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Measurement of the scales of inhomogeneity in the placement of etch dislocation holes in digital images of GaAs single crystalsN. Y. Komarovskiy^{1,2,*}, S. N. Knyazev¹, E. A. Sokolovskaya², A. V. Kudrya²,
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The homogeneity of the properties of semiconductor single crystals has been a key trend in the industry's recent development. However, there is currently no consensus on the degree of property heterogeneity or methods for its assessment. A method for analyzing the heterogeneity of dislocation etch pit distributions is proposed. This method is based on solving the problem of the proximity of binary image objects by partitioning the space into Voronoi polyhedra. Object size filtering was performed taking into account the known nature of the measured objects. A statistical filtering threshold of $585 \mu\text{m}^2$ was calculated using a histogram of the distribution of dark object areas. The nature of image noise was clarified through direct observation of etch pits using scanning electron microscopy, which allowed us to estimate the physical filtering threshold at $16 \mu\text{m}$. It was found that groups of dark objects identified by "statistical" and "physical" filtering, in the "object area S – its perimeter P " coordinates, corresponded to different slope values of the approximating curves: 0.33 and 0.85, respectively. It was shown that, to obtain objective results in digital image processing, it is necessary to accumulate representative measurement statistics – the size of the measurement area when studying the dislocation structure of GaAs should be at least 0.42 mm^2 .

Keywords: Czochralski method; GaAs; etching patterns; dislocations concentration; light microscopy; digital image processing; Voronoi polyhedral.

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1. Introduction

In the course of the last years, the main trend in development of the solid-state field is to achieve the required level of the single crystals structural uniformity that is determined by concentration of dislocation etching pits and their arrangement geometry [1]. In gallium arsenide single crystals grown by the liquid-encapsulated Czochralski method, the dislocation density (N_d) is $\sim 10^4 \text{ cm}^{-2}$. Their impact of dislocations on electrophysical parameters of the final integrated circuit is ambivalent: on one hand, they create

additional levels in the band gap; on the other hand, they are a sink of point defects [2]. Dislocations concentration level in case of growth according to the Czochralski method is foremost determined by the value of thermoplastic deformation relaxation, and lays within N_d : 10^2 (InSb) ... $8 \times 10^4 \text{ cm}^{-2}$ (GaAs) interval for semiconducting compounds of $A^{\text{III}}B^{\text{V}}$ group [3, 4].

Non-uniformity of dislocations arrangement depends on the gradient of voltages occurred during growth and is primarily justified by non-uniform temperature field of a furnace [5]. In case

of high-temperature semiconducting compounds of $A^{III}B^V$ group (InAs, GaAs), the process is additionally complicated by the need of a molten mass liquid encapsulation which in turn may contribute into non-uniformity of temperature field in the crystal-melt interface. The non-uniformity of the thermal field can be mitigated by adjusting the temperature and dynamic parameters of the growth process – ξ . For instance, varying the temperature of the background heater (within the tolerance range) can reduce heat loss from the surface of the growing ingot [6]. However, it is difficult to assess the effectiveness of such adjustments in practice due to the lack of a measure for structural non-uniformity. Calculating this measure is even more complicated by the presence of objects (noise) on the thin-section surface, the formation of which is unrelated to the material's dislocation structure, as well as by the absence of unified guidelines regarding the required measurement scope.

The objective of this work is to study the nature of noise occurring during formation of etch pit images and to develop a methodology for calculating the non-uniformity of their distribution.

2. Object of the Study

GaAs single crystals alloyed with Te, 40 mm in diameter, were grown with the use of Czochralski method with liquid encapsulation of LEC molten mass in crystallographic direction

[100]. The plates cut perpendicularly to the growth axis on the disk-cutting machine were used as the samples. The plates surfaces were ground, chemically polished in acid solution with the following ratio – H_2SO_4 , H_2O_2 , H_2O (3:1:1) – during 25 seconds at the room temperature, and preferentially etched in KOH alkali melt at 450 °C during 7 minutes [7, 8].

