

ProTemp Furnace SOP

1 Scope

1.1 This SOP provides instructions to operate the ProTemp Atmospheric Furnaces.

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3 Reference Documents

3.1 External Documents

- 3.1.1 SMBB Lab User Guide

4 Equipment and/or Materials

- 4.1 ProTemp Oxidation Furnaces
- 4.2 Quartzware
- 4.3 Boat fork
- 4.4 Monitor wafers
 - 4.4.1 Phosphorous Doping: <100> P-Type bare silicon
 - 4.4.2 All Other Processes: <100> N-Type or P-Type bare silicon
- 4.5 Filler wafers

5 Safety

- 5.1 Follow all Nanofab safety procedures.
- 5.2 Ensure all Cooldown cycles are complete before handling any quartzware, wafers, or samples.



CAUTION



All recipes include the necessary cool down time to ensure the quartzware, wafers, and samples are cool enough to be touched and handled.

Do not touch or handle quartzware, wafers, or samples unless the current run is COMPLETE.



CAUTION



6 General Information

- 6.1 Ensure all samples are authorized to be loaded into a furnace.




!!! WARNING !!!




No photoresist, kapton tape, metals, glass, or other foreign material is allowed in any furnace.

Only quartz, silicon, silicon carbide, silicon dioxide, silicon nitride, and/or Polysilicon are allowed without PRIOR approval of lab staff.



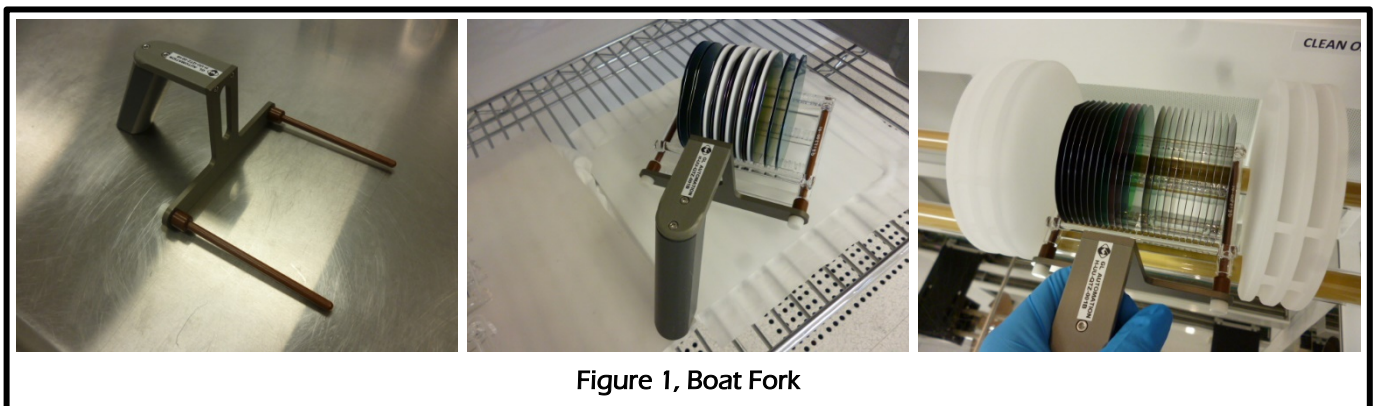



!!! WARNING !!!






- 6.1.1 Only the following substrates or samples are allowed in the **Clean Ox** furnace:
- Bare silicon wafers that have received NO prior processing
 - Wafers with an oxide that was thermally grown in the Clean Ox furnace
 - Wafers with LPCVD Undoped Polysilicon deposition
 - Wafers with LPCVD Nitride deposition
- 6.1.2 The following substrates or samples are allowed in the **Doped Ox** furnace:
- All samples approved for processing in the Clean Ox furnace
 - Wafers that have been processed through PECVD Oxide deposition
 - Wafers that have been processed through PECVD Polysilicon deposition
 - Wafers that have been processed through PECVD Nitride deposition
 - Wafers that have been processed through LPCVD Doped Poly Deposition
 - Wafers that have been processed through LPCVD LTO Deposition
 - Wafers that have been processed through LPCVD PSG Deposition
 - Wafers that have been doped by any method
- 6.1.3 **For any substrates or samples not listed above**, contact Staff to determine the authorized furnace.
- 6.2 Ensure all samples are clean, dry, and particle-free.
- 6.3 Place quartzware on a quartz plate, silicon carbide cantilever paddle, or a cleanroom wiper.
- 6.3.1 Do NOT directly place quartzware on stainless steel.
- 6.4 Do NOT touch boats or boat covers with your hands.
- 6.4.1 Use the boat fork (see **Figure 1, Boat Fork**) to move boats and boat covers.
- 6.4.1.1 Insert the tines of the fork into the pick-up tubing on the boat or boat cover.



- 6.5 Before touching a quartz baffle, put on a second pair of clean blue nitrile gloves.
- 6.6 Solid Source wafers that have been out of the furnace more than one (1) hour need a dehydration bake per section 9 Solid Source Instructions.

