# Technical Specifications for Fabrication, Supply, Installation and Commissioning of Liquid silicon infiltration (LSI) furnace

## 1. Introduction

C-C-SiC composites offer distinct advantages over C-C composites namely shorter lead time and superior oxidation resistance. These composites have potential for use as throat inserts, thermo structural re-entry components, Liquid and cryo engine divergent, substrate for optical mirrors, etc. Liquid Silicon Infiltration (LSI) is a widely used process for realization of these composites. It is proposed to set-up an LSI facility of **retort size 1100 mm dia x 1200 mm height** at CMSE.

## 2. Vacuum Chamber Details

| Sl. No. | Description | Specification |
|---|---|---|
| **2.1** | **Configuration** | Vertically mounted cylindrical double walled chamber |
| **2.2** | **Vacuum Chamber Size** | **1300 mm Dia x 1350mm Ht. (Approx.)** |
| **2.3** | **Material Of Construction** | Stainless Steel 304 L |
| **2.4** | **Chamber Cooling** | Double walled, water cooled guiding baffles shall beprovided in the annular space of the chamber cylindrical, top dish and bottom dished ends for effective water circulation. |
| **2.5** | **Chamber Top Side** | Topside of the chamber shall be welded with flange to achieve vacuum sealing with top dished lid. |
| **2.6** | **Chamber Bottom Side** | Bottom side of the chamber shall be provided with demountable dished end which shall be coupled with bottom flange with bolts. |
| **2.7** | **Top Dished Door / Lid** | Top dished lid shall have swivel arrangement for opening and closing by hydraulic lifting mechanism supported on the cylindrical side of the chamber with suitable brackets. |
| **2.8** | **Ports** | Ports shall be provided for evacuation, electrical feed-through, gas inlet port, thermocouple feed through vent and view port etc. |

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| **2.9** | **Design Code** | Chamber shall be fabricated as per ASME CODE SEC VIII,DIV I. |
| **2.10** | **Welding** | Chamber and sub-assemblies shall be welded using argon arc welding technique for leak tight joints. |
| **2.11** | **Surface Finish** | Inner surface of the chamber shall be polished for low out gassing rate. |
| **2.12** | **Leak rate** | All the individual components shall be leak-checked by using helium mass spectrometer leak detector to an individual leak rate of 1x $10^{-6}$ m.bar lit/sec. |
| **2.13** | **Support Structure** | Mild steel elegant tubular rigid support structure shall be provided with a staircase in the front for easy access to the top side of chamber.<br>Adequate clearance and arrangement shall be provided at the bottom for repair and maintenance of the furnace, by removal of the bottom dished end. |
| **2.14** | **SEALING** | Suitable sealing with adequate water cooling shall be provided to seal the chamber flange with top lid flange, all the feed-through, hydraulic job loading-unloading shaft, etc. |

**3. Monorail Crane:**

Suitable Monorail crane equipment having capacity for lifting and lowering minimum of 1500kgs weight for crucibles, porous preforms and raw materials shall be provided.

**4. HOT ZONE DETAILS :**

| Sl. No. | Description | Specification |
|---|---|---|
| 4.1 | **Retort size (Graphite)** | **1100mm OD x 1200mm Ht. (Approx).** |
| 4.2 | **Mother crucible size (Graphite)** | **1000 mm OD x 500mm (Ht.) with additional graphite cylinder to extend height by 200 mm** |
| 4.3 | **Job crucible size (Graphite)** | **900 mm O.D X500mm(Ht.)** |
| 4.4 | **Maximum Design Temperature** | **1800° C** |

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| 4.5 | **Working Temperature** | **1700°C** |
| 4.6 | **Temperature Uniformity** | ± 10 °C in the effective hot zone above 600°C |
| 4.7 | **Heating Rate** | 1 °C –10°C/min from ambient to 1700 °C |
| 4.8 | **Cooling** | Natural vacuum cooling (or) under inert atmosphere of argon partial pressure. |
| 4.9 | **Method Of Heating** | Heating elements shall be made by silicon resistant grade graphite from **SGL/Schunk/Mersen**. |
| 4.10 | **Heat Insulation** | Graphite rigid board based Heat Insulation shall be provided from **SGL/Schunk/Mersen**. |
| 4.11 | **Graphite Retort** | Graphite retort shall be made in 4 rings placed one above the other matching to the retort dimension. Top of the retort shall be provided with lid to prevent escape of Silicon Vapours to the heating elements side by assisting in the differential pressure between inside Vs outside of retort. Differential pressure control system is described in Section 8.5. |
| 4.12 | **Mother Crucible** | Mother crucible shall be made of 2 rings with dimensions as given in Sl.no 4.2. It may be noted that the additional 2nd ring will be used only in case of components that requires height greater than 500mm. |
| 4.13 | **Job Crucible** | Graphite crucible of size 900 mm OD x 500mm (Ht.) – 1 No. shall be provided. |
| 4.14 | **Charge Hearth** | Charge Hearth shall be made of graphite of suitable thickness to withstand weight of 750 kgs which include weights of crucible, job, retort with lid**.** |
| 4.15 | **Job loading-unloading** | Suitable Motorized/Hydraulic lifting mechanism |

