Carbon Nanotube Catalysed Chemical Vapour Deposition
Process Information (version 2.1)

1 Location

Cleanroom (611B), Level 6 Rankine Building, Oakfield Avenue.

2 Processes

The tube furnace (4" diameter process tube) will be used for three independent purposes:

1. Carbon nanotube growth on silicon wafers.
2. Dry oxidation of silicon wafers.
3. Wet oxidation of silicon wafers.

Feedstock Gases

Carbon nanotube growth:	Hydrogen, 99.995 % (4 % of total flow), <175 bar, reduced to 1 bar by regulator (LH thread fitting) Argon, 99.999 %, <200 bar, reduced to 1 bar by regulator (RH thread fitting) Argon bubbled through Ethanol
Silicon oxidation (Dry):	Oxygen, 99.999 %, <200 bar, reduced to 1 bar by regulator (RH thread fitting)
Silicon oxidation (Wet):	Argon, 99.999 %, <200 bar, reduced to 1 bar by regulator (RH thread fitting)

All cylinders have a 50 L capacity at pressure.

The system is designed so that oxygen can NEVER be introduced to the furnace during carbon nanotube growth.

3 Electrical Equipment and Requirements

Item	Lindberg/Blue-M Three Zone Tube Furnace
Model	STF55666C
Dimensions (cm)	137.2 x 55.9 x 66 (W x F-B x H)
Power Requirements	11000 W, 240 VAC 50/60 single phase
Weight	75 kg

Item	Dual Channel Digital Power Supply and Readout Module PR4000 x 2
Model	MKS Dual Channel PR4000F5V1N
Dimensions	(half 19-inch rack mount), 24 x 9 x 23 (cm)
Power Req.	115/230 VAC, 50/60 Hz

Item	Mass flow controller x 3
Model	MKS 1179A 5000 sccm
Dimensions	15 x 15 x 5 (cm)
Power Req.	Supplied by Dual Channel Digital Power Supply and Readout Module PR4000

Item	Mass flow controller x 1
Model	MKS 1179A 500 sccm
Dimensions	15 x 15 x 5 (cm)
Power Req.	Supplied by Dual Channel Digital Power Supply and Readout Module PR4000

4 Operation Procedures

Only users who have been trained and demonstrated the safe operation of the furnace are allowed to use the tube furnace. Users MUST sign the page at the end of this document to confirm that they have been trained and have read this leaflet. Users are also required to read the School safety and chemical safety manuals that can be found on the School safety website:

http://www.gla.ac.uk/schools/engineering/studentstaff/safety/

Additional risk assessments (e.g. JWNC laboratory safety, COSHH forms) must be completed as required for sample preparation procedures.

4.1 Entering the Nanotube Growth Laboratory

Users should respect the dress code of the cleanroom and wear appropriate shoe covers, clean suit, safety glasses and latex gloves as appropriate to the work being carried out by others in the lab

4.2 Installing a Process Tube

The furnace must only be operated when a process tube is in place. The process tube is made from quartz with dimensions of 4" diameter, 2.5 mm wall thickness, 66" length. The furnace is fitted with tube adaptors to accommodate the process tube. Installation of the furnace process tube and tube adaptors can generate ceramic fibres/air borne particles which can result in the following:

- May be irritating to skin, eyes, and respiratory tract.
- May be harmful if inhaled.
- May contain or form crystalline silica at high temperatures (above 871 ° C) which can cause severe respiratory disease
- Possible cancer hazard.

Measures to minimise exposure include:

- Keep personnel not involved in the installation out of the area
- Use a good vacuum cleaner to clean area and equipment
- Do NOT use compressed air
- Wear long sleeve overalls, gloves, and eye protection to minimise eye and skin contact.
- A face mask should be worn.
- Overalls should be laundered after installation.

4.3 Carbon Nanotube Growth

The operator **MUST** be present at all times during the nanotube growth procedure when hydrogen is flowing or argon is passed through the ethanol bubbler.

