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*COMMUNICATIONS*

# DETERMINATION OF THICKNESS PROFILE OF AlGaAs/GaAs STRUCTURES

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The paper presents a procedure for determination of the thickness profile of semiconductor structures. It is based on estimating the layer thicknesses on a set of samples chosen in the desired direction. Samples are bevelled at an angle in the range of approximately $10^{-4}$ rad. After visualizing the layer interfaces on the bevelled surface and measuring the bevel profile, layer thicknesses are estimated. The procedure was used for an AlGaAs/GaAs structure but it is convenient also for other III-V materials.

K e y w o r d s: layer thickness, bevel, GaAs, AlGaAs, III-V semiconductor

## 1 INTRODUCTION

The performance and reliability of III-V semiconductor devices are known to depend on the perfection of the material and its control during technological steps. The most important factors to be controlled are the stoichiometric composition, concentration of dopants, thicknesses of layers and their uniformity in the wafer [1, 2, 3].

For many years various forms of wafer cross sectioning with etching or staining have been used to view junctions and heterojunctions in semiconductors. The main appeal of wafer cross sectioning as a means to view layers and junctions in semiconductors is its convenience, simplicity and speed; also wafer cross sectioning is a useful technique to complement or to guide more refined measurements employing higher resolution instruments (SEM, TEM, AES) [4, 5]. Direct vertical cross sectioning of a semiconductor wafer and measurement with an optical microscope becomes difficult for layer thicknesses approaching 0.5–1.0 $\mu$m. In the past angle-lapping or polishing (1–5°) was used to resolve dimensions down 50–100 nm. Recently, a technique for beveling by chemical etching [6, 7, 8] was developed to resolve and study semiconductor layers with thicknesses below 10 nm [8, 9].

In this paper we present a procedure for determination of layer thicknesses and thickness profiles of GaAs/AlGaAs structures on bevelled samples. The experiments were performed on a photodiode structure with layer thicknesses in the range 100 nm. We will show that this procedure is applicable also for thinner layers and for other III-V semiconductor structures.

## 2 EXPERIMENTAL

The following procedure for determination of the thickness profile on a 40 mm diameter wafer with a prepared GaAs/AlGaAs heterostructure was used. Ten samples ($4 \times 9$ mm in size) were cleaved from the wafer in the direction perpendicular to the facet (Fig. 1). This is [110] direction. Each sample was bevelled at an angle approximately as low as $10^{-4}$ rad. The $H_3PO_4/H_2O_2/H_2O$ etchant was used for bevel preparation. The depth profile of the bevel (further only bevel profile) was determined from the measurement of the step between the etched and non-etched (masked) surface in different positions throughout the bevel length using a Talystep profilometer. Details are presented in [6, 7]. Different AlGaAs layers were visualized on the bevel by wet chemical etching in the solution 1 $NH_4OH : 10\,H_2O_2$ at 45 °C. In the past this etchant for preparation of deep holes in GaAs substrate was used [10]. AlGaAs has acted as a stop layer. From the measured bevel profile and visualized boundaries between the layers on the bevel, the thicknesses of all layers were estimated. We have obtained ten values of layer thicknesses throughout the wafer in the direction perpendicular to the facet. This constitutes a thickness profile of the layers in this direction.

$Al_{0.25}Ga_{0.75}As$/GaAs structure prepared on a (001)-oriented $n^+$-type GaAs substrate was used for experiments. The following layers were grown by MBE: A 200 nm thick $n^+$ GaAs buffer layer followed by a 660 nm thick $n^+$-AlGaAs layer and a 500 nm thick $n$-AlGaAs layer and capped by a 300 nm thick $p$-AlGaAs layer. The structure was prepared on an intentionally non-rotated substrate.

Chemical beveling of the samples has been carried out in a system described in ref. [6, 7] and consisting of two tanks. The first one is used as a reservoir for the etchant and in the second one the beveling procedure is performed. The two tanks are connected through a tube equipped with a valve for fine control of the etchant flow speed in the range 0.01–1.0 mm s$^{-1}$. The samples for beveling were glued in vertical position to the sample holder. A part of the sample surface is protected by a

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black wax stripe to keep a reference for angle measurements. Before starting the etching procedure the tank for bevel etching was filled with deionized water (2 cm high column) to protect the surface from being damaged by the etchant vapor and to prevent formation of a meniscus during the process. Bevel preparation has been carried out at room temperature. A qualitative analysis of the bevel surfaces has been performed by means of optical interference microscopy.

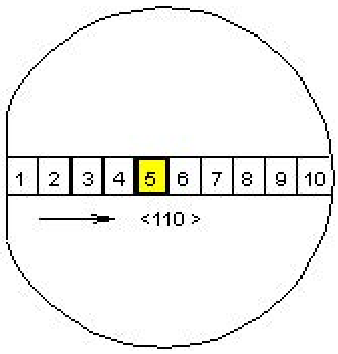


**Fig 1** Position of the samples on the wafer for layer thickness determination. The arrow indicates the direction in which the thickness profile was measured. Numbers denote order numbers of the samples.

