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Research Article

Methods of dislocation structure characterization in $A^{III}B^{V}$ semiconductor single crystals

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Abstract

The development pace of advanced electronics raises the demand for semiconductor single crystals and strengthens the requirements to their structural perfection. Dislocation density and distribution pattern are most important parameters of semiconductor single crystals which determine their performance as integrated circuit components. Therefore studies of the mechanisms of dislocation nucleation, slip and distribution are among the most important tasks which make researchers face the choice of suitable analytical methods. This work is an overview of advanced methods of studying and evaluating dislocation density in single crystals. Brief insight has been given on the main advantages and drawbacks of the methods overviewed and experimental data have been presented. The selective etching method (optical light microscopy) has become the most widely used one and in its conventional setup is quite efficient in the identification of scrap defects and in dislocation density evaluation by number of etch pits per vision area. Since the introduction of digital light microscopy and the related transfer from image analysis to pixel intensity matrices and measurement automation, it has become possible to implement quantitative characterization for the entire cross-section of single crystal wafers and analyze structural imperfection distribution pattern. X-ray diffraction is conventionally used for determination of crystallographic orientation but it also allows evaluating dislocation density by rocking curve broadening in double-crystal setup. Secondary electron scanning electron microscopy and atomic force microscopy allow differentiating etch patterns by origin and studying their geometry in detail. Transmission electron microscopy and induced current method allow obtaining micrographs of discrete dislocations but require labor-consuming preparation of experimental specimens. X-ray topography allows measuring bulky samples and also has high resolution but is hardly suitable for industry-wide application due to the high power consumption of measurements.

Digital image processing broadens the applicability range of basic dislocation structure analytical methods in materials science and increases the authenticity of experimental results.

Keywords

semiconductor single crystals, electronics materials, dislocation density, digital light microscopy, X-ray and electron microscopy

1. Introduction

Dislocation density is one of the main structural parameters of semiconductor single crystals. Dislocations have direct and indirect effects on the electrophysical properties of materials [1–4].

The direct effect of dislocations is the formation of broken bonds at boundaries of two-dimensional imperfections, leading to the generation of more carriers. The presence of unsaturated covalent bonds is the origin of the acceptor nature of dislocations. The indirect effect of dislocations is the generation of chemical potential gradient which in turn accelerates diffusion in defect plane, resulting in the formation of impurity atmospheres (Cottrell clouds).

There are two main dislocation structure formation mechanisms during single crystal growth:

1. Dislocation inheritance directly from the seed. The dislocation multiplication activation energy is in this case far lower than the dislocation nucleation energy [1].
2. Dislocation nucleation during growth due to induced thermal stresses and energetically favorable stress relaxation through dislocations. The Czochralski method cannot completely avoid the generation of temperature gradients (radial and axial). The radial temperature gradient is determined by the diameter of the growing ingot and is among the main factors causing problems in the growth of large-diameter crystals (150+ mm). The axial gradient is determined by the length of the ingot and increases as the ingot grows longer [2].

By analogy with impurity atoms and intrinsic point defects, dislocations can produce additional electron states in the band gap, this accounting for their direct effect on the performance of product ICs. The electrical activity of dislocations typically exerts a negative effect on the properties of semiconductor devices, e.g. by causing premature breakdown in device regions where dislocations cross a *p–n* junction. Dislocations also affect the lifetime of free carriers. In pure crystals they often limit the lifetime of nonequilibrium carriers [3–8].

It was shown for single crystal Ge that a decrease in the dislocation density from 10^5 to 10^3 cm^{-3} leads to a substantial increase in the transistor current gain (approximately twofold) [9].

Along with dislocation density, dislocation distribution inhomogeneity may have a great effect on the performance of product ICs as was shown earlier for semi-insulating GaAs [10]. High-temperature growth dislocations have the greatest effect on the properties of single crystals due to the high diffusion mobility of nonequilibrium intrinsic point defects. X-ray topography of silicon wafers showed that the scrap percentage is the highest for transistors made from peripheral parts of a single crystal wafer [11]. It was noted that the dislocation structure of the substrate is inherited during the synthesis of an epitaxial layer, with the inhomogeneity of the dislocation distribution in the substrate being retained throughout further IC fabrication process stages [10–12].

Dislocation identification is quite a power-consuming process and nowadays the high development of analytical methods allows one to use either qualitative or quantitative approaches. By and large all the existing methods can be divided in 1) methods used in quality control at industrial factories; 2) research methods.

The former category includes the selective etching method which is based on the assessment of dislocation etch pit density **(EPD)** and is rather a qualitative one in its conventional variant. This method also allows solving some research tasks, e.g. studying the effect of annealing on the dislocation structure of single crystals [2]. X-ray diffraction is conventionally used for the determination of crystallographic orientation but it also allows evaluating dislocation density by rocking curve broadening in double-crystal setup.

Research methods include scanning and transmission electron microscopy, X-ray topography and the induced current method. Thanks to their high resolution methods of this category allow studying the interaction between single dislocations, as well as plastic deformation mechanisms.

The aim of this work is to systematize available information on the advantages and drawbacks of each specific method for generating an algorithm of further research steps.

2. Selective etching method

The selective etching method is the most streamlined one and indispensable in industrial conditions since it allows solving a wide range of tasks in the quality control of as-grown semiconductor single crystals. At a preliminary qualification stage this method can be used for structural defect control aiming at the detection of such defects as low-angle boundaries, polycrystalline regions, macroscopic pores and cracks [5–7].

