

Semiconductors

DEVICE NANOSTRUCTURES

■ Introduction. The purpose of the investigation

A wide set of modern electronic and light-emitting devices, e.g. transistors or injection laser diodes, is based on the semiconductor heterostructures.

These heterostructures contain various potential barriers- n⁺-n, n-p junctions etc.- whose position should be known with a high precision relative to the nanometer-thin layers the heterostructure may consist of. Thus, to characterize the heterostructures constituting the base elements of the devices or the whole devices, one should measure the thickness of heterolayers and determine the localization of the potential barriers within the heterostructure. These objectives may be reached via SPM studies on the structure cleavages.

What is attractive and of paramount importance, the SPM studies can be carried out not only under equilibrium conditions, but also under applied external bias, when high current flows through the operating devices. In this note we present several examples of the semiconductor device heterostructures characterization in different SPM modes with the use of NT-MDT tools.

■ Getting ready the measurements Sample preparation

The semiconductor heterostructures are usually epitaxially grown on thick substrates (a few hundredths of microns) and appear on the sample cleavages in the close proximity to their edges (1 or 2 microns or less). Optical viewing system of SPM device is necessary to position the tip of the SPM cantilever on the heterostructure layers of interest. The heterostructure samples of different semiconductor systems were studied: ZnSSe/GaAs, ZnBeMnSe/GaAs, GaAlAs/GaAs and GaInSbAs/GaSb. The samples with freshly

prepared cleavages were gripped in a miniature vice or fixed by a double-side scotch on the vertical side of the small block. In the case of GaAs-based laser diodes, the freshly cleaved rectangular resonators (typical sizes 100x300x1000 microns) were soldered on In-coated heat sinks. If necessarily, an external bias was applied between p- and n-contacts of laser diodes, both external power supply and SPM setup having common earth. A crucial point for successful SPM studies of heterostructure samples is preparation of the mirror-like cleaved faces (for details see results on topography study of ZnSSe and ZnBeMgSe-based laser structures).

■ SPM mode choice

Both contact and resonant modes were used for the topography acquisition. In many cases the surface relief of the heterostructure cleavage contains sufficient information to measure the thickness of individual layers and the positions of the heterointerfaces. Additional data of the heterostructure inspection with high lateral resolution permitted the usage of the local elasticity mode (force modulation technique). To directly demonstrate chemical contrast of the constituent layers we applied lateral force imaging. The information of practical significance for the device structures- electrostatic potential distributions - was obtained by the combination of the above-described approaches with two pass techniques such as SKM and EFM. A separate attention, from the vantage point to grade up the instrumental function in electrostatic measurements, was given to the EFM data in contact mode obtained by the measurements of vibration amplitude of electrostatically excited cantilever (force modulation technique).

▣ Cantilever selection

The majority of results were obtained by contact cantilevers CSC12. These soft sensors have rectangular shape and long (~15 micron) tip and are made of highly doped Si of p-type conductivity. Rectangular shape and small force constant of these cantilevers make them excellent candidates for friction measurements, while slim, long and conductive tip permits to detect electrostatic forces with minimal influence of the parasitic capacitance of the tip side and of the flat part of the cantilever. When applying two pass techniques such as SKM and EFM, we used resonant cantilevers NSG11/Pt of similar design.

▣ Special conditions of measurements

To suppress 50 Hz interference, when measuring the potential distributions by SKM or EFM with applied to the device contact biases, an external power supply and SPM should have common earth. To avoid coupling between topography relief and SKM or EFM signals the cleavage surface of the studied device should be almost atomically flat. Studying an operating light emitting device when high current flows through a sample, the cantilever may get warm, that results in the shift of the resonant frequency. One should wait until new resonance is stabilized.

▣ Measurements realization

Optical viewing system of SPM device should be used to position the tip of the SPM cantilever on the heterostructure layers of interest.

On the very edge of the cleavage the cantilever tip can be easily damaged due to large height variation in surface relief. To save the tip and time, one should choose a scan frame with one side almost parallel to the edge and use slow scan rates (about one micron per second).

To study the potential distributions on the cleavage of device heterostructure, first the SKM mode should be applied with its ability to measure absolute value of the contact potential difference (CPD). Then, to determine the individual peculiarities in the CPD profile with better lateral resolution, a non-contact EFM may be used.

▣ Obtained results: analysis, processing, representation and application

The obtained data on topography, lateral force and local elasticity studies illustrate wide abilities of SPM for layer thickness characterization in semiconductor heterostructures. We also present results that permit to consider SKM and EFM modes as powerful tools for the investigation of the electrostatic potential distributions in the layered semiconductor devices. More detailed discussion of the obtained results can be found in scan descriptions.

▣ Acknowledgments

We are very grateful to our colleagues Dr V.P. Evtikhiev, Dr S.V. Ivanov and Dr V.V. Lundin from A. F. Ioffe Institute, as well as Dr H. Riechert from Infineon Technologies AG (Germany) for providing the samples for the investigations. We would particularly mention Dr V.P. Evtikhiev who has, by his discussions about the operation of high power laser diodes, maintained our interest in the subject of the presented work.

▣ Device nanostructures

- Topography acquisition. ZnSSe/GaAs laser structure

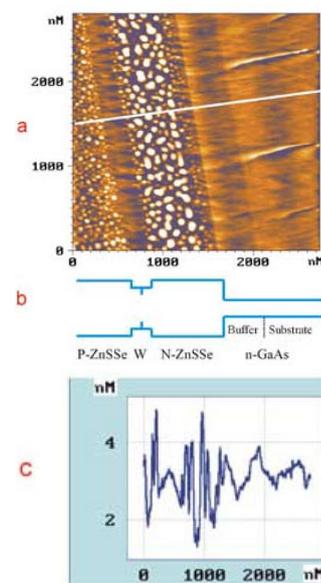


Fig. 1(a-c)

Relief peculiarities of individual layers on cleavages with rough surface. The samples of heterostructures based on 3-5 or 2-6 compounds, that have zinc blend crystalline structures, are easy to cleave to form almost atomically flat cleavage surface, on which the constituting layers come out.

Rather conventionally, we can imagine two possible ways of cleavage proce-

cedure of semiconductor wafer: like chopping a wood plank or like breaking a cracker. As a rule, the fist

way gives the mirror-like surface; the second way leads to the stepped and rough surface. Nevertheless, the last case may be interesting, since a cleavage crack propagates perpendicular to the interfaces of the layered structure, and surface morphology of adjacent layers may change notably due to different mechanical behavior of those layers during the cleavage development.

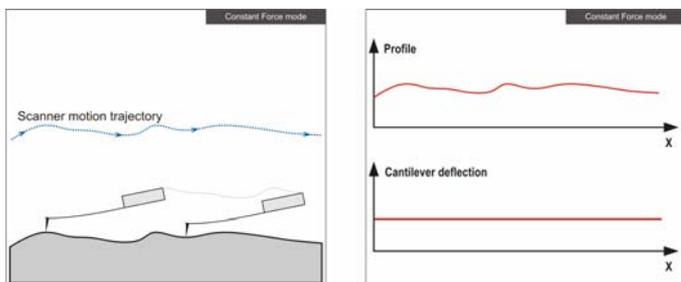
In Fig. a) a contact AFM topography image of such prepared cleavage of ZnSSe/GaAs laser heterostructure is presented. Six regions of different surface morphology formed on the cleaved surface describe the history of the cleavage crack propagation. Indeed, relatively flat GaAs surface implies good condition for the cleavage development, whereas granular morphology of ZnSSe emitters reflects process of rather plastic rupture, than cleavage. This agrees well with more ductile behavior of ZnSe-based materials in reality, compared to the brittle GaAs. As one can see all the layers constituting the laser structure may be identified, in the agreement with the heterostructure energy band diagram (Fig. b). It is interesting to note, that, besides, a buffer GaAs region and an additional interface in N-ZnSSe emitter due to growth interruption are revealed in the image.

