Paper #T14

A Promising New Valved Source for Ga or In Evaporation

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ABSTRACT

A new valved source for evaporation of Ga or In has been demonstrated. It's performance in terms of controlling the group III flux has been shown to be very good to excellent in terms of response time (2-3 seconds), reproducibility (~1%), stability (~0.1%), and shut-off ratio (at least 30:1). In addition, this cell has produced GaAs, InGaAs, and InAlAs layers with material quality comparable to other state-of-the-art sources in terms of uniformity, surface morphology, background doping, carrier mobility, and PIN device performance. Finally, it has been demonstrated that the graphite-based material used for the crucible and valve in this source do not generate any extra carbon doping compared to the usual Pyrolytic Boron Nitride (PBN) based crucibles.

I. Introduction

MBE growers have long desired a Group III source whose flux is controlled by mechanical means, such as a valve, rather than by temperature changes. Ideally, such an effusion cell will reproducibly generate the same flux as a function of valve position, will allow the flux to be varied from the maximum desired value to as close to zero as possible, and will produce material quality that is at least as good as that from other state-of-the-art Group III sources. Aside from the ability to create compositionally graded layers, such a source would greatly increase system up time. This increase would be due both to conserving the material in the cell, and to the reduction (or even elimination) of Ga and In shutter sticking events. In addition, if the flux vs. valve position were sufficiently reproducible, daily calibration and start-up times for the MBE would be greatly reduced.

An early attempt at varying the flux mechanically did not make use of a valve, but rather mounted a standard effusion cell on a linear motion feed-through, and changed the flux by varying the source to substrate distance (1). This approach only produced In flux changes of about a factor of 2. A more recent attempt at mechanical flux variation involved using 2 heated PBN disks with aperture holes at the mouth of the cell. One of the disks could be rotated to allow the aperture holes in the disks to vary from completely aligned to not overlapping at all (2). This design achieved a Ga flux variation of a factor of 8. However, material quality suffered, both in terms of increased oval defect density, and decreased photoluminescence intensity.

We present here a new design of valved effusion cell for either Ga or In, which has produced good results both in terms of valve performance and material quality.

II. Effusion cell design

The Titan valved source consists of the crucible assembly, the valve mechanism, and the surrounding heaters, thermocouples, and heat shielding. The crucible, nozzle and valve assembly are made from a graphite-based material with a proprietary coating. This material has been previously demonstrated to provide advantages over Pyrolytic Boron Nitride (PBN) based crucibles. See Reference 3 for more information on this crucible design.

The crucible assembly consists of two parts: the material reservoir and the conical distribution nozzle. The valve is located between the reservoir and distribution nozzle. When closed, the orifice of the material reservoir becomes the sealing surface (valve seat) for the valve. As the valve opens, it moves up slightly into the cone of the distribution nozzle. When full open, the valve moves 200 mil, or 0.2 inches. The valve itself is a disk with two "ears" to which two push rods are attached. The rods penetrate the sides of the distribution nozzle through holes with a very close tolerance, so as to prevent any reflected group III material from reaching the heater filaments or thermocouples. The valve drive mechanism has a minimum number of linkages, and incorporates a spring assembly that allows some sealing force to be applied to the valve while protecting it from over torque conditions. The cell has two heater filaments and two thermocouples; one set each for the base (material reservoir) and for the tip (distribution nozzle). The cell used in this study was built on a 4.5 inch diameter flange. However, there is no reason that it cannot be scaled up to virtually any larger flange size. A schematic drawing of the valved Titan is show in Figure 1.

III. Experimental Conditions

The valved Titan was installed on a Veeco GENIII MBE system used primarily for the growth of GaAs, InGaAs, and InAlAs. The source flange of this system is vertical, with 12 source ports. The bottom 6 ports are upward looking, and are used for Group III materials, arranged in mirror symmetry to the vertical centerline of the source flange – i.e., the bottommost 2 ports (sharpest upward looking) have In sources, the next 2 ports up each have Ga, the shallowest upward looking 2 have Al. This means that when comparing In or Ga cells of different design, a true "apple to apples" comparison can be made in terms of system geometry. All of the Group III effusion cells were initially Veeco Sumo cells.