The second stage is etch pattern (pit) counting at dislocation emergence sites. Dislocation etch pits are pinnacled cavities the symmetry of which is determined by the crystallographic orientation of the surface. This fact originates from the difference in the etching rates for different crystallographic planes having different atomic layer packing densities [6].

Dislocation density (N_D) in semiconductor single crystals is measured by visually counting the number of etch pits under an optical microscope in several vision fields. The choice of vision fields is based on the crystallographic orientation of the wafer which may have either a relatively homogeneous dislocation density distribution with a maximum at the periphery of the wafer or a complex omega-shaped dislocation density distribution [2]. When counting etch pits one should only take into account pinnacled etch pits having similar shapes and sizes and

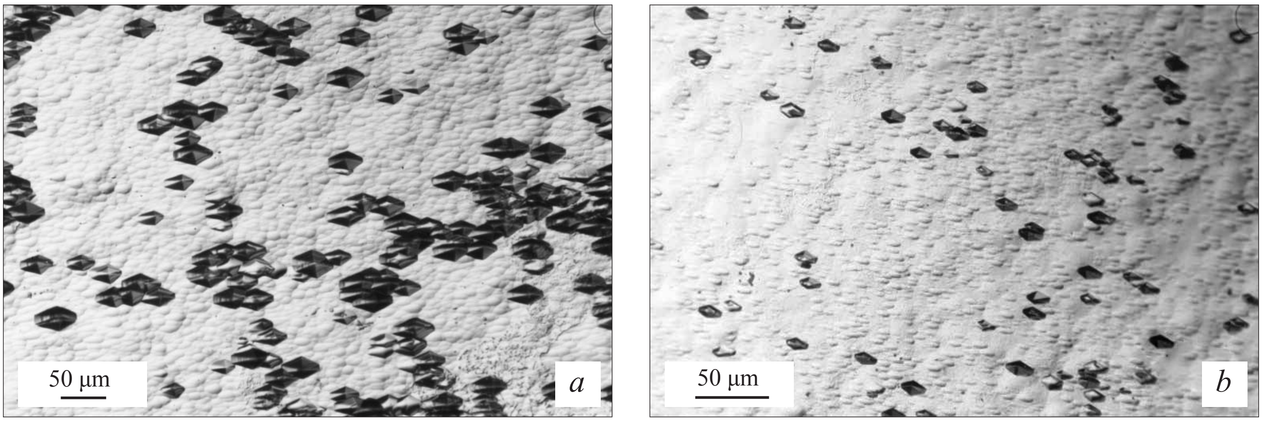

Figure 1. Etch patterns on {100}GaAs surface: (*a*) dislocation etch pits; (*b*) non-dislocation etch pits

forming at non-degenerate dislocation emergence sites (Fig. 1 *a*). Etch pits having flat or dish-shaped bottoms as well as etch pits whose dimensions are far smaller than those of dislocation etch pits (microdefect-related etch pits, see Fig. 1 *b*) should be ignored.

Optical light microscopy can provide for either qualitative structure characterization or structure comparison with reference scales, and therefore no quantitative measures can be introduced in the description of structural inhomogeneity with this method.

Currently, structural inhomogeneity is one of the most important criteria in the choice of semiconductor wafers by potential consumers, but the use of existing methods often yields contradictory results due to certain subjectivity in the selection of information-bearing features in images [13].

Potential solution to this problem can be qualitative metallography based on the measurement of the number of structural features in digital images (in the form of pixel intensity matrices, or the image brightness field) [14, 15]. This approach allows one to analyze individual frames or their panoramic clips on a specimen (product) scale, e.g. for characterizing etch pattern distributions in semiconductor single crystal wafers (Fig. 2).

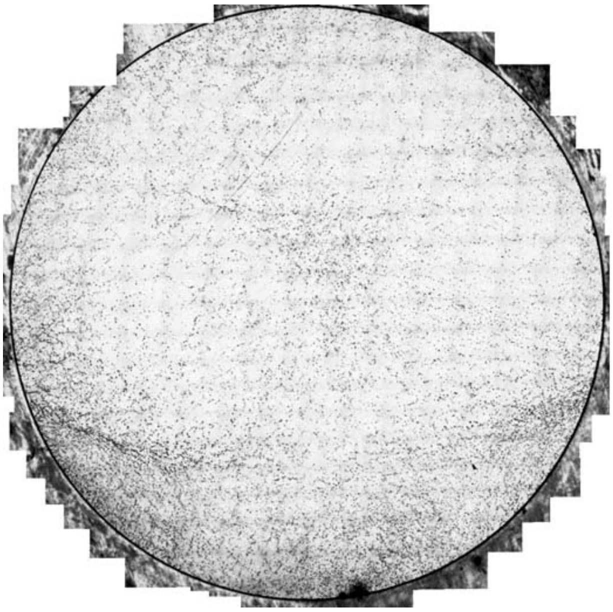

Figure 2. Panoramic image of dislocation etch pits distribution in single crystal {111} InAs

During the formation of a panoramic image (clipping of single frames), a frequent occurrence is a dark “grid” forming at superimpositions of individual frames and potentially causing errors to the overall pixel intensity matrix for the whole panoramic image. Correction of these defects often requires deep understanding of the nature of the test object and the role of individual structural components in the formation of its properties [14].