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| | **arrangement** | shall be provided for loading and un-loading the Job in cold condition. To achieve this, the Mother and job crucible shall be lifted to a1400 mm height from the base in cold condition, and after loading the jobs from the top, the crucibles shall be lowered to its original down position. The heat treatment and LSI process shall be conducted in mother and job crucibles in down condition. Suitable sealing mechanism shall be provided between the lifting shaft and the vacuum chamber, to avoid any leakage due to the Hearth movement. In spite of regular UP and Down operation of the Charge Hearth, the desired level of vacuum and leak tightness shall be achieved as mentioned in Section 2.12 above.<br><br>Note: The lifting mechanism shall be designed to function for at least 50,000 cycles of lifting and lowering operations. |
| 4.16 | **Heater Feed Through** | Heater feed through made of electrolytic copper shall be provided with water cooling facility |
| 4.17 | **Graphite properties and Heater properties** | Heating elements, Retort, Mother crucible and job crucible shall be made from the Graphite<br><br>1. Bulk density 1.7g/cc (min)<br>2. Compressive strength 30 Mpa (min)<br>3. Thermal conductivity 120 W/mk (min)<br>4. Ash <0.07%<br>having the following properties. |

**5. HEATING SYSTEM POWERSUPPLY**:

The Furnace Heating System shall comprise of 415V AC, 50 Hz, 3 Phase low tension transformer of suitable capacity which shall be controlled through Thyristor Power Controller.

**6. THERMOCOUPLES:**

Total 6 Nos. of Tungsten, 5% Rhenium Vs Tungsten 26% Rhenium thermocouples shall be provided of which 3 Nos. shall be used for Temperature measurement and control and other 3 Nos. shall be used for over temperature controller.

**7. Optical Pyrometer**

Optical Pyrometer for the range of 1000°C to 2000°C shall be provided for temperature measurement.

**8. VACUUM PUMPING SYSTEM:**

**8.1. ROTARY VACUUM PUMP:**

Single stage rotary piston pump having a pumping speed of 250 $m^3$/hr(min.)shall be provided to achieve desired vacuum level within 30 min for evacuation of the chamber in conjunction with roots pump. **Make: Edwards / Pfeiffer / Leybold / Busch.**

**8.2. ROOTS PUMP**

Mechanical Booster Pump having pumping speed of 1500 $m^3$/hr(min.)shall be provided to achieve desired vacuum level within 30 min for evacuation of chamber in conjunction with rotary piston pump. **Make: Edwards / Pfeiffer / Leybold / Busch**

**8.3Water Cooled Condenser:**

Water Cooled Condenser made of stainless steel with inner cooling coil made of copper shall be provided in the Vacuum Line for cooling the hot gases coming out from the Retort.

**8.4ISOLATION VALVES:**

Electro pneumatically operated angle valve shall be provided in the evacuation line to isolate the chamber from the roots vacuum pump.

**8.5Differential Pressure Control System:**A differential pressure needs to be maintained during process, to avoid escape of silicon vapors out of retort and deposition on heating elements. A suitable arrangement shall be made to achieve, measure and monitor a pressure difference of 2-4 mbar (approx.). This shall be achieved through control of vacuum at the two locations through motorized throttle valves with PID Controller and capacitance manometers, etc.

**8.6 Vent Valve:** An Electro Pneumatically operated right angle valve shall be provided to vent the Chamber at the end of the process either by admitting air or by connecting dry Nitrogen Gas Source. This valve operation shall be interlocked with pressure transmitter.

**8.7 Vacuum piping lines:** Vacuum piping lines made of stainless steel shall be provided to connect the pumping system with the Vacuum Chamber.

## 9. VACUUM MEASUREMENT:

### 9.1. COMPOUND DIAL GAUGE:

A Dial type Mechanical vacuum gauge having a measuring range of -760 to +760 mm of Hg shall be provided.

### 9.2. VACUUM MEASURING GAUGEIN VACUUM LINE:

Pirani sensors for measuring vacuum in the range of 1000 m.bar to $10^{-3}$m.bar shall be provided near the vacuum pumps.