3 rounds of experiments were conducted. As_4 from a valved As source was used for all growths. In round 1, the valved Titan was loaded with In, and put in place of an In Sumo cell. The shutter of the remaining In Sumo cell jammed shortly after the valved Titan was installed, and no comparisons could be made. During this round we mainly studied valve performance (reproducibility, stability, and dynamic range). Uniformity was studied by using the valved Titan plus a Ga Sumo cell to grow a 1µm thick InGaAs layer nominally lattice matched on a 3-inch diameter InP substrate. A Bede D1 high-resolution x-ray diffractometer (HRXRD) was used to measure the composition.

In round 2, the valved Titan crucible and valve were replaced, it was loaded with Ga, and the cell was substituted for a Ga Sumo cell. During round 2 comparisons were made of GaAs uniformity and defect density between the mirror image Ga Sumo and Ga valved Titan cells. 3-inch diameter substrates were used. To measure uniformity, 2 wafers were grown with a 20 period superlattice of alternating 1,000Å GaAs and 100Å

AlAs layers. The structure was chosen to minimize the effect of any variations in the AlAs thickness on the superlattice period. The Bede D1 was used to acquire the superlattice satellite peaks, and Bede RADS Mercury modeling software was used to extract the GaAs and AlAs layer thicknesses across the wafer. In a separate experiment, defect density was measured with a Tencor Surfscan. For this experiment, 2 wafers were grown with 1µm of undoped GaAs, using a growth rate of 1µm/hr. Valve performance was also studied in this round.

In round 3, the initial crucible and valve used for In from round 1 were replaced in the valved Titan, heat shielding was added to the lip of the distribution nozzle, and the cell was put back into its initial In port. Also, before round 3, the growth chamber (including source flange) was cleaned, and repairs and preventive maintenance were done. Further assessment of valve performance was conducted. In addition, since the In Sumo cell was now working properly, extensive comparisons were made between the 2 In cells of material quality for InGaAs and InAlAs, grown lattice matched on InP. Surface morphology, background (unintentional) doping, and mobility were studied by growing a variety of InGaAs and InAlAs layers on 2-inch diameter substrates. Surface morphology in this round was assessed by using a Nomarski contrast microscope, at magnifications of 325X and 800X. Background doping and mobility were measured at room temperature with an Ecopia Hall Effect Measurement system. Finally, InGaAs/InAlAs PIN photodetector structures were grown with each In cell on 3-inch diameter substrates. The thickness of the intrinsic (undoped) InGaAs absorbing layer was 2µm, sandwiched between a 0.7µm N+ InAlAs layer on the bottom, and 0.2µm P+ InAlAs layer on top. When two thin digitally graded layers (between the InGaAs and the

InAlAs layers), and a thin InGaAs cap layer are included, the total thickness of the PIN layers is ~3.0µm. These wafers were used to compare uniformity, surface morphology, and dark currents. Dark currents were measured on unpassivated mesa diodes with a diameter of 100µm.

IV. Results

A. Valve and Flux Behavior

Flux was measured using a nude Bayard-Alpert ion gauge as a Beam Flux Monitor (BFM), and so is actually Beam Equivalent Pressure (BEP). All pressures reported in this paper are in Torr. The cell was run with almost all the power to the tip heater filament, which was controlled with the tip thermocouple. During rounds 1 and 2 (prior to adding a heat shield at the lip), it was found that large valve movements produced a slight fluctuation in the base temperature, which translated into flux variations. The flux stabilized more quickly if the base temperature controller was set to hold the base thermocouple a few degrees above the temperature it would equilibrate at with no base power. This resulted in the power to the base being only ~1-2% of the total power. Under these conditions, when moving the valve from closed to near full open, the flux would start out ~5% high, then stabilize in ~1 minute. After heat shielding was added to the tip of the cell (round 3), the stabilization time became about the same as the gauge response time of ~2-3 seconds.

