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# STUDY OF GaInAsSb EPILAYER BY SCANNING ELECTRON ACOUSTIC MICROSCOPY

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Scanning electron acoustic microscopy (SEAM) is a new experimental tool. SEAM study has been performed on GaInAsSb epilayer, which was grown upon 2°-off (1 0 0) towards ⟨1 1 0⟩ GaSb substrate by metalorganic chemical vapour deposition (MOCVD). Comparison of scanning electron microscopy (SEM) and SEAM images show that the two techniques provide different information. SEAM images can give information on growth process of pyramidal hillocks during the deposition of GaInAsSb epilayer.

Keywords: A. semiconductors, B. surface and interface, C. grain boundaries.

## 1. INTRODUCTION

III–V ANTIMONIDE based compounds have several important applications, including optical communications employing fluoride-based fibres [1], lasers, radar exploiting atmospheric transmission windows, remote sensing of atmospheric gases and molecular spectroscopy. Since the quaternary GaInAsSb alloy system has a direct band gap adjustable in wavelength from 1.7 to 4.3 $\mu$m when grown lattice-matched on GaSb, InAs and InP substrate, it may provide the basis for emitters and detectors over this entire region.

Scanning electron acoustic microscopy (SEAM) was developed in 1990 [2] and has been mainly used in the last few years in the characterization of thermal, elastic and pyroelectric properties on a microscale resolution. The mechanism of the electron acoustic signal generated by SEAM is different for different kinds of materials. It has been successively reported as a new experimental tool for the study of polarization distribution, phase transitions, subgrain boundaries and domain structure in polar materials and non-destructive observation of internal phenomena in many other materials and devices. The present work is a preliminary study of the capabilities of SEAM in the characterization of the GaInAsSb alloy; some observations are briefly compared with SEM.

## 2. EPITAXIAL GROWTH

The GaInAsSb layer was grown on GaSb substrate by MOCVD using a conventional atmospheric pressure horizontal reactor. The sources of Ga, In, Sb and As were trimethylgallium (TMGa), trimethylindium (TMIn), trimethylantimony (TMSb) and arsine ($AsH_3$) diluted to 10% in hydrogen, respectively. TMGa, TMIn and TMSb were held at −14°C, 16°C and −10°C respectively by using temperature baths and carried by Pd-diffused hydrogen into the reactor. The substrates were *n*-type GaSb oriented 2°-off (1 0 0) towards ⟨1 1 0⟩. GaSb substrates were chemically polished by a solution of $NHO_3$: $HCl:CH_3COOH = 0.2:2:20$, before being put into the reactor. The growth temperature was 640°C. The III/V ratio is 0.759, TMGa/(TMGa + TMIn) ratio is 0.745, TMSb/(TMSb + $AsH_3$) ratio is 0.868. The growth time was 90 min.

## 3. SEAM EXPERIMENTAL

SEAM observations were performed in a modified KyKy-1000B microscope made by a China–U.S.A. joint-venture company [3]. It was constructed using conventional scanning electron microscopy to which several newly designed parts, a flexible plug-in beam blanking system, an opto-electric coupler and a spring

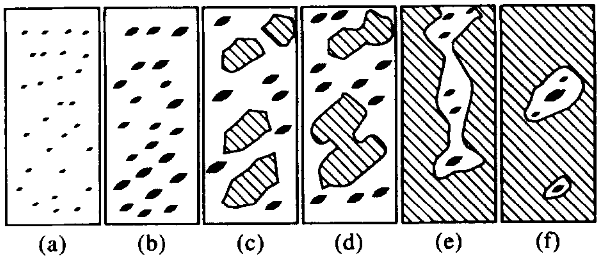



Fig. 1. Schematic diagram of the initial growth stage.

loaded and metal shielded PZT electron-acoustic signal detector were attached. It uses a chopped electron beam to generate an acoustic signal in the specimen surface due to the primary electron beam. This signal is usually detected by a piezoelectric transducer attached to the bottom surface of the sample and used to form a scanned image. This electron acoustic signal was detected at the reference frequency, $f$, generated by a function generator and amplified by a pre-amplifier installed inside the electron gun. It was then fed to a lock-in amplifier operating at frequency $f$ and a video amplifier for recording and displaying. The electron beam characteristics were chopping frequency from 30–500 KHz, duty ratio of 50%, acceleration voltage of 20–30 KV, and maximum beam current of $4 \times 10^{-6}$ A.

## 4. RESULTS AND DISCUSSION

Many experiments demonstrated quite clearly that the initial stages of epitaxial growth consisted of three-dimensional nuclei before there is enough deposit present to form a single monolayer covering the substrate surface [4, 5]. Nuclei shape and size varies from one system to another and also depends on the deposition conditions such as temperature and growth rate [6]. When two growing nuclei coalesced