### 9.3. CAPACITANCE MANOMETERON FURNACE:

- Capacitance Manometer having measuring range of 0.1 m. bar to 1000 m. bar shall be provided.
- Capacitance Manometer having measuring range of 0.01 m. bar to 100 m. bar shall be provided.

### 9.4. VACUUM CHARACTERIZATION:

#### 9.4.1 ULTIMATE VACUUM:

In clean, cold, empty, dry degassed chamber vacuum of better than 1 x $10^{-2}$m.bar in15 minutes shall be achievable after back filling with dry Nitrogen.

**9.5. LEAK RATE:**

In clean, cold, empty, degassed condition, Leak Rate less than 1 x $10^{-3}$m.bar lit / sec shall be achieved under pressure rise method under valve off condition.

**10.0CONTROL INSTRUMENTATION AND CONTROLCONSOLE:**

The furnace shall be provided with following instrumentation and control system for complete operation of the system both vacuum cycle and temperature cycles in auto mode.

**10.1Temperature Programmer Controller :**

A microprocessor based Digital Temperature Programmer Controller of **M/s.EUROTHERM/Siemens/ Yokogawa** shall be provided for temperature measurement and control in conjunction with PID Controllers and thermocouples.

**10.2 PID Controller.**

**M/s. Eurotherm /Siemens / Yokogawa** Make PID Controller shall be provided for temperature measurement and control also works in conjunction with master temperature programmer controller.

**10.3 Over Temperature Controller.**

Over temperature controller of **M/s. Eurotherm /Siemens/Yokogawa** make PID Controller shall be provided to cut-off the power supply to the heating element in the event of any malfunctioning of temperature programmer controller or if temperature level shoots beyond the normal operation temperature.

**10.4 Programmable Logic Controller (PLC):**

Programmable Logic Controller shall be provided in the system for complete automation of both temperature cycle as well as the vacuum cycle to achieve various interlocks provided in the system.

**10.5 AUTO / MANUAL VACUUMCONTROLLER:**

Auto/manual vacuum controller with manual override facility consisting of mimic diagram with indication lamp, ON/OFF Switches, utility Failure Indications with alarm shall be provided.

## 10.6 INDUSTRIAL PC WITH SCADA SOFTWARE:

Industrial PC with Supervisory Control and Data Acquisition System (SCADA) shall be provided

- Real time Graphic operator interface and furnace monitoring in pre-defined format.
- Multilevel access control for operational security.
- Display of analog inputs & output groups in tabular or bar graph format.
- Display of controller monitoring groups in bar-graph format containing set point and process variables.
- Discrete I/O monitoring groups in panel style status display.
- Alarm monitoring through an alarm line display that displays most recent, most severe active and acknowledged alarm.
- Programming of furnace in vacuum mode (or) inert gas partial pressure mode of operation with predefined temperature cycle, vacuum cycle and inert gas cycle.
- Data logging and storage as per specified time intervals.
- Multi-level logging to handle different critical levels of parameters.
- Storage of all data on computer hard disk
- Retrieval and display of all stored data from computer hard disk for reviewing purpose.
- Acquisition & display of job temperature and pressure (vacuum / partial pressure) in hot zone at predefined time intervals, storage of acquired data on computer hard disk, reviewing of acquired data.
- Export of data in ASCII text file format for future data analysis.
- Printing of data in tabular and graphical form to a printer ( Colour printer to be provided).
- On - field HMI panel for control operations shall be provided.

## 11. CONTROL CONSOLE:

The control cubicle shall be made out of sheet steel with IP-54 construction accommodating the following:

(i) Auto / manual controller
(ii) Vacuum gauge controller
(iii) Temperature programmer controller
(iv) Over temperature controller

All the electrical switch gear like control transformers contactors, relays, fuses, timers, and motor starters, etc., shall be mounted on a plate and fitted vertically for the convenience of maintenance. The control console shall have full front doors opening for easy approach of internal components.

A power panel shall be provided in the control console which houses RYB indicator, fuses, selector switch, main isolator switch, current and voltmeters etc.

The above control console shall be fully wired to operate 415V AC, 3 phase, 50Hz mains power supply along with power neutral and ground connection.

**12 SAFETY DEVICES:**

The furnace shall be provided with number of safety devices to protect the system and the operator from mal-function and possible operator's errors.

**12.1 OVERLOAD PROTECTION:**

The motor of the Rotary Vacuum Pump and Roots Pump shall be fitted with thermal overload protection facility to protect the motor from drawing excessive current due to overload.

**12.2 COOLING WATER LINES:**

Water lines for different systems of the furnace shall be taken from a central manifold distributing them into various circuits providing each with a control valve for independent operation.