Reproducibility of flux vs. valve position has proven to be excellent, and is within the accuracy of the BFM (~ ± 1%), as shown in figure 2. This data was collected during the third round of experiments, using the valved cell for In. The BEP with the valve full open ($2.0\text{X}10^{-7}$) gives a growth rate of InP on an InP substrate of ~0.65mL/s

(monolayers/second). Stability for a set valve position is also excellent, as shown by the growth of a 9µm thick InGaAs layer. This growth took 7.1 hours, and the composition varied by less than 0.1% In, as show in figure 3. Another 9µm thick InGaAs layer grown with the Sumo In cell had similarly good stability, as judged by the lack of compositional variation.

The flux range (change between full open and closed) found during round 1 was only about a factor of 10. However, this proved to be due to a nut which held one of the push rods not having been tightened properly, leading to the valve not sitting flush with the valve seat when closed. This was correcting for round 2 (using Ga), and the range became over 400. With valve full open producing a BEP of $2.0X10^{-7}$ (which produces a growth rate for GaAs on a GaAs substrate of 1mL/s), closing the valve produced a BEP of $4.5X10^{-10}$. During round 3 (switching back to In), the range became ~30. With valve full open producing a BEP of $2.05X10^{-7}$, closing the valve produced a BEP of $6.8X10^{-9}$. One possible explanation for the decrease in range between rounds 2 and 3 is that cell was subjected to a high temperature outgassing while empty, and the spring which provides some of the sealing force may have annealed and lost some of it strength. Another possibility is that there is some difference in the crucible/valve set which was used for Ga and the set that was used for In. However, a 30:1 flux range is still very good, and perfectly acceptable for many applications.

B. Uniformity

The 1µm thick InGaAs layer grown with the valved Titan in round 2 showed no change in composition from the center out to 35mm, meaning that any change in In flux across the wafer exactly matched that of the Ga cell. The Bede D1 is sensitive to changes

of In content of less than 0.1% In. The GaAs thickness was measured out to 32mm from the center of the superlattice wafers grown with the Sumo and valved Titan cells in round 2. The thickness variation for both was very close: the Sumo cell produced a 2.0% thickness drop, and the valved Titan produced a 1.7% drop. The PIN photodetector wafers grown in round 3 showed no change in InGaAs composition, and a 0.1% In rise for the InAlAs layers from the center out to 35mm for both wafers.

C. Surface Morphology

The Surfscan measurements of the 1μm thick GaAs layers grown in round 2 showed little difference between the 2 wafers. The valved Titan layer had 284 defects/cm^2, and the Sumo layer had 225 defect/cm^2. It should be pointed out that both of these growths were done relatively soon after opening the growth chamber to switch the valved cell from In to Ga. The valved Titan growth was the 3rd one and the Sumo growth was the 5th one after opening. The relatively high defect density for both wafers may well be due to insufficient chamber clean-up and/or insufficient Ga cell degassing. However, it is still clear that the valved Titan does not create significant extra defects compared to the Sumo. In fact, the slightly lower defect density for the Sumo growth may be due to a slight extra clean up of the growth chamber between the 3rd and the 5th growth.

The InAlAs and InGaAs layers grown during round 3, and examined with Nomarski contrast microscopy at magnifications of 325X and 800X, showed no difference between the Sumo and valved Titan In cells. The thickest layers grown were 9μm thick, undoped InGaAs layers. Both growths had very few medium to large defects, but a relatively high density of small (~1μm), very shallow defects. The PIN

photodetector wafers grown in round 3 (with a total thickness of ~3.0μm) also looked no different from each other when examined at these magnifications, and had an overall good morphology (no roughness and low defect density).