7 Pre-Clean

- 7.1 **For new, unused bare silicon wafers**, a Pre-Diffusion Clean prior to processing through any furnace cycle is recommended.
- 7.2 **For all other samples/substrates/wafers**, a Pre-Diffusion Clean prior to processing through any furnace cycle is **REQUIRED**.
- 7.3 Complete the Pre-Diffusion Clean in the Pre-Diffusion wet bench.
NOTE: The dedicated baths should be used for whole wafers, since the SC-1 bath includes a megasonic clean for improved particle removal. However, for partial wafers the specified mixtures may be used in smaller beakers/containers, if necessary.
- 7.4 Refer to **TABLE 1, FURNACE PRE-CLEAN** for the correct mixtures, temperatures, and times.
- 7.5 If the Organic Clean bath temperature is above 100°C and has not been used within the last 2 hours, add 100ml of Hydrogen Peroxide.
 - 7.5.1 Wait at least 5 minutes before using the bath.

7.6 Organic Clean Bath Preparation

- 7.6.1 **If using an alternate bath container**, ensure the container is compatible with temperatures up to 200°C (e.g., pyrex, quartz, teflon).
- 7.6.2 Add the necessary amount of Sulfuric Acid to an empty container.
- 7.6.3 Turn on the heater.
- 7.6.4 Wait for the heater to exceed 100°C.
- 7.6.5 Add the necessary amount of Hydrogen Peroxide.
- 7.6.6 Wait until the bath temperature has reached setpoint.
- 7.6.7 Wait at least 5 minutes before using the bath.

7.7 SC-1 Bath Preparation

- 7.7.1 **If using an alternate bath container**, ensure the container is compatible with temperatures up to 100°C (e.g., pyrex, quartz, teflon).
- 7.7.2 Add the necessary amount of Ultra Pure Water (UPW) to an empty container.
- 7.7.3 Add the necessary amount of Ammonium Hydroxide.
- 7.7.4 Turn on the heater.
- 7.7.5 Wait for the heater to exceed 65°C.
- 7.7.6 Add the necessary amount of Hydrogen Peroxide.
- 7.7.7 Wait until the bath temperature has reached setpoint.
- 7.7.8 Wait at least 5 minutes before using the bath.

7.8 Oxide Removal Bath Preparation

- 7.8.1 **If using an alternate bath container**, ensure the container is compatible with Hydrofluoric Acid (e.g., plastic, teflon).
- 7.8.2 Add the necessary amount of Ultra Pure Water (UPW) to an empty container.
- 7.8.3 Add the necessary amount of Hydrofluoric Acid.
- 7.8.4 Wait at least 5 minutes before using the bath.

7.9 SC-2 Bath Preparation

- 7.9.1 **If using an alternate bath container**, ensure the container is compatible with temperatures up to 100°C (e.g., pyrex, quartz, teflon).
- 7.9.2 Add the necessary amount of Ultra Pure Water (UPW) to an empty container.
- 7.9.3 Add the necessary amount of Hydrochloric Acid.
- 7.9.4 Turn on the heater.
- 7.9.5 Wait for the heater to exceed 65°C.
- 7.9.6 Add the necessary amount of Hydrogen Peroxide.
- 7.9.7 Wait until the bath temperature has reached setpoint.
- 7.9.8 Wait at least 5 minutes before using the bath.

Step	Description	Container Type	Ratio ¹	Chemical	Quantity (ml)	Temp (°C)	Time (mm:ss)
1	Organic clean (pirahna)	Pyrex Quartz Teflon	5	Sulfuric Acid	2500	120	10:00
			1	Hydrogen Peroxide, 30%	500		
2	Rinse			Ultra Pure Water (UPW)			5:00
3	SC-1	Pyrex Quartz Teflon	5	Ultra Pure Water (UPW)	6250	75	10:00
			1	Ammonium Hydroxide	1250		
			0.4	Hydrogen Peroxide	500		
4	Rinse			Ultra Pure Water (UPW)			5:00
5	Oxide Removal	Plastic Teflon	50	Ultra Pure Water (UPW)	2000		01:00
			1	Hydrofluoric Acid, 49%	40		
6	Rinse			Ultra Pure Water (UPW)			5:00
7	SC-2	Pyrex Quartz Teflon	6	Ultra Pure Water (UPW)	3900	75	10:00
			1	Hydrochloric Acid, 37%	650		
			1	Hydrogen Peroxide	650		
8	Rinse			Ultra Pure Water (UPW)			5:00
9	Spin-Rinse Dry			As programmed			