Similarly the outlet water from different systems of the furnace shall be connected to a common outlet manifold. Water flow switches shall be provided on the outlet of each system so that the water flow rate of each of the system can be sensed and OK signal is given to operate respective power controls.

In case of water supply failure or reduced rate of water flow these switches should de-energize the electrical circuit and give alarm showing the status of the system through indicators.

**12.3 PRESSURE SWITCHES:**

Two pressure switches one in pneumatic line and the other in the argon gas line shall be connected to alert the operator by alarm in case pneumatic supply drops below the required operating pressure and to automatically stop argon pressure inside the furnace chamber when it reaches the operating pressure.

**12.4 PIRANI CONTROLLER (PART OF VACUUM GAUGE):**

Pirani vacuum controller shall be incorporated operating in the range of 0.5 to 1 x $10^{-3}$m.bar through a pirani gauge head. The vacuum controller shall sense the vacuum of respective places and energise the relay when the preset vacuum is reached.

**12.5 PRESSURE RELIEF VALVE:**

Pressure relief valve shall be provided in the chamber to protect the chamber from being exposed to high pressure while filling it with inert gas. This relief valve shall open and vent the extra gas to atmosphere in case the chamber pressure rises beyond the set value.

**12.6 OVER TEMPERATURE ALARM:**

Over Temperature controller is provided in the circuit, so that when temperature over-shoots from the set safety level, it cuts-off the power to furnace and gives over temperature alarm.

**13.0 SAFETY INTERLOCKS AND ALARM FOR THE FURNACE:**

a) During Power failure, all the vacuum valves shall close and all pumps and motors shall be de-energized.
b) Power supply to the hot zone shall be de-energized if the cooling water under reduced flow of water to the vacuum chamber or if the water flow fails.
c) In emergency, push button switches shall be provided to de-energize Rotary Pump, Close the vacuum valves and all gas inlets. This can be done by de-energizing power to the control circuit.
d) A pressure relief valve shall be provided on the water jackets to bypass the water to the drain if the pressure exceeds the preselected limits.
e) A Over temperature controller shall be provided in the system with a thermocouple/pyrometer. If the temperature exceeds the pre-selected limit, Furnace power shall be switched-off and an alarm shall be triggered.
f) A pressure switch shall be provided in the pneumatic line to alert the operator if the pressure falls below the preset value.
g) Safety critical interlocks shall be ensured during various modes of operation (manual/semi-automatic mode).

**14Design Document**

Detailed design drawing along with required technical design reports and instrumentation diagram should be submitted by party to VSSC for verification and approval. The party should modify the design as per recommendation given by VSSC review committee, and re-submit the design drawings for final approval**. Fabrication of items should be initiated by the party after formal approval from VSSC.**

**15. Pre-delivery Inspection**

a) VSSC Engineer shall carry out pre-delivery inspection at party's site before dispatching the items to VSSC. The party shall produce the following items during pre-delivery inspection at party's site (i) Test Certificates of Materials used for fabrication (ii) Verification of DP test reports of all weld joints (iii) Verification of bought-out items.

b) The Party has to intimate VSSC regarding readiness for pre-delivery inspection at party's site.

c) The party has to submit all test reports certificates during the pre-delivery inspection.

d) Conformance to specification with regard to components/subsystems shall be verified by the inspection team of VSSC.

**16.Acceptance Criteria**

The party has to demonstrate the performance of the system by operation trials such as:

a) All the designed safety interlocks shall be demonstrated.

b) All individual instruments like PID controller, thermocouples, vacuum gauges etc. shall be calibrated by accredited agencies with traceability to national standards and the calibration certificates are to be supplied along with the equipment.

c) The party shall demonstrate one process cycle during commissioning phase.

d) The equipment should be calibrated to show the requisites like temperature uniformity and temperature accuracy within limits as one time calibration after commissioning.

e) Party shall also submit budgetary offer for all essential spare items with make and specifications.

**17.Delivery, Transportation, Installation and commissioning**

a)Party has to demonstrate the full working of the system at VSSC including the performance of the subsystems.

b) The delivery of the total system to CMSE Stores, Vattiyoorkavu and installation and commissioning of the system at VSSC shall be completed within 6 months from the date of release of Purchase order.

c) The party shall be responsible for transportation of all items to CMSE Stores, Vattiyoorkavu along with necessary documents.

**18 Documents to be provided by the party while supplying the items**

• Layout drawing for accommodating the process equipment and auxiliaries

• Brochures/ Catalogues of all the equipments/instrumentation

• Material safety data sheets

• Comprehensive wiring/ cabling and interface schematics of whole system

• Operational and installation manual during the installation phase