3. Scanning electron microscopy and atomic force microscopy

Along with conventional optical light microscopy, scanning electron microscopy **(SEM)** and atomic force microscopy **(AFM)** can also be used for the identification of etch patterns forming in the course of selective etching. These approaches allow analyzing etch pits of different sizes due to their high resolution [16].

Arbitrarily, etch patterns can be categorized by sizes as small (30–50 nm) pinnacled faceted cavities forming at edge dislocation emergence sites, medium-sized etch pits (50–150 nm) formed by mixed-type dislocations with a predominant screw component and large etch pits (150–200 nm) forming at screw dislocation pile-up emergence sites (nanotubes) as demonstrated earlier [17, 18] for GaN. Figure 3 shows a secondary electron SEM micrograph of etch patterns.

As can be seen from Fig. 3, due to the high resolution of the method, study of secondary electron scanning electron microscopic microroughness patterns allows analyzing etch patterns of different nature and different sizes, up to less than 1 mm sized etch pits. In this case the etch pits are hexagonal which is typical of hexagonal GaN.

Analysis of individual pixel brightness intensity distribution along the cross-section (the black line in Fig. 3) allows plotting the etch pattern profile. However characterization of the typical facet shape of the etch pits and determination of the bottom size limit for etch pit profile reconstruction require brightness field analysis for the entire image which can be facilitated by image digitalization.

AFM methods are used in the studies of the dislocation structure of semiconductor single crystals for the characterization of the surface profiles of etch patterns with a resolution of tens of nanometers or greater (Fig. 4).

Characterization of etched surfaces of single crystal wafers using this method allows analyzing the effect of different parameters on the formation of etch pit profiles as shown earlier [18]. Figure 5 shows reconstruction of etch patterns in single crystal GaN.

It can be seen from Fig. 5 that AFM allows detailed characterization of actual etch pit profiles unlike SEM which only allows quasi-profile characterization (Fig. 3).

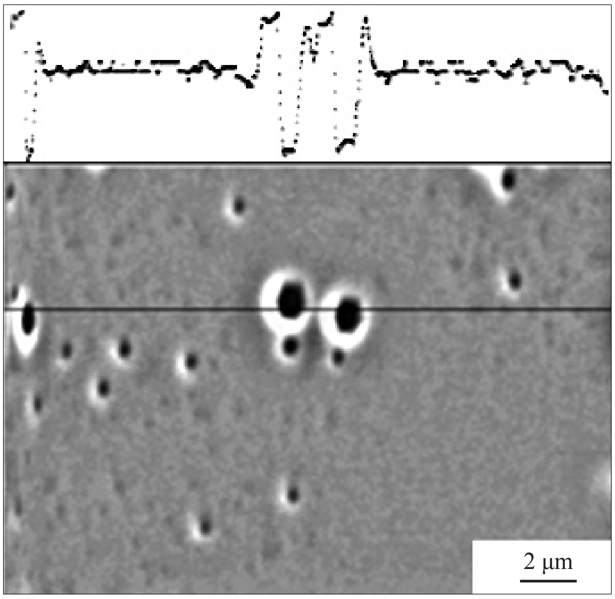

Figure 3. Secondary electron SEM micrograph of etch pits [17]

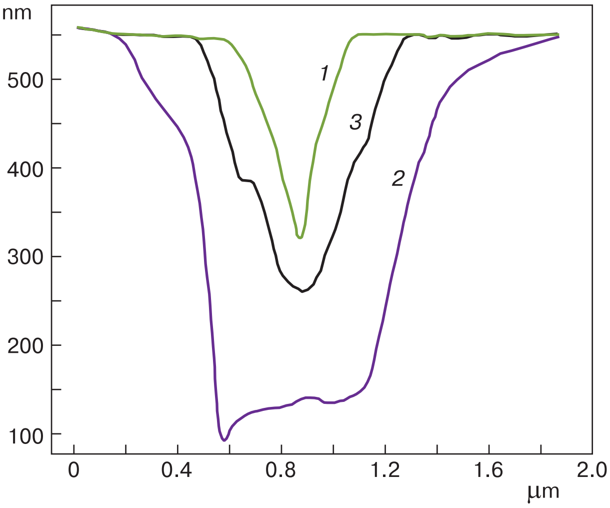

Figure 4. Reconstruction of etch pattern profiles in GaN [18]: (*1*) edge dislocation profile; (*2*) screw dislocation profile; (*3*) mixed dislocation profile

4. X-ray diffraction method

X-ray structural analysis is conventionally used in the metallurgy of semiconductor materials for the precision determination of as-grown single crystal end surface orientation [20]. Additional possible tool is point-wise analysis of rocking curve broadening and intensity allowing comparison between distributions of structural imperfections, e.g. stresses (microcrystalline deformation) and dislocations.

All crystalline materials contain various types of structural imperfections which exert an extremely strong effect on all the properties and processes taking place in the crystals. Structural imperfections can be produced by various lattice distortions that lead to the formation of different types of features in diffraction patterns [21]. Medium-sized mosaic blocks produce sharp reflection curves whereas small-block aggregations broaden the diffraction peak (crystal mosaic structure). Furthermore rocking curve asymmetry depends on the orientation of small-block aggregations.