AFM images are obtained by commercial SPM P4 with CSC12 cantilevers.

On the next pages description of the used mode, details and grounds of image obtaining are presented.

Additional information about laser structures were received with usage of Lateral Force, Force Modulation, Contact EFM, Kelvin modes

SPM mode



In Contact mode of operation the cantilever deflection under scanning reflects repulsive force acting upon the tip.

Repulsion force F acting upon the tip is related to the cantilever deflection value x under Hooke's law: $F = -kx$, where k is cantilever spring constant. The

spring constant value for different cantilevers usually vary from 0.01 to several N/m.

In our units the vertical cantilever deflection value is measured by means of the optical registration system and converted into electrical signal DFL. In contact mode the DFL signal is used as a parameter characterizing the interaction force between the tip and the surface. There is a linear relationship between the DFL value and the force. In Constant Force mode of operation the deflection of the cantilever is maintained by the feedback circuitry on the preset value. So vertical displacement of the scanner under scanning reflects topography of sample under investigation.

Constant Force mode has some advantages and disadvantages.

Main advantage of Constant Force mode is possibility to measure with high resolution simultaneously with topography some other characteristics - Friction Forces, Spreading Resistance etc.

Constant Force mode has also some disadvantages. Speed of scanning is restricted by the response time of feedback system. When exploring soft samples (like polymers, biological samples, Langmuir-Blodgett films etc.) they can be destroyed by the scratching because the probe scanning tip is in direct contact with the surface.

There unto under scanning soft unhomogeneous samples the local flexure of sample surface varies. As a result acquired topography of the sample can prove distorted. Possible existence of substantial capillary forces imposed by a liquid adsorption layer can decrease the resolution.

References

Magonov, Sergei N. Surface Analysis with STM and AFM. Experimental and Theoretical Aspects of Image Analysis. VCH 1996.

Practice details

Topography acquisition. ZnSSe/GaAs laser structure.

Preparation steps for topography study of the heterostructure on the cleavages

a) A 50 mm scanner, an optical viewing system, and x-y translation stage may be necessary to have comfortable conditions of measurements.

b) The samples with freshly prepared cleavages may be gripped in a miniature vice or fixed by a super glue on the vertical side of the small block. A double-side scotch can be used instead of glue, but when the linear sizes of the sample are less than 1 mm, the scotch is often not effective means for good fixation.

c) The cantilever beam should be parallel or perpendicular to the cleavage edges to facilitate the search of the edge in the scan area.

d) Before approach, the tip should be placed in the center of the scanning area, and approximately 15-20 mm away of the edge of the cleavage (for the reference, when using the optical viewing system, the width of the cantilever NSG11 is about 50 mm).

e) When the edge has been found, first we move the tip several microns away of the edge, then withdraw the tip from the sample (switch out a feedback loop), close a microscope by the protective cover, suspend the whole system, and finally switch on a feedback loop again.

No specific recommendations for the cantilever type can be given. Any new and good contact or resonant cantilever permits to get good topography data on usually atomically flat cleavages. The only demand is not to ruin the cantilever in search of the sample edge.

Specific parameters of the scan can be found in http://ntmdt.ru/Appl_Notes/Dev_nanostr/fl_opt.zip and

http://ntmdt.ru/Appl_Notes/Dev_nanostr/fl_mdt.zip AFM images are obtained by commercial SPM P4 with NSG11 cantilevers.

Additional information about laser structures were received with usage of Contact EFM, Kelvin mode.

Basics

Topography acquisition. ZnNeMgSe/GaAs laser structure.

References

1. V. Ankudinov, A. N. Titkov, T. V. Shubina, S. V. Ivanov, P. S. Kop'ev, H.-J. Lugauer, G. Reuscher, M. Keim, A. Waag, G. Landwehr, Applied Physics Letters, October 1999, Volume 75, Number 17, pp. 2626-2628, "Cross-sectional atomic force microscopy of ZnMgSSe- and BeMgSSe-based laser diodes".

(http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/1.pdf).

2. AFM images are obtained by commercial SPM P4 with NSG11 cantilevers.

Additional information about laser structures were received with usage of Contact EFM, Kelvin mode.

- Topography acquisition. ZnBeMgSe/GaAs laser structure.

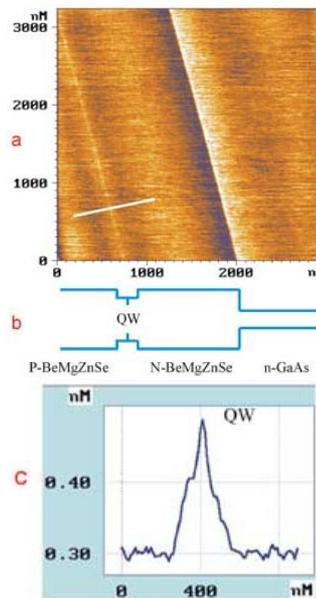


Fig. 1(a-c)

Influence of strains at the layer interfaces on the relief of atomically flat cleavages.

In Fig. 1a topography of the cleavage of ZnBeMgSe/GaAs laser heterostructure is presented (cantilever CSC12 was used). In this case the surface is atomically flat, and the topography information is not sufficient to detect all the layers of the laser. There are two

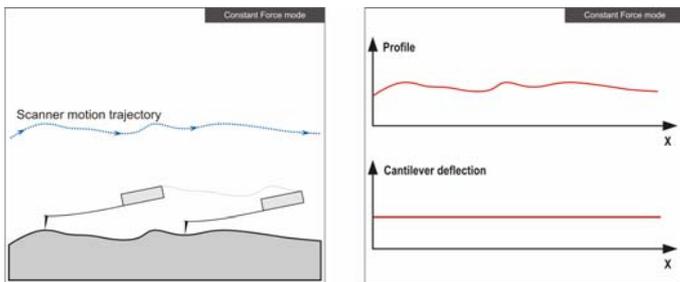
meaningful features: a white 2-angstrom-height ridge corresponding to the compressed ZnCdSe quantum well (QW) and 6-angstrom-height step at the interface with GaAs substrate. The ridge corresponding to QW (see a height profile in fig.c) resulted from the compensation of bulk mechanical strains by the surface tension; the step reflects non-ideal structural quality of the first interface. Detailed analysis of the factors responsible for the

surface peculiarities on the cleavages of strained heterostructures may be found in [1].

AFM images are obtained by commercial SPM P4 with CSC12 cantilevers.

On the next pages description of the used lateral force mode and the details of image obtaining are presented. For more information see also paper NN. Additional information about laser structures were received with usage of Contact EFM, Kelvin mode.