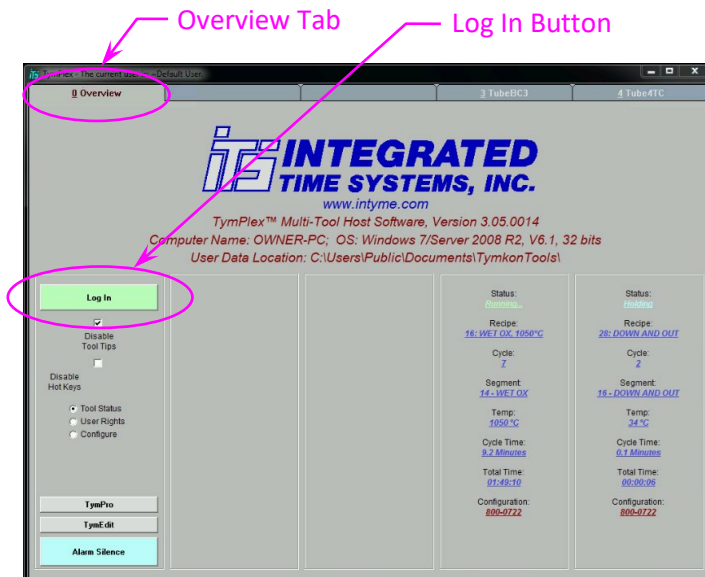
¹Maintain this ratio when using the alternate bath containers.

²If the Organic clean bath temperature is greater than 100°C and has been unused for more than 2 hours, add 100ml of Hydrogen Peroxide prior to use.

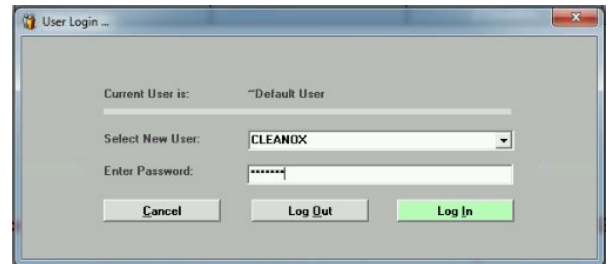
8 Furnace Operating Procedures

8.1 Log On

- 8.1.1 From the ProTemp host computer, select the Tymplex program.
- 8.1.1.1 **If necessary**, open the Tymplex program.
- 8.1.2 From the **Q Overview** screen, click the **Log In** button.
- 8.1.3 Type the Username and password (in CAPS) for the applicable furnace. (See **TABLE 2, LOG IN INFORMATION.**)
- 8.1.4 From the **User Login ...** dialog box, click the **Log In** button.



Furnace	Clean Ox	Doped Ox
Username	CLEANOX	DOPEDOX
Password	CLEANOX	DOPEDOX



8.2 Enable Coral

- 8.2.1 Enable the furnace in Coral.

8.3 Check Furnace Status

- 8.3.1 Select the main tab for the desired furnace.
- 8.3.2 Select the Operate subtab.

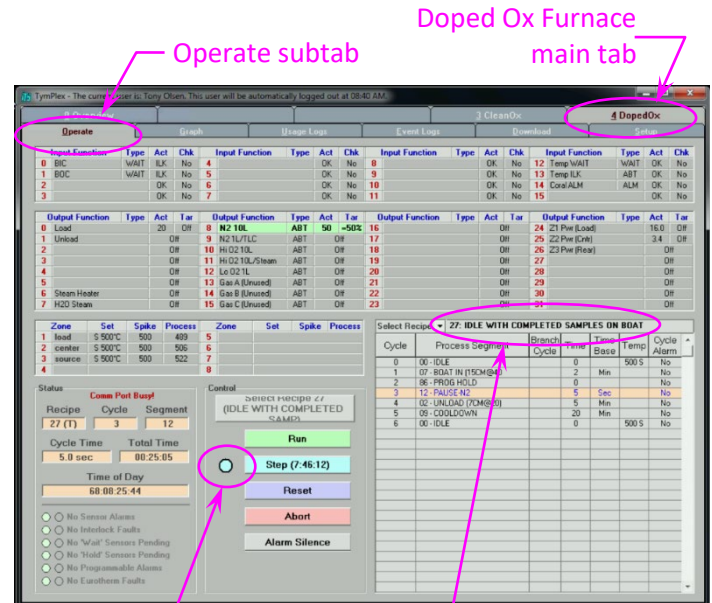
8.3.3 Review Current Status

NOTE: If the current recipe is "IDLE WITH COMPLETED SAMPLES ON BOAT", samples from the previous run have not been removed from the boat.

- 8.3.3.1 **If the current recipe is "IDLE WITH COMPLETED SAMPLES ON BOAT"**, do the following:
- 8.3.3.1.1 **If there are no instructions from the previous user**, contact that user before removing any samples and monitor wafers.
- 8.3.3.1.2 **If the instructions from the previous user are "Do Not Disturb"**, do not proceed.

8.3.3.1.3

If the instructions from the previous user authorize you to handle their samples, continue.



