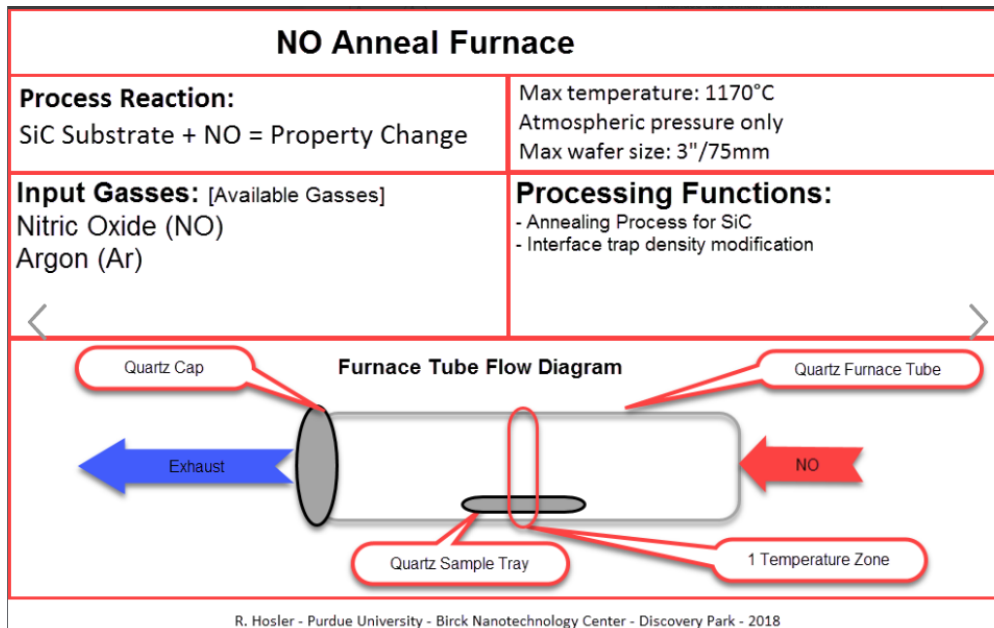


Figure 2 – NO Anneal Capabilities

3.1 System Capabilities

Current process capabilities include NO annealing over the range from room temperature to 1175°C. Manually controlled regulators and a three way valve dispense either Argon, Nitric Oxide, or no gas. Each user group maintains their own furnace tube to prevent cross contamination. To switch a tube, contact the research engineer in charge of the tool.

4 Operation

4.1 Logging In

Enable the tool in iLabs by creating a reservation and activating it. Ensure that the reservation covers all preparation and cooldown time for the furnace during which any gas is flowing or any setpoint above room temperature is programmed.

4.2 Sample Preparation

Samples must be able to fit on the sample tray installed in the tube. Samples and wafers must be cleaned according to the procedures outlined by your group for your tube. Do not use another group's tube. TAI (Toluene, Acetone, IPA) process followed by either Piranha or RCA cleaning as appropriate for sample compatibility are recommended. See the [wiki - Cleaning](#) for more information on sample cleaning and preparation to avoid tool contamination.

4.3 Loading Procedure

Visually inspect the tube before opening. **Is there apparent brown gas or any other colored gas present through the tube cap? If so, do not proceed and immediately turn off the NO regulator valve. Then promptly notify a staff member.**

Ensure that the setpoint and actual temperature on the furnace is below 140°C. Heat-resistant gloves are available if needed and are recommended. Verify that the tube installed is the proper one for your group. This information is posted on the front of the furnace hood.

Typically groups first purge the tube with argon for at least 20 minutes to displace any atmosphere in the tube. Fully close the argon regulator by turning it clockwise until it stops. Then move the 3 way valve to the Argon position. Slowly open the regulator valve and establish 5 SCFH of flow.

If the regulator is turned on too quickly or valved while under a high setting it can push the front tube cap off.

After the tube is purged, open the tube by first disconnecting the tube clamp which evacuates the vessel from the front. Separate the ball joint and set the clamp aside. While bracing the portion of the tube that protrudes from the furnace, slowly remove the tube cap. This may prove difficult but very little force should be used otherwise the cap can break. If the cap does not move then slightly tap forwards long the edges to try and loosen the seal. Once the tube cap is removed, set it aside on a clean wipe.

Remove the pull rod from the right-hand container marked NO ONLY. Be very careful not to move up or down while removing the rod, it is very fragile. Only touch as far forward as you have to in order to prevent contamination. Use the hooked end to grab the ring on the end of the sample tray and pull it forward to a position where you can load your sample.

Place your samples on the tray with clean tweezers and use the rod to push the tray to the approximate middle of the furnace. Carefully place the pull rod back into the proper container. Place the cap back on the furnace tube gently, no significant force should be required. Re-connect the ball joint for the exhaust tube and place the clamp back over the joint.

4.4 Process Execution

Once the sample is loaded, adjust the temperature on the single-zone furnace panel using the up and down arrows. Once the target setpoint has been selected, wait a moment and the furnace will begin to heat up. This is indicated by the OP1 LED which shows that power is being applied to the furnace coil.

Once your desired setpoint is reached and the sample has had time to stabilize at this temperature, set the Argon regulator to zero and ensure that the NO regulator valve is also fully turned clockwise. Switch the 3-position valve over to NO and slowly adjust the regulator to the desired flow rate. This is typically 0.3 SLPM on the indicator but may vary for your group. Verify that brown gas is filling the tube and the temperature remains steady. When your annealing time is completed, turn off the NO gas regulator and switch the position over to Argon. Slowly adjust to 5 SCFH to purge the tube for a minimum of 20 minutes. Adjust the furnace temperature back to 0° C. Wait until the temperature is below 140° C and verify that brown gas is not present in the tube before opening and removing your sample.

Remain within visual and audio range of the tube during the ENTIRE time that NO gas is being dispensed.

If at any time you see brown gas escaping from the tube, hear the local gas alarm, or smell anything abnormal, immediately close the regulator and switch it to OFF if this is practical to do without endangering yourself.

If the gas is escaping the hood, immediately evacuate the area and hit the TGMS alarm in the hall.

It is suggested to perform this procedure with another trained NO anneal tube user who can provide coverage if you need to take a break. If you cannot remain at the tube due to any circumstance, turn off the NO gas regulator and move the position to OFF if you plan to return shortly. If not, set the tube to argon and allow the tube to purge the NO gas before leaving.

The importance of this rule is that the local detection system is not tied into the TGMS system, so in the event of an alarm a user must be present to evaluate the situation and possibly hit the TGMS alarm to evacuate the building.

**This gas can be FATAL.
Treat it and your labmates with respect by safely operating this furnace.**

5 References

5.1 [Birck Nanotech Wiki – Cleaning Page](#)

5.2 [MSDS for Nitric Oxide](#)

6 REVISION RECORD

Reason for Revision	Date of Revision	Person Responsible
Initial Release	10/26/2022	Rich Hosler