

# Studying the Effect of Geometric Parameters of Oriented GaAs Nanowhiskers on Young's Modulus Using Atomic Force Microscopy

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**Abstract**—A technique for determining Young's modulus of oriented nanowhiskers using atomic force microscopy is developed. Results of studying the effect of geometric parameters on Young's modulus of oriented gallium arsenide nanowhiskers are presented. Young's modulus value has been found experimentally for the GaAs nanowhiskers, which varied from 9 to 143 GPa depending on their aspect ratio. It is shown that Young's modulus of the GaAs nanowhiskers depends on their aspect ratio and can exceed Young's modulus of the bulk GaAs. The results can be used in the development of technological processes for forming structures of the nano- and microsystem hardware and the nano- and microelectronics based on oriented nanowhiskers, in particular arsenide gallium nanowhiskers, as well as in the development of techniques for the nanodiagnostics of filamentary structures.

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## INTRODUCTION

Now one of basic tendencies in the development of contemporary machinery and technology is the miniaturization of functional devices and the development of instruments based on new materials [1]. Semiconductor nanowhiskers are promising material for creating new instruments and devices of micro- and nanoelectronics and micro- and nanosystem hardware (CMOS schemes, sensors, field emitters, probes for atomic force microscopy (AFM), etc.) [2, 3].

Nanowhiskey-based instruments often function under conditions of high local loads and bending deformations, evoking increased interest in studying the mechanical properties of such structures and their dependence on geometric parameters. It is assumed that reducing single-crystal sizes will lead to a substantial increase in their strength and elasticity [4, 5]. However, experimental investigations into the dependence of the mechanical properties of nanowhiskers on their geometric parameters yield contradictory results [5–7]. For example, in [5], growth in the Young's modulus value of a GaAs nanowhiskey was observed from 118 to 183 GPa with a reduction in its diameter from 150 to 55 nm, while this dependence was not revealed in [6]; moreover, a measured Young's modulus value for the GaAs nanowhiskey of  $42 \pm 5$  GPa was considerably less than the Young's modulus of the bulk GaAs. An inverse trend was observed for SnO<sub>2</sub> nanowhiskers: Young's modulus reduced with a decrease in the nanowhiskey diameter [7]. Thus, studying the effect of geometric parameters of GaAs

nanowhiskers on their mechanical properties is an urgent problem.

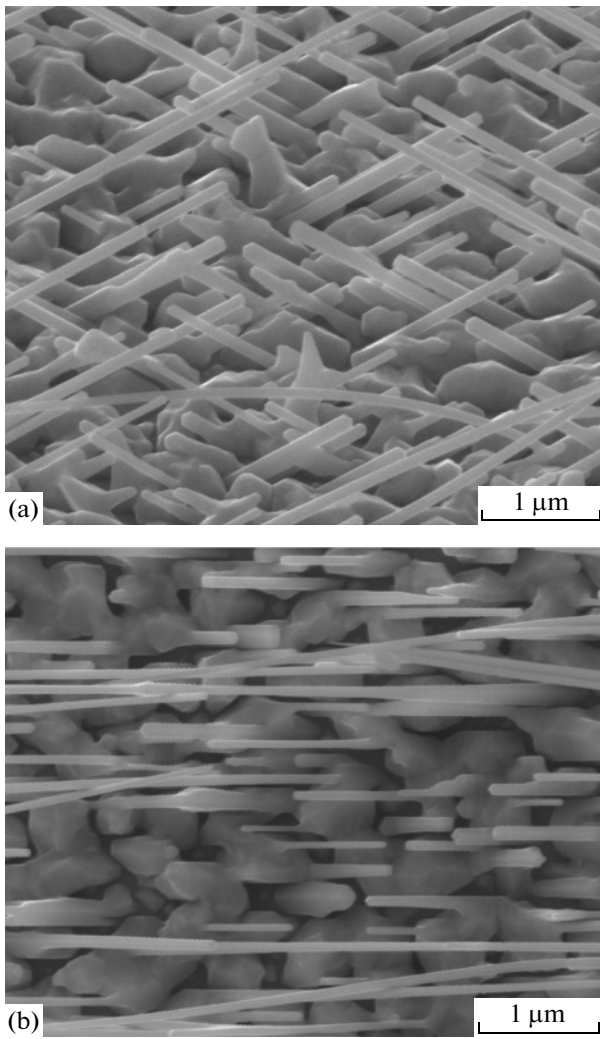
One of the promising methods for studying nanowhiskers is AFM. This method makes it possible not only to determine geometric parameters, but also to investigate mechanical, electrical, and other properties of the samples without special sample preparation, which enables the complex study of properties of an individual nanowhiskey in the course of a single experiment and is an important advantage in studying the effect of geometric parameters of GaAs nanowhiskers on their Young's modulus.