The PR4000 Controllers are connected to the MFCs as follows:

"Controller 1"	FL1 "Ar MFC"	FL2 "O_2 MFC"
"Controller 2"	FL1 "Ar/EtOH MFC"	FL2 "H_2 MFC"

1. Check to ensure there is adequate exhaust extraction and then turn ON the furnace enclosure heat extract (set to high (H)).
2. Check that the flexible piping is connected between the "Exhaust Line" located to the rear of the furnace and the "Ar/H_2/EtOH" gas line.
3. Check that the bubbler contains ethanol, turn ON the ethanol bubbler chiller and set the appropriate temperature (in the range 0 – 30 °C).
4. Turn ON both of the MFC PR4000 power supplies ("Controller 1" and "Controller 2").
5. Check to ensure the three-way valve is set to flow through the large furnace.
6. OPEN the cylinder valve on the Argon cylinder and adjust the regulator valve to read 1 bar.
7. OPEN ball valve ("Ar Valve 1") between the Ar regulator and the "Ar MFC" and "Ar/EtOH MFC".
8. Set the Argon flow rate setpoint for both the "Ar MFC" and "Ar/EtOH MFC" to 300 sccm and open the "Ar/EtOH Valve 2" then "Ar/EtOH Valve 1". Press "ON" on the two controllers to purge the gas line and deoxygenate the ethanol in the bubbler for 10 mins.

*** The valves MUST be opened in this order to prevent pressure build up in the glass bubbler***

9. To isolate the bubbler, close the "Ar/EtOH MFC" by setting the setpoint to 0 and then pressing "ON" on the controller, then close the "Ar/EtOH Valve 1" followed by "Ar/EtOH Valve 2".

*** The valves MUST be opened in this order to prevent pressure build up in the glass bubbler***

10. OPEN the cylinder valve on the Hydrogen cylinder and adjust the regulator valve to read 1 bar.
11. OPEN ball valve ("H_2 Valve 1") between the H_2 regulator and the "H_2 MFC".
12. Set the Hydrogen flow rate setpoint (Controller 2, FL2) to 10 sccm and press "ON" on the controller to flow for 5 mins.
13. Close the "H_2 MFC" by setting the setpoint to 0 (Controller 2, FL2) and pressing "OFF" on the controller, continue to flow Argon for a further 5 mins.
14. Close the "Ar MFC" by setting the setpoint to 0 sccm (Controller 1, FL1) and pressing "OFF" on the controller.
15. Change the connection from the "Exhaust Line" to the "Furnace Line".
16. OPEN the "Ar MFC" by setting the setpoint to 2000 sccm (Controller 1, FL1) and pressing "ON"on the controller, flow for 10 mins. This should be sufficient time for the Ar to fill the process tube by 150 % to remove air in the tube.
17. While the Ar is flowing, open the exhaust box, remove the endcap assembly and take out the radiation shield boat using the quartz push rod.
18. Load the sample wafer onto the sample boat and inert in to the process tube using the push rod to the "centre line" on the quartz push rod to ensure that the wafer boat is centred in the furnace.
19. Replace the radiation shield, the endcap (locking the endcap in place with the clip), shut the exhaust box and lock with the wing nuts.
20. Turn ON the furnace power supply switch (on the wall).
21. Turn ON the furnace power switch (furnace front panel)
22. Program the temperature eurotherm for all three zones of the furnace.

23. Program the MFCs.

24. The parameters are anticipated to be as follows:

Stage #	Temperature	Duration	Process Gases
1	25 – 700 °C	10 mins	Ar, 4% H_2
2	700 – 850 °C	10 mins	Ar, 4% H_2
3	850 °C	5 mins	Ar, 4% H_2
4	850 °C	20 mins	Ar, 4% H_2, Ar/Ethanol
5	850 – 25 °C	~2 hours	Ar,

The example given below shows a growth scheme with a total flow of 2000 sccm with an Ar/EtOH flow rate of 200 sccm. Hydrogen concentration is kept at 4% throughout the run.

