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Electrochemical defect profiling for semiconductor heterostructures

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Abstract

A special selective electrochemical etching-based apparatus is presented which is appropriate for use in the *in situ* observation of the defects in heterostructures. The working of the set-up is demonstrated on InGaAs/GaAs(001) heteroepitaxial systems where the epitaxial layer thickness is above the critical layer thickness. With incremental layer removal, the depth profile of the dislocation density is mapped and the measured density of defects which is inversely proportional to the layer thickness is in agreement with the theoretical model.

1. Introduction

It is of great interest to investigate the defects induced by the lattice misfit in mismatched heteroepitaxial systems [1]. The defects in the layers can be observed by different methods. One common one used for investigating defect distributions is x-ray topography. Its main advantage is that the different kinds of defect can be investigated in a non-destructive way; the disadvantages are that the investigation is confined to just the lateral direction and the measurement is very time consuming. Another commonly used method is transmission electron microscopy. The defect distribution in the cross-sectional direction can be investigated in high resolution with this method; however, the investigation is again limited to one direction and the sample preparation is onerous and time consuming. We show in this work an electrochemical method-based apparatus, which is appropriate for use in simple and quick investigations of crystal defects in lateral and vertical directions.

Using electrolyte on the semiconductor surface, a controlled electrochemical layer removal can be carried out. Selective electrochemical etching is a method used for the investigation of defects in epitaxial layers and substrate material. One of the advantages of this method is that it is appropriate for obtaining defect distributions in lateral [2] and vertical [3] directions; another is that it does not share the disadvantages of the other methods: i.e. it is fast, with good resolution and the set-up is simple.

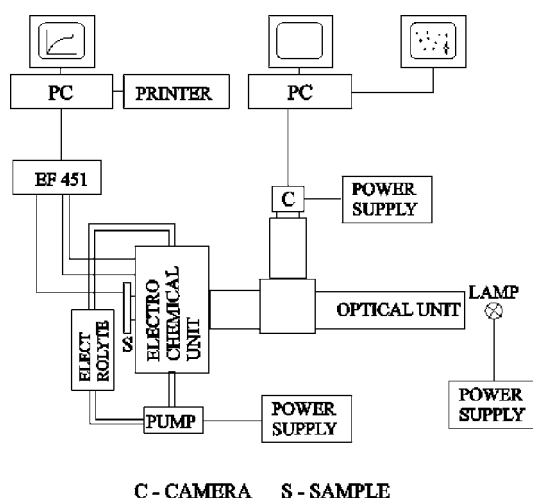


Figure 1. The block scheme of the electrochemical defect profiler.

In this study, we investigate the threading defects in $\text{In}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$ heterostructures grown by molecular beam epitaxy (MBE) by using selective electrochemical etching. The electrochemical defect profiling is carried out with the help of a newly developed electrochemical defect profiler. By means of incremental etching, we map the depth distribution of the defects. The results obtained are compared with a theoretical model.

2. Experimental details

2.1. The electrochemical defect profiler

Our novel electrochemical defect profiler consists of two main parts. One of them is the electrochemical part and the other is the optics. The block scheme of the whole set-up is shown in figure 1. The electrochemical part consists of a specially constructed electrochemical cell. The electrochemical cell is connected to a computer-controlled potentiostat, type Elektroflex EF451. The current can be measured in eight ranges from 2 A to 25 pA. The potentiostat is controlled entirely digitally and the status of the equipment is displayed by light-emitting diodes on the front panel. The control software (in Turbo Pascal) makes more working modes possible—such as cyclic voltammetry, impedance analysis and integration of current. The program automatically saves the measuring protocol and adds the corresponding setting parameters of the set-up.

One of the most important parts of the set-up is the specially designed electrochemical cell. The cell must be narrow; that is, the sample has to be close to the window to allow optical observation. The construction is also special because of the small cross-section. The cell is cut from a plexiglass block. The working electrode is the semiconductor sample, the counter-electrode is made of carbon and platinum wire and the reference electrode is a saturated calomel electrode. The sample is held against a rubber ring. The cross-section of the cell is shown in the lower part of figure 2.

Because the cell is narrow, a special method is needed for the removal of bubbles and cleaning electrolyte from the chamber. Because of the small size of the cell, the fresh electrolyte which is needed for the reaction is transported by circulation with the help of an attached pump.