Blue dot

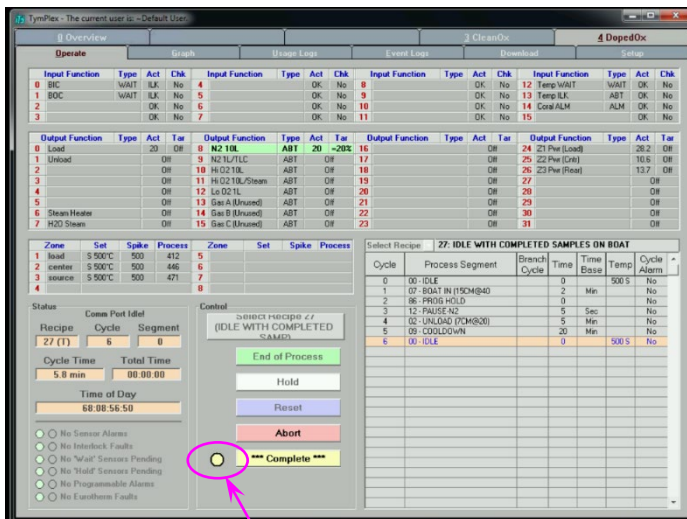
8.3.3.2 If there is a blue dot next to the **Step** button, the furnace is on Hold.

8.3.3.2.1 Click the **Run** button.

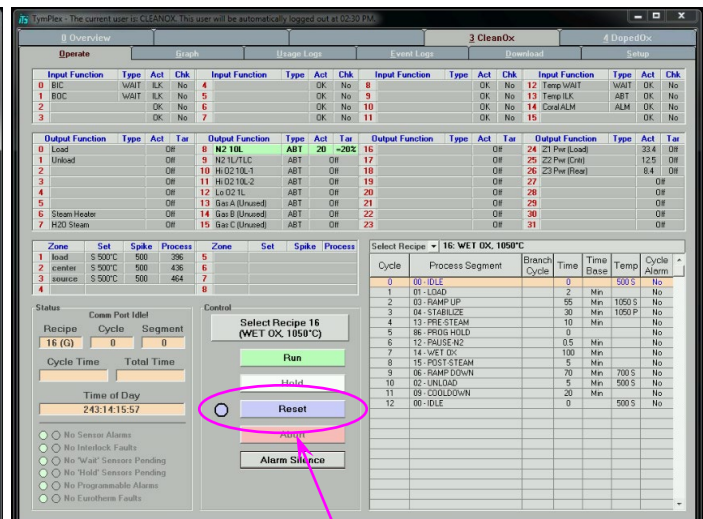
8.3.3.2.2 Wait for the current run to complete.

NOTE: The **Alarm Silence** button will be replaced by a yellow ***** Complete ***** button with a yellow dot.

8.3.3.3 If there is a yellow dot next to the ***** Complete ***** button, the current recipe has completed.



Yellow Dot



Reset button

- 8.3.3.3.1 Click the ***** Complete ***** button.
- 8.3.3.3.2 Click the **Reset** button.
- 8.3.3.4 **If the cantilever is in and there is a blue dot next to the **Reset** button,** download and run the "BOAT OUT" recipe per paragraphs 8.7 Download Recipe File through 8.9 Run Recipe.
- 8.3.3.5 **If the cantilever is in and there is a yellow dot next to the ***** Complete ***** button,** click the ***** Complete ***** button.
- 8.3.3.5.1 Download and run the "BOAT OUT" recipe per paragraphs 8.7 Download Recipe File through 8.9 Run Recipe.
- 8.3.3.6 **If the cantilever is out,** the furnace is ready for use.
- 8.3.3.7 **If samples from another member are loaded on the boat AND he/she has authorized you to handle their samples,** follow their instructions to remove the samples.

8.4 Load Boat

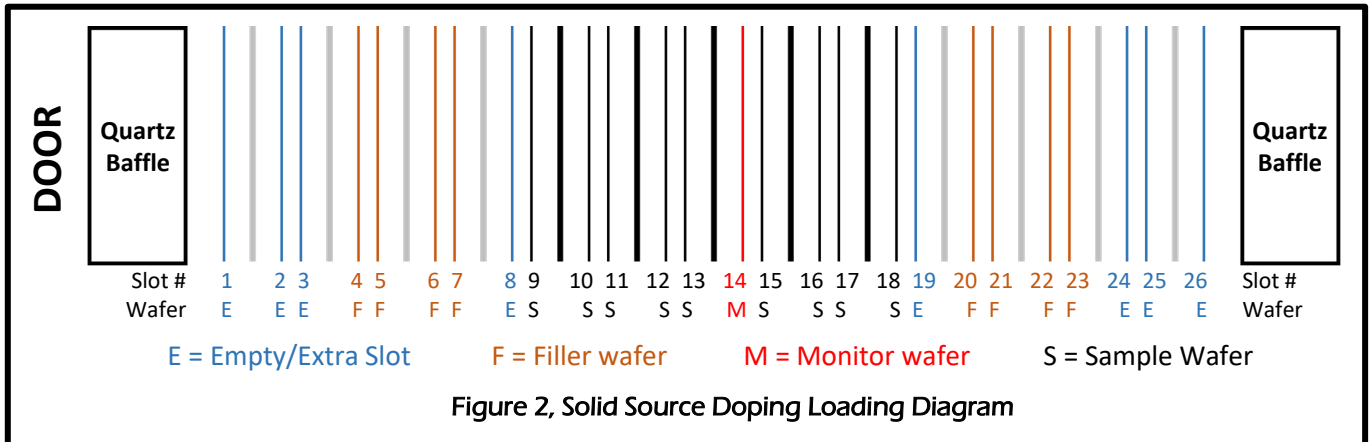
8.4.1 Load Wafers

	!!! WARNING !!!	
	No photoresist, kapton tape, metals, glass, or other foreign material is allowed in the furnace.	
	Only quartz, silicon, silicon carbide, silicon dioxide, silicon nitride, and/or Polysilicon are allowed without <u>PRIOR</u> approval of lab staff.	
		