Stage	Duration (mins)	Furnace Temp	MFC (setpoint)	Valves Open	Valves Closed	Notes
1	10	25 – 700 °C	Ar "Controller 1", FL1 = 1920 O_2 "Controller 1", FL2 = 0 Ar/EtOH "Controller 2", FL1 = 0 H_2 "Controller 2", FL2 = 80	Ar Valve 1 H_2 Valve 1	Ar/EtOH Valve 1 Ar/EtOH Valve 2 O_2 Valve 1	Catalyst reduction/ Temperature ramp
2	10	700 – 850 °C	Ar "Controller 1", FL1 = 1920 O_2 "Controller 1", FL2 = 0 Ar/EtOH "Controller 2", FL1 = 0 H_2 "Controller 2", FL2 = 80	Ar Valve 1 H_2 Valve 1	Ar/EtOH Valve 1 Ar/EtOH Valve 2 O_2 Valve 1	Catalyst reduction/ Temperature ramp
3	5	850 °C	Ar "Controller 1", FL1 = 1920 O_2 "Controller 1", FL2 = 0 Ar/EtOH "Controller 2", FL1 = 0 H_2 "Controller 2", FL2 = 80	Ar Valve 1 H_2 Valve 1	Ar/EtOH Valve 1 Ar/EtOH Valve 2 O_2 Valve 1	Catalyst reduction/ Temperature stabilisation
4	20	850 °C	Ar "Controller 1", FL1 = 1720 O_2 "Controller 1", FL2 = 0 Ar/EtOH "Controller 2", FL1 = 200 H_2 "Controller 2", FL2 = 80	Ar Valve 1 H_2 Valve 1 Ar/EtOH Valve 1 Ar/EtOH Valve 2	O_2 Valve 1	Nanotube growth
5	~120	850 - 25	Ar "Controller 1", FL1 = 2000 O_2 "Controller 1", FL2 = 0 Ar/EtOH "Controller 2", FL1 = 0 H_2 "Controller 2", FL2 = 0	Ar Valve 1	H_2 Valve 1 Ar/EtOH Valve 1 Ar/EtOH Valve 2 O_2 Valve 1	Cool down

25. Close the hydrogen cylinder valve at the end of Stage 4.
26. Once the furnace temperature decreases to 200 °C, open the exhaust box, remove the endcap assembly and take out the radiation shield boat using the quartz push rod. Remove the sample boat using the push rod, replace the radiation shield and then the end cap. Finally close the exhaust box.
27. Change the gas supply connection from the "Furnace Line" to the Exhaust Line".
28. Turn off the chiller.
29. Open the H_2 Valve 1, and set the hydrogen (Controller 2, FL2 = 80 sccm) to flow until the H_2 flow reading shows 0. Close "H2 Valve 1" and set Controller 2, FL2 = 0.
30. Close the Hydrogen Regulator Valve.
31. Close the Argon cylinder valve.
32. Continue to allow the Argon to flow until the Ar flow reading shows 0. Close "Ar Valve 1" and set Controller 1, FL1 = 0.
33. Close the Argon Regulator Valve.
34. Check all set points = 0, Turn off PR4000 controllers.
35. Turn off heat extractor
36. Turn off furnace at front and then at wall.

4.3.1 Carbon Nanotube Growth Emergency Shut Down

In the case of a moderate emergency –

1. Turn OFF the furnace using the power switch at the front of the furnace
2. Turn OFF both MFC controllers
3. Turn OFF the Hydrogen and Argon gas cylinders
4. Warn any other cleanroom users and leave the room via an appropriate emergency exit ensuring that the door is closed once everyone has left the room.
5. Report the fault/emergency to John Weaver (ext 5656) or Phil Dobson (ext 4314).

In the case of a severe emergency -

1. Warn other people in the cleanroom and leave the room via the emergency exit immediately, ensuring the door is closed and everyone has left the room.
2. Raise the alarm and report the emergency by ringing 4444 on any internal telephone.

4.4 Silicon Oxidation (Dry)

The PR4000 Controllers are connected to the MFCs as follows:

"Controller 1"	FL1 "Ar MFC"	FL2 "O_2 MFC"
"Controller 2"	FL1 "Ar/EtOH MFC"	FL2 "H_2 MFC"

