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Application Note 01: **Growth of CNT Forests**
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This application note describes a process for growing CNT forests using the SabreTube, using a catalyst-coated silicon substrate in a CVD atmosphere of $C_2H_4/H_2/He$. A similar procedure is described in related journal publications [1-3]; however, this note details the full process which has been tested using the commercially-available SabreTube.

This procedure assumes the user has the following equipment and materials:

- SabreTube desktop thermal processing system.
- SabreTube gas pre-heater, with power supply and controller.
- Gases: C_2H_4 (99.5+%), H_2 (99.999+%), and He (99.999+%).
- Appropriate gas flow meters/controllers (customer supplied and connected, Fig. 1).
- Silicon wafer growth substrates (thickness up to 1 mm), coated with 1/10/100 nm $Fe/Al_2O_3/SiO_2$. The SiO_2 is thermally-grown, and the thickness can exceed 100 nm. Fe/Al_2O_3 is deposited by e-beam evaporation, and the thicknesses should be within 10% of the specified values.
- Cap substrates, which are ideally single-side polished (SSP) Si_3N_4-coated Si wafers, but may also be SiO_2-coated Si, bare Si, or quartz.

Before operation, ensure that:

- All gas connections have been leak-checked.
- The heater substrate (1 cm wide) is installed and functioning properly.
- All surfaces which contact the reaction gases are clean and free of debris.

The procedure is as follows:

Substrate installation

1. Ensure power and gas flows to the SabreTube are off.
2. Remove the safety shield and retract the quartz tube.
3. Place a 1x1 cm sample (or desired size, not exceeding the width of the heater) of catalyst substrate on the heater, with the catalyst-coated side facing upward. Alternatively, a catalyst-coated heater may be installed.
4. Place a cap substrate on top of the catalyst substrate. The cap substrate should be the same size as the catalyst substrate, and ideally the non-polished side of the cap should contact the catalyst surface. The cap substrate acts to thermally-shield the catalyst from radiative losses, and to moderate flow of high-activity hydrocarbons to the catalyst. The configuration is shown in Fig. 2.
5. Close the quartz tube and install the safety shield.

CNT growth processing

1. With power to the substrate and pre-heater remaining OFF, flush the tube with 1000 sccm He, and wait for 3 minutes.
2. While maintaining the He flow, power ON the pre-heater, and begin heating the pre-heater to 1020 ^{o}C. The substrate should remain at room temperature.
3. After the pre-heater reaches the setpoint temperature (approximately 10 minutes), change the flow to 310/120 sccm H_2/He, and wait 2 minutes
4. Maintaining this flow, power ON the substrate and heat to 825 ^{o}C. Wait for two minutes.
5. Change the flow to 120/310/120 sccm C_2H_4/H_2/He, and wait for the desired growth time.
6. After the desired growth time, switch the gas flow to 1000 sccm He, and power OFF the substrate and pre-heater. Wait for five minutes.
7. Reduce the He flow to a trickle (~10 sccm), and wait until the chamber has cooled.
8. Remove the safety shield, open the chamber, and remove the substrate.

Note that the time/temperature sequences can be programmed into the pre-heater and substrate controllers if desired. The process sequence is summarized in Table 1.

Time [min]	**Flow [sccm]**	**T_{ph} [^{o}C]**	**$T_{substrate}$ [^{o}C]**
0	1000 He	RT	RT
3	1000 He	Ramp to 1020	RT
13 (approx.)	310/120 H_2/He	1020	Ramp to 825
15	310/120 H_2/He	1020	825
17	120/310/120 C_2H_4/H_2/He	1020	825
17 + t_{growth}	1000 He	OFF	OFF
22 + t_{growth}	~10 He	OFF	OFF

Table 1. Time, flow, and temperature sequence for CNT forest growth

Troubleshooting notes

1. The efficacy of growth strongly depends on characteristics of the catalyst substrate. The parameters specified here have been specifically tuned to the catalyst specifications mentioned in the introduction. In all cases, the growth rate and catalyst lifetime are very sensitive to the preheater temperature.
2. If efficacy of growth degrades over time, it may be necessary to replace the inner preheater tube and/or clean the components inside the chamber. The heater substrate may be cleaned by heating it to 900 ^{o}C in air for 1-5 minutes.
3. If the quartz reactor tube becomes dirty over time, it should be cleaned (e.g., using an acetone wipe), as this can contaminate the growth process and affect the IR temperature measurement.

Notes on Flow meters

1. Manual or electronic flow meters can be used.
2. Manual flow meters are typically “floating ball” type flow meters (sometimes called rotameters). Manual flow meters fluctuate slightly, and their output flow changes according to the input pressure (so the flow changes as the tank empties). Two common vendors are Swagelok and Matheson Trigas.
3. Electronic flow meters are typically “mass flow controllers” (MFCs). They are more expensive, but much more accurate and stable than manual flow meters. Two common vendors are Aalborg and MKS.

4. Both manual and electronic flow meters should be calibrated for the appropriate gas. Calibration can also be performed (with less accuracy) using air or N_2 and then applying a correction factor determined from a standard table.

