Rapid Thermal Chemical Vapor Deposition Hybrid System

NRL requires a rapid thermal Chemical Vapor Deposition (CVD) hybrid system for the growth of heterostructures consisting of two dimensional (2D) materials, specifically including but not limited to graphene and hexagonal boron nitride (h-BN), with ferromagnetic metals. The system shall provide separate vacuum chambers for CVD growth and for electron beam deposition, with *in situ* vacuum transfer of the 2" diameter substrate wafer between chambers. The system shall provide for rapid changes of sample temperature over the range 25°C to 1100°C for CVD growth at operating pressures from 10^{-2} Torr to 760 Torr in gas atmospheres which include 20 Torr of methane or hydrogen. It shall be capable of growth upon 2" diameter substrate wafers and shall include four gas channels for CVD growth. The electron beam (e-beam) chamber shall provide for room temperature deposition at an operating pressure of 10^{-6} to $5x10^{-8}$ Torr of six separate materials onto the 2" substrate wafer – typical examples include iron, cobalt, nickel, permalloy, tantalum and gold. The system shall include a sample load lock for introduction of the 2" diameter substrate wafers from atmosphere and onto the sample manipulator in the e-beam chamber within 30 minutes while allowing a maximum e-beam chamber pressure of 1 mTorr (mTorr = 10^{-3} Torr). The CVD chamber may be an extension of the load lock chamber. The system shall include all instrumentation, power supplies, vacuum pumps, flanges, viewports and cabling to meet the performance specifications.

NRL shall provide all necessary electrical, compressed air, water and gas requirements, and a single new dry scroll pump (ISP-250C-SV, 10.6 CFM, 12 mTorr base pressure) for rough pumping.

General system features

- Stainless steel cold wall chamber
- Copper gasketed Conflat style flanges
- quartz window for viewing sample during growth and sample transfer
- quartz window with shutter for viewing e-beam hearths
- Fast digital PID sample temperature controller
- One purge gas line
- Four stainless steel process gas lines with independent digital mass flow controllers
- Pressure measurement
- Downstream pressure control for CVD growth processes
- Oil-free high vacuum turbo molecular pump and dry backing pump
- Floor standing system with wheels and feet with all equipment integrated within the system frame (not including roughing pump and sources for process gases, water, air and electrical power)
- Optical access for future in-situ optical monitoring of sample during growth

Vacuum and gauging

- E-beam chamber base pressure of $5x10^{-8}$ Torr or less
- Vacuum gauging with digital readout to monitor pressure from system base pressure to atmosphere

- At least one vacuum gauge in the CVD chamber shall be insensitive to gas composition over the range 1 mTorr to 760 Torr. An acceptable example of such a gauge is a capacitance manometer.
- Pumping system: the vacuum pumps must be oil free. The main high vacuum pump shall be a turbo molecular pump of at least 350 liter/sec speed (N_2) and capable of achieving a pressure in the e-beam chamber of 10^{-7} Torr after a dry nitrogen backfill to atmosphere within 12 hours, and a pressure of less than 10^{-6} Torr after sample transfer from the load lock within 3 hours.
- Vacuum valving: gate valves shall isolate the e-beam chamber from the vacuum pumps and the load lock.
- Venting the chamber: the system shall include a means to automatically vent the e-beam chamber or load lock independently at the user's discretion by backfilling the chamber or load lock with dry nitrogen. The dry nitrogen source will be supplied by NRL.
- Operating pressures during growth (typical ranges)
  CVD: 10 mTorr to 760 Torr
  E-beam: $1x10^{-6}$ to $5x10^{-8}$ Torr

Load lock / sample introduction and transfer

The system shall include a load lock chamber and sample transfer assembly attached to and isolated from the e-beam chamber by a gate valve for the introduction of 2" diameter substrate wafers. The load lock shall be pumped independently of the e-beam chamber through either separate pumps or appropriate isolation valves. It shall enable the user to introduce a new substrate wafer from atmosphere and load it onto the e-beam sample manipulator within 30 minutes while allowing the e-beam chamber pressure to remain below 1 mTorr. The load lock chamber may be an extension of the CVD chamber.

