Eur. Phys. J. Appl. Phys. **27**, 407–409 (2004)
DOI: 10.1051/epjap:2004119

THE EUROPEAN PHYSICAL JOURNAL
APPLIED PHYSICS

AFM and XRD studies of GaAs surface after anisotropic etching

N. L. Dmitruk[1,a], V. P. Kladko[1], R. V. Konakova[1], and A. V. Karimov[2]

[1] Institute for Physics of Semiconductors of the National Academy of Sciences of Ukraine, 45 Prospect Nauki, 03028, Kyiv-28, Ukraine
[2] Physical-Technical Institute of the Scientific Association "Physics-Sun" of the Academy of Sciences of Republic of Uzbekistan, Tashkent, Uzbekistan

Received: 4 July 2003/ Accepted: 31 December 2003 – © EDP Sciences

Abstract. A technology of anisotropic etching of GaAs (100) samples with two different types of geometric disorder (dendrite, quasi-grating) has been elaborated. The geometric statistical parameters of micro(nano)relief were investigated using bearing analysis. The measurements of the radii of curvature have shown that the samples were rather deformed, with nonuniform strain lateral distribution over the wafer areas. The radii of curvature were different for different samples, depending on surface treatment. The X-ray diffraction data indicate that the subsurface region of anisotropically etched GaAs crystal and epitaxial films grown on such substrates are not worse than those of a crystal with smooth chemically polished surface. Geometric disorder of such surfaces leads to enhancement of the diffuse contributions on the rocking curve tails.

PACS. 61.72.-y Defects and impurities in crystals; microstructure – 81.40.-z Treatment of materials and its effects on microstructure and properties

1 Introduction

Semiconductor textured surfaces and interfaces are used to reduce light reflection and increase photocurrent in solar cells and photodetectors. However, formation of textured surface is often accompanied by its damage. That is why formation of microrelief surfaces by chemical anisotropic etching is particularly attractive: it produces surfaces with appointed geometrical relief (and, maybe, even improved structural properties) without damaging the subsurface layer [1]. Here we present the results of our investigations of surface topography with atomic force microscopy (AFM) and structure perfection with X-ray diffraction (XRD), made for three different types of single-crystalline GaAs surfaces, as well as for AlGaAs epitaxial films grown on these substrates.

2 Experimental procedures

We investigated single-crystalline n-GaAs (100) samples 350 μm thick. The donor (Sn) concentration was $(2{-}4)\times10^{17}$ cm^{-3}. Surfaces of three types were obtained: one after mechano-chemical polishing and two other with microrelief (dendrite- and quasi-grating-like). They were prepared using selective etching in concentrated HNO_3 and $2HF{:}2H_2SO_4{:}1H_2O_2$ mixture, respectively. The AFM patterns of flat and textured surfaces were obtained using a Nanoscope Dimension 3000 with a silicon nitride tip.

The crystallographic quality of GaAs samples was determined with a high-resolution diffractometer equipped with a 400 GaAs monochromator. The characteristic Cu_{K_α}-radiation was used. The rocking curves (RCs) were taken using a detector with a system of slits before it. The samples were scanned close to the exact Bragg position over the range of about 1° in the so-called ω- and $\omega/2\theta$-modes. In the second case the reflected beam intensity was recorded using a detector with a system of cracks. The measurements were carried out in the discrete angular mode, the interval being $2''$. To analyze the experimental data obtained, a comparison between the experimental and calculated RCs was carried out using fitting according to the so-called χ^2-procedure [2].

3 Experimental results and discussion

Shown in Figure 1 are the microphotographs and depth histograms of the surfaces studied. One can see that the mean roughness height is 0.4 nm, 0.2 and 0.6 μm for the flat, quasi-grating-like and dendrite-like surfaces, respectively. The so-called quasi-grating-like surface is a set of V-grooves oriented along the [110] direction. The grooves at the front and back surfaces are perpendicular.

