Procurement of reactor for the chemical vapor deposition of epitaxial 4H and 6H silicon carbide

Background

The Naval Research Laboratory plans to conduct research into the epitaxial crystalline growth of 4H and 6H silicon carbide. These semiconductors are of interest to the US Navy as their properties are desirable for high voltage, high current solid state switches. This procurement is directed to obtaining a dual chamber chemical vapor deposition (CVD) reactor that will enable NRL to perform research and development in the growth of desired epitaxial device structures with those properties that are envisioned for the target electronic devices.

General Specifications

The reactor must consists of two reduced pressure, horizontal gas flow, hot-wall process tubes, i.e., the tubes must not be water-cooled, they must be serviced by a common source gas handling, vacuum, and RF heating systems, and capable of n and p-type doping. The process must be computer controlled and the reactor must have an integral safety system which must also communicate to the NRL laboratory.

Detailed Reactor Specifications

1. Process Tubes and susceptors (common): The cylindrical, quartz process tubes must be configured such that a tube will be optimized for current state of the art silicon carbide epitaxial growth technology (Process tube one) and that new processes will be developed in the other tube (Process tube two).
    a. Both tubes must be at least 250mm in diameter.
    b. Both process tubes must contain a coated, e.g., TiC, ultra pure graphite susceptor which is designed to contain the substrate carrier. The susceptor must be designed so that the wafer is simultaneously heated on the bottom and top to the same temperature.
    c. The process tubes must be capable of supporting 6 different kinds of interchangeable substrate carrier configurations, composed of coated ultra pure graphite: a single 4 inch substrate, a single 3 inch substrate, a single 2 inch substrate, 3 different 2 inch substrates, a single quarter-wafer sized 3 inch substrate and a single quarter-wafer sized 2 inch substrate. The source gas flows horizontally over the substrate.
    d. The susceptor and carrier must be surrounded by ultra pure graphite insulation – in this manner, the quartz process tube must not be subjected to high temperatures.
    e. The process tubes must be held in place by leak tight flanges that contain the gas delivery system on one end and the exhaust system on the other.
    f. In addition, the exhaust side must have a loading port that is also leak tight so that the substrate carrier can be introduced to the susceptor, by means of a transfer arm or fork, and placed appropriately in the susceptor.
    g. For both process tubes a quartz window must be mounted on one flange so that a pyrometer can view the graphite susceptor for temperature control purposes.
    h. Two pyrometers must be supplied with the equipment. To insure reproducibility of pyrometer readings, these devices must be mounted on an x-y positioning device.
2. Process tube one
    a. must be configured so that the substrate(s) in the interchangeable substrate carrier rotate at a uniform rate of between 1 and 100 rotations per minute.
        i. Rotation must be accomplished by a non-mechanical frictionless system, preferably through the mechanism of gas flow on and through coated graphite parts underlying the substrate.
        ii. The offeror shall provide a single 3 inch substrate carrier compatible with substrate rotation and a single quarter-wafer sized 3 inch substrate carrier.
    b. The graphite parts and hence the substrate, must be heated to a temperature up to 1600 C
3. For process tube two
    a. both flanges must also contain a quartz window that is mounted in a flange of at least 2 ¾ inch diameter such that the a laser beam passing through the center of the window can be

specularly reflected from the center of the wafer in the susceptor and emerge at the center of the window on the other flange.

   b. The graphite parts and hence the substrate, must be heated to a temperature up to 1600 C
   c. The offeror shall provide a single 2 inch substrate carrier and a single quarter-wafer sized 2 inch substrate carrier
   d. This tube must also be capable of being heated to 1800C, therefore a special customized susceptor kit, including a single 2 inch substrate carrier, with insulation suitable for high temperature processing (maximum 1800C) is also required.

