

## LAYERED SEMICONDUCTOR InSe AS A STANDARD NANORELIEF IN THE METROLOGY OF NANOOBJECTS

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### ABSTRACT

Intensive development of nanotechnology requires high-precision measurements on atomic and molecular levels by creating the metrological system for measurements, at the first turn, the length in the nanometer scale. Such measurements require standard samples of nanorelief surface.

High chemical stability and the absence of dangling bonds (defects), low surface roughness of cleaved surface allows to use the Van der Waals surface of InSe single crystals as the standard nanorelief. This is evidenced by the research<sup>1</sup>es of morphology for Van der Waals surface by scanning tunneling microscopy in which images with atomic resolution were obtained. A single crystal InSe, was grown by vertical Bridgman-Stockbarger method. The crystal has  $\gamma$ -polytype rhombohedral structure. with parameters  $a = 4.003 \text{ \AA}$ , and  $c = 24.9553 \text{ \AA}$  (in hexagonal axes).

Thus InSe is appropriate standard nanorelief for probe microscopy.

**Keywords:** standard of nanorelief, layered semiconductor InSe, Van der Waals surface

### INTRODUCTION

Creation of new high technology and new materials with fundamental shifts in the structure and technical level of production and output of production at world level, is largely ensured by the level of metrological support.

The leading countries of the world, occupying key positions in microelectronics (Japan, USA, Germany, Great Britain, etc.) pay the highest attention on achievement of metrology for linear measurements in micro-and nanometer range. USA has a national program of metrological provision of microelectronics (National Semiconductor Metrology Program). Program, including, involves the creation standards of the unit of length in the nanometer range.

Natural arrangement of atoms in the lattices of crystals, as well as specially created relief on single crystal surfaces are currently used as standards. Review of the principles, methods and problems of nanometrology is presented in [1]. One of the natural standards is a nanorelief of surface for highly oriented pyrolytic graphite (HOPG). Simultaneously with the scanning of an unknown

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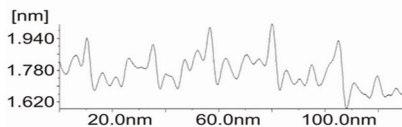
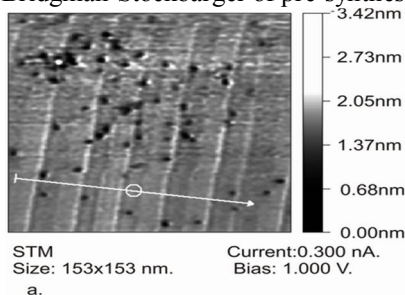
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surface the scan of the well-known surface of graphite (the period of the hexagonal crystal lattice:  $a = 2,464 \pm 0,002 \text{ \AA}$ ) is carried out. Software recognition of atoms image of the graphite allows to generate a control signal for correcting the microscope manipulator. When cleaving HOPG many stages with dimensions less than  $2 \times 2 \text{ mm}$  are formed. They are used as a model of nanorelief. They appear at fracture of graphite planes, oriented not toward cleavage. Graphite planes have structural defects associated with dislocations. Different types of dislocations manifest themselves in the topography of the surface in many ways. Over time, the accumulation of the adsorbate on the surface, results in poor quality images. Van der Waals (VdW) surfaces of InSe single crystals can serve as analogue of HOPG surface.

Semiconductor InSe belongs to a group of layered compounds  $A_3B_6$ . Each layer of this crystal consists of atomic groups Se-In-In-Se with strong covalent bonds in the layer. Se atoms of neighboring layers are linked by weak VdW forces and form VdW surface. Absence of free chemical bonds in VdW surface promotes its high electrochemical stability.

### EXPERIMENTAL METHOD

Monocrystalline layered semiconductor InSe, grown by the vertical Bridgman-Stockbarger of pre-synthesized ingot.



**Fig. 1** – STM image of the InSe (0001) VdW surface, obtained by blade cleaving and exposed in air about 2 min. a – corrugated (0001) plan with defects; b - range of heights along the line indicated in Fig. 1a

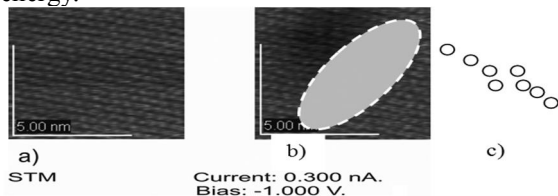
Weissenberg method revealed that the crystal has a rhombohedral structure of  $\gamma$ -polytype with periods of the unit cell and  $= 4.003 \text{ \AA}$ ,  $a = 24.9553 \text{ \AA}$  (in hexagonal axes). As STM technique UHV ( $10^{-8} \text{ Pa}$ ), scanning probe tunneling microscope JSPM-4610 has been used. The probe radius was not more than 5 nm.

### RESULTS AND DISCUSSION

The morphology of VdW surface for InSe layered single crystal undergone by different kinds of its treatment was researched by STM methods. It was supposed that the VdW surface prepared by adhesive tape oxidizes in open air due to hemisorbtion of acid agents by broken In and Se

bonds. The kind of Volt-Ampere characteristics allows to affirm, that the surface oxide layer is a mixture of  $\text{In}_2\text{O}_3$  and wide-gap selenium oxides [2].

The tunnel microscope scanning of InSe surface, prepared by sliding and further exposure on an air during 2 min reveals the surface ordering in the form of a corrugation of a complicated profile having fine structure. (*Fig. 1*) The latter reflects the redistribution of charge density after chemisorption of gas molecules from air and surface relaxation to the state with minimal energy.



**Fig. 2.** – STM image of the InSe (0001) VdW surface, obtained by blade cleaving in an atmosphere of anhydrous nitrogen: a - area without defects; b - the surface with shaded area of the defect; c - the scheme of arrangement of atoms in the defect area, marked by the cycle in Fig. 2.b

Point defects disturb a periodic crystal potential. This perturbation has dimensions up to four lattice periods and looks like shaded area (*Fig. 2b*).

## CONCLUSIONS

Only the VdW surface InSe (0001) of rhombohedral structure prepared by cleaving the upper layers in the absence of oxygen, reveals atomic resolution on STM image of the crystal with period  $a = 4.003 \text{ \AA}$ .

Thus InSe is appropriate standard nanorelief for probe microscopy.

In studies of the derivative of the current-voltage characteristics of tunnel current flowing between the probe of STM and the surface being studied in its various locations, it was found that the composition of the natural oxide is a result of oxidation of the components of the crystal and represents a mixture of nitric oxides  $\text{In}_2\text{O}_3$  and wide selenium. The fine structure of STM image reflects the corrugation of VdW surface InSe at the atomic scale level, the redistribution of charge density on it after chemisorption of gas molecules from the air and the relaxation of this surface to the state with minimum energy.

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## REFERENCES

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The atomic resolution is observed (*Fig. 2a*) on the VdW surface (0001) for InSe single crystal, prepared by sliding in oxygenless media. The surface corrugation is absent. Point