D. Background doping and mobility.

Hall effect measurements at room temperature on the 9μm thick, undoped InGaAs layers grown in round 3 produced the following results for carrier concentration and mobility (μ): the valved Titan produced $n = 5.3 \times 10^{14}$ carriers/cm^2, and $\mu = 5{,}000$ cm^2/V-sec, while the Sumo gave $n = 6.5 \times 10^{14}$ carriers/cm^2, and $\mu = 4{,}600$ cm^2/V-sec. These results are assuming a surface depletion of 1μm, and no depletion at the epi-layer/substrate (InGaAs/InP) interface. Even if the depletion depth were different from this assumption, it would only change the absolute carrier concentrations by about the same amount for both layers. The mobilities for these doping levels would be in the range of ~10,000 –12,000cm^2/V-sec if the compensation levels were low (4,5), indicating that there is significant extra scattering in the layers grown for this experiment. This could either be from impurities diffusing out from the Fe-doped InP substrates, or from p-type compensation during growth, such as C incorporation. However, judging from both the carrier type and concentration, and from the mobilities, the layer grown with the valved Titan does not have any more p-type compensation than the Sumo, and in fact has slightly less. It should also be noted that the Sumo layer was grown after the Titan layer (with a 1 week hiatus between the two), and so could have been expect to be slightly better than the Titan layer, which is opposite from the observed result. The most important conclusion from this comparison is that the graphite-based crucible of the valved Titan is not introducing extra carbon into the epitaxial layers.

E. PIN dark currents.

The dark currents were measured at a bias of 9V on test diodes with a diameter of 100µm. Previous experience at Picometrix with similar PIN devices has shown that at this bias, a dark current less than 50nA is very good, and anything less than 20nA is excellent. The wafer grown with the Sumo cell produced a dark current of 13nA, while that grown with the valved Titan gave 16nA.

V. Summary.

A new valved source for evaporation of Ga or In has been demonstrated. It's performance in terms of controlling the group III flux has been shown to be very good to excellent in terms of response time (2-3 seconds), reproducibility (~1%), stability (~0.1%), and shut-off ratio (at least 30:1). In addition, this cell has produced GaAs, InGaAs, and InAlAs layers with material quality comparable to other state-of-the-art sources in terms of uniformity, surface morphology, background doping, carrier mobility, and PIN device performance. Finally, it has been demonstrated that the graphite-based material used for the crucible and valve in this source do not generate any extra carbon doping compared to the usual Pyrolytic Boron Nitride (PBN) based crucibles.

Acknowledgements.

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Figures

1. Schematic of the valved Titan effusion cell.
2. In BEP vs. valve position from the 3rd round of experiments. The valve stated at 0 (closed), then was cycled 5 times between: 200, 60, 0, 200, 0, 60, and finally closed at the end. The 3 valve positions gave BEP's of $2X10^{-7}$ (corresponding to a growth rate of ~0.65mL/s InP on InP), $1X10^{-7}$, and $\sim 7X10^{-9}$ torr. The cell shutter was left open the entire time.
3. HRXRD of 9µm thick InGaAs layer growth with the In valved Titan. The InP substrate peak is barely visible as a shoulder on the left side of the InGaAs peak. If the composition of the layer had varied by as much as $\pm 0.1\%$, the peak would have been broadened out to the vertical dashed lines marked in the figure.