	!!! WARNING !!!	

- 8.4.1.1 **Unless otherwise noted,** load all wafers with the polished side toward the door of the furnace.
- 8.4.1.2 Load the wafers with the wafer flat up.
- 8.4.1.3 Load one wafer per slot.
- 8.4.1.4 Solid Source Doping**
- NOTE: Solid Source wafers that have been out of the furnace more than one (1) hour need a dehydration bake per section 9 Solid Source Instructions.*
- NOTE: The solid source wafers should always have a filler or sample wafer in the adjacent slots – even when stored.*
- 8.4.1.4.1 Remove the necessary filler wafers (adjacent to the solid source wafers) and place them in empty slots at either end of the boat.
- 8.4.1.4.2 Load all sample and monitor wafers with the polished side (side to be doped) toward the solid source wafer.
- 8.4.1.4.3 Load a monitor wafer onto the boat in slot 14 (see **Figure 2, Solid Source Doping Loading Diagram**).

NOTE: Monitor wafers for Phosphorous Doping are <100> P-Type bare silicon. Monitor wafers for Boron Doping are <100> N-Type bare silicon.

- 8.4.1.4.4 Load sample wafers in slots 9-13 and/or 15-18, starting at the center.
- 8.4.1.4.5 Load filler wafers in slots 4-7, 20-23, and all unused sample slots.

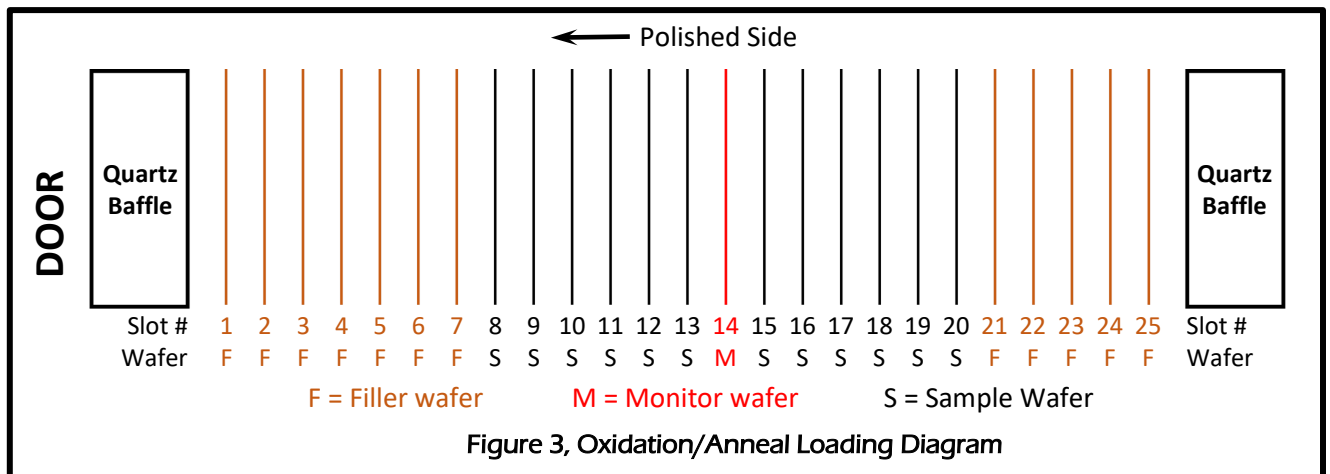


8.4.1.5 Oxidation/Anneal

- 8.4.1.5.1 Load a monitor wafer onto the boat in slot 14 (see **Figure 3, Oxidation/Anneal Loading Diagram**).