The objectives of this work are nanowhiskey research using AFM, the development of a technique for determining Young's modulus of oriented GaAs nanowhiskers by AFM, and an investigation into the effect of geometric parameters of GaAs nanowhiskers on their Young's modulus.

## EXPERIMENTAL PROCEDURE

An experimental sample with arsenide gallium nanowhiskers oriented towards the substrate surface was produced using the molecular-beam epitaxy at the NANOFAB NTK-9 nanotechnological complex (NT-MDT, Russia) [8–10]. The (100) GaAs plate was used as a substrate. The GaAs nanowhiskers grew along the [111] crystallographic directions towards the most packing density of the sphalerite-type lattice [11].

A preliminary estimation of the geometric parameters of the sample was performed based on an analysis



**Fig. 1.** SEM images of the experimental sample of gallium arsenide nanowhiskers: (a) view at an angle of 52°; (b) top view.

of the research results obtained using the Nova NanoLab 600 scanning electron microscope (SEM) (FEI Co., the Netherlands). Figure 1 presents the SEM images, an analysis of which has demonstrated that there are GaAs nanowhiskers on the sample surface which have a length of 1.2 to 6  $\mu\text{m}$ , a diameter of 100 to 200 nm, and an angle of inclination with respect to the substrate from 30 to 50° depending on the nanowhisker length. The angle of nanowhisker inclination was measured using the application software package supplied together with the Nova NanoLab 600 SEM taking into account the geometry of the sample and detector layout. Since the geometric parameters of the nanowhisker array are inhomogeneous and the SEM method does not make it possible to determine the geometric parameters and the Young's modulus of a nanowhisker during a single experiment without an additional apparatus, the geometric parameters of the sample were also investigated by AFM.

The geometric parameters (length and diameter) of nanowhiskers, as well as the angle of inclination of an individual GaAs nanowhisker with respect to the substrate, were measured using AFM at the Ntegra probe nanolaboratory (PNL) [10]. The measuring results are presented in the table. Initially, the geometric parameters of GaAs nanowhiskers were investigated in the AFM semicontact mode. However, during the scanning, significant distortions of geometric parameters of nanowhiskers were detected which were probably connected with the oscillations of nanowhiskers due to their interaction with the vibrating cantilever; this effect was also observed in [12]. In this case, the GaAs nanowhisker diameter determined in the AFM semicontact mode exceeded five- to tenfold the nanocrystal diameter defined from the SEM data. This trend was observed while using both the NSG 20 cantilever [10] with an aspect ratio of 3 : 1 (Fig. 2a) and the HA\_NC dedicated whisker cantilever [10] with an aspect ratio of 5 : 1 (Fig. 2b).

To minimize the interaction between the cantilever and the GaAs nanowhiskers during scanning, the AFM contactless mode was used, which made it possible to determine the length and diameter of nanowhiskers (Fig. 2c) without a distortion of their size and with a high resolution. The NSG 20 silicon cantilever was applied as a probe.

The mechanical parameters of GaAs nanowhiskers were determined at the Ntegra PNL using AFM force spectroscopy, in which the dependence of the cantilever bend value (DFL signal) on the degree of protrusion of the scanner's  $z$ -piezo tube (Height signal) was taken during the direct and inverse strokes of the cantilever. The resulting dependences make it possible, taking into account the cantilever stiffness  $k$ , to calculate a force acting on the nanowhisker at the given point  $F = k\Delta\text{Height}$ , and the deflection of the GaAs nanowhisker under effect of this force will be equal to the AFM probe bend and to the appropriate change in the Height signal.