1. Check to ensure there is adequate exhaust extraction and then turn ON the furnace enclosure heat extract (set to high (H)).
2. Check that the flexible piping is connected between the "Exhaust Line" located to the rear of the furnace and the "Oxygen" gas line.
3. Check to ensure the three-way valve is set to flow through the large furnace.
4. Turn ON the MFC PR4000 power supply ("Controller 1").
5. OPEN the cylinder valve on the Oxygen cylinder and adjust the regulator valve to read 1 bar.
6. OPEN ball valve ("O_2 Valve 1") between the oxygen regulator and the "O_2 MFC".
7. Set the oxygen flow rate setpoint (Controller 1, FL2) to 500 sccm press "ON" on the controller, flow for 5 mins.
8. Close the "O_2 MFC" by setting the setpoint to 0 sccm (Controller 1, FL2) and pressing "OFF"on the controller.
9. Change the connection from the "Exhaust Line" to the "Furnace Line".
10. Open the exhaust box, remove the endcap assembly and take out the radiation shield boat using the quartz push rod.
11. Load the sample wafer onto the sample boat and inert in to the process tube using the push rod to the "centre line" on the quartz push rod to ensure that the wafer boat is centred in the furnace.
12. Replace the radiation shield, the endcap (locking the endcap in place with the clip), shut the exhaust box and lock with the wing nuts.
13. OPEN the "O_2 MFC" by setting the setpoint to 2000 sccm (Controller 1, FL2) and pressing "ON" on the controller, flow for 10 mins. This should be sufficient time for the O_2 to fill the process tube by 150 % to remove air in the tube.
14. Turn ON the furnace power supply switch (on the wall).
15. Turn ON the furnace power switch (furnace front panel)
16. Program the temperature eurotherm for all three zones of the furnace.
17. Program the O_2 MFC to flow 500 sccm (Controller 1, FL2).
18. The parameters are anticipated to be as follows:

Stage #	Temperature	Duration	Process Gases
1	25 – 700 °C	10 mins	O_2
2	700 – 1000 °C	15 mins	O_2
3	1000 °C	mins to hrs	O_2
4	1000 - 25 °C	2 hrs	O_2

19. Once the furnace temperature decreases to 200 °C,
20. Close the oxygen cylinder valve.
21. Close "O_2 Valve 1".
22. Change the gas supply connection from the "Furnace Line" to the Exhaust Line".
23. Open "O_2 Valve 1", and set the oxygen (Controller 1, FL2 = 500 sccm) to flow until the O_2 flow reading shows 0. Close "O_2 Valve 1" and set Controller 1, FL2 = 0.

24. Close the oxygen regulator valve.
25. Close "O_2 Valve 1".
26. Check all set points = 0, Turn off PR4000 controller.
27. Open the exhaust box, remove the endcap assembly and take out the radiation shield boat using the quartz push rod. Remove the sample boat using the push rod, replace the radiation shield and then the end cap. Finally close the exhaust box.
28. Turn off heat extractor
29. Turn off furnace at front and then at wall.

4.4.1 Silicon Oxidation (Dry) Emergency Shut Down

In the case of a moderate emergency -

1. Turn OFF the furnace using the power switch at the front of the furnace
2. Turn OFF both MFC controllers
3. Turn OFF the Oxygen gas cylinder
4. Warn any other cleanroom users and leave the room via an appropriate emergency exit ensuring that the door is closed once everyone has left the room.
5. Report the fault/emergency to John Weaver (ext 5656) or Phil Dobson (ext 4314).

In the case of a severe emergency

3. Warn other people in the cleanroom and leave the room via the emergency exit immediately, ensuring the door is closed and everyone has left the room.
2. Raise the alarm and report the emergency by ringing 4444 on any internal telephone

4.5 Gas Leaks

4.5.1 Emergency Shut Down (Ar or O_2 leak)

1. Turn OFF the furnace using the power switch at the front of the furnace
2. Turn OFF both MFC controllers
3. Turn OFF all gas cylinders
4. Leave the room via the emergency exit and ensure that the door is closed once everyone has left the room.
5. Report the fault/emergency to John Weaver (ext 5656) or Phil Dobson (ext 4314)
6. The room should be left for at least 1 hour before returning to check the oxygen levels. This should only be carried out by an appropriate member of staff, wearing the appropriate safety equipment. Given the small amount of gas present in the pipework this should be more than sufficient time for it to be removed by the ventilation system.