The pan views of dislocation structures on GaAs plates obtained using Axio Observer D1m Carl Zeiss microscope with 50-fold magnification by linking individual frames linking in Thixomet [9] software package were used as the study object.

3. Nature of Image Noises

To detect the nature of noises formation on 2D images obtained using the light microscope, morphology of the etching pits structure was studied by means of observation using the scanning electron microscopy (Fig. 1a). The structure of pits on their binary views (based on the approaches proposed in the paper) was similar to the morphology of the etching pits observed by means of the light microscopy (Fig. 1b).

For example, the pits with open contour were observed. The slope of dislocations towards the thin section plane could result in their formation on the image, the bottom of such pits was displaced relative to their geometrical center to distance d – Fig. 2a.

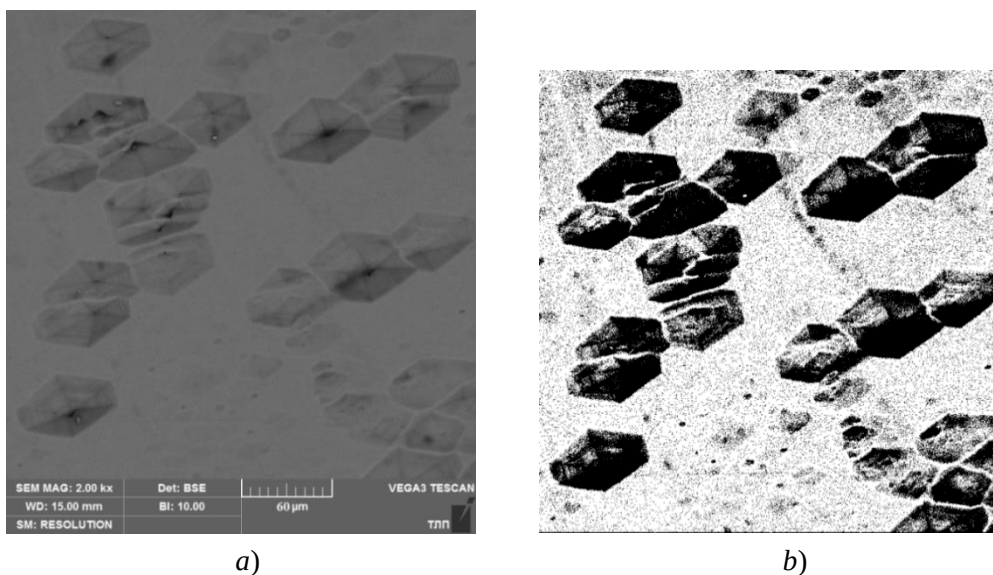


Fig. 1. Initial image of etching pits in the scanning electron microscope (a) and after binarization (b)