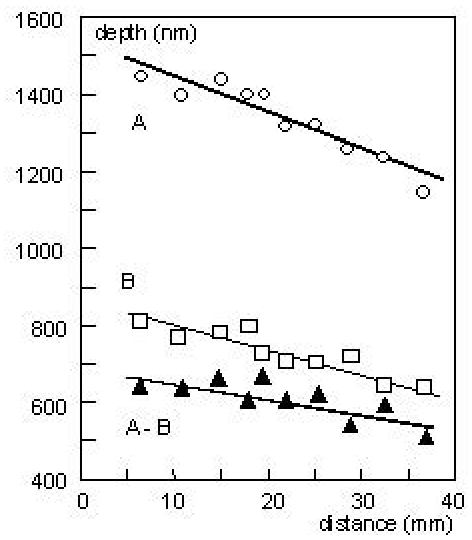


**Fig 2** Bevel profile of the sample in the middle of the wafer (sample No. 5). The length on the bevel surface is measured from the bevel edge.

## 3 RESULTS AND DISCUSSION

All ten samples were bevelled in the direction of the longer side of the sample. Bevel profiles were determined using a Talystep profilometer. The bevel profile of the sample in the middle of the wafer (No. 5 in Fig. 1) is shown in Fig. 2. The bevel was prepared by 7 : 3 : 3 composition of the etchant. We see two linear slopes on the bevel profile. The upper slope with bevel angle of $2.7 \times 10^{-4}$ rad is from the etching of GaAs (substrate and buffer layer). The lower part of the bevel profile with the angle of $4.2 \times 10^{-4}$ rad stems from the etching of AlGaAs layers. Different values of bevel angles are caused by different etching rates for GaAs and AlGaAs materials.

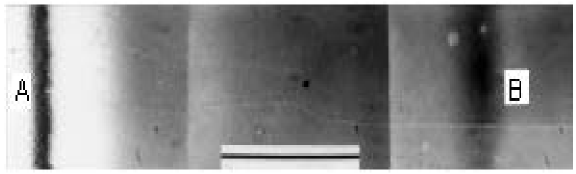


**Fig 3** The surface of the bevel after visualization of the layer boundaries by wet chemical etching. The regions left from A band and right from B band are the $n^+$ GaAs buffer layer and the $n$-AlGaAs layer, respectively. The region between bands A and B is the $n^+$ AlGaAs layer. The marker denotes 0.5 mm.

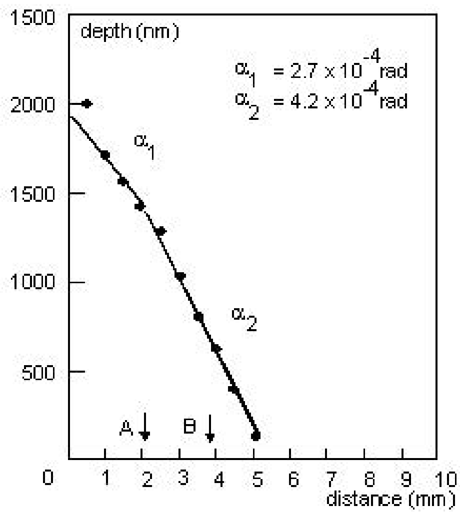


**Fig 4** The dependence of the depth of boundaries A and B in the samples (measured from the sample surface) and the thickness of $n^+$ AlGaAs layer (marked as A–B) on the distance of the sample from the facet in the chosen direction.

The surface of the bevelled samples was smooth. It was not possible to estimate the positions of layers by optical microscopy there. After etching in $1\,NH_4OH : 10\,H_2O_2$, the boundaries between layers were visible. In Fig. 3 there is a part of the bevelled sample after etching. Two narrow bands across the bevel are seen in the picture. The vertical band marked by A is the visualized boundary between the GaAs buffer layer and the n+ AlGaAs layer. The black vertical band marked as B is the visualised boundary between $n^+$ AlGaAs and $n$ AlGaAs layers. The width of this boundary is about three times broader than the boundary A. This is due to the lower etching selectivity of $n^+$ and $n$ AlGaAs layers than for GaAs and AlGaAs layers. From the position of the visualized boundary on the bevel and using the bevel profile (Fig. 2) we estimated the depth in which this boundary in the structure is situated. In this manner the depth positions of all boundaries and then the thicknesses of layers were estimated. In the case of a constant etching rate of the layer, its thickness could be easily calculated by multiplying the value of the bevel angle with the width of the visualized layer on the bevel surface. As an example, we present calculations of the layer thickness on the $n^+$ AlGaAs layer in these two ways:

i) Positions of visualized boundaries A and B of $n^+$ AlGaAs on the bevel surface were 2.1 and 3.7 mm. From Fig. 2 we see that it corresponds to the depth of 1400 nm and 730 nm in the structure. The layer thickness is the difference of these two values. It is 670 nm.
ii) The distance between the visualized boundaries A and B is 1.6 mm (Fig. 3) and the bevel slope $\alpha_2$ in the part of AlGaAs layers is $4.2 \times 10^{-4}$ rad. Multiplication of these two values gives the thickness of layer as high as 672 nm.