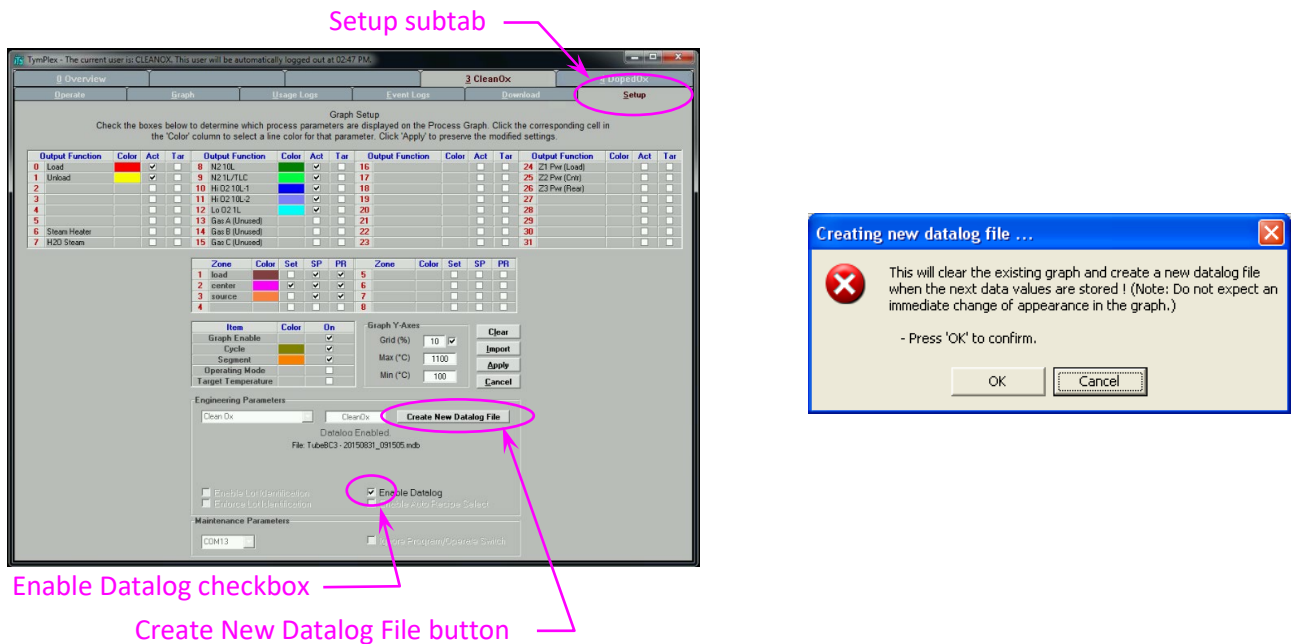
NOTE: Monitor wafers are bare silicon. Samples that are bare silicon may be used as monitors.

- 8.4.1.5.2 Load sample wafers in slots 8-13 and/or 15-20, starting at the center.
- 8.4.1.5.3 Load filler wafers in slots 1-7, 21-25, and all unused slots.
- 8.4.1.5.4 Ensure there is a sample, monitor, or filler wafer in every slot.



8.5 Initialize Graph

- 8.5.1 Select the **Setup** subtab.
- 8.5.2 Ensure the **Enable Datalog** check box is checked.
- 8.5.3 Press the **Create New Datalog** File button.
- 8.5.4 At the **Creating new datalog file ...** box, click **OK**.



The screenshot shows the 'Setup' subtab in the ProTemp software. The 'Enable Datalog' checkbox is checked, and the 'Create New Datalog File' button is highlighted. A dialog box titled 'Creating new datalog file ...' is open, displaying a warning message: 'This will clear the existing graph and create a new datalog file when the next data values are stored ! (Note: Do not expect an immediate change of appearance in the graph.) - Press 'OK' to confirm.' The dialog box has 'OK' and 'Cancel' buttons.

