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Growth of InGaAs structures using *in situ* electrochemically generated arsine

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AT&T Bell Laboratories, Murray Hill, New Jersey 07974

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The use, transportation, and storage of the hazardous gas, arsine, raise serious safety issues. Consequently, there is considerable interest in the generation of arsine on demand from less hazardous substances. We report the first use of *in situ* generated arsine for III-V epitaxy. The gas has been generated electrochemically at an arsenic cathode in an aqueous electrolyte and used to supply a hydride vapor phase epitaxy reactor. InGaAs/InP test structures were grown on InP substrates and were similar to comparison structures grown using tank arsine. Recessed-gate enhanced Schottky metal-semiconductor field-effect transistors were fabricated and exhibited well-behaved current-voltage characteristics.

Arsine (AsH_3) is commonly used as a source of arsenic in III-V epitaxy for photonic and electronic device applications. Owing to its extreme toxicity and high vapor pressure at ambient temperatures,[1,2] the use, transportation, and storage of this gas in significant quantities raise serious safety issues.[3–8] Increasing public concern and the possibility of more stringent environmental regulations[9–11] also raise questions concerning the handling and even the availability of this gas in the future. For these reasons there has been considerable interest in alternative sources,[2,12–14] in atmospheric pressure storage systems[15] and in the generation of arsine on demand from less hazardous substances.[16,17]

It has been shown recently[16] that arsine can be generated electrochemically with high current efficiency at an arsenic cathode in an aqueous electrolyte and this is the basis for a compact arsine generator system.[17] Using this generator, we have demonstrated for the first time the use of an *in situ* arsine source for III-V epitaxy. We report the growth of InGaAs/InP structures in this manner using a hydride vapor phase epitaxy (VPE) reactor and the fabrication of field-effect transistors (FETs).

The present study utilized a multichamber hydride VPE reactor which has been previously described.[18] Substrates consisted of (100) indium phosphide intentionally misoriented 3° towards the nearest [110] and were either *n* type (sulfur or tin doped) or semi-insulating (iron doped) as required. Prior to growth, each substrate was sequentially etched in H_2SO_4:H_2O_2:H_2O and 1% bromine in methanol. A phosphine partial pressure of approximately 5×10^{-5} atm was used to minimize thermal decomposition of the indium phosphide prior to growth[19]

The experimental configuration is depicted schematically in Fig. 1. Arsine is electrolytically generated at an arsenic electrode in the electrochemical reactor (A). The process essentially consists of the cathodic reduction of elemental arsenic in an aqueous potassium hydroxide solution according to the overall reaction[16]

$$As + 3H_2O + 3e^- = AsH_3 + 3OH^-.$$

The rate of generation is determined by the current applied from the power supply (B). The generator system is controlled by the microprocessor-based electronic module (C). The arsine is passed through a drying column (D) and into the reactor through mass flow controller MFC1 when the Nupro air-operated valves *V*1 and *V*3 are open. The system can also be purged through the charcoal scrubber by closing *V*3 and opening *V*4. Comparison runs using arsine from a commercial cylinder (2% in hydrogen) were made by closing *V*1 and opening *V*2.

InP/InGaAs/InP structures were grown using both cylinder arsine and *in situ* generated arsine. The nominal

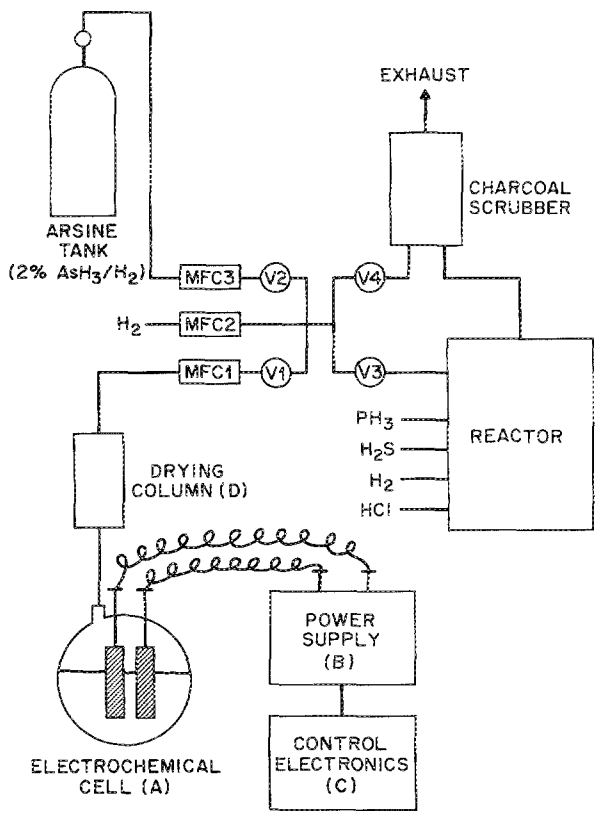

FIG. 1. Schematic of reactor configuration.

