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Growth of extremely uniform layers by rotating substrate holder with molecular beam epitaxy for applications to electro-optic and microwave devices

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Integrated optics and integrated microwave circuits require extremely uniform epitaxial layer thicknesses, composition profiles, and doping profiles. With a sample rotating mechanism, molecular beam epitaxy can for the first time prepare GaAs and Al, Ga1-x As layers with thickness variation of less than 1% over a lateral dimension of 5 cm. The variation of AlAs mole fraction of the AlxGa1-x As over a 10-cm² area was less than 0.4%. The variation of the "pinch-off" voltage for a field effect transistor structure was less than 1.4% over a 10-cm² wafer. These results represent the most uniform epitaxial layers ever prepared with any crystal growth technology.

PACS numbers: 81.15.Ef, 73.60.Fw, 72.80.Ey, 81.10.Bk

The development of microelectronics depends in large measure on advancement in the preparation of thin epitaxial semiconducting layers. As the device becomes more miniaturized, the requirements for the film material become more stringent. Many devices require abrupt doping profiles,1-3 precise film thicknesses,4-7 and excellent surface morphology in order to be compatible with fine-line lithography. The prerequisite for the development of integrated optics is the availability of extremely uniform multilayered structures so that the mesa-etching process can be performed in a controllable manner. Similarly, for the fabrication of integrated microwave circuits, one requires extreme uniformity in doping profiles in addition to the uniformity in layer thickness so that all the identical devices will perform identically with the same biasing voltage. Even for discrete devices, uniformity is extremely important for the yield in fabrication.

In the past, although molecular beam epitaxy (MBE) (Ref. 8) can prepare epitaxial layers in atomic dimensions, the uniformity of the layer over a large area was limited by the inherent intensity profile of the molecular beam at the substrate surface. The profile may follow a cosine distribution or a modified intensity9 depending on the effusion cell geometry. We have overcome this difficulty, and this work describes the excellent uniformity of the growth of GaAs and Al, Ga1-x As epitaxial layers with thickness variations less than 1% over a wafer 5 cm in diameter using a rotating substrate holder in the MBE system.10

Figure 1 shows the top view of the MBE system. The substrate holder can accommodate wafers 5 cm in diameter, and it can rotate continuously at a speed of from 0.1 to 5 rpm. The results reported in this letter were obtained with a rotating speed of 2 rpm. All the effusion cells are mounted between 5 and 32° from the horizontal plane. They have apertures 2.5 cm in diameter and are located 12 cm from the substrate. A liquid-nitrogen-cooled shroud is used to enclose the entire growth area in order to minimize the residual wa-

ter vapor and carbon-containing gases in the vacuum chamber during epitaxy.

Three separate experiments were performed to evaluate the uniformity of the epitaxial layer: The first measured the cleaved cross section of the GaAs and Al, Ga1-x As layers with a Normarski interference microscope after they were stained in a solution composed of HNO₃ : H₂O = 1 : 3. This measurement will determine the uniformity in growth rates. The second measured the wavelength of the photoluminescent peaks of Al, Ga1-x As layers across the entire wafer in order to determine the uniformity in chemical composition. The third measured the distribution of the carrier concentration and the pinch-off voltage of a thin n-type layer grown on a semi-insulating substrate. This will determine the uniformity of impurity incorporation and the feasibility for application to integrated circuits.

The sample preparation is similar to that described earlier.5 The epitaxial growth was carried out at 580 °C after a

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FIG. 1. Schematic of the MBE system viewed from the top. The rotating sample holder has a variable speed from 0.1 to 5 rpm.
flash desorption of the surface oxide at 620 °C. The Ga effusion cell was heated to 1060 °C to give a growth rate of 2.25 μm/h. The As effusion cell was heated to 350 °C to permit growth under an As-stabilized condition,\textsuperscript{11} and the As background pressure in the growth chamber during epitaxy was 6 \times 10^{-11} Torr. In the case of growing Al\textsubscript{0.3}Ga\textsubscript{0.7}As, the additional Al effusion cell was heated to 1140 °C for a growth rate of 3.22 μm/h of Al\textsubscript{0.3}Ga\textsubscript{0.7}As.

The thickness distribution across a 5-cm wafer showed a variation of larger than 8% when the substrate was not rotated and the variation reduced to less than 1% when the substrate was rotated with a speed of 2 rpm. Figure 2 shows a variation of larger than 8% when the substrate was not rotated and the variation reduced to less than 1% when the substrate was rotated with a speed of 2 rpm. This may be converted to variation of the AlAs mole fraction of 0.4%.