4.5.2 Emergency Shut Down (H2 leak)

1. Do NOT switch electrical appliances ON or OFF.
2. Set the H_2 MFC setpoint to 0.
3. Turn OFF all gas cylinders
4. Leave the room via the emergency exit and ensure that the door is closed once everyone has left the room.
5. Report the fault/emergency to central services on extension 4444. Contact John Weaver (ext 5656), or Phil Dobson (ext 4314)
6. The room should be left for at least 1 hour before returning to check the oxygen levels. This should only be carried out by an appropriate member of staff, wearing the appropriate safety equipment. Given the small amount of gas present in the pipework this should be more than sufficient time for it to be removed by the ventilation system.
7. In the event of a hydrogen leak, the roof space above the false ceiling must also be checked for excess hydrogen/depleted oxygen, since hydrogen would tend to rise, pass through the false ceiling and collect in the roof space. Once these checks have been satisfied, the leak(s) must be found, sealed and tested by a qualified member of staff before any further use of the tube furnace.

5 Chemicals and Reagents

COSHH forms completed for nanotube growth laboratory are:

- Operation of Tube Furnace for Carbon Nanotube Growth/Silicon Oxidation
- Use of Latex Gloves.

6 Risk Assessment

6.1 Risk of Burns (Thermal)

Some of the exposed quartz at either end of the furnace will become hot during operation. It is not necessary to touch these areas and access is largely restricted by the furnace enclosure. The only pieces of glassware that do need to be touched are the pyrex/aluminium endcap assembly, sample boat, radiation shield boat and push rod. The endcap at the exhaust end of the process tube will become hot during the process although this has been minimised by the use of radiation shields inside the process tube during operation and the high air flow in the exhaust box. Heat resistant gloves are provided, and should be used if necessary.
The furnace is well insulated and designed to allow the operator to use the front control panel at all operating temperatures (0 – 1100 °C). However, the top casing can become quite warm during operation. The operator should not touch nor place anything on the top of the furnace.

The inside of the furnace tube will be very hot during operation (≤1100 °C). The operator must not attempt to insert hands or the push rod in to the furnace while it is hot. Samples are to be loaded at room temperature and only unloaded once the furnace reaches <200 °C. A long quartz push rod is provided to insert and withdraw the sample boat from the centre of the process tube. The part of the quartz push rod inserted into the hot furnace will become hot (<200 °C) after being used. Care should be taken not to touch the hot section of the rod and it must be placed on the heat resistant perforated steel table.

Risk controlled by safety equipment and by training

6.2 Risk of Burns (Chemical)

No corrosive chemicals will be used during the process.

6.3 Risk from Noise

Regulations set a maximum daily 'dose' of sound energy ($L_{EP,d}$) of 90 dB which should not be exceeded. The *Health & Saftey Executive* have produced the following list of (L_{Aeq}) over exposure period exposure time to reach an $L_{EP,d}$: 87 dB(A) 16 hours, 90 dB(A) 8 hours, 93 dB(A) 4 hours, 96 dB(A) 2 hours, 108 dB(A) 7.5 minutes. Noise in the room is primarily generated by the HEPA hoods and extraction system (~50 dB(A)). However this is well below the limits set by the HSE. *Therefore, this does not present any significant risk.*

6.4 Risk of Electrical Shock

All electrical equipment is fully grounded. Disconnect electrical equipment from the power supply before servicing.
Risk is controlled by equipment construction and training.

6.5 Risk Assessment of Creation of Explosive Mixtures in the Tube Furnace

The maximum flow rate of each gas is limited by the appropriate MFC.

The maximum flow rate for Oxygen is 5000 sccm. } Controlled by PR4000 #1
The maximum flow rate for Argon is 5000 sccm. } Controlled by PR4000 #1

The maximum flow rate for Hydrogen is 500 sccm. } Controlled by PR4000 #2
The maximum flow rate for Argon/Ethanol is 5000 sccm. } Controlled by PR4000 #2

To eliminate the possibility of oxygen being introduced to the chamber at the same time as hydrogen, two gas supply lines are present; one line with only oxygen (for oxidation) and a second line with a gaseous mixture of argon/ethanol, argon and hydrogen (for nanotube growth). The furnace endcap has only one inlet adaptor. The chosen gas line and the furnace are connected using flexible stainless steel tubing fitted with quick-fit adaptors.

The nanotube growth process only requires a hydrogen gas fraction that is below the combustion limits of hydrogen (<4.1% in air). The carbon source is ethanol vapour carried by argon. To prevent the influx of air into the process tube an endcap specified to provide a gas tight seal suitable for atmospheric and vacuum applications has been employed for the tube inlet. The exhaust end is situated in a separate chamber which has an independent extraction system providing a high flow of air extraction, designed to rapidly cool and dilute the exhaust.