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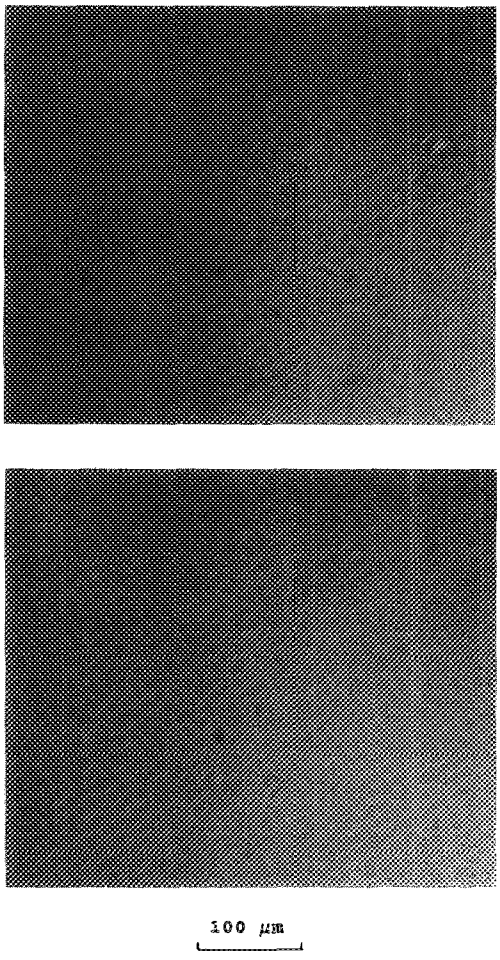

FIG. 2. Nomarski micrographs showing surface morphology of InGaAs/InP structure grown using the arsine generator (top) and tank arsine (bottom).

concentrations of reactants were similar in both cases: typically the III/V ratio was 5.5 and an In/Ga ratio of approximately 12.5 was required in each case to obtain the lattice-matched composition $In_{0.53}Ga_{0.47}As$. These values are typical for hydride VPE and no special changes were necessary to accommodate the arsine generator. After initial calibration runs to optimize composition, run-to-run variations in lattice mismatch were small ($<0.05\%$). The growth rate was typically 8.5 μm h^{-1}. The morphology obtained is shown in Fig. 2. Clearly the material grown using *in situ* generated arsine is comparable to that obtained from tank arsine. Typical background carrier concentration in both cases was 10^{16} cm^{-3} (n type) and is believed to be dominated by an as yet unidentified impurity from a source other than the arsine. It is planned to use the generator shortly on a higher purity system in order to better assess the inherent purity of the arsine. Intentional doping of the InGaAs layers was achieved using a H_2S source. Carrier concentrations in the 10^{16}–10^{18} cm^{-3} range could be routinely obtained.

Recessed-gate enhanced Schottky metal-semiconductor field-effect transistors (MESFETs) were fabricated using a four-layer growth sequence. First a 3000 Å undoped InP buffer layer was grown on a semi-insulating iron-doped InP substrate. An n-type (2×10^{17} cm^{-3}) InGaAs channel, nominally 1000 Å thick, was grown next followed by a 300 Å undoped InP barrier enhancement layer and a 1000 Å n-type (2×10^{17} cm^{-3}) InGaAs contact cap. Devices were mesa isolated by etching nonselectively through the conducting layers. The end point was determined by measuring the total sheet resistance. Source and drain contacts were formed using AuGe/Ni/Au evaporation, lift-off, and rapid thermal annealing. The gate region was defined lithographically, and the InGaAs cap layer was removed by a selective wet chemical etch. The InP barrier layer was then treated with a ruthenium-containing solution to enhance the barrier height.[20] The gate metal consisting of 2000 Å of gold was then evaporated and the pattern lifted off. Schottky contacts on undoped InP formed in this way have given barrier heights of 0.75–0.80 eV.

TABLE I. Material characteristics of InGaAs MESFET channel layers.