- The chamber shall be a stainless steel cold wall chamber with conflat style flanges. If it is an extension of the CVD growth chamber, the walls shall be water cooled.
- The chamber shall include the following ports
  - A port for introduction of samples, which shall have a hinged o-ring sealed flange providing access at least 6" in diameter to the sample area
  - A port for the CVD sample manipulator
  - A port for the transfer arm assembly
  - A port with a viewport for optical monitoring the sample surface during growth
  - an auxiliary port with a 4.5" or 6" OD flange for an optional upgrade to a turbo molecular pump.

Chemical Vapor Deposition (CVD) growth

Chamber: the chamber shall be a stainless steel water cooled cold wall chamber with conflat style flanges separated from the e-beam chamber by a gate valve. It may be an extension of the load lock chamber. If it is a separate chamber, it shall include the following ports:

- A port for the CVD sample manipulator

- A port with a viewport to facilitate sample transfer from the load lock arm onto the CVD growth manipulator
- A port with a viewport for optical monitoring of sample surface during growth
- an auxiliary port with a 4.5" or 6" OD flange for an optional upgrade to a turbo molecular pump.

Gas handling: the system shall provide for introduction of 4 separate gases either individually or any combination thereof into the growth chamber at a user selected flow rate for each gas and user selected total growth chamber pressure.

- Mechanism: electronically controlled mass flow controllers with individual digital readout
- Channels: a minimum of 4 independently controlled channels for 4 gases
- Gases: methane, hydrogen, argon, and borazine.
- Flow rates and accuracy: 1% of full scale accuracy and 1% of reading. Individual controllers will have full scale flow rates selected from 10 – 1000 sccm (specified at time of order)

Pressure control during growth: active downstream pressure control to maintain user selected growth chamber pressure for CVD growth for a user-selected gas flow process. Example: a graphene CVD growth process with 10 sccm CH4 and 100 sccm Ar/H2 at a total pressure of 10 Torr. An acceptable example of such downstream control is an adjustable throttle valve at the vacuum pump port with suitable feedback to the pressure management hardware and electronics.

Operating pressures during growth (typical):

CVD: 10 mTorr to 760 Torr

Atomic hydrogen

The system shall include capability to generate atomic hydrogen near the sample surface for pre-cleaning the growth substrate, facilitating growth of graphene, h-BN and heterostructures thereof with ferromagnetic metals, and etching the graphene and h-BN after growth.

Sample stage for CVD growth

Sample size: the sample stage shall hold substrates ranging in size from 1 cm x1 cm up to 2" in diameter. A typical substrate would be a Si(001) wafer. The sample stage shall securely hold the sample for heating as specified below.

Sample temperature: the system shall include a sample heater, power supply, temperature measurement device, and digital PID controller system to meet the following performance specifications. The sample heater shall be capable of meeting these specs from the system base pressure to atmosphere in a gas environment of up to 20 Torr of methane or hydrogen.

Range: 25 – 1100°C

Rate: 50°C/min over the range 100-900°C. The sample shall reach a temperature of 1000°C from 25°C within 30 minutes at a pressure of 1 mTorr

Uniformity: ± 5°C over the 2" diameter substrate wafer after a settle time of 10 minutes at the user-selected temperature

Accuracy: ± 5°C after a settle time of 10 minutes at the user-selected temperature

Stability: ± 5°C over a 30 minute period after a 10 minute settling time

Electron Beam Evaporation

The electron beam deposition chamber shall be separated from the CVD / load lock chamber by a gate valve to prevent cross contamination, and pumped by the main turbo-molecular pump. It shall include the following capabilities.