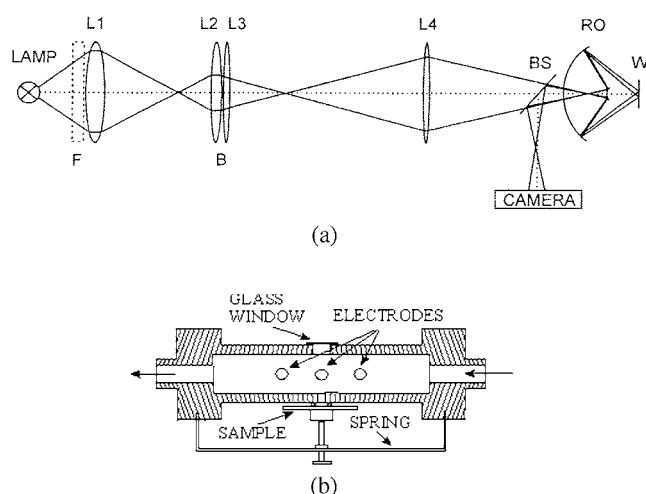


Figure 2. Lower part: the cross-section of the specially designed electrochemical cell. Upper part: a schematic drawing of the optical system, where L1–L4 are lenses, RO is the microscope objective and BS is the beam splitter.

The pump serves furthermore to effect (i) the removal of the bubbles which have become stuck in the sealing ring and (ii) the washing of the cell with distilled water.

The optical part consists of a lens system illuminating part, a microscope objective and a beam splitter to which is attached a camera. The case of the optical system was turned from an aluminium block. The setting of the optics can be carried out with the help of screws. The optical layout of the illuminating and viewing system is shown in the upper part of figure 2. The attached camera is connected to a computer with two monitors (one of them provides the menu of the image processing program and the other serves to display the surface investigated). The picture can be saved as a bit-map file (*.bmp). Either broadband or narrow-band illumination can be applied to the wafer. The spectral limiting medium of the broadband illumination is the liquid in the etching chamber: if it is water based, the transmission is between 0.3 and 1.4 μm , which is much narrower than that of the glass material of the lenses and that of the light source. About 10 mm depth of water absorbs the radiation above 1.4 μm drastically, and the transmission is only a fraction of one per cent. This spectral range is only slightly wider than the sensitivity range of the Si CCD camera. Within this spectral range a narrow band can be selected by inserting an interference filter, to investigate the effect of etching as a function of the illuminating wavelength.

The camera is a black-and-white one having 512×512 pixels. It is connected to a frame grabber that allows further experiments—for example, comparison of images and convolution. It permits simultaneous display of the buffer frame and an overlayable colour graphics plane, as well as management of programmer access. It has online preprocessing units through its look-up tables, located at the input and output of the video channels. A real-time conversion is provided by a flash converter having a resolution of eight bits.

2.2. Dislocation profile in the sample

The electrochemical layer removal is carried out under two different sets of conditions. One of them is the so-called polishing or non-selective conditions where the bias is chosen on the low-slope part of the polarization curve with optical stimulation. The other is selective etching,

which can be carried out with very small polarization and a lightly illuminated surface or at large polarization in darkness [2]. The crossed charges are integrated during the etching, so the removed layer thickness was calculated from Faraday's law, where the parameters entering are the physical (material density, valence number etc) and geometrical data of the system. The etched depth is controlled by an alpha-step profiler.

The (001)-oriented GaAs wafers used in the experiment were Zn doped ($p = 4 \times 10^{18} \text{ cm}^{-3}$) and the etch pit density (EPD) was $<5 \times 10^4 \text{ cm}^{-2}$. The In content in the MBE-grown layer was 30% and the layer thickness was $1.2 \mu\text{m}$. The sample preparation is detailed in [4].

3. Results and discussion

Using electrochemical etching, we found that the undoped layers were slightly of p type. The Matthews–Blakeslee (equilibrium) critical layer thickness for the misfit-generating dislocation [1] is about 8 nm for the sample ($\text{In}_{0.3}\text{Ga}_{0.7}\text{As}/\text{GaAs}$; lattice misfit $f = 0.024$); that is to say, our layer is much thicker than the critical layer thickness.

The etching parameters (e.g. bias voltage) were optimized by using a bare GaAs substrate. According to our earlier experiments, 10% NH_4OH is a suitable aqueous electrolyte for defect etching [5, 6]. Besides the type of electrolyte, the surface morphology depends on the bias voltage too. After etching, the surface is well structured when an anodic bias larger than 0.5 V (defect etch conditions) is applied. At lower bias voltage, the surface remains mirror-like (polish etch conditions). The etching current, when the bias ranges between 0.5 and 0.8 V, is stable, i.e., increases only slowly with depth or time. The etching current is large at higher doping in this bias range, and the etch rate is low. In this range we can remove thick layers and detect the location of the etched surface relative to the interface or substrate. For anodic bias larger than 1 V, the etching current becomes unstable and increases strongly with time/depth. This etching current range for the undoped layer is larger than that for the bare substrate. The slope of the I – V characteristic of the epitaxial layer above 0.5 V bias is larger than the slope of that of the substrate. This can be explained by the presence of defects. At higher EPD, the current variation versus time/depth is steeper, because at higher bias voltage, current filaments are formed at the imperfections [2].