Besides, the excess of surface area over projected one for the flat (microrelief) surface was no more than 0.6%

[a] e-mail: nicola@dep39.semicond.kiev.ua

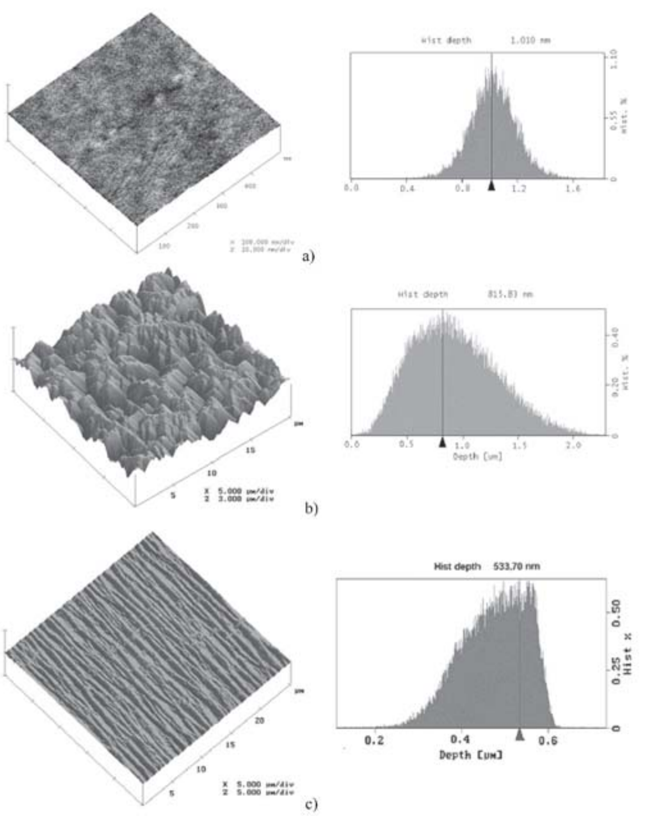

Fig. 1. AFM images (left) of GaAs with flat (a), dendritic (b) and quasi-grating (c) surfaces. On the right are histograms of depth distribution (bearing analysis).

(∼ 30–50%). Figure 1 also presents roughness height histograms for the areas ∼ $20 \times 20\ \mu\text{m}^2$. One can see that microroughness distribution is Gaussian for the flat surface, while for the textured surfaces it departs from Gaussian.

To make numerical estimation of the intrinsic stress level for all the samples studied (both flat and microrelief), we measured macroscopic bending. The mean radii of curvature at typical sample treatments are given in Table 1.

Table 1.

Sample number and type of surface	Radius of curvature, m
1—flat	77–150
2—dendrite	14.9–25
3—quasi-grating (1 min.)	6.3–8.5
4—quasi-grating (3 min.)	0.63–2.17

The bending forms and values for the samples from different batches but exposed to the same type of treatment are approximately the same. However, bending was more complicated for the samples whose structure was of the first type.

This seems to be related to a nonuniform distribution of growth defects. An analysis of the results obtained indicates at bending relief over the wafer surface, i.e., macroscopic bending of the structures studied is modulated. This modulation depends on the initial nonuniformity of the elastic strain field in material, as well as on nonuniformity of intrinsic strains induced by treatment. The surface profiles for structures of different types but exposed to the same treatment are close to each other.

One can see from Figure 2 that RC half-widths are 16, 27 and 46″ for the samples with flat, quasi-grating-like and dendrite-like surfaces, respectively. These RCs differ from one another by their shapes at tails. This fact indicates at variations in the intensity and spatial distribution of X-ray diffuse scattering from the crystals studied. The samples 2 and 3 demonstrate stronger diffuse scattering than that observed from the sample 1. For the sample 2 (dendrite-like surface) diffuse component of X-ray scattering exceeds those for the rest of the samples studied.

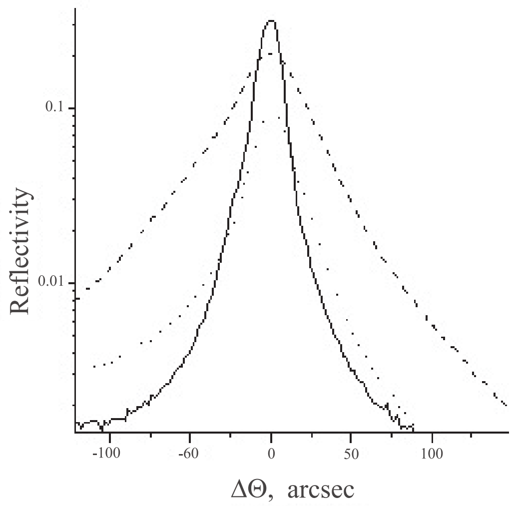

Fig. 2. The rocking curve (004 reflection) for GaAs single crystal before anisotropic etching (solid line, sample 1) and after it (dashed line, sample 2; dotted line, sample 3).