To calculate the Young's modulus and the bending stiffness of nanowhiskers, a technique based on the beam theory has been developed according to which a nanowhisker is regarded as a flexible beam with a length  $L$  and an effective bending stiffness  $(EI)_{\text{eff}}$ , the deflection of which under the effect of an external force  $F$  can be calculated as [13]

$$w = \frac{T}{P} \left( \frac{\tan(kL)}{k} - L \right), \quad k = \sqrt{P/(EI)_{\text{eff}}}, \quad (1)$$

where  $T$  is the external force component acting perpendicular to the nanowhisker axis;  $P$  is the external force component acting parallel to the nanowhisker axis. The external force component  $T$ , applied to the nanowhisker, evokes its deflection from the equilibrium state, while the external force component  $P$  causes the probe sliding over the nanowhisker surface.

Geometric and mechanical parameters of GaAs nanowhiskers obtained using atomic force microscopy

No. of GaAs nano-whisker	Length of GaAs nano-whisker $L$ , nm	Diameter of GaAs nano-whisker $D$ , nm	Aspect ratio of GaAs nanowhisker	Angle of nanowhisker inclination $\theta$ , deg	Deflection of GaAs nanowhisker $w$ , nm	Force $F$ , $\mu\text{N}$	Bending stiffness of GaAs nanowhiskers $(EI)_{\text{eff}}$ , $\text{N nm}^2$	Young's modulus of GaAs nano-whisker $E$ , GPa
1	6218	233	26.69	27.21	105	5.04	900.43	137.37
2	3050	175	17.43	27.66	101	4.85	117.85	86.52
3	1406	135	10.42	32.24	85	4.08	15.63	54.22
4	1641	166	9.88	28.42	104	4.99	19.91	31.84
5	2173	156	13.93	28.89	116	5.57	47.6	69.24
6	3647	196	18.61	30.63	105	5.04	246.7	107.8
7	3072	176	17.45	27.47	114	5.47	118.67	85.03
8	2728	156	17.48	26.10	103	4.94	72.02	83.45
9	1232	216	5.71	29.62	71	3.41	9.14	8.84
10	1596	177	9.02	32.73	57	2.74	22.91	31.07
11	1390	216	6.43	34.33	78	3.74	16.28	13.95
12	1947	157	12.4	28.27	54	2.6	32.15	51.20

Taking into account the angle of inclination of the GaAs nanowhisker with respect to the substrate surface  $\theta$  and the angle of tilt of the cantilever holder, which is  $20^\circ$  in the given AFM design, the components of the external force are (Fig. 3) as follows:

$$T = F \cos(\theta - 20^\circ),$$

$$P = F \sin(\theta - 20^\circ).$$

Calculating the value of bending stiffness  $(EI)_{\text{eff}}$  based on Eq. (1) and assuming the moment of inertia of the nanowhisker to be equal to the moment of inertia of a rectangle with respect to the axis perpendicular to the nanocrystal deflection, we can find Young's modulus of the nanowhisker as

$$E = \frac{(EI)_{\text{eff}}}{I} \approx \frac{12(EI)_{\text{eff}}}{LD^3}, \quad (2)$$

where  $D$  is the diameter of the nanowhisker.

Expressions (1) and (2) make it possible to calculate the bending stiffness and the Young's modulus of an individual nanowhisker and investigate the dependence of mechanical properties of nanowhiskers on their geometric parameters.