- the sample stage shall receive the 2" diameter sample substrate from the transfer mechanism and hold the sample for e-beam deposition. No heating or cooling is required. A typical substrate would be a 2" diameter Si(001) wafer. Three 2" diameter metal plates with retaining clips for holding smaller samples shall be included, and hold samples ranging in size from 1 cm x 1 cm up to 3 cm x 3 cm.
- The e-beam system shall include 6 independent sources with water cooled hearths with at least 7 cc source capacity and 6 kW power: an acceptable arrangement would be a 6-pocket rotary source
- Deposition rate at sample: 0.3 nanometer / second for iron
- rate monitor: a measurement instrument shall be included within the e-beam chamber to measure the e-beam deposition rate during deposition with a measurement range from 0.01 to 1 nanometer/sec and an accuracy of ±0.01 nm/sec. An acceptable instrument would be a quartz crystal microbalance.
- shutters: the system shall include 2 shutters: a sample shutter within 1" of the sample face which shields the sample from the e-beam hearth, and a shutter directly above the e-beam hearth so that the e-beam rate can be stabilized using the rate monitor at a user-selected rate before the sample shutter is opened for actual deposition on the sample. Both shutters shall be electro / pneumatically controlled.
- Control instrumentation: the system shall include the necessary power supplies and instrumentation to meet the above e-beam evaporation performance specs and control e-beam rate, e-beam shutter and sample shutter to deposit predetermined user-selected thickness of film deposited from e-beam source.
- Operating pressure during e-beam deposition (typical): $1x10^{-6}$ to $5x10^{-8}$ Torr
- The following specific conflat flanged ports shall be included
  - Two viewports to monitor the sample surface during e-beam deposition
  - A viewport to facilitate sample transfer from the load lock transfer arm onto the e-beam growth manipulator
  - A viewport with a shutter to see the e-beam hearth
- Additional conflat flanged ports shall be included as necessary to meet overall system specifications for vacuum, gauging, venting, pumping, etc

Computer interface

- all pumps, valves, mass flow controllers, gauging and other instrumentation shall include an electronic interface to enable future upgrade to computer control. Acceptable output interfaces are RS232, GPIB, USB, or Firewire.

Computer: No computer or software will be supplied with this procurement.

Installation and training
The vendor shall assemble and install the system at NRL and provide a hour training session of at least 4 hours to a group of 4 users.

Drawings: vendor shall submit drawings of proposed system design with bid illustrating compliance with specifications above, and full chamber drawings for approval by end user before fabrication.

Demonstrated growth performance and acceptance specs

- **Growth of graphene:** The system shall have demonstrated growth of graphene on copper foil and nickel foil to a thickness of 1-3 layers within a 30 minute growth time
- **Graphene quality:** The system shall have demonstrated growth of graphene on copper foil and/or nickel foil with a Raman "D" defect peak intensity less than 10% of the "G" peak intensity for graphene films 1 layer thick on copper and up to 1-10 layers thick on nickel. Raman measurements will be performed onsite by NRL using 488 or 532 nm laser excitation at room temperature.
- Process must guarantee generation of atomic hydrogen and C-H radicals for graphene film formation over 2 inch wafer.

Warranty : 1 year parts and labor

OPTIONS
The vendor is requested to provide pricing for the following options which may or may not be included in the order. Options should be available and pricing should be effective up to 6 months after system installation at NRL.

OPTION 1. Computer and Control software and hardware to provide the following functions:

- Simultaneous control of sample temperature, gas flow rates for all 4 gas channels, and operating pressure to accomplish the CVD growth of graphene films. For a given growth run stream, the control software shall provide 20 separate user-defined segments. In each segment, the user shall be able to specify sample

temperature, temperature ramp rates and soak times, system pressure and gas flow for each of the 4 gas channels.

- Deposition control: control e-beam rate, e-beam shutter and sample shutter to deposit predetermined user-selected thickness of film deposited from e-beam source.
- Automatic venting and pump down of growth chamber or load lock

OPTION 2. Turbo pump on load lock

- An additional turbo pump with at least a 50-80 liter/sec pumping speed (N2) and gate valve shall be mounted on the load lock to enable a base load lock pressure of 10^{-6} Torr within 6 hours of venting and pumping load lock.

OPTION 3. Additional mass flow controllers

- Two additional mass flow controllers (MFCs), control electronics and gas plumbing / valves from MFC to growth chamber.

OPTION 4. Two-color infrared (IR) pyrometer with LED digital display for measuring and monitoring sample and filament temperature through an optical fiber.