The furnace is located in an extraction hood which generates a high flow of air above the furnace primarily designed to remove the heat load of the furnace from the room, but additionally ensures that in the event of a gas leak from the process tubes, process gases are rapidly diluted and removed from the hot regions of the furnace. In the unlikely event of an Argon supply failure coinciding with failure of the inlet endcap seal, air might enter the process tube. This gas mixture could explode and perhaps shatter the quartzware and process tube. However, the extent of any explosion that might occur will be relatively minor, short lived and entirely contained within the furnace and the operator will be protected by the enclosure screens.

Risk of explosion and consequences of any explosion are minimised by system design and eliminated by the operator training.

The operator must follow the procedures for operation of the nanotube growth furnace given in section 4.3. If the operator does not follow the procedures there is some scope for the creation of explosive mixtures inside the process tube.
Risk associated with the use of hydrogen in a high temperature tube furnace is reduced by the following:

- The gases are only used in one location.
- The point-of-use is a very well ventilated room.
- The hydrogen and oxygen lines are fitted with flash-back arrestors.
- Regulators are fitted to all cylinders at the cylinder.
- The gas pressure is reduced to 1 bar by the gas regulators at the cylinders.
- The hydrogen composition is kept below the combustion limit (<4.1 %) in the process tube by operator training and by hardware.
- The equipment is electrically grounded.
- The mass flow controllers always fail to “shut” on loss of electrical power.
- Gas pipe line quick connectors fail to “shut” when disconnected.
- Operation is restricted to trained users.

- The endcaps are designed to provide a gas tight seal suitable for atmospheric and vacuum applications has been employed for the tube inlet.
- The furnace will be operating at atmospheric pressure.
- The furnace is enclosed in a frame with protective screens to shield the operator in the unlikely event of explosion.
- The operator will always be in the room while hydrogen is flowing in the system or Ar is passing through the ethanol bubbler.

Any risk associated with the creation of explosive mixtures in the process tube is therefore controlled by equipment and by training.

6.6 Risk Assessment of Gas Supply Failure/Leaks

6.6.1 Risk Assessment of Process Gas Supply Failure

Carbon Nanotube Growth

In the most extreme case, failure of the Argon supply would lead to the hydrogen composition in the process tube increasing above the 4% concentration normally used. Although the flow of hydrogen will be relatively small (typically < 250 sccm), this could create a potentially explosive mixture. To reduce the risk of this happening the following measures have been made:

- The operator will check the supply of process gases at the beginning of each process run as indicated by the gas cylinder regulator.
- The user will monitor the MFC flow readings during the process. The Ar flow is indicated by the "Ar MFC" and "Ar/EtOH MFC" flow reading on "Controller 1 FL1" and "Controller 2 FL1", respectively.

Risk is reduced by equipment and training.

Silicon Oxidation (Dry)

The process can continue safely without the continuous flow of oxygen.

Silicon Oxidation (Wet)

The process can continue safely without the continuous flow of Ar/H_2O vapour.

6.6.2 Risk Assessment of process gases (H_2, Ar, O_2) leaking from the cylinders or pipelines

Gas leaking from the cylinders could in the most extreme case give rise to explosive gas mixtures forming in the room or asphyxiation of the operator. To reduce the risk of this happening the following measures have been made:

- The room is very well ventilated
- Gas cylinders are securely fixed to the wall.
- Leak detector will be frequently used with Argon in the system to test cylinders and pipeline Swagelok connections to ensure no leaks have developed. This will take place every 10 runs.

- Where it has been specified by the manufacturer that P.T.F.E (Teflon) tape is require to seal joints, degreased oxygen safe P.T.F.E. tape has been used.

Risk is reduced by equipment and training.

6.6.3 Risk Assessment of Process Gases Leaking at the Endcap Connections

The endcap at the inlet end of the process tube is securely fixed with an air tight seal. The inlet end cap is designed to provide an airtight seal for processes at atmospheric pressure and vacuum. The endcap at the exhaust end is easily removed to allow for loading and unloading of samples. The endcap at the exhaust end does not provide an air tight seal but is located in the centre of the exhaust box to ensure that all exhaust gases are rapidly removed by the extraction system.