SPM mode



In Contact mode of operation the cantilever deflection under scanning reflects repulsive force acting upon the tip.

Repulsion force F acting upon the tip is related to the cantilever deflection value x under Hooke's law: $F = -kx$, where k is cantilever spring constant. The spring constant value for different cantilevers usually vary from 0.01 to several N/m.

In our units the vertical cantilever deflection value is measured by means of the optical registration system and converted into electrical signal DFL. In contact mode the DFL signal is used as a parameter characterizing the interaction force between the tip and the surface. There is a linear relationship between the DFL value and the force. In Constant Force mode of operation the deflection of the cantilever is maintained by the feedback circuitry on the preset value. So vertical displacement of the scanner under scanning reflects topography of sample under investigation.

Constant Force mode has some advantages and disadvantages.

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Constant Force mode has also some disadvantages. Speed of scanning is restricted by the response time of feedback system. When exploring soft samples (like polymers, biological samples, Langmuir-Blodgett films etc.) they can be destroyed by the scratching because the probe scanning tip is in direct contact with the surface.

There unto under scanning soft unhomogeneous samples the local flexure of sample surface varies. As a result acquired topography of the sample can prove distorted. Possible existence of substantial capillary forces imposed by a liquid adsorption layer can decrease the resolution.

Practice details

Topography acquisition. ZnBeMgSe/GaAs laser structure.

See [practice details](#) of topography acquisition, ZnSSe/GaAs laser structure.

Specific parameters of the scan can be found in http://www.ntmdt.ru/App1_Notes/Dev_nanostr/f2opt.zip, http://www.ntmdt.ru/App1_Notes/Dev_nanostr/f5opt.zip and http://www.ntmdt.ru/App1_Notes/Dev_nanostr/f2mtdt.zip, http://www.ntmdt.ru/App1_Notes/Dev_nanostr/f5mtdt.zip.

Basics

References

1. V. Ankudinov, A. N. Titkov, T. V. Shubina, S. V. Ivanov, P. S. Kop'ev, H.-J. Lugauer, G. Reuscher, M. Keim, A. Waag, G. Landwehr, Applied Physics Letters, October 1999, Volume 75, Number 17, pp. 2626-2628, "Cross-sectional atomic force microscopy of ZnMgSSe- and BeMgSSe-based laser diodes". (http://www.ntmdt.ru/App1_Notes/Dev_nanostr/1.pdf).
2. AFM images are obtained by commercial SPM P4 with NSG11 cantilevers. Additional information about laser structures were received with usage of Contact EFM, Kelvin mode.

- Corrosion influence. ZnSSe/GaAs laser structure

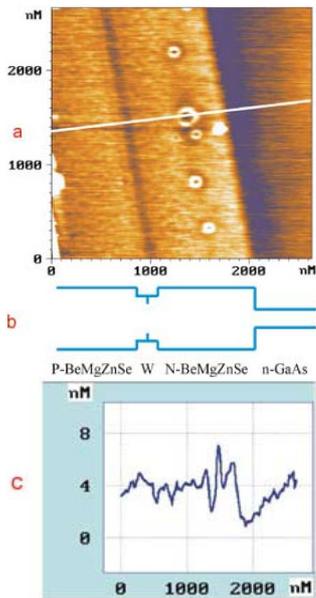


Fig. 1(a-c)

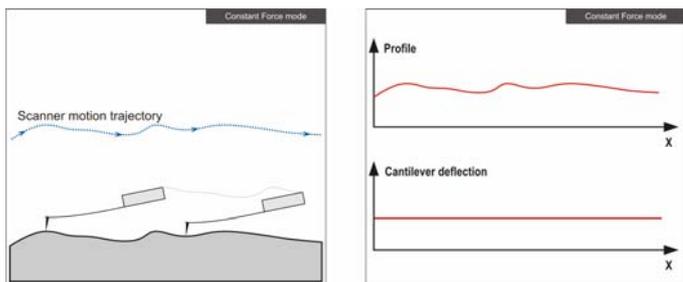
Relief modifications caused by corrosion. It is worth noting that surface corrosion and oxidation may cause local modification of the cleavage surface topography. These processes take place in air and may create individual relief for the layers, as a consequence of their different chemical activity. In fig.a topography of the corroded cleavage is shown.

The enhanced corrosion of ZnSe-based layers contrasting stable GaAs surface is evident from the image (see also a height profile in fig.c). Additionally, in comparison with freshly cleaved surface, an undoped waveguide region (W) is resolved as two 100 nm wide stripes. The corrosion of ZnSe-based materials is, however, rather slow under ambient condition, and it takes more than one month to wait single-monolayer-changes in the surface morphology.

[2, http://ntmdt.ru/App1_Notes/Dev_nanostr/2r.pdf] AFM images are obtained by commercial SPM P4 with CSC12 cantilevers.

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Practice details

See practice details of topography acquisition, ZnSSe/GaAs laser structure.

Some products of the corrosion may be weakly bounded to the surface. Therefore to minimize their damage by AFM probe in contact mode, we applied small error signals for the topography acquisition. (Setpoint values of the vertical deflection of the SPM laser beam from the cantilever were chosen very close to the initial deflection from the cantilever far away from the surface).

Specific parameters of the scan can be found in f3.opt and http://www.ntmdt.ru/Apppl_Notes/Dev_nanostr/f3m dt.zip. On the next page description the grounds of image obtaining are presented.

AFM images are obtained by commercial SPM P4 with NSC11 cantilevers. Additional information about laser structures were received with usage of Contact EFM, Kelvin mode.

Basics

References

1. A. V. Ankudinov, M. S. Dunaevskii, V. A. Marushchak, A. N. Titkov, S. V. Ivanov, S. V. Sorokin, T. V. Shubina, P. S. Kop'ev, A. Waag, G. Landwehr, Physics of the Solid State, October 2000, Volume 42, Issue 10, pp. 1927-1934, "Morphology of (001) and (110) Surfaces of Crystal Layers in Solid Solutions of II-VI Compounds with a High ZnSe Content under Atmospheric Conditions". (http://www.ntmdt.ru/Apppl_Notes/Dev_nanostr/2r.pdf)

AFM images are obtained by commercial SPM P4 with NSG11 cantilevers.

Additional information about laser structures were received with usage of Contact EFM, Kelvin mode.

- Oxidation influence. GaAlAs/GaAs laser structure

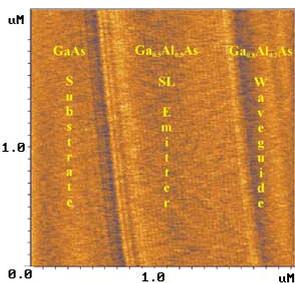


Fig. 1

Relief modifications caused by oxidation. For GaAs-based materials, second process, heterogeneous oxidation of layers, may predetermine a certain surface relief

[3, http://ntmdt.ru/Apppl_Notes/Dev_nanostr/3r.pdf]. At the first moments after cleaving the oxidation develops quickly, then it saturates, leaving stable in time oxidized surface relief.