4. The gas delivery system must be configured to deliver the chemical sources required for the process; these sources are:
   a. high-purity hydrogen as a carrier gas, silane (as a less than 2% gas mix in hydrogen), propane, nitrogen as an n-type dopant gas, trimethylaluminum as a p-type dopant gas, and Argon.
   b. In conjunction with the gases listed above, the system must also be capable of delivering HCl and Silicon tetrachloride gas sources. Therefore the process tubes and graphite parts described above, as well as the gas delivery system and exhaust gas pumping system (both to be described) must be compatible with these source gases.
   c. Also, the reactor must be configured with a separate, spare gas source line, blanked off.
   d. The requirements for the gas delivery system are that all gases can be directed into process tube one or two via the means of leak tight vent-run valving systems, whereby if the gas is not directed into a process tube, then it is directed into an exhaust line which leads to a connection to an existing fan/duct exhaust system in the laboratory.
   e. The entire gas delivery system and flanges must be constructed of 316L stainless steel.
   f. Each type of doping sources must consist of two separate gas handling systems.
      i. The first must be a standard handling system which directs the gas directly into the vent-run valving system.
      ii. The second must be a double dilution handling system whereby the dopant gas (either n-type or p-type) is mixed with a controlled amount of hydrogen and then a portion of the mix is then directed into the vent-run valving system; the balance must be directed away into an exhaust line.
   g. All input doping sources, i.e., nitrogen, HCl, silane (gas mix), etc., must also have in-line particle filter before the vent-run valving systems.
   h. Due to the required SiC epitaxial growth operating conditions, the following capabilities must be met. NRL facilities will supply feedstock hydrogen and argon to the machine. The reactor must have porting which will allow connection of the feedstock gases and direct them to purifiers contained within the machine and shall be supplied by the offeror (Pd-based for Hydrogen and getter-based for Argon) which must then be condition the feedstock gas so that state-of-the-art gas purity is obtained. The purifiers in the reactor must be capable of supplying these pure gases with maximum flows of 100 standard liters per minute for hydrogen and 25 standard liters per minute of argon to the vent-run valving systems. Each gas source line flow must be metered by mass flow controllers of the analog-type and metal-sealed variety.
   i. In addition, porting must be made available so that NRL can supply connections to nitrogen n-type dopants and an HCl pure gas source, which must then be metered by the machine via metal-sealed mass flow controllers (up to 1000 standard cubic centimeters per minute for nitrogen and up to 100 standard cubic centimeters per minute for HCl) and directed to the vent-run valving systems. In the case of the double dilution lines, additional metal-sealed, analog-type mass flow controllers are required for the hydrogen dilution gas.
   j. The reactor must contain internal connections for two trimethylaluminum bubblers and one Silicon tetrachloride bubler of a type commonly supplied by BOC gas or Praxair. These valved bubbler connections must consist of a hydrogen supply metered by metal-sealed mass flow controllers (100 standard cubic centimeters per minute typical), a by-pass line with isolation valving, and porting for leak detection to insure proper attachment of the bubblers.

5. The reactor must also have 3 thermostatic or constant temperature baths so that the trimethylaluminum and silicon tetrachloride bubblers' vapor pressure can be accurately controlled. The offeror shall supply one bubbler containing 100g of electronic grade trimethylaluminum.
6. Method of wafer heating
    a. Huettinger type RF generator of at least 40 KW power.
    b. NRL requires that the RF generator unit be configured to operate using 208V.
    c. The induction coil must be designed and positioned to meet the performance specifications and temperature control for each process tube to be accomplished by PID feedback using the pyrometer reading.
    d. A switch must be in-place so that process tube one or two can be RF heated.
7. The process tubes pressure must be controlled by vacuum pump- proportionating valve system whereby a pressure gauge supplies a PID signal to the proportionating valve.
    a. The vacuum pump must be a dry pump of the Ebara 25S type or type with similar or better operational capability such that the reactor can operate under growth conditions at a pressure of 100 to 300 mbar.
    b. A particle filter must be in-place between the process tubes and the proportionating valve.
    c. The exhaust must be ported such that NRL can connect it to an existing fan/duct exhaust system.
    d. The process tubes must be also pumped by a turbo molecular drag pump, ca. 500 liters per minute, with a backing pump to facilitate pump down during loadings and unloadings, and bake-out. This must also include the necessary pressure sensors and controllers.
    e. The pump connections must also include a CF port, blind capped, so that NRL may attach an RGA unit.
8. Reactor enclosure: The entire reactor must be appropriately enclosed in steel and/or polycarbonate and act as a primary barrier to leaks similar to standard gas cylinder storage cabinets; all doors must be sealed to prevent leaks to the outside.
    a. The enclosure must contain ports for connections to an existing NRL fan/duct system.
    b. The electronic controls for the reactor such as the programmable logic controller for the system control and interlock handling, DC supply, interface units, ground leakage protection, and safety devices must all be housed in a separate electronic cabinet.

Operational Control

1. The reactor must be computer controlled via a programmable logic controller that must also be interfaced to an IBM-compatible computer system; the computer system must have the following properties:
    a. PC-based Pentium 4 level computer system with
        i. DVD read/write drive,
        ii. a hard disk memory storage of at least 100 Gbytes,
        iii. system memory of at least 512 Mbytes,
        iv. and at least a 19 inch color monitor (flat panel preferred),
        v. running XP Professional
        vi. an ink-jet or color laser printer.
        vii. <u>SEPARATELY PRICED OPTION</u>: The computer system must be provided with an uninterruptible power supply that will protect the computer and send a signal to the reactor that will ensure proper system shutdown in the event of power failure
    b. Running on the computer system must be a custom designed, dedicated software package for control and monitoring the process,
        i. including data logging and recipe functions.
        ii. This software must allow complete control of each aspect of the growth process, including, temperature, gas delivery through valves and process pressure.
        iii. Feedback between the various sensors (temperature, pressure, mass flow and pumping speed) must be integral to the computer-based software control.