A rocking curve is a curve showing the dependence of the reflection intensity (*I*) on the primary beam incidence angle onto the specimen at a constant angle between the radiation source and the detector [22]. Typical rocking curve parameters are intensity, halfwidth and angular position of the diffraction peak.

If a crystal contains mutually misoriented mosaic blocks, the overall rocking curve will show the rocking curves of individual blocks shifted relative to each other through the block misorientation angle. Rocking curve broadening allows one to draw a conclusion as to the development degree of the mosaic structure in the test single crystal, i.e., the degree of disorder in the crystal lattice of the test material. If multiple diffraction patterns are taken at different points of the single crystal, the array

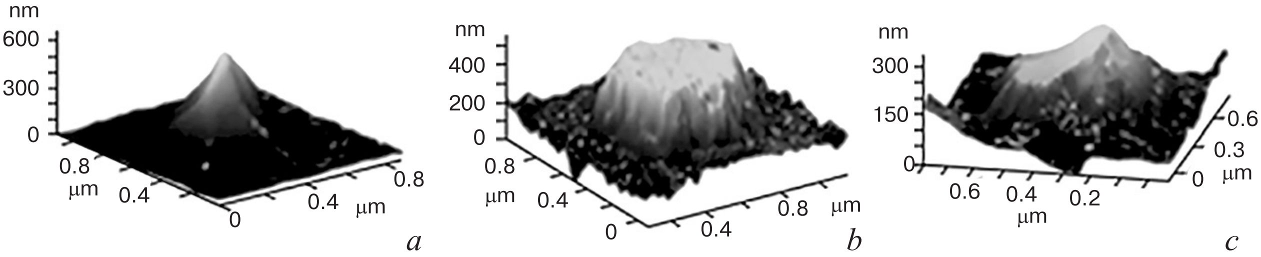

Figure 5. Reciprocal atomic force microscopic 3D image of etch pits [18]: (*a*) edge dislocation etch pits; (*b*) screw dislocation etch pits; (*c*) mixed dislocation etch pits

of the experimental data will allow characterization of the mosaic structure and micro-block pattern got the entire specimen surface.

Test single crystal specimens can be divided in three groups by diffraction peak broadening magnitude [21]:

Group 1: average rocking curve halfwidth within 4 arc min;

Group 2: rocking curve halfwidth from 4 to 6 arc min;

Group 3: average rocking curve halfwidth (averaged by several crystal surface points) above 6 arc min, as well as block-containing crystals.

In industrial conditions, analysis of structural imperfections in as-grown semiconductor single crystals is preceded by an optical light microscopy study which allows fast assessment of dislocation pile-up density and distribution pattern, as well as judging about the presence of a mosaic block structure in the test ingot. If the test sample does not contain block boundaries, it will be appropriate during further rocking curve pattern analysis to correlate the observed rocking curve broadening with dislocation density and thus to calculate the dislocation density. Figure 6 shows a typical rocking curve and a dislocation density distribution in the cross-section of a GaAs single crystal wafer.

From the classical viewpoint [23] this calculation method is unacceptable for low-dislocation crystals ($<10^8$ cm^{-2}) but it was reported [24] that for single crystals with a dislocation density of about 10^5–10^6 cm^{-2} this method can yield plausible results correlating with data of other materials science approaches.

5. X-ray topography

Diffraction topography (microscopy) holds a special place among nondestructive characterization methods of