[1] Hart AJ, Slocum AH. Flow-mediated nucleation and rapid growth of millimeter-scale aligned carbon nanotube structures from a thin-film catalyst. Journal of Physical Chemistry B. 2006;110:8250-7.
[2] Hart AJ, van Laake L, Slocum AH. Desktop growth of carbon nanotube monoliths with in situ optical Imaging. Small. 2007;3(5):772-7.
[3] van Laake LC, Hart AJ, Slocum AH. A suspended silicon platform heater for rapid thermal control of surface reactions with application to carbon nanotube synthesis. Review of Scientific Instruments. 2007;78:083901.

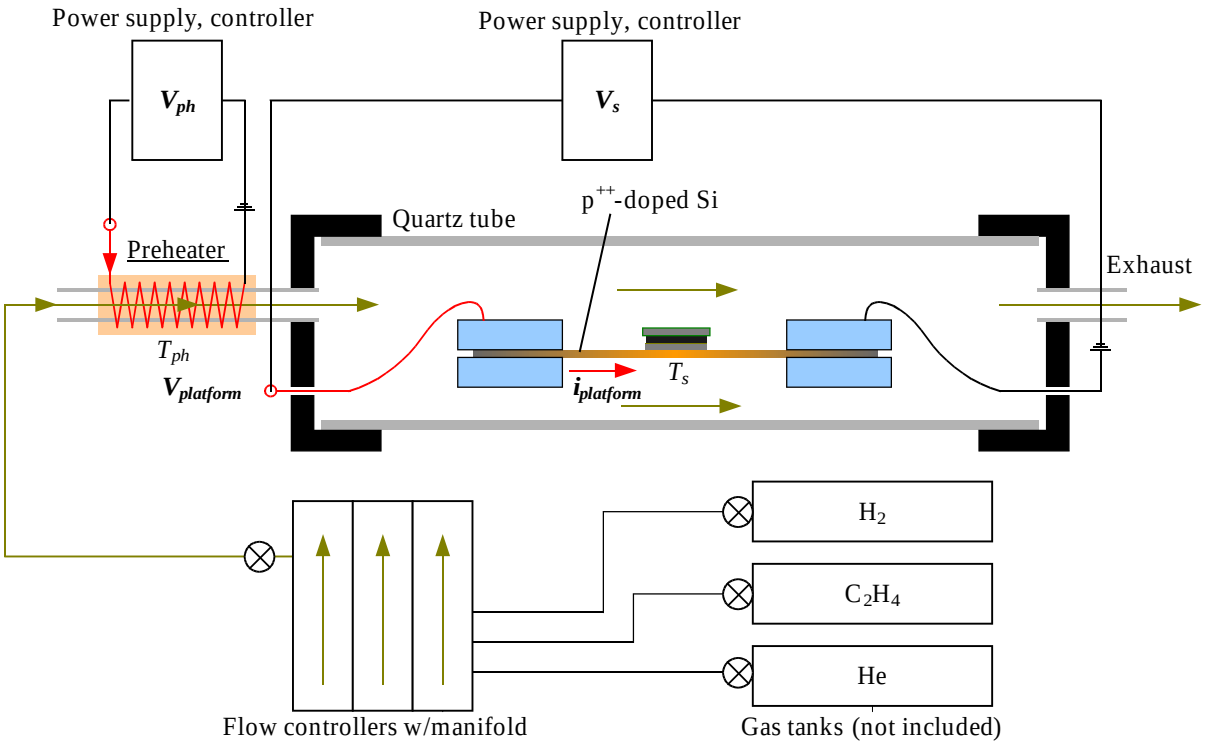

Figure 1. Simplified diagram of SabreTube setup with gas tanks, flowmeters, and power supplies.

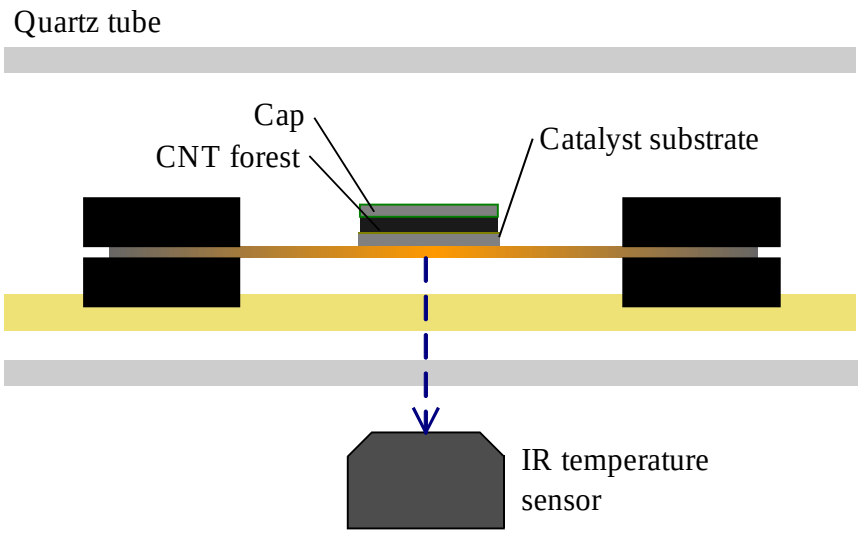

Figure 2. Heated platform and growth substrate configuration.