In Fig. 1 contact AFM topography image of GaAlAs/GaAs laser cleavage is shown. GaAlAs emitter was grown as a superlattice (SL) by insertion of GaAs layers into GaAlAs matrix. Since Al is very actively oxidized in ambient atmosphere, Al-containing layer has thicker oxide and, therefore,

may elevate over the surface level of the Al-free layer with thin oxide

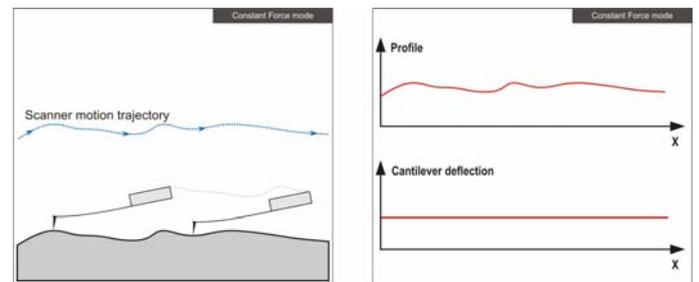
[3, http://ntmdt.ru/Apppl_Notes/Dev_nanostr/3r.pdf]. This fact is very important and permits to identify in AFM topography all the layers of the GaAlAs/GaAs based laser heterostructures, what can be used to link the other SPM signals (e.g. SKM and EFM) to the device design [4,5].

AFM images are obtained by commercial SPM P4 with NSG11 cantilevers.

On the next pages description of the used lateral force mode and the details of image obtaining are presented.

Additional information about laser structures were received with usage of Contact EFM, Kelvin mode.

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Practice details

See practice details of topography acquisition, ZnSSe/GaAs laser structure.

Specific parameters of the scan can be found in f4.opt and

http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/f4m dt.zip.

References

1. A. V. Ankudinov, V. P. Evtikhiev, V. E. Tokranov, V. P. Ulin, A. N. Titkov, Semiconductors, May 1999, Volume 33, Issue 5, pp. 555-558 "Nanorelief of an oxidized cleaved surface of a grid of alternating Ga_{0.7}Al_{0.3}As and GaAs heterolayers".

http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/5.pdf.

Basics

References

1. A. V. Ankudinov, V. P. Evtikhiev, V. E. Tokranov, V. P. Ulin, A. N. Titkov, Semiconductors, May 1999, Volume 33, Issue 5, pp. 555-558 "Nanorelief of an oxidized cleaved surface of a grid of alternating Ga_{0.7}Al_{0.3}As and GaAs heterolayers".

http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/3r.pdf

AFM images are obtained by commercial SPM P4 with NSG11 cantilevers.

Additional information about laser structures were received with usage of Contact EFM, Kelvin mode.

- Measurements of contact potential differences and external voltage drop profiles

The unique information of practical significance for the device structures may be obtained by the combination of the above-described approaches with two pass techniques such as SKM and EFM. We used for these modes Cantilevers NSG11/Pt. These sensors has slim and long tip that is optimal design decreasing the parasitic capacitance of the tip side and of the flat part of the cantilever.

In Fig. 1 AFM/SKM study of GaAlAs/GaAs laser diode is presented. Topography (left image) and SKM data in figure below were obtained under equilibrium conditions, when both n- and p-contacts of the laser diode were grounded (n-p junction of the device is closed). Data in fig.1c were taken under external positive bias applied to p-contact (n-p junction of the device is open). In topography image (see fig.1a) all the layers of the laser diode are resolved. There are two bright horizontal lines in topography image between n-GaAs substrate and n-GaAs buffer and in the middle of the undoped waveguide. The upper line corresponds to the peripheral thin GaAlAs layer used to adjust the growth parameters of the following Ga_{0.5}Al_{0.5}As emitters and Ga_{0.8}Al_{0.2}As waveguide. The lower line is compressively strained InGaAs QW.

SKM images in Fig. 1 permits to localize n-p junction as the place when the contact potential difference (CPD) undergoes the main changes. SKM data on Fig. 1 demonstrate unambiguously that the surface electric field of n-p junction is inverted when applying high forward biases. The bulk electric field must have, however, the same sign for any biases and only reduce its absolute magnitude under forward biases. To find quantitative correlation between obtained SKM data and potential distribution in the bulk, generally, one have to account for the profile of the surface band bending across the structure. Assuming that the surface band bending does not depend significantly on the bias conditions, it is possible to exclude the band bending contribution in CPD by subtraction equilibrium SKM data from SKM data taken under applied bias. As a result of this procedure, a surface voltage drop profile will be obtained that have to conform to the bulk voltage drop profile.

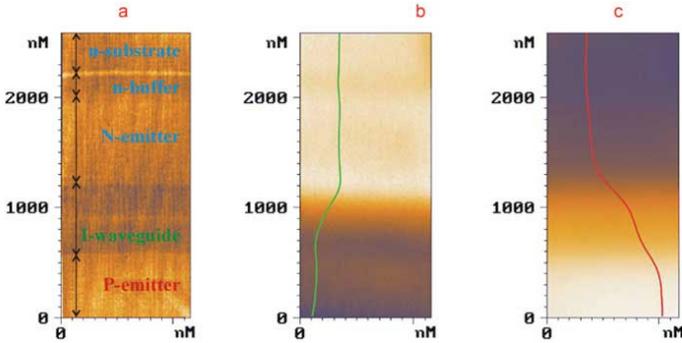


Fig. 1 Topography (a) and SKM data images of GaAlAs/GaAs laser diode (b - nonbiased, c - forward biased)

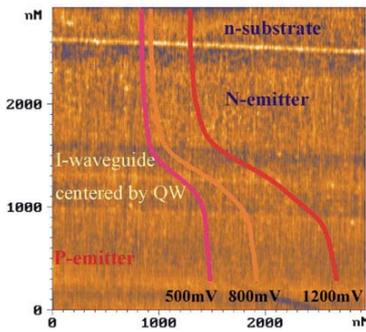
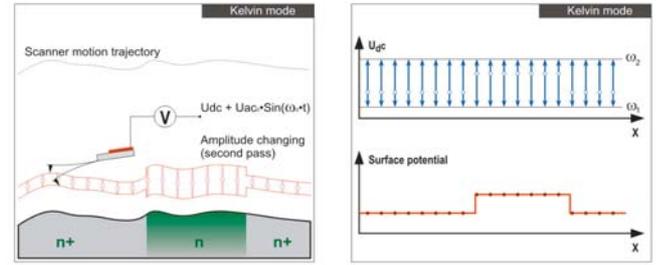


Fig. 2 Surface voltage drop profiles

In Fig. 2 the surface voltage drop profiles are displayed over the cleavage surface topography image of the studied device. Three experimental profiles obtained for 500, 800 and 1200 mV biases increase monotonously in the waveguide region, that is in a good agreement with bulk voltage drop modeling (for simulations one can use device simulator SimWindows available by internet). One should underline, however, that the measured surface voltage drops are about half of the applied biases. Besides, experimental profiles reveal a nonzero potential on the grounded n-substrate and some voltage drop in both highly doped emitters. These deviations from the expected picture of the voltage drop results from the convolution of the instrumental function with true voltage drop. AFM images are obtained by commercial SPM P47H with CSC12 cantilevers.

On the next pages description of the used lateral force mode and the details of image obtaining are presented. For more information see also paper NN. Additional information about laser structures were received with usage of Contact EFM, Kelvin mode.