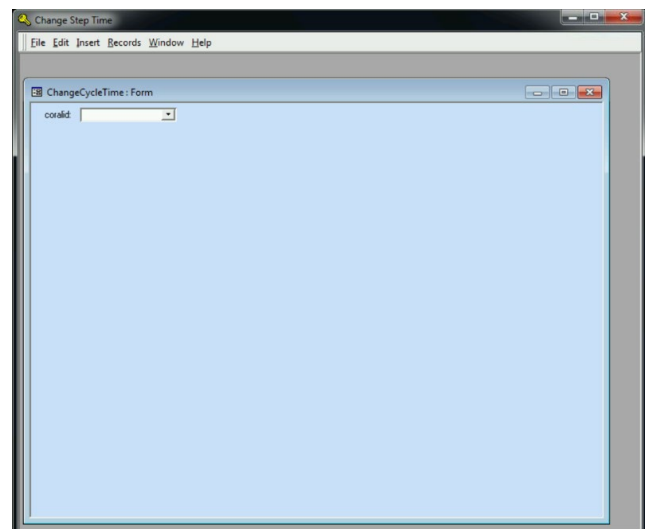
Setup subtab

Enable Datalog checkbox

Create New Datalog File button

8.6 Change Oxidation Time

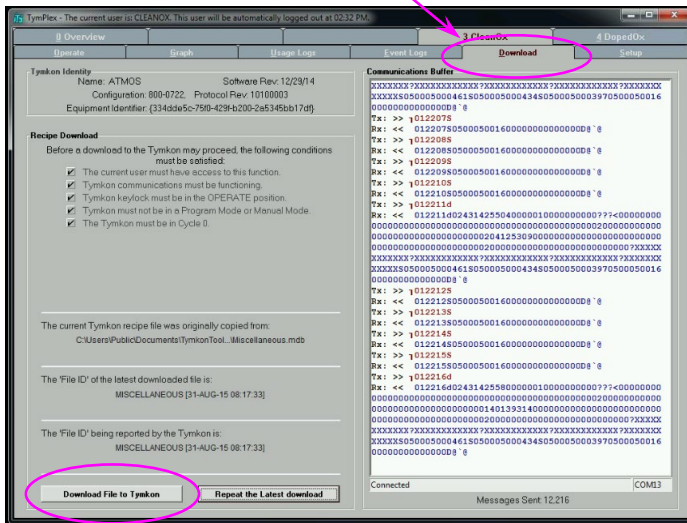
- 8.6.1 From the desktop, open the "Change OxTime" utility.
- 8.6.2 Enter/select your Coral ID.
- 8.6.3 Following the prompts, change the oxidation time, if needed.



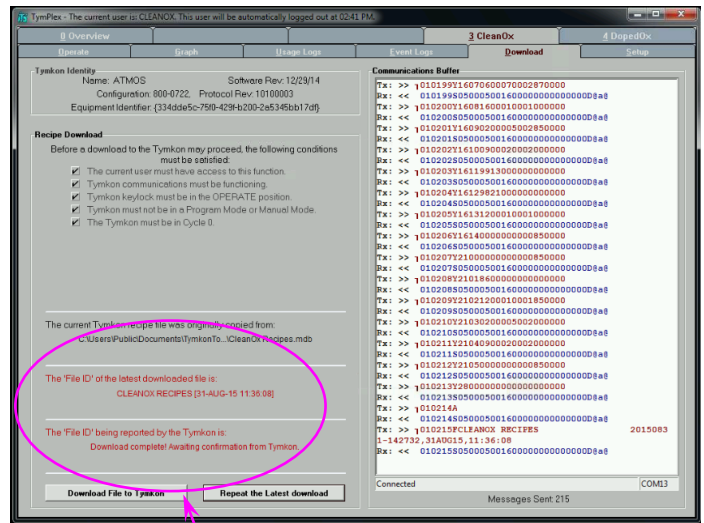
8.7 Download Recipe File

- 8.7.1 To download a new recipe file, select the **Download** subtab.
- 8.7.2 Click the **Download File to Tymkon** button.
- 8.7.3 From the dialog box, locate and select the correct recipe file (see **TABLE 3, RECIPE FILE LOCATION**).
- 8.7.4 From the **Ready to Download** dialog box, click the **Begin Download** button.
- 8.7.5 Wait for the recipe to complete downloading. (Sections will change from red to black text.)

Download subtab



Download File to Tymkon button



This text changes from red to black when download is complete.

TABLE 3, RECIPE FILE LOCATION

Furnace	Path	Recipe Type	Recipe File
CleanOx	[MainPath]\CleanOx\CleanOx Recipes	Dry Oxidation	Clean DryOx Recipes.mdb
		Wet Oxidation	Clean WetOx Recipes.mdb
DopedOx	[MainPath]\DopedOx\DopedOx Recipes	Anneal	Doped Anneal Recipes.mdb
		Boron Doping	Boron Solid Source Recipes.mdb
		Dry Oxidation	Doped DryOx Recipes.mdb
		Phosphorous Doping	Phos Solid Source Recipes.mdb
		Wet Oxidation	Doped WetOx Recipes.mdb

[MainPath] = C:\Users\Public\Documents\TymkonTools\UserData

8.8 Select Recipe

- 8.8.1 Select the **Operate** subtab.
- 8.8.2 Click the **Select Recipe** pull down box.
- 8.8.3 Select the desired recipe.
- 8.8.4 Activate the recipe by clicking the **Select Recipe # (recipe name)** button.

Select Recipe pull down box

Select Recipe button

8.9 Run Recipe

- 8.9.1 From the **Operate** subtab, click the **Run** button.

Run button

8.10 Wait for Run Completion

8.10.1 Wait for the cooldown cycle to complete.

NOTE: The boat will be out, the current recipe will be "REMOVE SAMPLES" and the current cycle will be "REMOVE SAMPLES". If this recipe is not reset within a programmed amount of time, the system will start the "IDLE WITH COMPLETED SAMPLES ON BOAT" and the load will push back into the furnace.

8.10.2 Click the **Reset** button.

8.10.3 At the **Reset the current recipe ?** dialog box, click the **OK** button.

8.11 Unload Boat

8.11.1 Ensure the run has completed.



CAUTION



All recipes include the necessary cool down time to ensure the quartzware, wafers, and samples are cool enough to be touched and handled.

Do not touch or handle quartzware, wafers, or samples unless the current run is COMPLETE.



