Wide-dynamic-range, fast-response CBr$_4$ doping system for molecular beam epitaxy

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Over the past several years, vertical-cavity surface-emitting lasers (VCSELs) have been the subject of intensive worldwide research due to their applications in optical interconnects and optical data links. To improve VCSEL performance, various bandgap-engineering schemes have been implemented in the distributed Bragg reflectors (DBRs) to simultaneously achieve low optical loss and low electrical resistance. This is especially important for p-DBRs due to higher free carrier absorption loss and lower mobility of holes. These bandgap-engineered DBRs usually have many different doping levels within a short DBR period. In addition, a very high doping level is needed to minimize the contact resistance. For molecular beam epitaxy (MBE), carbon doping using carbon tetrabromide (CBr$_4$) has been shown to produce material with better properties than doping with Beryllium, another commonly used p-dopant. However, most CBr$_4$ doping systems currently available cannot quickly and precisely switch between multiple doping levels, which is required for VCSEL growths. Here we report a custom-designed CBr$_4$ doping system that is suitable for growing sophisticated structures such as VCSELs.

All CBr$_4$ doping systems mainly perform two basic functions. One is to control the CBr$_4$ base vapor pressure, which is several hundred millitorr at room temperature, by either directly regulating the vapor pressure or indirectly regulating the CBr$_4$ temperature. The other is to reduce the CBr$_4$ vapor pressure, usually by several orders of magnitude, before injecting into the growth chamber to have the desired doping levels. Fig. 1 shows the schematic of our CBr$_4$ doping system, which consists of the thermoelectric cooler (TEC) system and parallel orifice valve (POV) system to perform these two functions.

The CBr$_4$ vapor pressure is temperature controlled by using four TECs [1]. One side of the TECs contacts the CBr$_4$ canister and the other side contacts a water-cooled copper block. The CBr$_4$ temperature is monitored using a thermocouple welded on the CBr$_4$ canister and controlled by a PID controller. A Baratron is used to monitor the vapor pressure. To characterize the TEC system, the CBr$_4$ temperature and vapor pressure were both recorded for three hours as shown in Fig. 2. The temperature was initially maintained at ~5°C, then was raised to ~20°C after one hour and lowered back to ~5°C after another hour. Once stabilized, the temperature and vapor pressure is nearly constant. In Region I, II, and III, the standard deviation for the temperature and vapor pressure is less than 0.1°C and 1% of the mean. The difference in vapor pressures for Region I and III is less than 2%, which indicates that the vapor pressure is fairly reproducible. From ~5°C to 20°C, the CBr$_4$ vapor pressure increases over 10 times. The rise time and fall time (10%-to-90%) for the vapor pressure is ~6.5 and ~1.5 min, respectively.

At a given CBr$_4$ temperature, the doping is controlled by the POV system, which currently has six different size orifices. These orifices, ranging from 50 to 250 µm in diameter, are used to reduce the CBr$_4$ vapor pressure injecting into the growth chamber. Upstream of each orifice is an air-operated pneumatic valve. By opening different combinations of pneumatic valves, the doping can be controlled digitally and reproducibly. Since the switching time for these valves is practically negligible, the doping can be changed rapidly. Ideally, if the conductance doubles for every other orifice, almost two orders of magnitude of the doping concentration ($2^4=64$) can be realized with six orifices. Combined with adjusting the CBr$_4$ temperature, three orders of magnitude of the doping concentration can be achieved in this system. To evaluate the POV system, a calibration sample which has eight layers with different valve opening combinations was grown, and Fig. 3 shows the doping profile measured by secondary ion mass spectrometry (SIMS). The CBr$_4$ temperature was maintained at ~5°C during the growth. The spike near the surface is due to the carbon contamination from the environment. The doping profile is relatively flat as shown in the figure. If the doping level for opening individual valve of C6, C5, and C4 are summed, the resulting doping level is $1.96\times10^{18}$ cm$^{-3}$, which is very close to the doping of $1.98\times10^{18}$ cm$^{-3}$ obtained from opening valves C4, C5, and C6 together. This implies that the doping contribution from each valve can indeed be added. The transient response is also evaluated by measuring the CBr$_4$ beam flux and shows that the typical transient time is less than 10 seconds.

In conclusion, a compact versatile CBr$_4$ doping system for MBE was designed and built. This system can achieve a wide doping range by using six different size orifices and adjusting the CBr$_4$ temperature. The doping is controlled by pneumatic valves and can be changed rapidly. These make the system suitable for growing complicated structures such as VCSELs.

Fig. 1: Schematics of the CBr₄ doping system

Fig. 2: The CBr₄ temperature and vapor pressure with the temperature setpoint changed between –5°C and 20°C

Fig. 3: Doping profile measured by SIMS with eight different valve combinations