SPM mode



Kelvin probe force microscopy (Kelvin mode of Scanning Probe Microscopy) was invented for measuring contact potential difference between the probe and the sample [1]. At present time Kelvin mode is based on the two-pass technique. In the first pass the topography is acquired using standard Semicontact mode (mechanical excitation of the cantilever). In the second pass this topography is retraced at a set lift height from the sample surface to detect the electric surface potential $\Phi(x)$. During this second pass the cantilever is no longer excited mechanically but electrically by applying to the tip the voltage V_{tip} containing dc and ac components

$$V_{tip} = V_{dc} + V_{ac} \sin(\omega t)$$

The resulting capacitive force F_{cap} between the tip and a surface at potential V_s is

$$F_{cap} = (1/2) (V_{tip} - \Phi(x))^2 (dC/dz)$$

where $C(z)$ is the tip-surface capacitance. The first harmonic force

$$F_{cap w} = (dC/dz) (V_{dc} - \Phi(x)) V_{ac} \sin(\omega t)$$

leads to suitable cantilever oscillations. The feedback then changes the dc tip potential V_{dc} until the w component of the cantilever (and accordingly w component of the tip-force) vanishes, e.g. $V_{dc}(x)$ became equal to $\Phi(x)$. So mapping $V_{dc}(x)$ reflects distribution of the surface potential along the sample surface. If no special tip-sample bias voltage is applied this distribution is Contact Potential Difference distribution.

References

1. Appl. Phys. Lett. 58, 2921 (1991).

Practice details

Preparation steps for the SKM study of the heterostructure on the cleavages

- a) A 50 mkm scanner, an optical viewing system, and x-y translation stage may be necessary to have comfortable conditions of measurements.
- b) The samples with freshly prepared cleavages may be gripped in a miniature vice or fixed by a super glue on the vertical side of the small block. A double-side scotch can be used instead of glue, but when the linear sizes of the sample are less than 1 mm, the scotch is often not effective means for good fixation.
- c) Long side of the cantilever beam should be perpendicular to the cleavage edge to facilitate the search of the edge in the scan area. This space arrangement of cantilever and sample will be also optimal to decrease the role of the parasitic capacitance, since it permits to have minimal area of overlapping between the surfaces of the cantilever and the sample.
- d) Before approach, the tip should be placed in the center of the scanning area, and approximately 15-20 mkm away of the edge of the cleavage (for the reference, when using the optical viewing system, the width of the cantilever NSG11 is about 50 m).
- e) When the edge has been found, first we move the tip several microns away of the edge, then withdraw the tip from the sample (switch out a feedback loop), close a microscope by the protective cover, suspend the whole system, and finally switch on a feedback loop again.
- f) The sequence of actions to adjust SKM mode (it can be found in the manual "SKM setting").
- g) To optimize the feedback action for contact potential difference (CPD) measurements we also may recommend to find the phase domain (180 degree) allowing negative feedback loop gain, and to adjust the phase shift of the input signal in the middle of that domain.

Specific parameters of the scan can be found in http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/f8opt.zip, http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/f9opt.zip and http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/f8mtdt.zip, http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/f9mtdt.zip. Lift values, applied for data acquisition in SKM mode, are also given in scan descriptions.

To extract the surface voltage drops profiles, two set of data were used: topography height profile and CPD profile (SKM data) taken under equilibrium, and those measured under some bias applied across the studied device.

To improve resolution the profiles may be averaged along the direction parallel to interfaces of the heterostructure. Using topography data we mark the heterostructure interfaces on the CPD profiles, then conform and align the equilibrium CPD profile to the CPD profile under some bias, and finally subtract the first profile from the second one to obtain the surface voltage drop profile.

Basics

As it was already noted, there are some deviations in measured surface voltage drops from actual voltage drop. The whole voltage drop between n- and p- sides of the structures is more than two times lower, than the actual drop. At higher biases (curve 1200 mV) the voltage appears to drop also on the well conductive emitters. As the bias grows, the SKM detects an increasing positive potential on the grounded n-substrate. All these effects can be explained taking into account the contribution of the capacitance of the probe sides into the SKM signal. To illustrate, let's we have absolutely flat probe (e.g. cantilever without a pyramidal tip). Then, the potentials measured on n- and p- sides of the structure would be almost equal to each other. The ideal situation (the measured by SKM CPD profile coincides with the actual surface potential profile) can be only realized when the contribution of the very end of the tip to the whole probe - sample capacitance is overwhelming. In reality the contribution of the different parts of the probe into the whole capacitance are comparable, and one needs to account for all those additional capacitance using a convolution procedure. However, the convolution procedure will hardly reveal reliably some additional features in the surface voltage drop profiles, since it will just improve spatial resolution and increase the amplitude of the features in the measured signal. Therefore we consider the approach to analyze the measured surface voltage drop as completely substantiated.

In the scan description, it was also assumed, that the bulk potential variations in semiconductor are traced by the surface potentials. Indeed, the local values of surface and bulk potentials of the

semiconductor are rigidly bound with each other through the magnitude of the subsurface barrier. Our assumption means that the profile of the subsurface barrier across the semiconductor structure is independent on the applied bias distribution. It can be considered as a first approximation, whose effectiveness is confirmed not only by the presented above results, but also by the following arguments.

The magnitude of the subsurface barrier measured in air for GaAs-based materials is $\Phi_B \sim 1V$. This barrier is determined by the charge trapped on the surface states, which must be equilibrated (screened) by the opposite charge of impurities in the depletion region of the semiconductor. For impurity concentrations in the range of $1-5 \times 10^{18} \text{ cm}^{-3}$ typical for highly doped conductive layers of a laser diode, the depth of the depletion region or the length in which the surface charges are screened is $l \sim 10^{-6} \text{ cm}$. The electric field in the subsurface region of a highly doped semiconductor is $E_S \Phi_B / l \sim 10^8 \text{ Vm}^{-1} = 10^6 \text{ Vcm}^{-1}$, and the corresponding density of the surface charge is $s_{SS} \sim \epsilon_0 \times E_S \sim 10^{-2} \text{ Cm}^{-2} = 10^{-6} \text{ Ccm}^{-2}$. At the same time, between two conductive (n- and p-doped layers) parts of the laser diode, separated by the isolating (undoped) waveguide region of width $L \sim 10^{-4} \text{ cm}$, there exists a similar potential difference $\Phi \sim 1V$. Due to the electric field in this region, the additional density of charge may be induced on the surfaces of the conductive layers of the diode, $s_A \sim \epsilon_0 \times \Phi / l \sim 10^{-8} \text{ Cxcm}^{-2}$. According to the estimation $s_A \ll s_{SS}$ we can conclude that, at least in the highly doped layers the subsurface barrier remains constant in the range of biases of a few volts. A more precise approach, extending beyond this approximation, is the object of further work. The proximity of the AFM tip to the semiconductor surface may influence the value of the subsurface barrier, although in air this effect seems not to be dominant for GaAs-based semiconductors having an extremely high density of the surface states. The subsurface barrier may also be changed in the light emitting structure due to the surface photo-voltage effect, or by a sizable variation of the screening length of the semi-insulating waveguide layer under injection of non-equilibrium carriers.