CAUTION

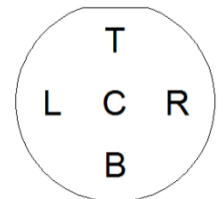
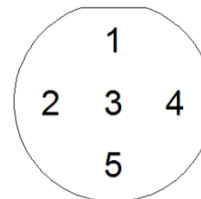


8.12 Unload Wafers

8.12.1 Remove the monitor wafers and samples from the boat.

8.13 Measure Monitor Wafers

8.13.1 Measure the thickness at 5 sites on each monitor wafer.



8.14 Place System in Standby

8.14.1 Select the "VACUUM IDLE" recipe.

8.14.2 Run the recipe.

NOTE: The cantilever will go in, the system will pump down, and go on hold.

8.15 Disable Tool in Coral

8.15.1 Disable the furnace in Coral.

8.15.2 Record the thickness measurements.

9 Solid Source Instructions

9.1 Install the desired boat

9.1.1 Ensure the unwanted quartz boat on the cantilever is sufficiently cool.

*NOTE: The programmed cooldown cycle will be finished and the host computer will show ***** Complete *****.*

- 9.1.2 **Using the boat fork**, carefully remove the unwanted boat and place it on a quartz plate for storage.
- 9.1.3 **Using the boat fork**, carefully place the desired boat on the cantilever.

9.2 Dehydrate Sources

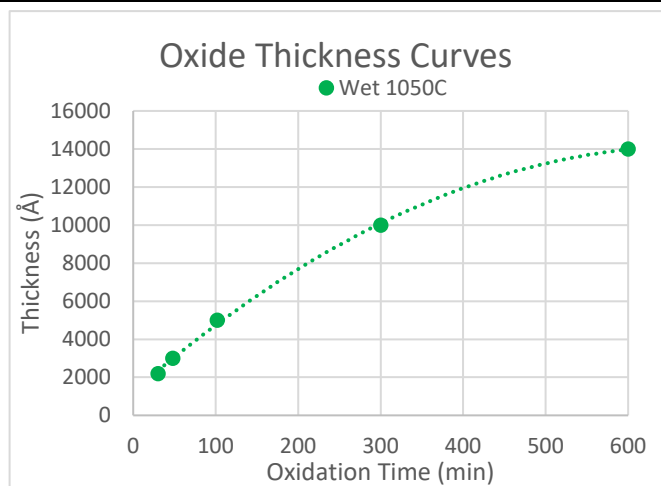
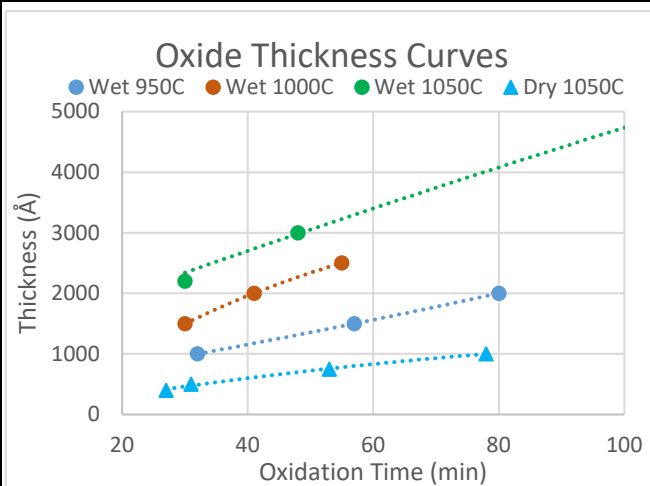
- 9.2.1 **If the solid sources have been out of the furnace for more than 1 hour**, dehydrate the sources.
 - 9.2.1.1 **Using the boat fork**, place the boat with the solid sources on the cantilever.
 - 9.2.1.2 Ensure each slot adjacent to a solid source wafer has a filler wafer.
 - 9.2.1.3 Process the "DEHYDRATE SOURCES" recipe.
 - 9.2.1.4 When completed, the sources are ready to be used for doping.

10 Process Notes

10.1 Process Summary

10.1.1 **TABLE 4, OXIDATION PROCESS SUMMARY DATA** lists the basic information for the various processes. Contact staff for help selecting a recipe and oxidation time.

Furnace	Recipe	N2 (slm)	O2 (slm)	N2/TLC (slm)	Temp (°C)	Approx. Time (min)	Thick (Å)	
CleanOx, Doped Ox	DRY OX (All temps)		4		1000	60	550	
					1050	27	400	
						31	500	
						53	750	
						78	1000	
	CLEAN OX (All temps)			3.5		850	100	800
						950	32	1000
							57	1500
							80	2000
						1000	30	1500
						41	2000	
						55	2500	
1050						30	2200	
						48	3000	
						102	5000	
	300	10000						
	600	14000						
Doped Ox	ANNEAL (All temps)	5						
	BORON DOPING	8						
	PHOS DOPING	8						



Note: Oxidation times can be approximated by this formula:

$$New\ Time = \frac{(Known\ Time) * (New\ Thickness)^2}{(Known\ Thickness)^2}$$

11 Revision History

Rev	Date	Originator	Description of Changes
3	10 Jun 19	T. Olsen	Update document to new format.
2	29 Jun 18	T. Olsen	General update and re-order sub-sections.
1	28 Mar 16	T. Olsen	Initial Release