Another critical evaluation of the uniformity is to study the impurity incorporation characteristics. An undoped Al\textsubscript{0.3}Ga\textsubscript{0.7}As 2-μm-thick layer was first grown as a buffer layer on a Cr-doped semi-insulating substrate followed by the growth of a Sn-doped n-type GaAs 6000-Å-thick (metallurgic thickness).\textsuperscript{12} Schottky-barrier diodes were formed over the wafer by evaporating Au dots through a mask with opening areas of 4 \times 10^{-7} cm\textsuperscript{2}. This permits the assessment of the net donor concentration, variation with distance both in the film growth direction and the lateral direction on the wafer, and the variation in pinch-off voltage. The pinch-off voltage referred to in this letter is defined as the voltage required to deplete the ionized donors to a value one order of magnitude below the doping level when measured with a doping profiler.\textsuperscript{13}

The depletion width \(l\) may be expressed as,\textsuperscript{14}

\[
I = \left[\frac{2e}{nq} (V_{bi} - V - kT/q)\right]^{1/2},
\]

where \(\varepsilon\) is the permittivity of the semiconductor, \(q\) is the electron charge, \(n\) is the donor impurity, \(V_{bi}\) is the built-in potential, and \(V\) is the bias voltage. It is clear from the above expression that the bias voltage used to pinch off the conducting layer is proportional to \(nl^2\). In other words, the distribution of the pinch-off voltage over the wafer is a measure of the variation of the product of the impurity concentration and the layer thickness squared. This parameter determines the yield in fabrication of the field effect transistor, which is one of the most versatile microwave devices because it can be used in a circuit of a mixer, oscillator, low noise amplifier, and high-power generator. Most recently, it is used in integrated circuits as a high-speed switching element. As we mentioned earlier, uniformity becomes even more important when integration of these devices on one chip is required.

Figure 3 shows five superimposed doping profiles measured over a 4-cm wafer. The reverse bias voltage required to deplete the layer where the net donor concentration decreased from 5 \times 10^{16} to 5 \times 10^{15} cm\textsuperscript{-3} was about 5 V. The variation of this pinch-off voltage is from 4.96 to 5.03 V over the entire wafer. This represents a variation in the \(nl^2\) of about 1.4% over a 4-cm wafer. The pinch-off voltage distribution measured in this manner actually represents the upper limit of the variation in \(nl^2\) because the performance of the ohmic contacts used in this measurement may not be identical. The ohmic contacts were formed by electric arching of five Au-2% Sn wires adjacent to the Schottky-barrier dots on the surface of the substrate. The uniformity of the net donor concentration over the wafer is illustrated by the coincidence of these traces in Fig. 3. The electrical thickness defined earlier\textsuperscript{12} as where the donor concentration decreased by a factor of 10 when measured with a doping profiler is 4750 Å. Results on this uniform layer suggest that the elec-
Photovoltaic detectors in SnS produced by Sb$^+$ ion implantation

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Np junction photovoltaic detectors in the p-type single-crystal SnS have been produced by Sb$^+$ ion implantation. Current-voltage and capacitance-voltage characteristics have been measured at 77 and 295 K. At 77 K, diodes had zero-bias resistance area products of $1.5 \times 10^3 \Omega \cdot \text{cm}^2$. The spectral response has been measured at 77 and 295 K. At 295 K, the peak responsivity was at a wavelength of 0.76 $\mu$m. At 77 K, the peak detectivity, which occurred at 0.62 $\mu$m, was $1.3 \times 10^{11}$ cm Hz$^{1/2}$ W$^{-1}$ with a peak quantum efficiency of 44%.

PACS numbers: 72.40.+w, 61.70.Tm, 73.40.Lq, 72.80.Ey

In this letter, we present some results on photovoltaic SnS detectors obtained by Sb$^+$ ion implantation. The p-type single-crystals used in these experiments were grown by the Bridgman method.

The SnS crystal is characterized by an orthorhombic double-layered structure (which can be described as a deformed NaCl structure) resulting in easy cleavage planes (001). SnS is a semiconductor with an indirect energy gap of 1.08 and 1.11 eV at 295 and 77 K, respectively.$^1$–$^5$

The Bridgman samples were cleaved into slices (15 x 10 x 1 mm) with a (001) surface plane. This was confirmed by x-ray diffraction. At 295 K, the value of specific resistance was 0.23 $\Omega$ cm. Hall-effect measurements with a bar measuring 11 x 9 x 0.3 mm showed a p-type carrier concentration of about $5 \times 10^{17}$ cm$^{-3}$ at 295 K.

After mechanical polishing (using Al$_2$O$_3$ powder 0.1 $\mu$m), the samples were electrolytically etched.$^6$ Mechanical copper implantation masks, 0.1 mm thick, were used. Apertures with a 1-mm diameter were opened in the masks by...