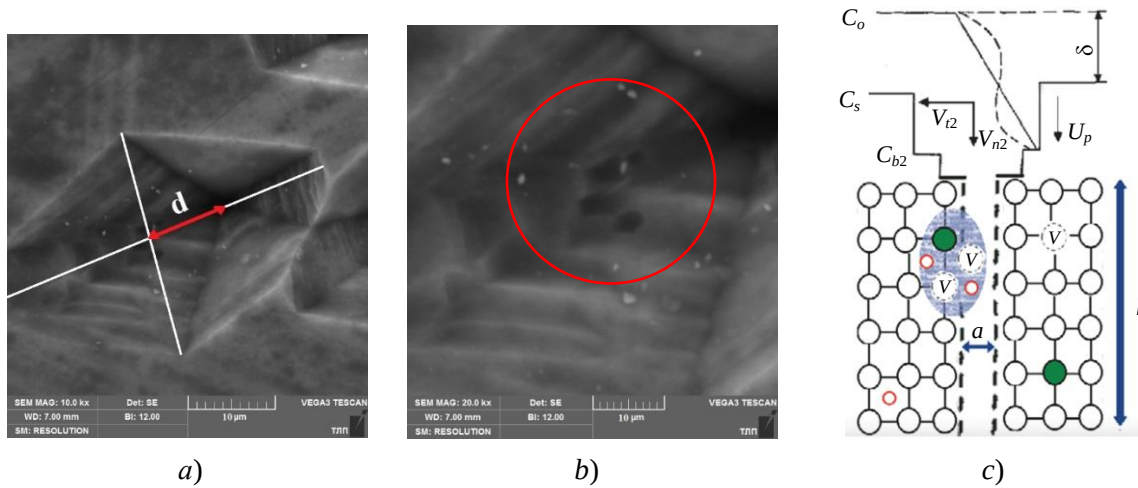


Fig. 2. Displacement of the etching pit bottom a); b) – etching pits caused by point defects; c) – scheme of the etching pit formation at the point of dislocation exposure with the Cottrell atmosphere

The sets of point defects constituting the Cottrell atmosphere along the dislocation line (l) of width (a) [10] could create etching pits of regular shape (Fig. 2c). Their area generally did not exceed $16 \mu\text{m}^2$, which determined the minimum (“physical”) filtration limit. The size of dark objects outlined in such manner was significantly lower than the “statistical” filtration limit ($585 \mu\text{m}^2$) that may be outlined based on histograms of distribution of the dark objects area values. Significantly, three groups of objects on

the image compliant with etching pits and background outlined based on physical and statistical filtration limits were consistent with different values of the approximating curves slope angle – 0.13, 0.85 and 0.33, respectively, within “object area S – perimeter P ” coordinates (Fig. 3). Linearity of the approximating curves is a sign of a physical phenomenon self-similarity – its persistency under similar fold change of S and P coordinates, which may reflect significant difference in the nature of the objects described [11].

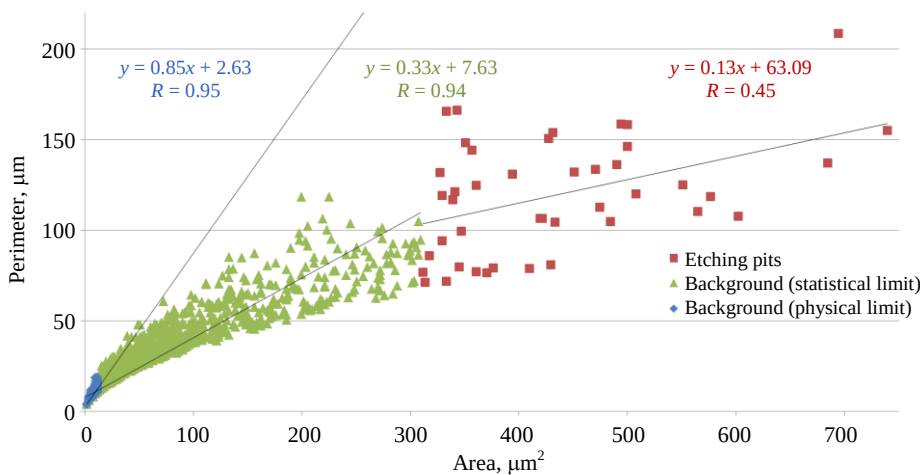


Fig. 3. Dark Objects Perimeter to Area Ratio

The difference in nature of the objects eliminated by physical and statistical filtration can be related to a specific feature of the contour formation process of pits which occur at the points of Cottrell atmosphere plate exposure – their size is determined by dissolution kinetics of dislocations, opposite to the pits formed in the point defects exposure areas [10].

4. Assessment of Non-Uniformity of Etching Pits Arrangement

Outlining the target objects on the binary view – etching pits related to the dislocation structure of a single crystal (using the statistical filtration – area less than $585 \mu\text{m}^2$) detected not only their size difference, but also a difference in arrangement on the thin section surface: presence of the areas of accumulation and sparcification (Fig. 4).