References

1. H.O. Jacobs, H. F. Knapp, A. Stemmer, Review of Scientific Instruments, March 1999, Volume 70, Number 3, 1756-1760, "Practical aspects of Kelvin probe force microscopy".
2. F. Robin, H. Jacobs, O. Homan, A. Stemmer, and W. Bachtold, Applied Physics Letters, May 15, 2000, Volume 76, Issue 20, pp. 2907-2909, "Investigation of the cleaved surface of a p-i-n laser using Kelvin probe force microscopy and two-dimensional physical simulations".

- Lateral resolution of surface potential measurements.

■ Lateral resolution of noncontact EFM versus SKM.

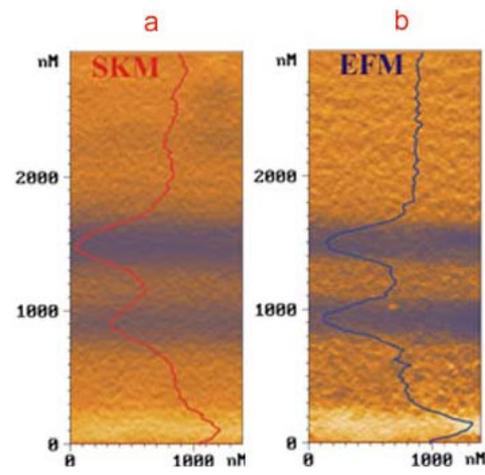


Fig. 1

To improve the instrumental function in electrostatic measurements, the ratio of the parasitic capacitance of the tip side and of the flat part of the cantilever to the useful capacitance of the tip apex should be minimized. EFM signals are proportional to the second derivative of the whole capacitance, unlike the first derivative dependence of SKM signals. Therefore the contribution of the tip apex in EFM mode is more substantial, than in SKM mode [10]. Fig.1 permits to compare lateral resolution of EFM with SKM. SKM and EFM data were taken from the same sample area and at the same bias conditions (1200 mV on p-contact) as data of Fig. 2c. The NT-MDT image processing software permits to represent data in different mode. We utilize in Fig. 1 2G images (gradient mode, that is convenient to annualize the surface electric field). The profiles of signals averaged along the direction

of the interfaces are displayed over the corresponding SKM and EFM images. SKM data were acquired at the negative lift value -20 nm, set point magnitude of cantilever vibrations for topography acquisition was ~40 nm. EFM data were measured at lift value -10 nm and free magnitude of cantilever vibrations reduced to ~30 nm. Comparison of fig.1a and fig.1b shows that, in spite of larger gap between the mean tip position and the surface, EFM demonstrates notably narrower spikes of surface electric field, than SKM.

- Force modulation by electrostatic excitation of the cantilever in contact mode (contact EFM).

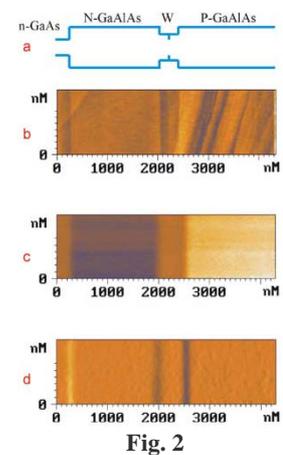


Fig. 2

An interesting possibility that may also grade up the instrumental function in electrostatic measurements is the application of force modulation by electrostatic excitation of the cantilever in contact mode. This mode appeared as dynamic contact electrostatic force microscopy (DC EFM) [11]. In contact mode, by the same reasons as

in noncontact mode, the electric forces act between the cantilever and the sample, and they can be measured in the same manner as in SKM. The current flow through the contact area may decrease those forces, but it is extremely small, since there are thick (a few nanometers) oxide layers on the tip and sample surface. The results of DC EFM study of another AlGaAs/GaAs based n-i-p laser are presented in Fig. 2. Along with the topography image in Fig. 2b, the simultaneously obtained first harmonic signal (phase-sensitive MagSIN) image (Fig. 2c) and the gradient of that signal in 2G image (Fig. 2d) are given. Comparison of Fig. 2d and Fig. 2b shows that measured surface electric field is mainly concentrated at the waveguide/emitters interfaces. Broadening of these spikes into emitters permits to evaluate lateral resolution on the level of 100 nm. Demonstrated high lateral resolution of electric forces is the consequence of the close proximity of the tip to the surface in contact mode, when the contribution of the tip apex capacitance is maximized. However energy resolution of DC EFM is moderate, since induced vibration of the

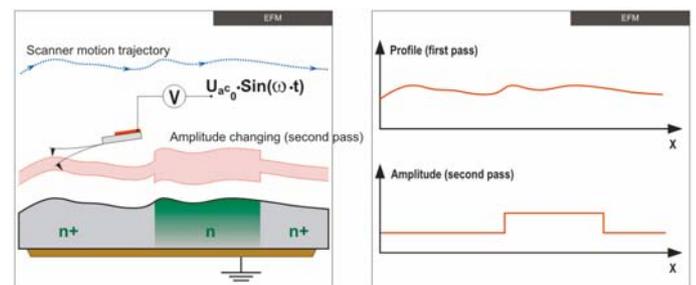
cantilever with both ends fixed are small. To reach satisfactory signal to noise ratio, one needs to apply rather high alternative voltage to the cantilever (3-4 V r.m.s. in comparison with ordinary 0.5-1 V r.m.s in two pass techniques). It is worth mention that DC EFM mode is possible on the first generation of NT-MDT tools, e.g. P-4-SPM with resonant block [4,5].

AFM images are obtained by commercial SPM P47H with CSC12 cantilevers.

On the next pages description of the used lateral force mode and the details of image obtaining are presented. For more information see also paper NN. Additional information about laser structures were received with usage of Contact EFM, Kelvin mode.

SPM mode

- Noncontact EFM



Generally Electric Force Microscopy (EFM) can be used in several modes, depending on the type of the sample under investigation and kind of the required information.

The most useful of them is Non-Contact EFM mode based on the two-pass technique. During the second pass the cantilever is piezodriven at resonant frequency and cantilever is grounded or biased by dc voltage V. Capacitive tip-sample electric force (or rather its derivative) leads to resonance frequency shift. Accordingly amplitude of cantilever oscillation decreases and phase of oscillation changes [1]. Both amplitude and (or) phase of oscillation deviations can be measured and electric potential distribution over the sample surface can be imaged.

This mode of operation has some advantages comparatively to Scanning Kelvin Microscopy (SKM). Registered amplitude or phase deviations images are determined by capacitive tip-sample electric force derivative, e.g. second derivative of tip-sample capacity. As a result Non-Contact EFM leads to higher resolution because the ratio of the parasitic capacitance of the tip side and of the flat

part of the cantilever to the useful capacitance of the tip apex should be minimized [2, 3].

References

1. J. Appl. Phys. 61, 4723 (1987).
2. Appl. Phys. Lett. 52, 1103 (1988).
3. Nanotechnology 12, 485 (2001).

Contact EFM

As in Noncontact EFM in Contact EFM the cantilever is biased directly by $V_{\text{tip}} = V_{\text{dc}} + V_{\text{ac}} \sin(\omega t)$, where V_{ac} is referred to as the driving voltage. Scanning is executed as in usual Constant Force mode and simultaneously the electric forces are measured. The capacitive force $F_{\text{cap}}(z)$ between the tip and a surface at potential V_s is

$$F_{\text{cap}}(z) = (1/2) (V_{\text{tip}} - V_s)^2 (dC/dz)$$

where $C(z)$ is the tip-surface capacitance dependent on tip geometry, surface topography and tip-surface separation z .