	Arsine generator	Tank arsine
Layer thickness (Å)	800	1200
Carrier concentration (10^{17} cm^{-3})	2.0	1.7
Hall effect measurements: 298 K		
Resistance (Ω/□)	658	459
Carrier density (10^{12} cm^{-2})	1.9	3.0
Mobility (cm^2/V s)	4880	4120
Hall effect measurements: 77 K		
Resistance (Ω/□)	372	338
Carrier density (10^{12} cm^{-2})	2.2	3.5
Mobility (cm^2/Vs)	7360	5250

Hall measurements were made on separate pieces of the same wafer by first selectively removing the cap and barrier layers and forming ohmic contacts on the InGaAs channel. Actual layer thicknesses were measured by selective etching and surface profilimetry. The results for room temperature and 77 K are shown in Table I. It is seen that the characteristics of the layer grown with *in situ* arsine are generally similar to those of the layer grown with tank arsine.

The direct current (dc) characteristics (drain current, I_D, versus drain voltage with gate bias as a parameter) for a 1.5 μm gate length and 100 μm width device are shown in Fig. 3. The numerical characterization is summarized in Table II. The devices show good saturation characteristics with low output conductance, clean pinch-off, and transconductance equivalent to heterojunction field-effect transistors (JFETS) of similar geometry.[21] Despite the relatively high channel doping, early gate-to-drain breakdown was not observed. Source resistance was somewhat high despite the quasi-self-aligned structure. This is primarily due to the undoped barrier layer and could be improved by raising the doping in the InGaAs cap or by doping the InP barrier itself or by a source/drain n^+ implant. There was no significant material-related difference between the devices grown using the arsine generator and those grown using tank arsine.

The arsine generator[17] used in this work is designed to interface with both atmospheric-pressure and low-pressure

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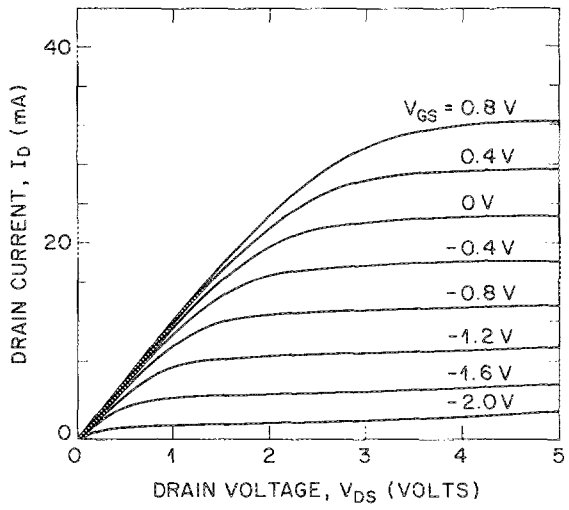

FIG. 3. Current-voltage characteristics of InGaAs MESFET grown using the arsine generator.

reactors. It is suitable not only for VPE but also for metalorganic chemical vapor deposition (MOCVD) and gas source molecular beam epitaxy (MBE) of III-V semiconductors and as a dopant source in silicon technology. Work on demonstrating MBE growth using the system is already in progress and it is planned to interface the system with a MOCVD reactor in the near future. Mass spectrometric measurements[16] indicate that the generator is capable of supplying stable and reproducible concentrations of arsine in hydrogen. While the growth of quaternary (InGaAsP) compositions was not examined in this preliminary study, the generator is designed to encompass such applications and InGaAsP growth experiments are planned as part of the ongoing study.

TABLE II. Characteristics of a typical MESFET grown using *in situ* generated arsine.

Gate length L_G	1.5 μm
Source resistance	5.7 Ω mm
Transconductance g_m	140 mS/mm
Saturation current I_{DSS}	240 mA/mm
Gate-to-source capacitance C_{GS}	2.5 pF/mm
Cutoff frequency f_t	8.9 GHz
Pinch-off voltage	3.0 V
Gate current at pinch-off	50 nA
Output conductance g_{out}	3.2 mS/mm
g_m/g_{out}	43

Both the arsine generator and liquid organoarsenic sources[2,12–14] such as *t*-butylarsine and trimethylarsenic possess the safety advantages of a low-pressure source. However, use of the arsine generator has the additional advantage that it involves no change from conventional process chemistry. A fully automated version of the generator is now available commercially.[22]

In summary, III-V epitaxy using an electrochemical *in situ* arsine source has been demonstrated for the first time. Enhanced Schottky InGaAs MESFETs were fabricated and exhibited good characteristics. No significant difference in material growth or properties or device charcteristics between *in situ* generated arsine and tank arsine was observed.

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