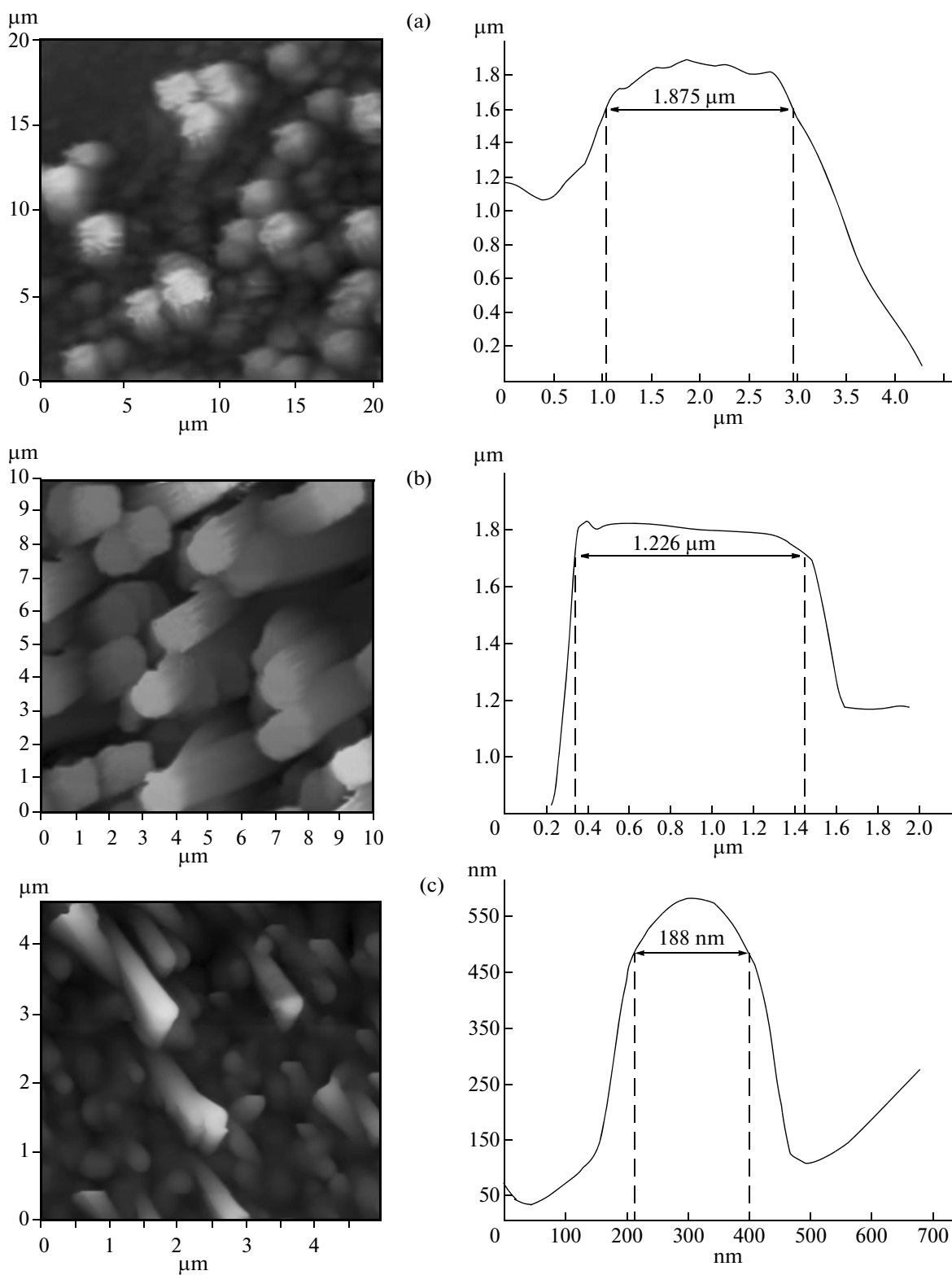
## DISCUSSION OF RESULTS

The bending stiffness and the Young's modulus were determined for 12 nanowhiskers of gallium arsenide with different geometric parameters using the developed technique and the results of studying the geometric parameters of GaAs nanowhiskers by AFM in the contactless mode. The AFM images of the first five nanowhiskers and the dependences obtained from the force spectroscopy for each of these nanowhiskers