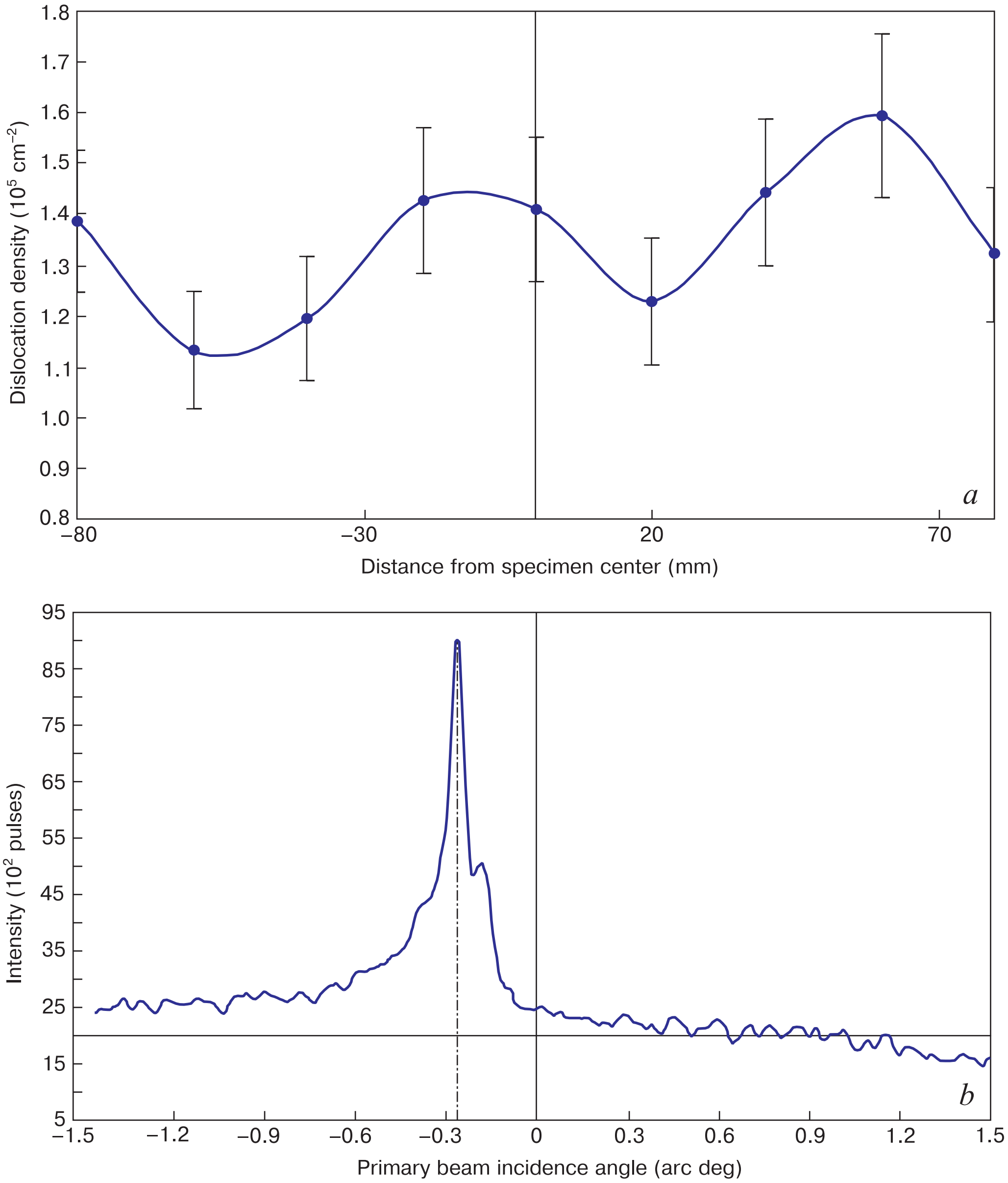

Figure 6. Results of X-ray characterization [22]: (*a*) dislocation distribution in specimen; (*b*) typical rocking curve

actual crystal structures. A distinctive feature of this approach is the possibility of characterizing relatively large specimens (with thicknesses on the order of 10 mm) and optically nontransparent single crystals and products. The high sensitivity of this method to lattice imperfections which allows characterization of block boundaries, microcracks, dislocations, domain boundaries and impurity segregation sites favors the wide application of X-ray microscopy techniques in various fields of science and engineering [25–27].

"The capabilities of X-ray topography include the determination of the type and spatial arrangement of dislocations in crystal bulk by transmittance topographic patterns obtained for two mutually orthogonal projections. Along with dislocations the method allows characterizing stacking faults, twin boundaries, growth layers caused by inhomogeneous distribution of impurities during crystal growth and point defect clusters. Analysis of contrast extinction for reflections from different types of planes allows judging about the type of lattice distortions" [28]. X-ray microscopic methods can be divided in the following groups:

1. Classical X-ray topographic methods: the Berg–Barrett method, the Schultz method, the Fujiwara method.
2. High resolution X-ray topography: the Lang method, image simulation and calculation methods;
3. Planar-wave topography, double- and triple-crystal setups;
4. Synchrotron radiation X-ray topography.

"For a two-beam case, X-ray wave field in a crystal is a superimposition of two types of Bloch waves having significantly different absorption coefficients. Therefore the images of dislocations will depend on whether both the types of Bloch waves contribute to the formation of the image and hence on the thickness of the crystal" [28].

The first systematized concepts regarding the formation of dislocation images in X-ray topographic patterns were put forward by A. Authier [27]. In accordance with his classification the image of a dislocation consists of three parts: the "direct" or "kinetic" image forming in a heavily distorted region of the dislocation elastic field due to the fact that the incident beam has a finite divergence and a specific spectral range, the "dynamic" image forming as a result of a redistribution of the wave field in the Bohrmann triangle and showing itself as a bright shadow in the topographic pattern, and finally the "intermediate" image resulting from the interference of the wave field propagating in the Bohrmann triangle with new wave fields generated in the heavily distorted region in the vicinity of the dislocation [27]. Figure 7 shows example of this image.

When carrying out digital image processing, one should contemplate the procedure of identifying information-bearing features in the image, e.g. dislocations, against surface texture features that are not of interest for the study. This can be implemented while designing the digital procedures of image binarization and filtration, taking into account the morphology of each specific structural feature. X-ray topography is indispensable due to its high resolution and the capability of characterizing massive specimens allowing one to study the target structure of the specimen and evaluate the length of discrete dislocations [29]. The main bottlenecks are the high cost of equipment and its low commercial availability.