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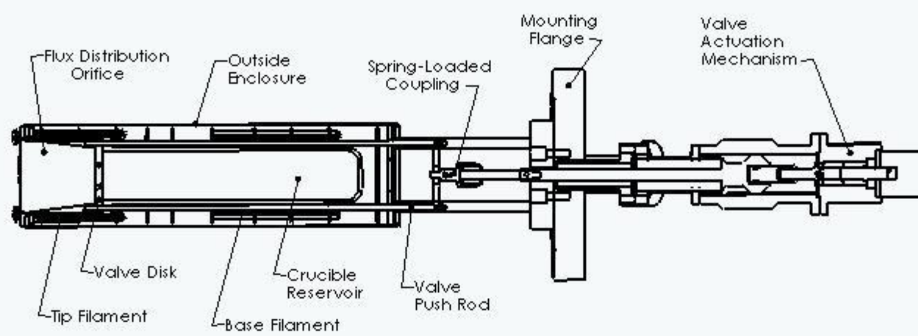
Figure 1. NAMBE paper #T14, R. N. Sacks and Craig Bichrt



Valved Effusion Cell

Figure 2. NAMBE paper #T14, R. N. Sacks and Craig Bichrt

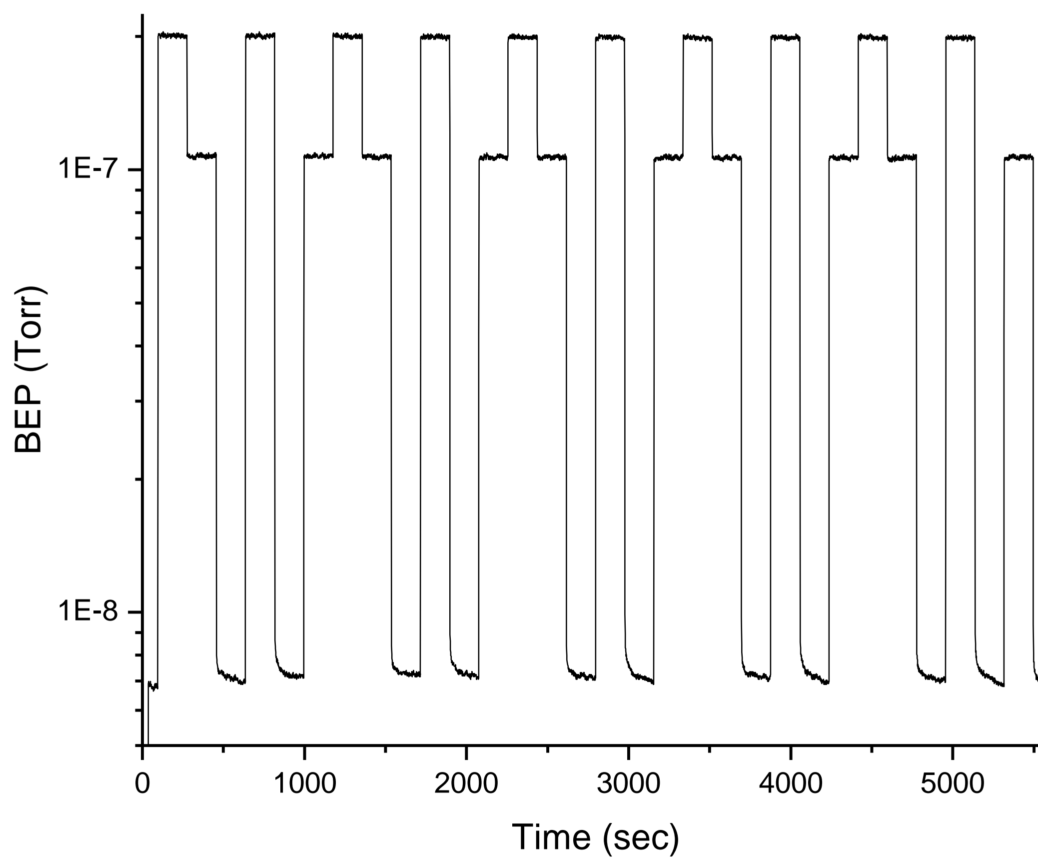
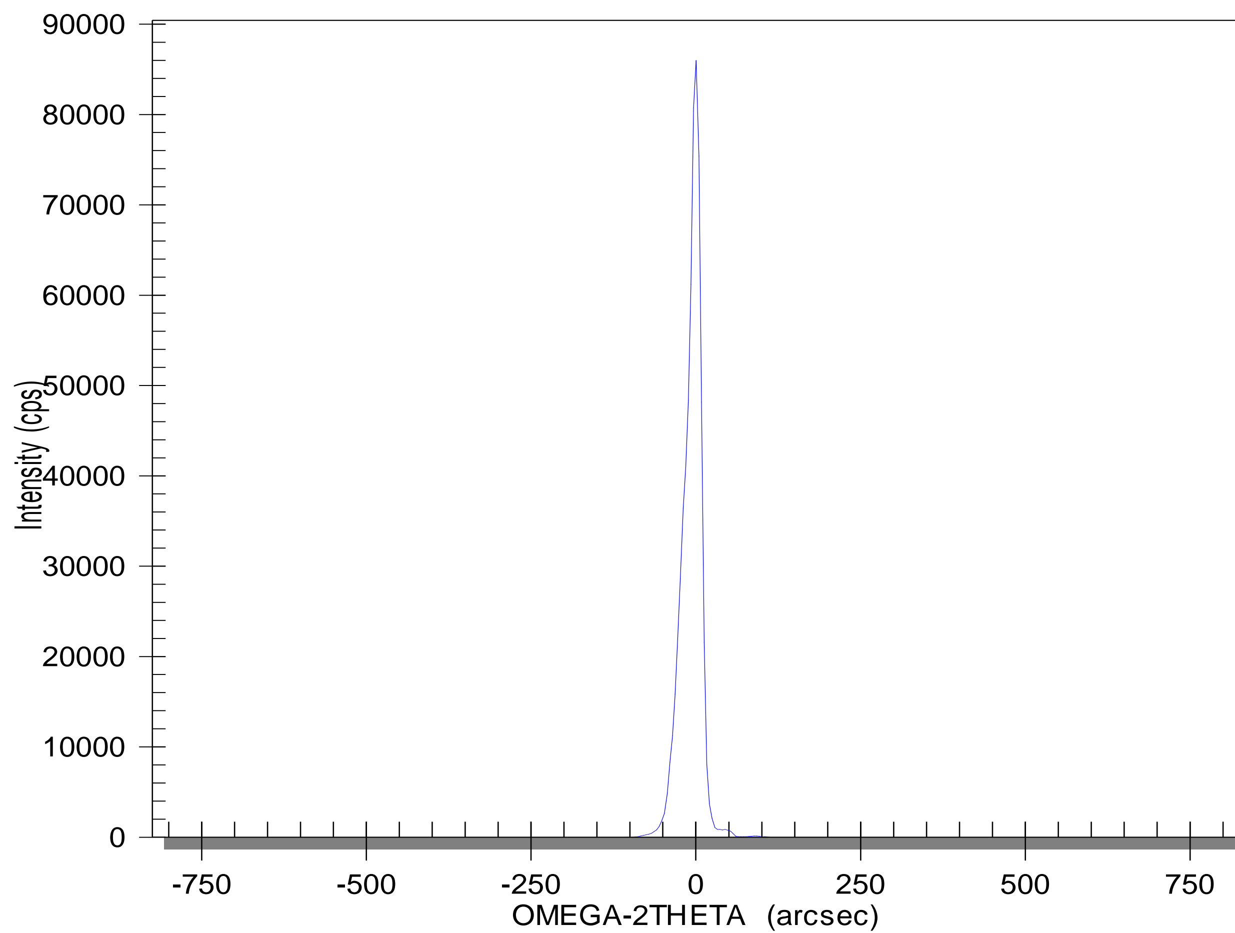