In contact EFM the influence of the first harmonic of $F_{\text{cap}}(z)$ on the cantilever oscillation is registered. Contact EFM can be applied to semiconductor structures. The current flow through the contact area may decrease those forces, but it is extremely small, since there are usually thick (a few nanometers) oxide layers on the tip and sample surface.

As an example data of GaAlAs heterostructures measuring are given.

References

1. Rev. Sci. Instr. 70, 1735 (1999).

Practice details

A noncontact EFM mode utilizes the phase shift measurements (absolutely in the same manner as in Phase Imaging mode). The name "noncontact EFM mode" is given to underline the electrostatic origin of the phase contrast.

See practice details of Voltage drops, Kelvin mode, and practice details Local elasticity, Force modulation mode. Specific parameters of the scan can be found in

http://www.ntmdt.ru/Applications/Dev_nanostr/f100pt.zip,
http://www.ntmdt.ru/Applications/Dev_nanostr/f110pt.zip and

http://www.ntmdt.ru/Applications/Dev_nanostr/f10mdt.zip,
http://www.ntmdt.ru/Applications/Dev_nanostr/f11mdt.zip

AFM images are obtained by commercial SPM P47H with NSG11 cantilevers.

On the next page description of the basics of the used mode are presented.

Additional information about laser structures were received with usage of Kelvin mode.

Basics

Lateral resolution improvement of surface potential measurements.

References

1. H. O. Jacobs, P. Leuchtman, O. J. Homan, and A. Stemmer, Journal of Applied Physics, August 1, 1998, Volume 84, Issue 3, pp. 1168-1173, "Resolution and contrast in Kelvin probe force microscopy". (http://www.ntmdt.ru/Applications/Dev_nanostr/7.pdf).
2. P. Girard, Nanotechnology, 2001, Number 12, pp. 485-490, "Electrostatic force microscopy: principles and some applications to semiconductors". (http://www.ntmdt.ru/Applications/Dev_nanostr/10.pdf).
3. U. Rabe, K. Janser, and W. Arnold, Review of Scientific Instruments, September 1996 Volume 67, Issue 9, pp. 3281-3293 "Vibrations of free and surface-coupled atomic force microscope cantilevers: Theory and experiment". (http://www.ntmdt.ru/Applications/Dev_nanostr/5.pdf).
4. J. V. Hong, Sang-il Park, and Z. G. Kim, Review of Scientific instruments, March 1999, Volume 70, Number 3, pp 1735-1739, "Measurements of hardness, surface potential, and charge distribution with dynamic contact mode electrostatic force microscope". (http://www.ntmdt.ru/Applications/Dev_nanostr/11.pdf).
5. A. Ankudinov, V. Marushchak, A. Titkov, V. Evtikhiev, E. Kotelnikov, A. Egorov, H. Riechert, H. Huhtinen, R. Laiho, Physics of Low Dimensional Structures, 2001, Issue ?, pp. 9-11, "Fine structure of the inner electric field

in semiconductor laser diodes under the applied bias studied by EFM”.

http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/4.zip

6. A. V. Ankudinov, E. Yu. Kotelnikov, A. A. Kantselson, V. P. Evtikhiev, A. N. Tikov, Semiconductors, July 2001, Volume 35, Issue 7, pp. 840-846, “Cross-Sectional Electrostatic Force Microscopy of Semiconductor Laser Diodes”.

(http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/12r.pdf in russian).

Additional information about laser structures were received with usage of [Kelvin mode](#).

- Laser structures Chemical Contrast Asquisition

For the atomically flat, featureless cleavages of heterostructures, additional chemical contrast of the constituent layers can be achieved in lateral force imaging (LFM) [1]. This SPM mode allows measuring friction variations on chemically nonidentical regions. An application of LFM permits, for example, to visualize a waveguide region of the ZnMgBeZe/GaAs laser structure already mentioned above (see upper figure). In figure below there are two LFM images measured in opposite fast scanning directions. The contrast reversal of the 200-nm wide stripe is observed when compare the left parts of images. That means different friction (reduced) on the material of the waveguide compared to the material of surrounding emitters.

The lateral resolution of the LFM mode may be very high, what is illustrated in next figure, where five 5nm-thick GaInSbAs QWs separated by 25nm-thick GaSb barriers are clearly seen as the lines of reduced friction.

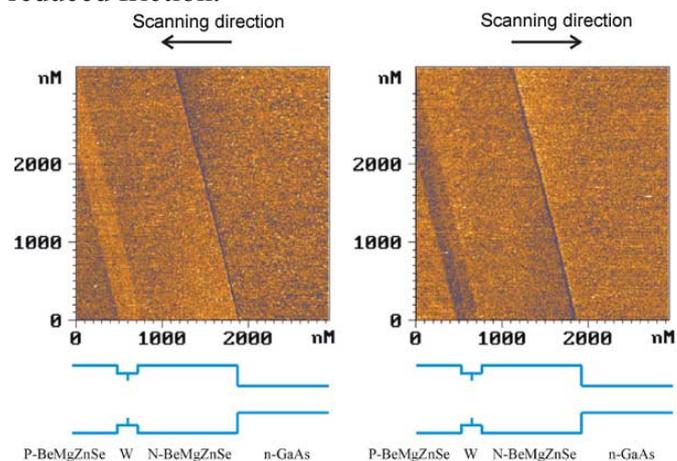


Fig. 1

AFM images are obtained by commercial SPM P4 with CSC12 cantilevers.

On the next pages description of the used lateral force mode and the details of image obtaining are presented.

Additional information about laser structures were received with usage of Contact EFM, Kelvin mode.

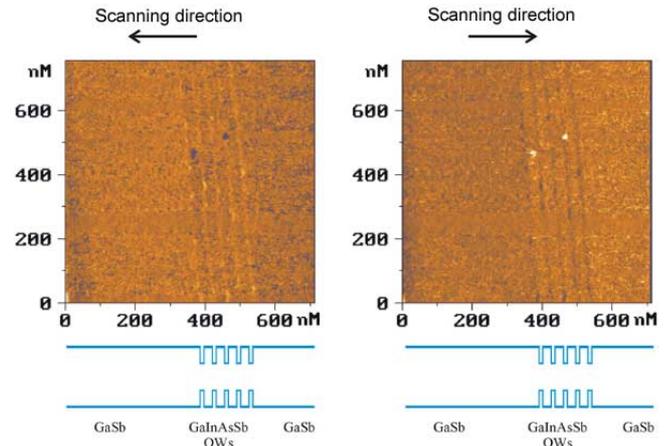
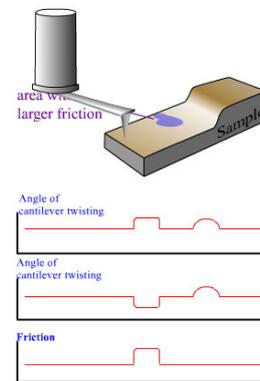


Fig. 2

SPM mode



Lateral Force mode allows to distinguish areas with different friction and also to obtain edge-enhanced images of any surface. This capability may be used in conjunction with topographical images during one scan to characterize your samples more completely.