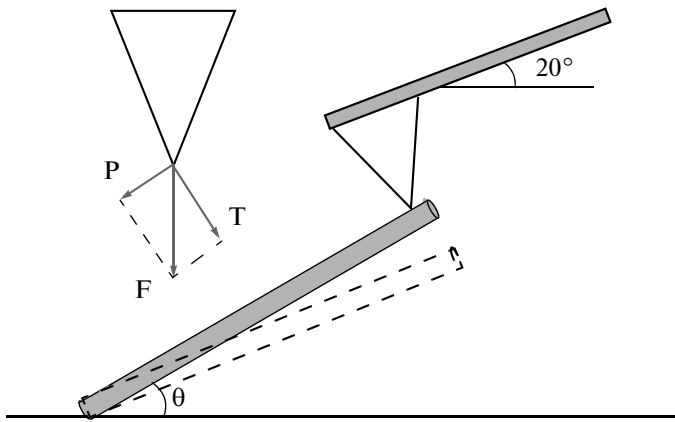
are presented in Fig. 4. It should be noted that the force spectroscopy of the GaAs nanowhiskers has to be performed only after scanning in the AFM contactless mode, because scanning in the semicontact mode strongly swings the nanowhiskers, which leads to the acquisition of unreliable  $DFL(\text{Height})$  relationships. Values of the geometric and mechanical parameters for all 12 GaAs nanowhiskers, which were determined based on the results of studying the GaAs nanowhiskers using AFM together with the developed technique, are presented in the *table*. The linear segment of the  $DFL(\text{Height})$  dependences, on which the deflection of nanowhiskers from the equilibrium position did not exceed 10% of the nanowhisker length, was used to calculate the mechanical parameters in order to satisfy the elastic interaction condition.

An analysis of the results has shown that the Young's modulus of the gallium arsenide nanowhiskers depends substantially on their geometric parameters, which is uncharacteristic of bulk material. It is established experimentally that the Young's modulus of the gallium arsenide nanowhiskers depends only on the aspect ratio of a nanowhisker and on its length (Fig. 5). The Young's modulus of the gallium arsenide nanowhiskers varied from 9 to 143 GPa, depending on its geometric parameters. In this case, with the aspect ratio more than 17 : 1, the Young's modulus of the gallium arsenide nanowhiskers exceeded the Young's modulus of the bulk gallium arsenide of 86 GPa [6].

It should be noted that the mean value of the cantilever stiffness  $k = 48 \text{ N m}$  was taken from the ratings declared by the manufacturer [10], because a more accurate determination of the  $k$ -parameter value requires additional independent studies. In this case,



**Fig. 2.** AFM images (on the right) and profilograms (on the left) of the experimental sample of GaAs nanowhiskers obtained in the AFM semicontact mode with (a) an NSG 20 cantilever and (b) a HA\_NC cantilever and (c) in the AFM contactless mode with an NSG 20 cantilever.



**Fig. 3.** Schematic of the AFM force spectroscopy of an oriented nanowhisker.

the calculated absolute values of Young's modulus include a systematic error (connected with the deviation of the AFM cantilever stiffness from the value used in the calculations and with the elastic bend of the AFM probe), which does not influence the behavior of the revealed dependence of Young's modulus of the GaAs nanowhiskers on their geometric parameters.

The observed dependence of the Young's modulus of a GaAs nanowhisker on the length and aspect ratio can be related to the growth in effect of the surface tension with the reduction in the ratio of the nanowhisker volume to its surface area. The effect of geometric parameters and the ratio of the nanowhisker

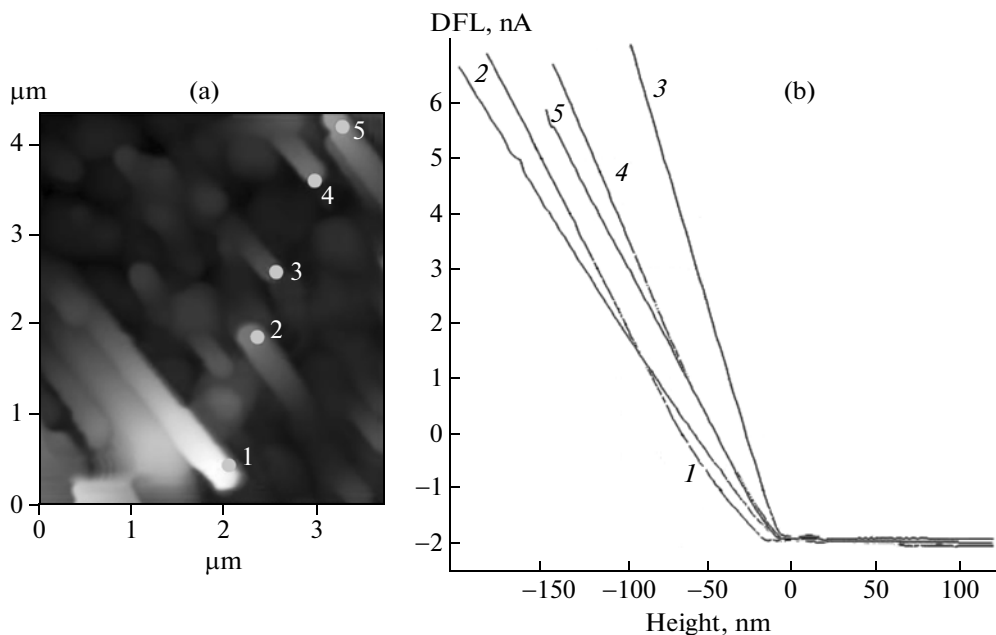
volume to its surface area on the Young's modulus are also observed for other nanowhiskers (lead, silver, zinc oxide, etc.) [14–16].