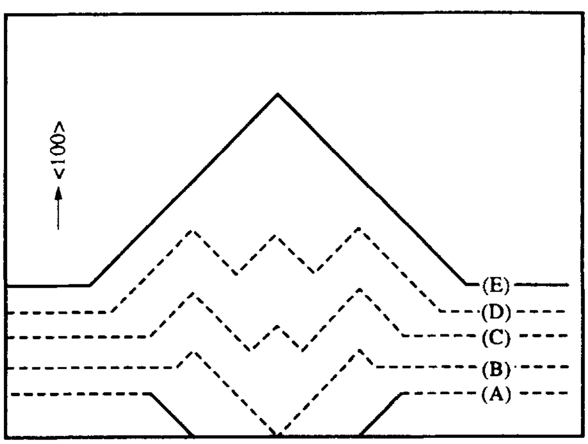
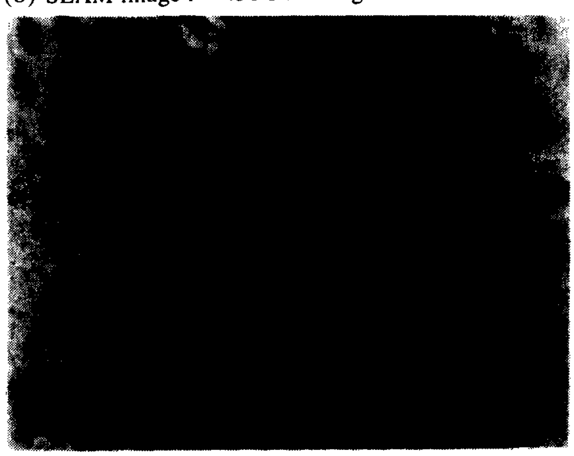



Fig. 2. Schematic diagram of the pyramidal growth sequences.

(a) SEM image

(b) SEAM image f = 456 KHz mag. x 200



(c) SEAM image f = 341 KHz mag. x 200

Fig. 3. SEM and SEAM images of a $Ga_{0.78}In_{0.22}As_{0.70}Sb_{0.80}$ epilayer.

they appeared to behave just like two liquid droplets, although the deposit temperature was below the melting point [6]. This mass transfer was explained in terms of a rapid surface diffusion process under the driving force of the surface energy minimization. The nuclei eventually joined together and formed a continuous deposit film, but there are many holes in the deposit film. These initial stages of growth are summarized schematically in Fig. 1. In [5], surface morphologies of MOCVD-growth strained-layer $In_{0.5}Ga_{0.5}As$ were observed by atomic force microscopy during initial stages of growth, the clear 3D islands and stripes between 3D islands were obtained in the surface morphologies. However any hole is surrounded by facets with higher index orientation and higher surface energy [7]. The implication is that growth occurs more rapidly on orientations with high surface energy and that large flat film surfaces with low energy orientations are left in their wake. These growth sequences are summarized schematically in Fig. 2.

Figure 3 shows the SEM image of a $Ga_{0.78}In_{0.22}As_{0.20}Sb_{0.80}$ epilayer and SEAM images of below the surface. The comparison of SEM and SEAM images shows that the two techniques provide different information. Figure 3(a) SEM image shows pyramidal hillocks paralleling to the $\langle 1\,1\,0 \rangle$ axis, Fig. 3(b) and (c) SEAM images show two morphologies of two growth stages. A reasonable hypotheses could be that GaInAsSb has a face centred cubic Bravais lattice. There is not piezoelectric coupling at $\langle 1\,0\,0 \rangle$ crystal orientation of substrate and epilayer [8]. If one assumes that the thermoelastic coupling is the main signal generation mechanism in GaInAsSb epilayer under the present observation conditions a rough estimation of the crystal slab thickness where the signal is generated can be made by calculating the value of the thermal decay length, $d$, at different frequencies of SEAM. By introducing the values of specific heat $C$ and thermal conductivity $K$ in the equation $d = (2K/f\rho C)^{0.5}$ ($\rho$ is the density and $f$ is the frequency) the values of 9.2 and 10.6 $\mu$m for $d$ at 456 and 341 KHz, respectively, are obtained. According to that, the image emerging at low frequencies would be situated deeper in the sample. In the usual route for growing GaInAsSb on top of (1 0 0) GaSb, the substrate is chosen with an offcut of 2°–3° in an attempt to reduce the density, or at least the height, of the pyramidal features, which are usually observed on the surface of GaInAsSb films. Figure 3(c) is an SEAM $f = 341$ kHz image corresponding to the area of Fig. 2(a), in the certain stage of the epitaxial growth, when there are many holes which are surrounded by facets with high index orientation. The size of every hole gradually decreases with increasing the thickness of the epitaxial growth. Figure 3(b) is an SEAM $f = 456$ KHz image corresponding to the area of Fig. 2(b); the size of the every hole in Fig. 3(b) is smaller than that in Fig. 3(c), which supports the former epitaxial growth model. Figure 3(a) corresponds to Fig. 2(c) and shows that the GaInAsSb film comprised elongated pyramidal subgrains whose major and minor axes are the directions $\langle 1\,1\,0 \rangle$ and $\langle 1\,\bar{1}\,0 \rangle$ respectively. In fact Fig. 3(c), (b) and (a) show the growth process of the pyramidal hillocks.

## 5. CONCLUSIONS

Electron-acoustic imaging by SEAM has definite advantages as compared with secondary electron imaging which was observed with SEM images. The experimental results shown in this paper indicate that SEAM reveals internal information of the specimen. The observation depth shown by SEAM is much deeper than that of SEM, which is especially useful for non-destructive analysis of devices for optics and electronics. The pyramidal hillocks growth process has been obtained.

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