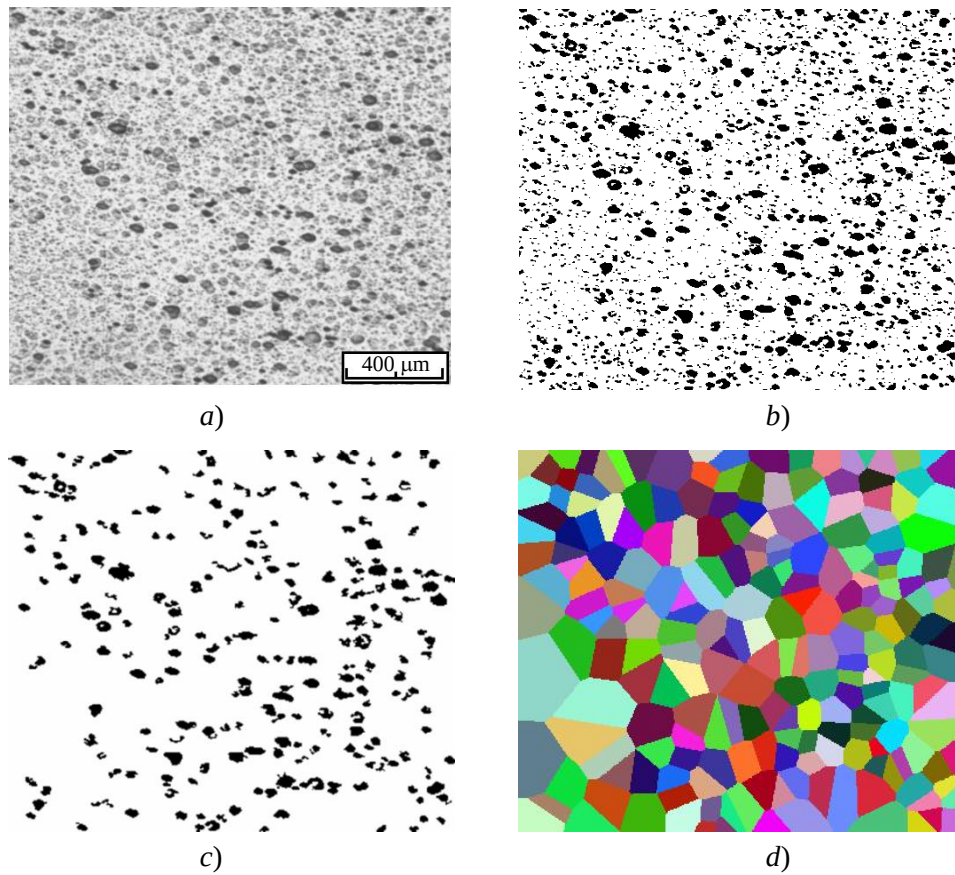


Fig. 4. Initial view (a), its binary representation (b), including the one after statistical filtration (c) and further image partitioning into Voronoi polyhedras (d)

To assess non-uniformity of etching pits arrangement, the space partitioning into Voronoi-Dirichlet polyhedras, which are polygons inside which all points (pixel) are located closer to their center than to the centers of adjacent

areas, was used (Fig. 4d) [17]. Such partitioning allows for intrinsic determination of a distance between the closest etching pits. Fig. 5 shows the histogram of distribution of distances between dark objects—polyhedras centers.

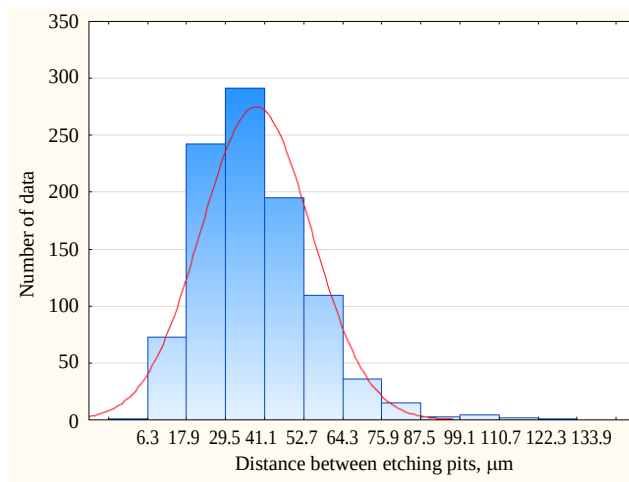


Fig. 5. Histogram of distribution of distances between Voronoi polyhedras centers (etching pits)

Distribution of the distances between the dark objects was different from regular

distribution (asymmetry coefficient – 1.12; excess – 2.39), which confirms non-uniformity of etching

pits arrangement visible on the image (See Fig. 4). Hence, for example, the image partitioning into four fragments of equal area (Fig. 6) provides for not only its own average distance between Voronoi polyhedras centers for each of them, but also allows for assessing a statistical nature of their distribution – Table. The obtained results show the need for justification of the minimum scope of experimental data for obtaining repeatable results of measuring etching pits concentration and arrangement on a thin section is raised.