The bending stiffness of the GaAs nanowhiskers, like the stiffness of the flexible beam, depended on the length, diameter, and other elastic properties of the nanowhisker.

## CONCLUSIONS

This work resulted in the development of a technique for determining the Young's modulus of oriented nanowhiskers using atomic force microscopy, which allows the Young's modulus and the bending stiffness of an individual nanowhisker to be calculated based on data from AFM force spectroscopy. Using the example of GaAs nanowhiskers, the applicability of AFM modes for studying the geometric parameters of oriented nanowhiskers is analyzed, and the AFM contactless mode is shown to be optimal for scanning these structures because it allows the required geometric parameters of nanowhiskers to be determined with a high resolution.

Experimental investigations into the effect of geometric parameters on Young's modulus of oriented nanowhiskers of gallium arsenide were performed using the developed technique. It is revealed that Young's modulus depends on the aspect ratio of the nanowhisker and exceeds the Young's modulus of the bulk material if the aspect ratio is more than 17 : 1. The behavior of the dependences of the mechanical properties of the gallium arsenide nanowhiskers on



**Fig. 4.** Results of experimental studies of GaAs nanowhiskers using AFM: (a) the surface topology obtained in the AFM contactless mode; (b) the  $DFL(Height)$  dependence from AFM spectroscopy.

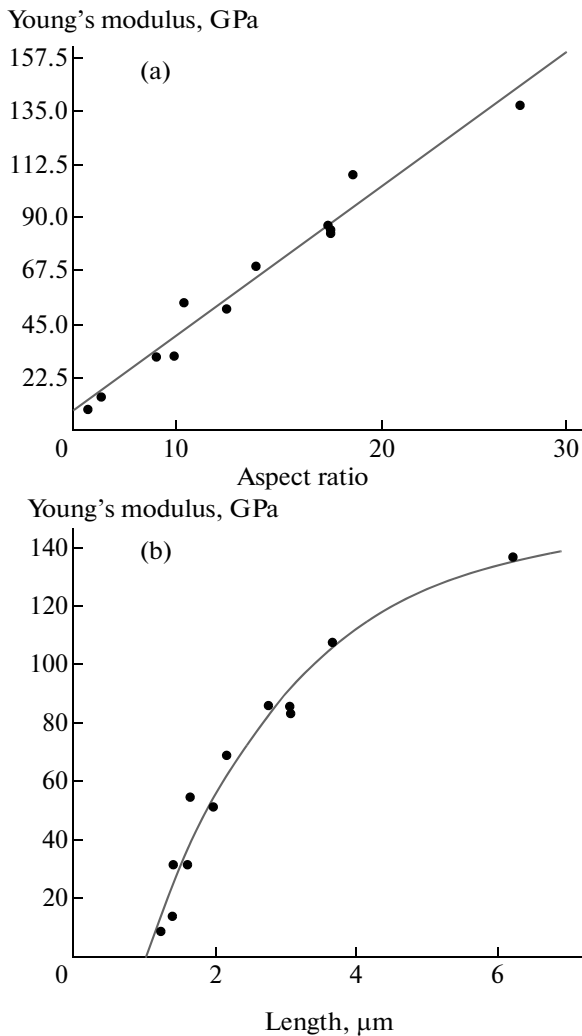


Fig. 5. The dependences of Young's modulus of GaAs nanowhiskers versus (a) the aspect ratio and (b) the length.

their geometric parameters is in agreement with published data for nanowhiskers made of other materials.