Figure 3 NAMBE paper #T14, R. N. Sacks and Craig Bichrt



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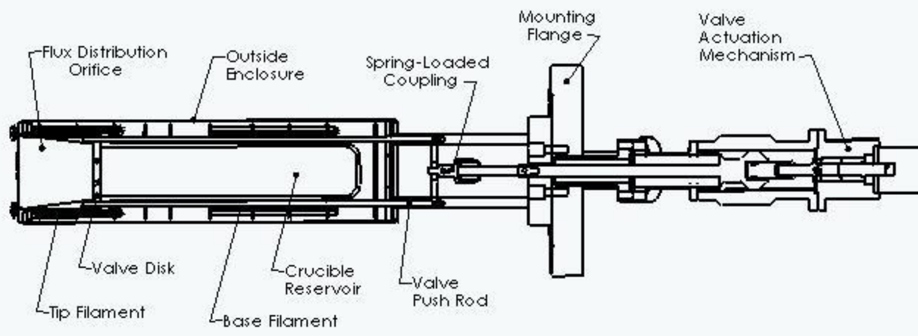

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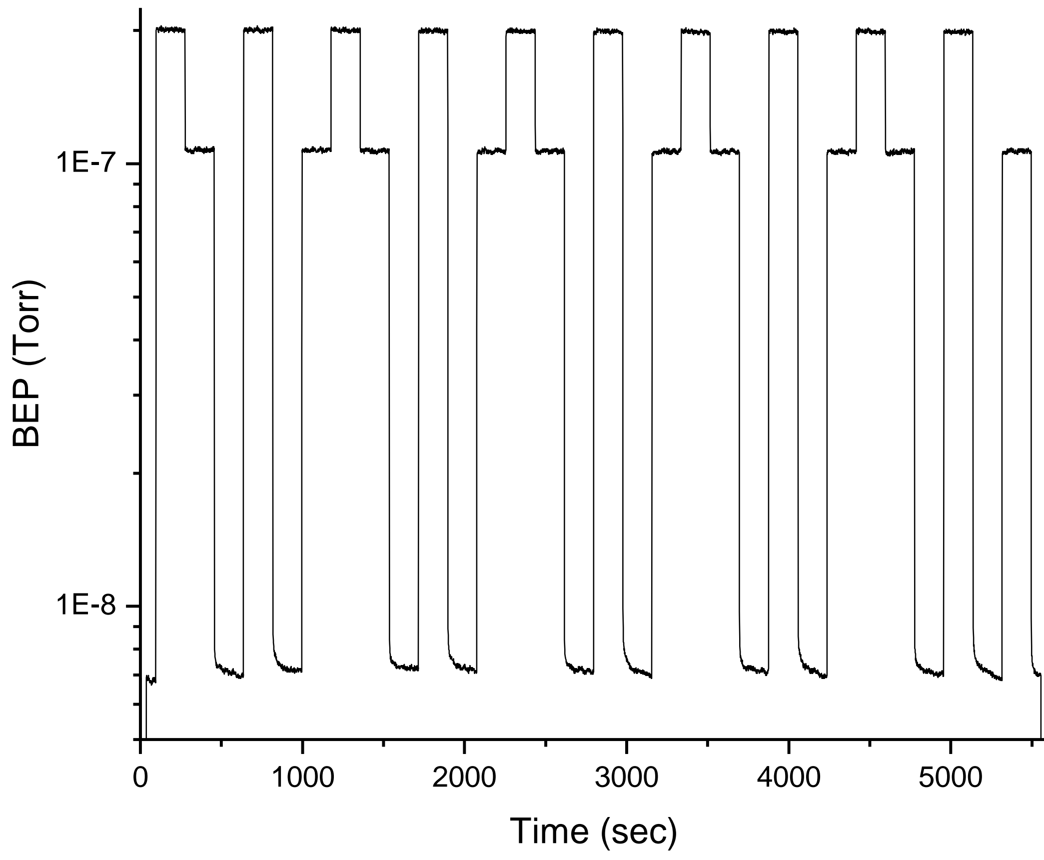

Figure 3 NAMBE paper #T14, R. N. Sacks and Craig Bichrt