6. Transmission electron microscopy

An electron beam propagating through a specimen undergoes scattering. Scattered electrons form an electron-optical image of the test object in the microscope. If the object is inhomogeneous its different regions will

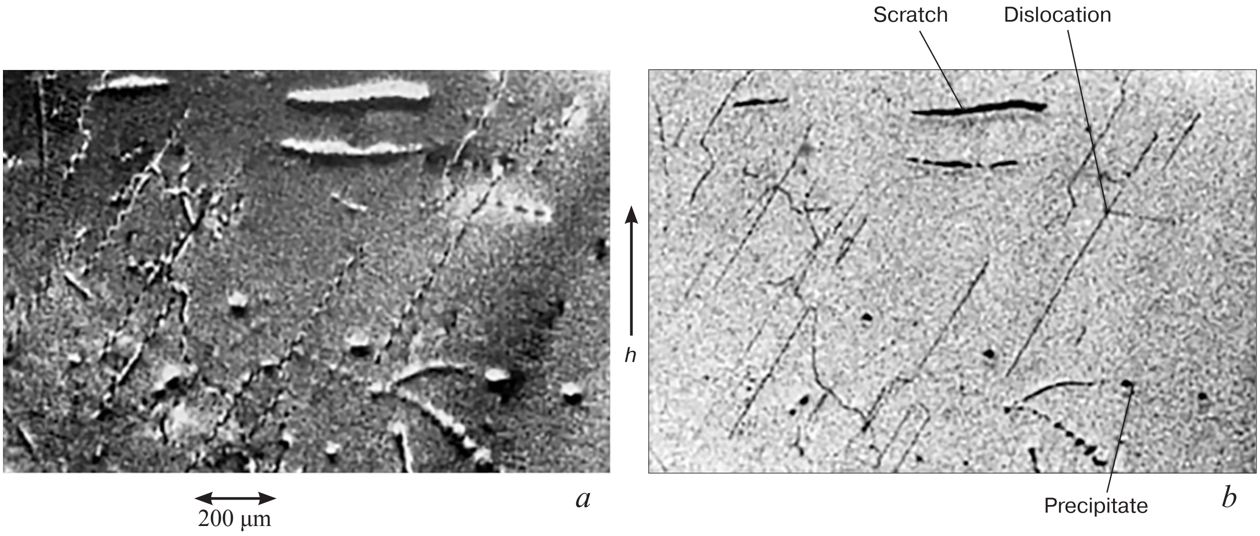

Figure 7. Synchronous double-crystal transmission topographic patterns of a (111) germanium single crystal, $\lambda \approx 0.035$ nm, (111) silicon crystal monochromator: (*a*) topographic pattern for a single crystal installed at the rocking curve peak angle; (*b*) topographic pattern for a single crystal installed at the rocking curve middle-slope angle. Dislocations, scratches and precipitates can be seen [20]

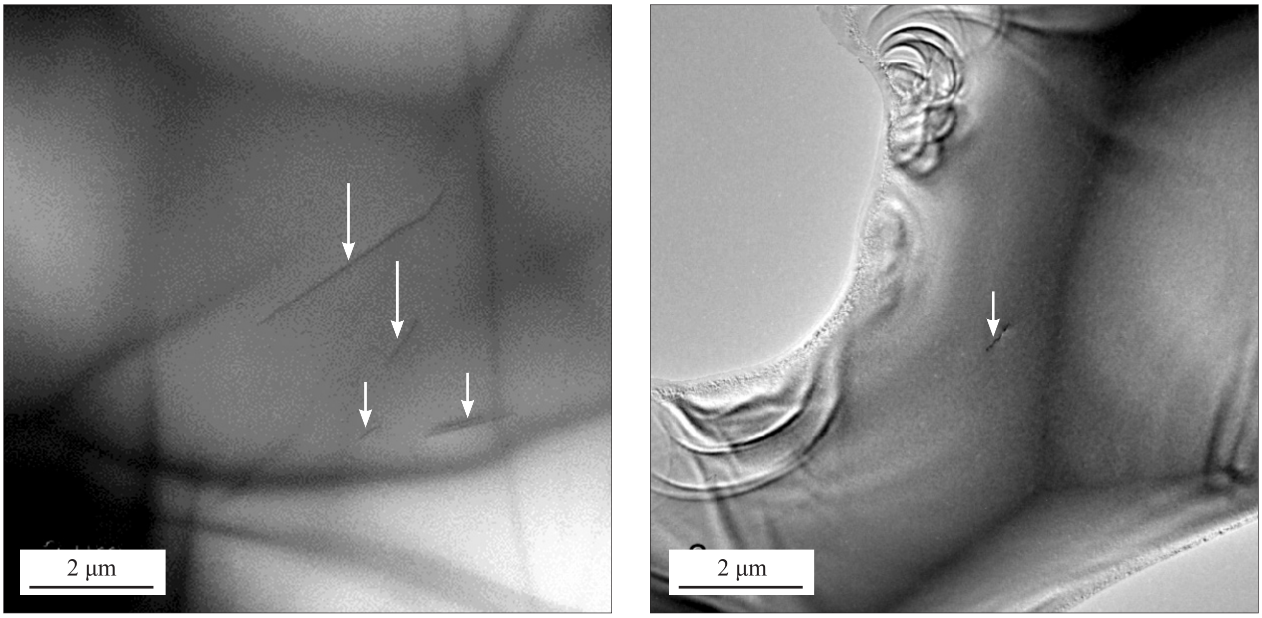

Figure 8. TEM micrographs of dislocations in GaAs [22]

scatter electrons differently. Thicker or denser regions of the specimen will scatter electrons stronger than thinner or less dense ones. There are several methods of obtaining and observing TEM images: bright- or dark-field microscope modes and observation of micro-diffraction contrast, the latter method however being of less importance for the characterization of the dislocation structure of single crystals [23].