iv. The software must be capable of dynamic process adjustment and emergency shutdown in the event of any process parameter moving out of a predefined window.
v. Also integral to the software must be a safety monitoring that must be capable of triggering a safety shutdown when there is a major system failure. .
vi. The system must be network capable and allow for remote monitoring of all aspects of a growth process and remote notification of any alarm and shutdown that may occur.

c. The offeror shall supply a set of parameters that must run on the dedicated software program for performing epitaxial growth using the reactor to grow 4H SiC to the specifications contained herein.
d. SEPARATELY PRICED OPTION: The computer system must offer an option for interfacing at least two operator-provided process monitors that must be voltage-based, e.g., a voltage from a signal generated by the detector of a laser reflection or optical probe system, into the data-logging system.

Documentation:

Also included upon delivery must be at least one complete document set written in the English language containing: operation manual, service manual, sub-suppliers manuals of the various subsystems components, list of components, flowcharts, wiring diagrams, list of interlocks, factory settings, alarm system overview, details on obtaining on-line or telephone assistance, and spare parts list

Safety

1. The reactor must have an alarm and interlock system that is sensitive to the following parameters:
   a. hydrogen detection through manufacturer installed Draeger-type or equivalent detectors in the main ventilation duct
   b. as well as from NRL detectors that are connected to the system,
   c. process cell overpressure,
   d. argon supply pressure low,
   e. hydrogen supply pressure low,
   f. cabinet ventilation low,
   g. pressurized air low,
   h. cooling water low,
   i. and an emergency STOP button.
   j. In particular, the reactor must be configured to accept at least 6 incoming alarm conditions supplied by NRL facilities:
      i. House ventilation,
      ii. Fire,
      iii. toxic gas leak,
      iv. general gas supply,
      v. Scrubber malfunction,
      vi. and Hydrogen detected.
   k. The reactor must be configured to provide 5 outgoing alarms to NRL facilities:
      i. Hydrogen detected (inside the reactor container),
      ii. activate emergency ventilation,
      iii. control system healthy/not healthy,
      iv. alarm levels of internal sensors,
      v. and an extra connection.
   l. All alarms must be presented by a sound and light signal identifying the alarm on the process display monitor and logged into the data logging portion of the dedicated operating software.
   m. For each alarm system, a special sequence must be performed, incorporating delays where needed such that source valves are closed, the heating is shut off, the rotation is disabled, etc, dependent upon the severity of the alarm signal and also so that little or no damage is sustained by the reactor to the alarm condition.

Installation

The installation of the tool shall be performed by engineers supplied by the offeror. NRL shall be responsible for providing the offeror with access to the space where the tool will reside, all of the required facility utilities, which include power, ventilation and cooling water supplies that will be prepared to the offeror's specification. NRL shall also provide all of the required gas sources and these will be in-place as per the offeror's specifications; the NRL-supplied feedstock Hydrogen and Argon will be consistent with the specifications as described in **Detailed Reactor Specifications 4 h**.

1. In order to facilitate this, at the time of the contract award, the offeror shall provide NRL with all the facility's requirements that are necessary so that all of the required utilities will be in-place prior to the time of the installation.
2. The offeror shall provide personnel who will install the tool into the NRL facility. The offeror shall inform NRL at the time of award of material and personnel requirements for the installation process. The offeror shall also oversee the unpacking of the tool and subsequent installation. The offeror shall be responsible for ensuring that safety procedures are followed during this process.
3. Upon completion of the physical installation, the offeror supplied personnel shall ensure that all components of the system are properly operating. The offer shall then proceed to ensure that all of the specifications are met as detailed under **Performance** in a timely manner.
4. A start-up kit must be provided and composed of a spare quartz process tube, ultra pure graphite insulation, any and all quartz liners, an ultra pure coated graphite susceptor, one interchangeable satellite of coated ultra pure graphite, and one set of tools and accessories as deemed appropriate by the offeror.

Training

The offeror shall provide training for NRL personnel on the use, maintenance and basic repair procedures for the reactor. Information must be provided regarding the various components of the reactor system and their function. As part of the training NRL personnel must be taught how to grow a state of the art SiC epitaxial layer. This training must include all of the procedures including wafer preparation, insertion in to the system, bring the system up to operating temperatures, performing the growth and subsequent system cool down, venting and wafer removal. This training must be done under the supervision of offeror supplied personnel. The training must be done in two stages, the first being at the offeror's location during the construction phase of the reactor and later at the NRL after the installation.