Fig. 7 shows the curves of average values of the dark object areas, the distance between them (polyhedras), numbers of their closest neighbors vs. the measurement scope varied within ~ 0.053 to 0.846 mm^2 (on the plate scale) for two independent parts of the panorama.

Following the obtained results, the measurement area increasing above 0.42 mm^2 provides for stabilization of the obtained characteristics values, regardless of the selected panorama section, thus ensuring their repeatability and compatibility. With the lower sample sizes, it is possible to obtain both identical and opposite results, meanwhile not consistent with the real values to be found. The obtained results confirm the need for obtaining a representative statistics of observations under digital measurements of the structures. It should be also noted that the areas different in terms of etching pits morphology can be observed in macroscale of a sample. Therefore, a comprehensive representation of the etching pits structure can be provided by measuring their geometry on the entire sample (product) scale.

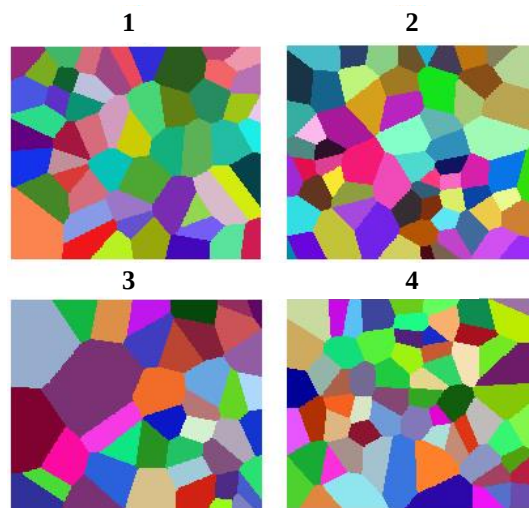


Fig. 6. Partitioning of four fragments of the initial image (Fig. 4) into Voronoi polyhedras

Table

Statistical characteristics of distribution of distances between etching pits for different image fragments shown in Fig. 6

Fragment No.		1	2	3	4
Average distance between polyhedras, μm		38.3 ± 1.1	35.7 ± 0.9	42.4 ± 1.7	36.2 ± 0.9
Coefficients of	asymmetry	1.12 ± 0.26	0.71 ± 0.24	2.13 ± 0.28	0.52 ± 0.24
	excess	1.85 ± 0.55	0.22 ± 0.48	7.54 ± 0.62	0.053 ± 0.49