A bright-field image is obtained if the diaphragm of the objective lens traps stronger scattered, i.e., strongly deflected electrons. In this case the electron flux will have the lowest density in the stronger scattering regions, i.e., thicker or denser ones. Image brightness depends on the quantity of electrons reaching the detector screen and therefore thicker and denser regions of the specimen will look darker in the image, whereas weaker scattering features of the specimen will on the contrary be visualized as brighter areas on the screen. The situation is the contrary for a dark-field image. The main hinders to the use of this method are the high cost of equipment and the necessity of labor-consuming specimen preparation: the specimens should be thinned to 100–150 nm. For single crystals this thinning is achieved by targeting an opening and studying its edges under a microscope.

This opens up the possibility of identifying different distortions of the crystalline structure (subgrains, stacking faults, dislocations) [23]. Figure 8 shows images of dislocations in single crystal {100} GaAs.

It can be seen from Fig. 8 that a TEM microscopic study can deliver images of discrete dislocations. Analyzing a large number of images and counting the number of identified dislocations per unit area of an image one can conclude on the density of structural imperfections in the single crystal. Along with the dislocations, the image forming in the electron microscope shows extinction lines. The origins of extinction may vary: extinction in the form of lines similar to low-angle boundaries is caused by variable thickness, while extinction located at edges of the targeted opening is produced by microdeformations [31]. Therefore there is the task to reliably identify information-bearing features in the image. This task can be solved by analyzing the regularities of brightness field formation.

7. Induced current method

Induced current operation mode of a scanning electron microscope **(ICM SEM)** can be successfully used for the identification of structural defects (stacking faults, dislocations, impurity segregation sites etc.) in semiconductors and dielectrics. The ICM SEM method allows one to determine the recombination rate and diffusion length in the vicinity of recombination centers in structures containing a *p*–*n* junction or a Schottky barrier. Figure 9 shows dislocation images in GaN with different donor impurity concentrations.

As can be seen from Fig. 9, this method can be used for studying the effect of doping on the formation of a dislocation structure. Along with the analysis of microscopic images this method can be used for the characterization of electrophysical properties of semiconductors and microelectronic devices since it allows identifying regions of local defects, leaks and breakdown locations and determining the parameters and location of *p*–*n* junctions. Furthermore the ICM SEM method shows good promise for fault detection in blocks or discrete elements of ICs and is widely used by many large companies for fast IC performance control [32].

Comparison between the micrographs obtained in induced current mode and the X-ray topographic patterns allows analyzing the effect of dislocations on the

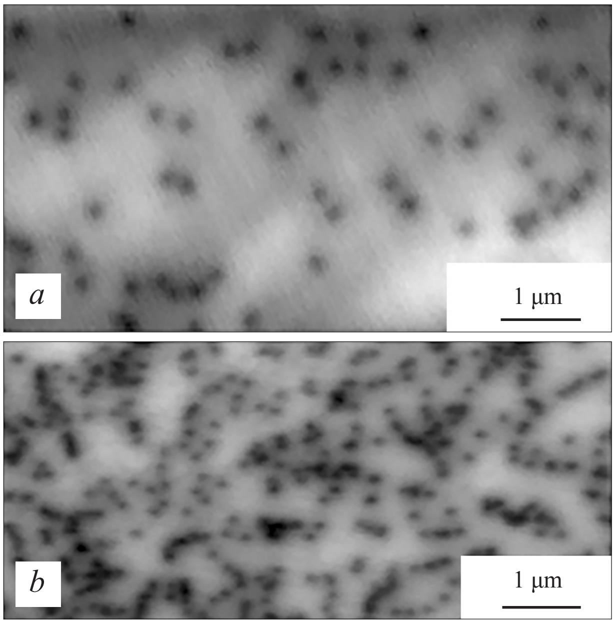

Figure 9. ICM SEM micrographs of dislocations [32]: (*a*) GaN with a donor concentration of 10^{15} cm^{-3}; (*b*) GaN with a donor concentration of 10^{17} cm^{-3}

electrophysical parameters of finite integrated systems [33]. The main disadvantage of the method is the necessity of labor-consuming specimen preparation for further studies: the specimen should contain a barrier structure (a Schottky barrier or a *p*–*n* junction) to produce a spatial charge region and act as a collector [34]. Apart from this one should draw attention to the insufficient sharpness of images which can influence the objectivity of the measurements. This entails the necessity of preliminary digital processing of the micrographs.

8. Digital image processing

Analysis of brightness field is in general a combination of morphological image processing operations and mathematical tools for data analysis, allowing one to divide image features into the test objects and the background. This approach is implemented by transiting from a digital micrograph to an analog image that can be represented as a discrete pixel intensity matrix [35]. Figure 10 shows reconstruction of a quasi-texture for the example of an etch pattern on a (100) GaAs wafer.