The training must include both the hardware and software aspects of the system. NRL personnel must be shown how to configure a growth run, shown which sensor readings are key to a good run, how to interpret the variations in sensor readings and when to stop a run. In addition, NRL personnel must be shown how to add NRL provided monitoring such as optical reflection as described in the **Operational Control part 1 d** SEPARATELY PRICED OPTION.

The training program must be for 3 NRL personnel with a <u>SEPARATELY PRICED OPTION</u> for a 4^{th}.

Performance Requirements

1. The offeror shall be able to document the successful manufacture, installation, and operation of a chemical vapor deposition reactor of the type described in the **General Specifications** for the epitaxial growth of 4H SiC to the performance specifications herein.
2. The offeror shall demonstrate the following after installation at NRL:
    a. No leaks in the reactor tubing and seals as detected by a calibrated helium leak detector (supplied by NRL) down to 2 x 10-8 mbar.l/s;
    b. Base pressure in the range of 10-6 mbar;
    c. Process gas flows stable within ± 2% at process related flows, for a period of two hours;
    d. Process pressure 250 mbar ± 2 mbar with a hydrogen flow of approx. 40 standard liters per minute for a period of two hours;
    e. Susceptor temperature 1600°C ±2°C with a hydrogen flow of approx. 40 standard liters per minute for a period of two hours.
3. In addition, the offeror shall demonstrate the performance of the reactor by growing epitaxial layers having the following properties:

For process tube one, using a 1x 3 inch wafer configuration with the substrate being rotated during growth, the offeror shall (all measurements are to be performed with an edge exclusion of 5 mm for pre-cleaned, 3 inch, 8 degree off-oriented 4H-SiC wafers of highest commercial grade material):

1. Demonstrate a growth rate of 4H-SiC that is greater than 3.5 μm per hour by growing an undoped 4H-SiC n-type epilayer with a thickness of 4 μm or greater on an n+-substrate (NRL will supply an FTIR-based system to measure epilayer thickness).

2. Demonstrate layer thickness uniformity of an undoped 4H-SiC layer grown on a n+-substrate with thickness of 4 μm or greater (NRL will supply an FTIR-based system to map epilayer thickness) where the σ/mean is less than or equal to 2%.

3. Demonstrate a background doping concentration of an undoped 4H-SiC epilayer having thickness of 4 μm or greater grown on a n+-substrate that is less than 1×10^{15} cm^{-3} as measured by a 9 point cross pattern by the Capacitance-Voltage technique. (NRL will supply the metallization and CV measurement).

4. Demonstrate an n-type doping uniformity measured by a 9-point cross pattern measured by the Capacitance-Voltage technique where the σ/mean is less than or equal to 10% using a 4H-SiC epilayer of at least 4 μm thickness doped by nitrogen to greater than 8×10^{15} cm^{-3} grown on a n+-substrate. (NRL will supply the metallization and CV measurement).

5. Using an n-type 4H-SiC epilayer with thickness 4 μm or greater grown on a n+-substrate that there is no backside deposition visible, as monitored by optical inspection.

In addition, the offeror shall provide 10 3-inch 6H-SiC wafers that are n+-type and 3.5 degree off axis and 5 3-inch 4H-SiC wafers that are n+-type and 8 degree off axis of a high commercial grade to perform the demonstrations on process tube one.

For process tube two using a 1 x 2 inch configuration, the offeror shall (all measurements are to be performed with an edge exclusion of 5 mm for pre-cleaned, 2 inch, 8 degree off-oriented 4H-SiC wafers):

1. Demonstrate a growth rate of 4H-SiC that is greater than 3.5 μm per hour by growing an undoped 4H-SiC n-type epilayer with a thickness of 4 μm or greater on an n+-substrate (NRL will supply an FTIR-based system to measure epilayer thickness).

2. Demonstrate layer thickness uniformity of an undoped 4H-SiC layer grown on a n+-substrate with thickness of 4 μm or greater (NRL will supply an FTIR-based system to map epilayer thickness) where the σ/mean is less than or equal to 7%.

3. Demonstrate a background doping concentration of an undoped 4H-SiC epilayer having thickness of 4 mm or greater grown on a n+-substrate that is less than 5×10^{14} cm^{-3} as measured at the center of the wafer by the Capacitance-Voltage technique. (NRL will supply the metallization and CV measurement).

4. The offeror shall demonstrate with an NRL provided laser that a beam can be reflected from the central portion of the surface of the wafer and recovered on the opposite end. This demonstration must use the optical ports that are configured by the offeror.

Warrantee

The offeror shall offer the government at least the same warrantee terms, including offers of extended warrantees, offered to the general public in customary commercial practice. The warrantee terms and options must be included in the system price. And the period of the warrantee shall begin at the time of final acceptance.