In view of these results, we carried out the defect etching at about 0.6 V bias, where the current density was less than 2 mA cm^{-2} . Prior to studying the substrate, the damaged surface layer was removed by non-selective etching. The position of the interface is indicated by the variation of current versus depth. The current density has a sudden increment at the interface, because the substrate has a higher charge-carrier concentration than the epilayer (see figure 2 in [3]).

A typical picture of the surface morphology for a sample after selective etching is shown in figure 3. The measured EPD of the substrate was about half of the value specified by the supplier. The substrate defect density was found to be non-uniform. This indicates that the epilayer dislocations originate also from sources other than the grown-in dislocations of the substrate. We further note that the non-uniform distribution of threading dislocations is inherent to the mismatched systems. The average measured EPD versus depth data are depicted in figure 4. The size of etch pits varies; larger pits indicate dislocations travelling further from the interface—this is indirect evidence of the depth inhomogeneity of the defect density. The elongated shape of the pits is due to the higher etch rate along one of the $\langle 011 \rangle$ axes.

Several theoretical models have been worked out to predict the threading dislocation density in mismatched heteroepitaxial systems [1]. The threading dislocation densities determined by defect development are generally smaller than the theoretically predicted

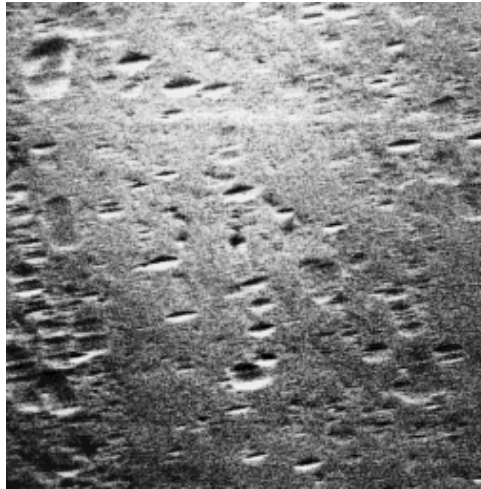


Figure 3. A view of the etched surface at $0.7 \mu\text{m}$ from the interface where the EPD is about $2 \times 10^8 \text{ cm}^{-2}$.

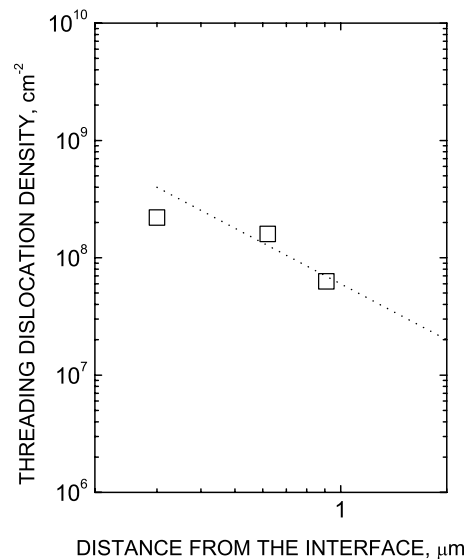


Figure 4. EPD as a function of etched depth for the measured sample with the model calculation.

values [3, 7]. Different equilibrium models exist yielding a critical layer thickness below which no plastic strain relaxation occurs and above which the layer is totally relaxed by the formation of misfit dislocations. However, this strain relief is a dynamic process depending on the effective stress, the temperature and the mechanical properties of the materials involved. Wehmann *et al* have introduced another model for the determination of the threading dislocation density which is based on fitting two unknown material parameters where the residual strain in the heteroepitaxial system depends on the layer thickness [8]. This model predicted values which agree very well with the measured EPD for the thick highly mismatched heteroepitaxial structures [7]. In our case the calculated curve is drawn as a dotted line in figure 4; it corresponds to the data depicted. The parameters of the system used in the dynamic model

are detailed below. The shear modulus, the Poisson ratio, the initial dislocation density, the activation energy for gliding and the activation energy for self-diffusion are 25.3 GPa, 0.33, 10^6 cm^{-2} , 1.7 and 3.85 eV, respectively.

4. Conclusions

We have presented a novel electrochemical apparatus for use in defect investigation. The set-up is suitable for observation of defect distributions in lateral and vertical directions. We gave details of the specially constructed electrochemical cell and optical system. The material system investigated was the thick InGaAs/GaAs heterostructure. We have shown that the set-up presented for the selective electrochemical etching is suitable for depth-dependent study of defect density in lattice-mismatched heteroepitaxial systems. The results agree with the general physical picture of heteroepitaxial growth and relaxation processes.

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