Analysis of the brightness field also allows one to systematize structural studies and hence improve the adequacy of experimental results. After digital processing the pixel intensity matrix finally acquires a form where the pixels in the micrograph areas corresponding to the background have a value of 255 (bright areas) and those corresponding to the test objects have a zero value (dark areas). Figure 11 shows the transformation of grayscale

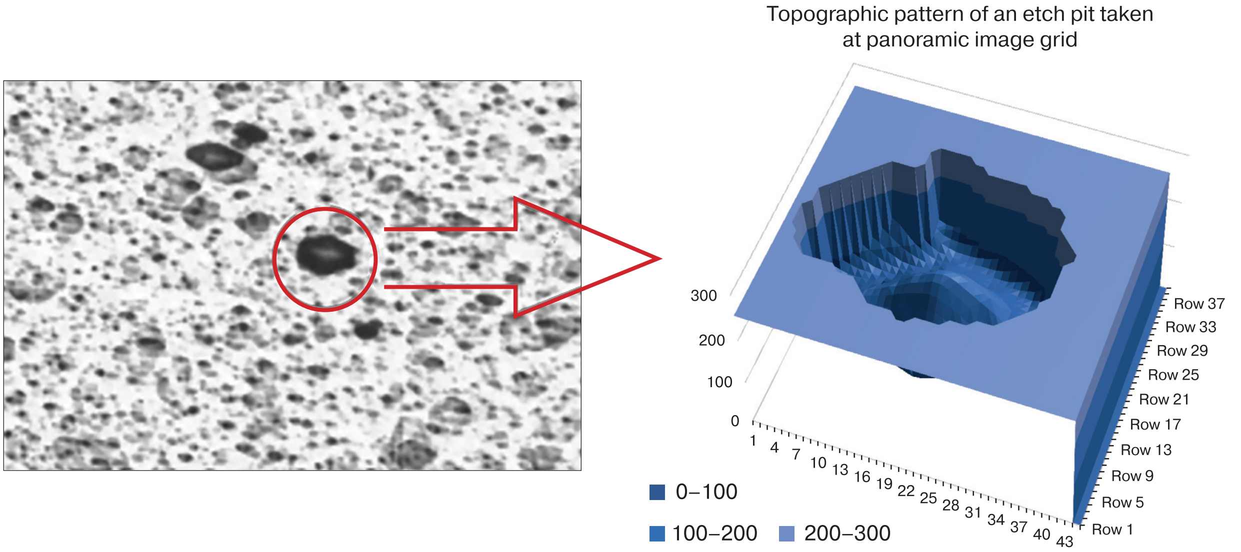

Figure 10. Reconstruction of etch pit profile obtained by optical light microscopy

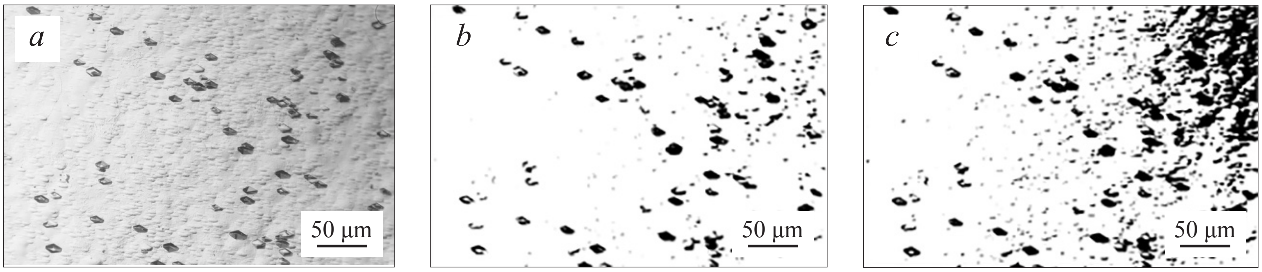

Figure 11. Effect of binarization with different thresholds on micrographs of (100) GaAs etch pits: (*a*) source image; (*b*) 160 threshold binarization; (*c*) 180 threshold binarization

etch pattern images taken from a (100) GaAs surface to binary (monochrome) mode, for different binarization thresholds.

As can be seen from Fig. 11, variation of the binarization threshold tangibly affects the morphology of the final binary image. This dictates the necessity of developing a binarization algorithm taking into account the physical regularities of the nucleation and distribution of dislocations, physicochemical features of etching for a specific semiconductor compound and brightness field analysis for the source image. Brightness field analysis can be used for the analysis of micrographs obtained with different methods. For example a method of separating binarized etch pit fragments on a single crystal wafer was developed for the example of TEM images of GaAs etch patterns [36, 37].

9. Conclusion

Study of the distribution and nature of dislocations in semiconductor single crystals is becoming an increasingly important task due to the rapid development of electronics and growing requirements to the structural perfection and homogeneity of electronics materials. The variety of existing analytical methods does not allow one to definitely select the optimum method for solving a wide range of research and applied tasks.

Methods delivering the highest resolution, i.e., SEM, TEM, ICM SEM, AFM and X-ray topography are indispensable for the achievement of different research goals. Due to the high power consumption of the characterization of structural imperfections which becomes quite tangible if the entire surface of a single crystal wafer is to be scanned, high-resolution microscopic methods have but limited application in industrial conditions. The selective etching method (optical light microscopy) does not deliver high resolution but is conventionally used in the quality control of as-grown single crystals and is rather a qualitative technique. In turn, X-ray diffraction allows evaluating the dislocation density by rocking curve broadening in double-crystal setup.

Digital image processing improves the objectivity of experimental results and broadens the capabilities of existing methods in the characterization of dislocation structures. This approach allows developing an algorithm of identifying information-bearing image features and as a result opens up the possibility of acquiring large arrays of measurement data on a specimen (product) scale for retrieving representative parameters. Accumulation of measurement data statistics in this field will provide for a deeper understanding of the formation regularities of structural inhomogeneities in semiconductor single crystals.

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