The physical basics of the Lateral Force mode are as follows. When scanning in the [Constant Force mode](#) perpendicularly to longitudinal axis of the cantilever, besides the cantilever's deflection in the normal direction, an additional torsion bending of the cantilever occurs. It is caused by the moment of forces acting on the tip. With minor deflections, the angle of torsion is proportional to the side (lateral) force. The cantilever's torsion bending is measured by the microscope optical recording system.

When moving over a flat surface with zones of different friction factors, the angle of torsion will be changing in every new zone. This allows measuring of the local friction force. If the surface is not absolutely flat, such an interpretation is

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complicated. To distinguish zones of different friction and relief influence one can utilize second pass on the same line in opposite direction. Nevertheless, this type of measuring allows obtaining images with clearly seen minor relief details and facilitates their search. In addition, the lateral force measuring mode easily provides the atomic resolution on mica and some other laminar materials.

Lateral Force mode has important usage for semiconductors, polymers, deposited films, data storage devices, investigative studies of surface contamination, chemical speciation and frictional characteristics, and a growing list of new applications.

References

1. Phys. Rev. Lett. 59, 1942 (1987). 2. Magonov, Sergei N. Surface Analysis with STM and AFM. Experimental and Theoretical Aspects of Image Analysis. VCH 1996.

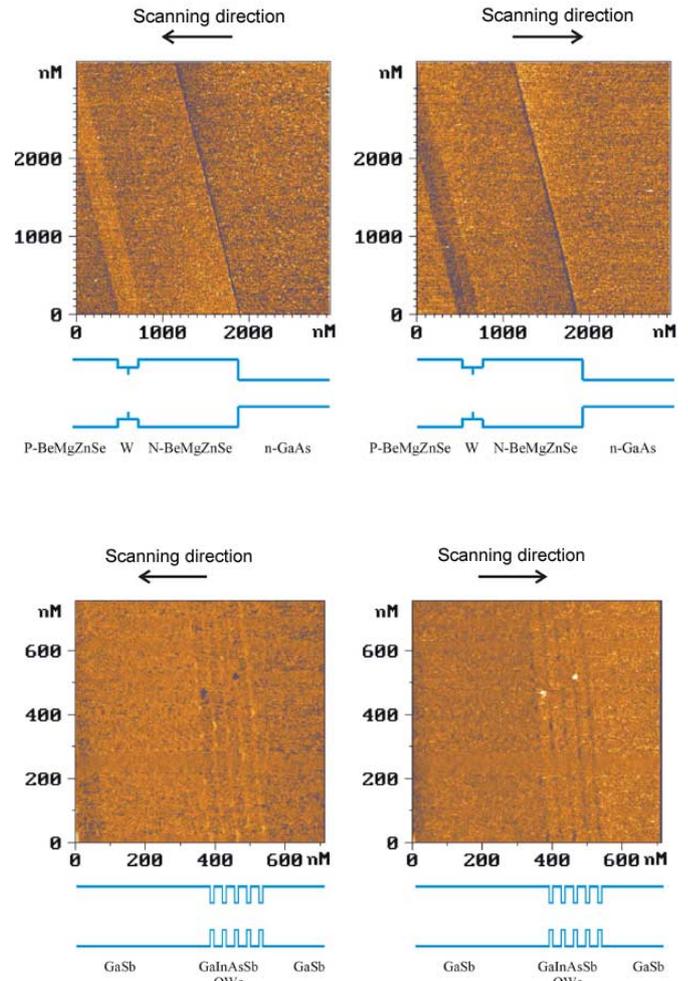
Practice details

Practice details of Chemical Contrast Acquisition.

See [practice details](#) of topography acquisition, ZnSSe/GaAs laser structure.

Cantilevers CSC12 with rectangular beam and small force constant were used. When studying the friction forces on the heterostructure cleavage, the long side of the cantilever beam should be set parallel to the sample edge. To increase the sensitivity of mode, the signal of lateral deflection of the cantilever must be chosen close to zero. To avoid artifacts in LFM image due to coupling between vertical and lateral cantilever deflection, slow scanning speed and high feedback gain are preferable.

Specific parameters of the scan can be found in http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/f5opt.zip and http://www.ntmdt.ru/Appl_Notes/Dev_nanostr/f5m dt.zip.



On the next page description of the used lateral force mode basics are presented.

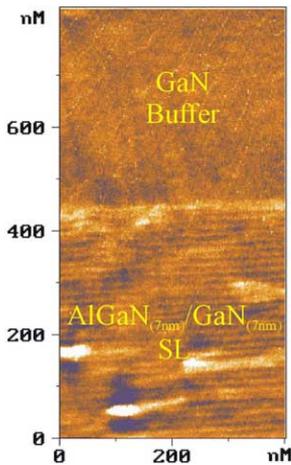
Basics

Grounds of Chemical Contrast Acquisition.

References

1. A. V. Ankudinov, A. N. Titkov, T. V. Shubina, S. V. Ivanov, P. S. Kop'ev, H.-J. Lugauer, G. Reuscher, M. Keim, A. Waag, G. Landwehr, Applied Physics Letters, October 1999, Volume 75, Number 17, pp. 2626-2628, "Cross-sectional atomic force microscopy of ZnMgSSe- and BeMgSSe-based laser diodes".

- Local elasticity imaging



In some heterostructure samples, for example hexagonal GaN-based heterostructures epitaxially grown on sapphire substrates, the cleavage plane of epitaxial layers does not coincide with the cleavage plane of the substrate.

On the cleavages of such samples the relief height variations may exceed several hundreds nanometers. A fruitful way to reveal different epitaxial layers is to apply local elasticity (force modulation technique) mode. A local elasticity image of the short-period AlGaIn(7nm)/GaN(7nm) superlattice is shown in figure. Both buffer GaN layer and the periodical structure of the superlattice are clearly resolved in image. AlGaIn corresponds to the higher signal (brighter contrast). Careful examination of the long-period AlGaIn/GaN superlattices (20, 40 and 80 nm) demonstrated strong correlation between topography and local elasticity signals [6,7]. Namely, low (high) local elasticity signal was observed at the relief elevations (depressions). Simple analysis shows decreasing of the tip-surface contact stiffness at elevations compared to depressions. We believe, therefore, that the local elasticity signal behavior on short-period superlattice has also mainly topographical origin. The interdependence of the topography relief and heterostructure composition, revealed by the local elasticity mode, has following reasons. Lattice constant of AlGaIn is less than that of GaN, therefore GaN layers are compressed, whereas AlGaIn layers are tensed in the interface plane. On the free surface the bulk periodic strain variations have to be balanced by the Laplas pressure under convex and concave relief: compressions create the convex relief elevations and tensions do the concave relief depressions.

AFM images are obtained by commercial SPM P4 with CSC12 cantilevers.

On the next pages description of the used [force modulation technique](#), its [grounds](#) and the [details](#) of image obtaining are presented.

Additional information about laser structures was received with usage of [Contact EFM](#), [Kelvin mode](#).

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