The results of this work can be used in the development of technological processes of forming structures of nano- and microelectronics and microsystem hardware based on oriented nanowhiskers.

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#### REFERENCES

1. B. Bhushan, *Springer Handbook of Nanotechnology*, 3rd ed. (Springer, 2010).
2. V. G. Dubrovskii, G. E. Tsyrlin, and V. M. Ustinov, "Semiconductor Filamentary Nanocrystals: Synthesis, Properties, Application," *Fiz. Tekhn. Poluprovodn.* **43** (12), 1585–1628 (2009).
3. Y. Li, F. Qian, J. Xiang, and C. M. Lieber, "Nanowire Electronic and Optoelectronic Devices," *Mater. Today* **9** (10), 18–27 (2006).
4. R. S. Ruoff, D. Qian, and W. K. Liu, "Mechanical Properties of Carbon Nanotubes: Theoretical Predictions and Experimental Measurements," *C. R. Physique*, No. 4, 993–1008 (2003).
5. Y. B. Wang, L. F. Wang, H. J. Joyce, Q. Gao, X. Z. Liao, Y. W. Mai, H. H. Tan, J. Zou, S. P. Ringer, H. J. Gao, and C. Jagadish, "Super Deformability and Young's Modulus of GaAs Nanowires," *Adv. Mater.*, No. 23, 1356–1360 (2011).
6. P. A. Alekseev, M. S. Dunaevskii, A. V. Stovpyaga, M. Lepsa, and A. N. Titkov, "The Way to Determine Young's Modulus for GaAs Obliquely Growing at the Substrate," *Fiz. Tekhn. Poluprovodn.* **46** (5), 659–664 (2012).
7. S. Barth, C. Harnagea, S. Mathur, and F. Rosei, "The Elastic Moduli of Oriented Tin Oxide Nanowires," *Nanotechnology* **20** (11), 115705 (2009).
8. O. A. Ageev, A. S. Kolomiitsev, V. A. Smirnov, et al., "The Way to Grow Nanosized Structures by Means of Nanotechnological NANOFAB NTK-9 Complex," *Izv. YuFU. Tekhn. Nauki*, No. 1, 109–116 (2011).
9. A. V. Rukomoikin and M. S. Solodovnik, "Forming and Investigation of Arsenide Gallium Nanostructure at Nanotechnological System NANOFAB NTF-9," *Izv. YuFU. Tekhn. Nauki*, No. 4, 237–238 (2011).
10. <http://www.ntmdt.ru>
11. E. I. Givargizov, *Filamentary Nanocrystals Growing from Vapor* (Nauka, Moscow, 1977) [in Russian].
12. O. A. Ageev, O. I. Il'in, A. S. Kolomiitsev, B. G. Konoplev, M. V. Rubashkina, V. A. Smirnov, and A. A. Fedotov, "Determining Geometrical Parameters of Vertically Aligned Carbon Nanotubes Using Atomic Force Microscopy," *Mikro-Nanosistem. Tekhn.*, No. 3, 9–13 (2012).
13. O. A. Ageev, O. I. Il'in, A. S. Kolomiitsev, B. G. Konoplev, M. V. Rubashkina, V. A. Smirnov, and A. A. Fedotov, "Development of a Technique for Determining Young's Modulus of Vertically Aligned Carbon Nanotubes Using the Nanoindentation Method," *Nanotech. Russ.* **7** (1–2), 47–52 (2012).
14. G. Wang and X. Li, "Size Dependency of the Elastic Modulus of ZnO Nanowires: Surface Stress Effect," *Appl. Phys. Lett.* **91**, 231912 (2007).
15. S. Cuenot, C. Frétiigny, S. Demoustier-Champagne, and B. Nysten, "Surface Tension Effect on the Mechanical Properties of Nanomaterials Measured by Atomic Force Microscopy," *Phys. Rev. B* **69**, 165410 (2004).
16. F. Song, G. L. Huang, H. S. Park, and X. N. Liu, "A Continuum Model for the Mechanical Behavior of Nanowires Including Surface and Surface-Induced Initial Stresses," *Int. J. Solids Struct.* **48**, 2154–2163 (2011).