X-ray diffuse scattering is known to occur from thermal vibrations of crystal lattice, as well as from surface imperfections and point and extended defects in the crystal bulk [3]. The above situation with diffuse background takes place for epitaxial films grown on these substrates (see Figure 3).

The X-ray RCs were taken for two cuts of the Ewald sphere in the reciprocal space [4]. We analyzed RCs for two scanning cuts in the reciprocal space for GaAs/n-GaAs epitaxial structures obtained on a flat (1) and quasi-grating-like (3) substrate surfaces. The slope values for log − log angular dependencies of X-ray diffusion scattering intensity for the above cuts are given in Table 2. From the tangent of slope angle one can determine type of predominant crystal imperfections: 3/2 – dislocation loops, 2 – cluster-like defects. From the data presented in Table 2 it follows that for the crystals studied the tangent of slope angle is close to 1.5.

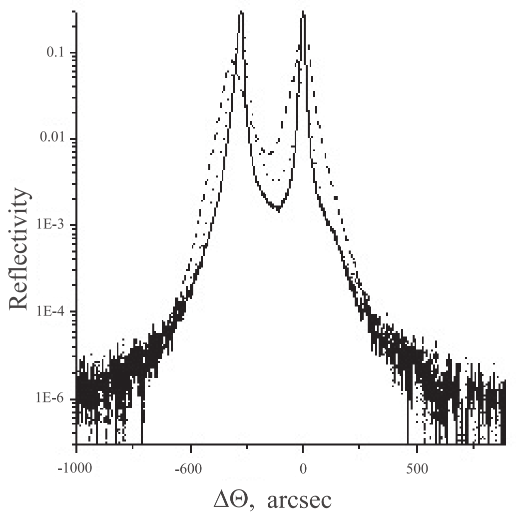

Fig. 3. The rocking curve (004 reflection, $\omega/2\theta$ scan) for AlGaAs/GaAs epitaxial structure before the anisotropic etching (solid line, sample 1) and after it (dashed line, sample 2; dotted line, sample 3).

Table 2.

Number of sample	Tangent of slope angle at ω-scanning	Tangent of slope angle at $\omega/2\theta$-scanning
1	1.53	2.13
2	1.59	2.12

For perpendicular directions the tangent of slope angle differs substantially from that at ω-scanning. This indicates at asymmetric diffuse scattering component distribution around the reciprocal lattice site 004. Judging from the results presented in Table 2, the defects (loops) of interstitial type are predominant in the sample 1. This conclusion is supported also by slower decay in that region. For the sample 3 defects of both types appear with almost the same probabilities.

In [5] it was shown that a periodic grating at the crystal surface results in appearance of satellites around a reciprocal lattice site (peak of reflection from crystal). In the case of a quasi-grating, however, this leads (along with appearance of diffuse scattering) to coherent peak broadening due to averaging of oscillations caused by presence of a periodic structure at the surface [6].

4 Conclusions

Surface texturing made to improve light absorption in GaAs solar cells has been investigated with atomic force microscopy and X-ray diffraction. We elaborated a procedure of anisotropic etching of GaAs (100) surfaces that provided two different types of surface geometry disorder (dendrite- and quasi-grating-like). By varying etching conditions (temperature and duration), we could change microrelief depth, as well as its statistical parameters.

The results of X-ray diffraction indicate that the near-surface region of etched GaAs crystals with quasi-grating-like surface microrelief is almost of the same high perfection as in crystals with chemically polished flat surfaces. The quality of epitaxial films grown on the substrate with quasi-grating-like microrelief is comparable to that of the films epitaxially grown on the flat substrate.

This work was supported by the Science and Technology Center in Ukraine (Project Uzb-56(J)).

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