In Fig. 4 there are the profiles of the depth of boundaries A, B and their difference A–B along the chosen direction marked in Fig. 1. All dependences are characterized by a decrease. This is caused by no sample rotation during the epitaxial growth. The upper dependence A presents the thickness profile of all three AlGaAs layers together. Dependence B in the middle presents the thickness profile of the sum of $n$ and $p$ AlGaAs layers and the lower dependence A–B presents the thickness profile of the $n^+$ AlGaAs layer. The decrease of the thickness of this layer is approximately 120 nm. It is then comprehensible that the decrease of the sum of the three layers together (dependence A) is biggest of all of these three dependences and it is approximately 300 nm.

The accuracy of the thickness estimation is affected by various factors:

i) At first, it is the linearity of the bevel profile and the accuracy of its measurement. An apparatus often used to measure the bevel profiles is the Talystep. We have used this type of apparatus, too. By using it, the step between the etched and unetched surfaces in different positions along the bevel is measured. In most cases this step is not rectangular and we have to take the value of a step far (sometimes 0.5–1.0 mm) from the etched edge. This value suffers from high inaccuracy due to the construction of the Talystep. It is then convenient to use another apparatus (*eg*, a 3-D profilometer).
ii) The visualization of the boundary positions is a very important part of this method. The cheapest and fastest way is to use wet chemical etching. We have to use a suitable etching solution. As one can see in Fig. 3, the width of visualized interfaces is relatively large. It corresponds to a 26 nm large interface region in GaAs/AlGaAs and 62 nm large interface region in $n^+$ AlGaAs/n AlGaAs junctions. This caused an error of 7 % in the estimation of the layer thickness. By decreasing the etching time or by using another more suitable etchant it is possible to make the visualizes boundaries narrower and to increase the accuracy of layer thickness estimation.

In the presented structure (in the middle of the wafer) we have measured the thickness of the $n^+$ AlGaAs layer as high as 670 nm. The predicted thickness for this layer by technology was 660 nm. The difference between these two values presents an error of 1.5 %. From Fig. 4 (A–B dependence) we see that the dispersion of values on $y$ axis is more than 1.5 %. It is about 45 nm. This is an error of 7 %. It is the same error as the previously estimated one in measuring the layer thickness by visualization of layer boundaries. Therefore we are able to conclude that the error of estimation of the position of the visualised layer boundaries caused the main contribution to the error of our procedure.

The presented procedure can be used also in the case of thinner layers. We must use a convenient etchant, which visualizes interfaces of very thin layers. By the above mentioned etchant, and short etching times (1–2 s) we visualized layers in a multiquantum well (MQW) GaAs/AlGaAs heterostructure [9]. The thicknesses of AlGaAs and GaAs layers were 3 and 9 nm, respectively. In this case the accuracy of layer thickness estimation was lower than in the structure presented here, of course. To increase the accuracy of position of the visualized layer boundaries, other methods could be used (*eg* SEM in different modes: low energy SEM [11], EBIC).

The presented procedure is not limited to GaAs/AlGaAs structures only. It is convenient to all semiconductor compounds, where bevel preparation is possible and where etchants for wet etching visualization or other methods for boundary visualization are known. It is suitable for InP based heterostructures, bevel preparation having been mastered by many laboratories [12, 13, 14, 15] and the etchant for visualization of layer interfaces is known as well [16].

## 4 CONCLUSION

We have developed a procedure for thickness profiling of epitaxial layers throughout the wafer. This procedure consists of an estimation of the thickness of layers on bevelled samples which were picked in desired directions from the studied wafer. After measuring the bevel angle and visualization the layers on the bevels it is possible to estimate the thickness of the layers on all samples with an error of 7 % and then to obtain a thickness profile of layers in the desired direction. The procedure was applied to a GaAs/AlGaAs structure. A decrease of the thicknesses in [110] direction was observed. It was caused by non-rotating the wafer during MBE preparation of the structure.

This procedure is suitable also for other semiconductor structures (especially for InP-based materials). It is a fast and cheap method but with limited accuracy. Using it, we first obtain rapid information about the thickness profile in the wafers. By additional methods (*eg*, various modes of SEM) for layer boundaries visualization the accuracy of the presented method is improved.

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