Risk is reduced by equipment and training.

6.7 Risk Assessment of Electrical Power Failure

Failure of power to the room will stop the air circulation in the room by turning off the HEPA filters, heat extraction and exhaust extraction. Some extraction for the exhaust will remain due to natural convection. The MFCs will default to the closed position and the gas supply to the furnace will cease.
In the event of electrical power failure to the Rankine building, in addition to the above, the furnace will shut down and cool down.

Failure of the electrical power supply poses no significant risk to the operator.

6.8 Risk Assessment of Exhaust Extract System Failure

The hot exhaust will tend to rise and is also directed towards the exhaust pipe by the endcap.
The exhaust system generates a distinctive sound and the extract can also be felt by hand. The operator will frequently check the extract during the process run.

Risk is reduced by equipment and training.

6.9 Risk Assessment of Heat Extract System Failure

The heat extraction is primarily used to remove excess heat from the tube furnace to prevent a significant increase in room temperature. However, if necessary, the furnace is designed to function without the need for an extraction hood.
The heat extraction system generates a distinctive sound and the extract can also be felt by hand. The operator will frequently check the extract during the process run.

Failure of the heat extractor poses no significant risk to the operator.

6.10 Risk Assessment of Process Products

6.10.1 Risk Assessment of Carbon Nanotubes

Carbon nanotubes have been reported to be potential carcinogens when produced in large quantities and dispersed in the air. Carbon nanotubes grown on silicon substrates are rigidly fixed to the silicon surface and therefore should not be considered as air borne particles nor a potential hazard. In addition the quantities produced using this method will produce less than a monolayer of carbon

nanotubes. The disposal of samples containing carbon nanotubes will require an oxygen plasma ash (5 min 200 W) prior to disposal in a contaminated sharps bin.

6.10.2 Risk Assessment of Silicon Dioxide

There is no risk associated with thin films of silicon dioxide.

6.10.3 Risk Assessment of Process Exhaust

The process tube exhaust will enter in to the fume extraction system in the ceiling. Exhaust will be combined with high air flow (0.228 m^3 s^{-1}) to dilute and cool the exhaust gases. Exhaust gases are not considered to be toxic and expected to contain no free nano particles
Risk is reduced by equipment and training.

6.11 Risk Assessment of Ethanol Bubbler

To prevent air entering the ethanol bubbler and creating a potentially explosive mixture when inside the furnace, the bubbler has been designed with air-tight seals. The procedure also includes a stage to remove any air present in the ethanol.
If the operator does NOT follow the procedure, there is some scope for the generation of pressure inside the bubbler that could give rise to the bubbler shattering and releasing highly flammable vapours. Measures made to reduce this risk include:

- Operators will be well trained and made aware of potential hazards
- A copy of the operation procedure (detailed in section 4.3) will be permanently located in the room.
- The volume of ethanol is only small (~100 cm^3).
- Refilling/cleaning of the bubbler is done away from ignition sources in a fume hood.
- The stock bottle of ethanol will be stored in a metal solvent cupboard located away from any ignition sources.
- The bubbler is immersed in a cooled glycol bath which will dilute any accidental release of ethanol.

Risk is reduced by equipment and training.

6.12 Further Safety Considerations

A CO_2 fire extinguisher is located near the main door of the room; a second is located at the emergency exit to room 607
A fire blanket is located near the main door of the room.
The fire extinguisher or fire blanket should only be used for very minor fires and only used by an appropriate member of staff. If the fire develops the operator should leave the room immediately using the emergency exit, close the door behind him/her and activate the nearest fire alarm while making their way out of the building via the stairs.
An emergency shower and drain is located near centre of the room, close to the system.
Only trained members of staff should move cylinders and fit regulators.

7 User Access

This equipment will be initially used by a limited number of named users (Phil Dobson, Yuan Zhang and John Weaver) and will **not** be considered to be open access for cleanroom users.
This emergency exit for the lab must be kept clear at all times.
All future users will be trained to operate the equipment according to an established procedure.

Print Name	**Group**	**Signature**	**Date**
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This document has been approved by:

PROF. JOHN WEAVER Signed_____ Date _____