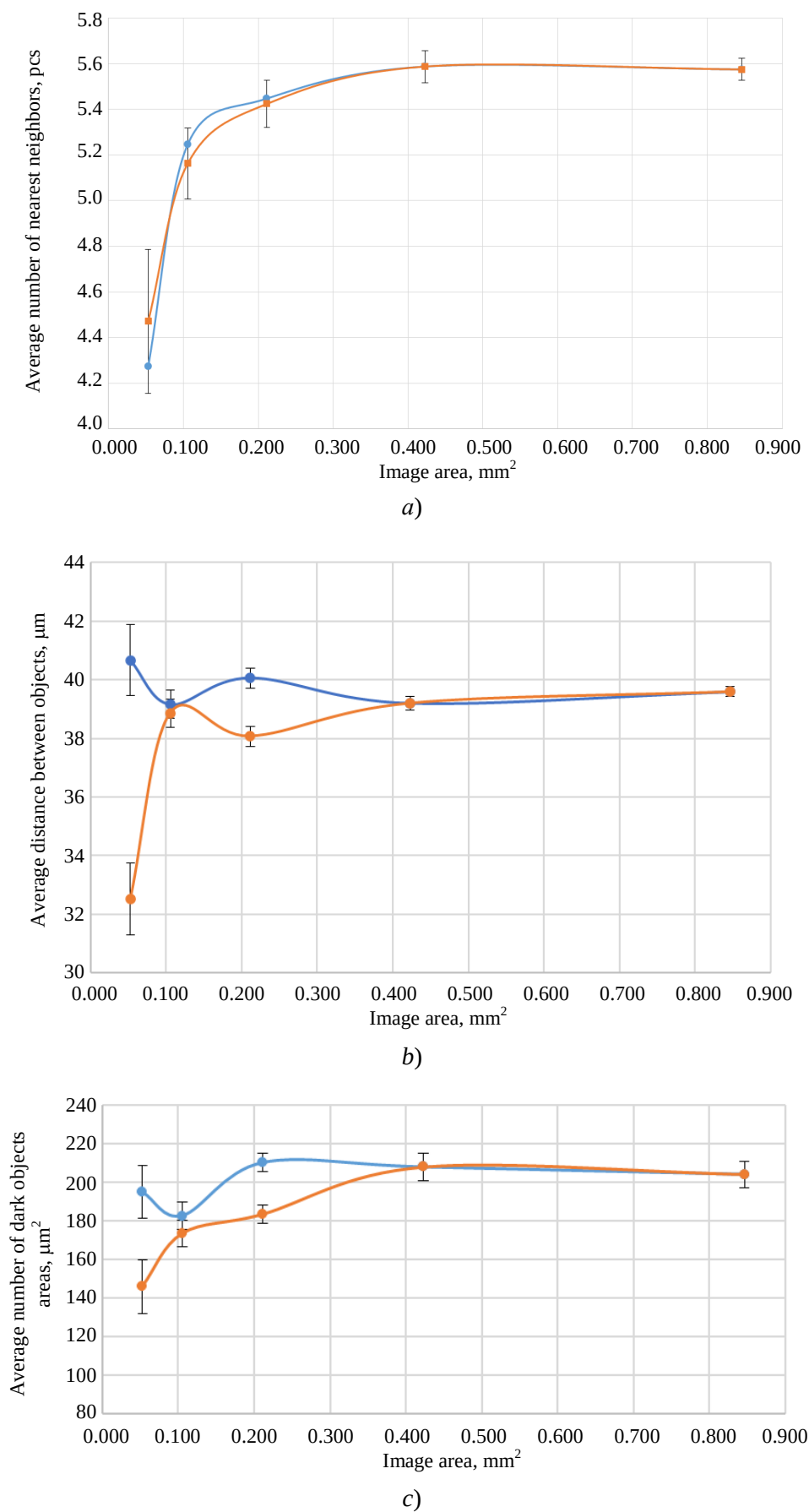


Fig. 7. Variation of average values of the number of nearest neighbors (polyhedras – dark objects) – (a), distances between them (b) and areas of dark objects (c) depending on the measurement scopes for two independent panorama fragments (–●–; –■–)

Conclusion

1. Based on the statistics of distribution of the dark objects areas for the pan view studied in the paper, the noises filtration limit was determined to be $585 \mu\text{m}^2$. It was demonstrated that the groups of dark objects outlined by “statistical” (based on areas distribution histograms) and “physical” (based on direct observations of etching pits morphology in the scanning electron microscope) filtration, within “object area S – perimeter P ” coordinates, were consistent with different values of the approximating curves slope angle tangenses: 0.33 and 0.85, respectively. Linearity of the curves reflects the self-similarity of the observed

physical phenomena, and the curves parameters reflect the difference in objects nature.

2. To assess non-uniformity of etching pits arrangement on the pan view, partitioning thereof according to Voronoi method was used, followed by assessment of statistics of the distances between dark objects (centers of Voronoi polyhedras) and the number of the nearest neighbors.

3. It was established that it is possible to obtain repeatable measurements results for etching pits morphology and regularity on GaAs Te (100) plates surface with the size of the studied area of 0.42 mm^2 and above (on the sample scale).

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