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Langmuir–Blodgett films and nanotechnology

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Abstract: In this article the prospect of nanotechnology development is analyzed. Nanotechnology development from science to industry is suggested. The reason for the high stability of Langmuir–Blodgett (LB) technology against external influences is due to the 'dead zone' under every layer of deposition. LB technology seems to be a technique for producing objects for nanotechnology. The results of conductive film modification by electric pulses from the STM tip are illustrated. The possibility of creating defects with sizes up to 5 nm is shown. The prospect of conductive and dielectric LB film applications as materials for nanoelement modulation is considered. © 1996 Elsevier Science Limited.

Keywords: Langmuir–Blodgett films, surfactant, resists, biosensors, surface energy, hydrophilic, liquid crystal, hydrophobic, wetting, contact angle, Langmuir–Blodgett trough, nanotechnology, tunnel and atomic force of microscopes, cantilever, megaprobe, molecule, viruses, microorganisms, periodical, highly oriented pyrolytic graphite (HOPG), modification, conductive layer

INTRODUCTION

As a result of the development of science in the 'nano' direction, Langmuir–Blodgett (LB) films have been attracting more and more attention. This interest is supported by the opportunity of forming similar layered structures by the consecutive deposition of monomolecular layers of various surface-active substances (SASs) (Adam, 1941; Blinov, 1988). Depending on the SAS chosen, the received structures can have the properties of a good dielectric (Myagkov, 1989), including pyro, piezo and ferroelectrics (Myagkov *et al.*, 1989; Roberts, 1989), conductors (Troitsky, 1991), various types of resists for microelectronics (Matveeva & Bokov, 1992), sensitive elements of gas sensors, and selective sensors on various organic compounds—so-called biosensors (Petty, 1991; Alexeev *et al.*, 1992).

The fact that the structure updating of a film by contact or electrical effect from a probe is possible is not in doubt, but if it is possible then how to use it for the creation of practically important electronic circuits remains unanswered. The purpose of this article is to try to designate experimental and technological approaches to this problem.

LB FILMS AND LB TECHNOLOGY

LB films are formed on the surface of firm plates by consecutive crossing or consecutive contact of a water surface on which a monomolecular layer of SAS has been formed previously. The method of deposition that consists of consecutive crossings by a plate of water surface with the Langmuir layer formed was devised by Blodgett and is refered to as the LB method or the method of vertical lift. The method of deposition by consecutive contact of surfaces was developed by Schaefar and is referred to as the Langmuir–Schaefer (LSh) method or the method of horizontal lift (Adam, 1941; Blinov, 1988; Adamson, 1976).

We shall analyze in more detail a procedure of film formation. First, a monomolecular layer of SAS is formed on a clean surface of water or water solution of salts. For this purpose one injects, drop by drop, on the water surface, a certain dose of solution of SAS which has been previously dissolved in a volatile solvent. The dose is chosen so that a densely compressed monomolecular layer can take approximately one-third of the water surface. The volatile solvent (hexane, benzene, chloroform, etc.) evaporates, and there is a rarefied layer of SAS on the surface. As a rule, it is advantageous for SAS molecules under these conditions to gather in microislands—surface steam. Thus the complete surface energy decreases because the system of broken hydrogenous bands is saturated on part of the free water surface. A balance with the hydrophilic plate is more often applied as a gauge of surface tension (the so-called Wilhemy method (Adam, 1941)) to register surface condition in LB technology. Thus the change of surface energy that occurs when forming microislands of SAS is not registered, but the change of angle of wetting of the hydrophilic plate is measured. The application of hydrophilic, completely wettable plate is registered, since otherwise the change of the condition of its surface due to the adsorption of SAS during the formation of a Langmuir layer on water is possible. The islands behave as a surface gas, and in the field of small pressures the appropriate equation of state is (Adam, 1941; Adamson, 1976):

$$\pi \cdot (S - S_0) = \mathrm{k} \cdot T \cdot c/n \qquad (1)$$

where π is the surface pressure in the monolayer, S is the area occupied by the layer, S_0 is the area of dense layer, k is Boltzmann's constant, T is temperature, c is the concentration of SAS and n is the number of molecules in the island.

Using Eq. (1), it is possible to determine the average number of molecules per island, if the system permits one to register surface pressure reliably, beginning with 0.01 mN/m. Depending on the SAS type, n can be from one up to several thousands. The large values of n are characteristic of firm films, and small (1–4) of liquid ones. The SASs that form firm films have small equilibrium pressure of spreading, π_{sp}, and are not able to relax in finite time if part of the monolayer is removed from the surface. That is why firm films can be deposited only by the LSh method. LB films of SASs that form 'liquid' surface layers can be received only when the chemical reaction of linkage, with the formation of a new complex with substrate substance, occurs as a result of deposition. Otherwise, on drawing the substrate upwards a layer slides back on the water because of the large value of spreading pressure. SASs that possess a mesomorphic phase on the surface are optimum for the deposition of LB films. The literary data on the results of research into spreading pressure are almost absent. Proceeding from the physical sense of spreading pressure, it is possible to assume that π_{sp} corresponds exactly to the maximum pressure of coexistence of the biphase area of the liquid and mesomorphic phases.

As the area occupied by the Langmuir film reduces, the phase conditions vary consistently from gas or vapor up to liquid/mesomorphic and firm. The dependence registered during compression of surface pressure π versus the area occupied by the layer is referred to as the π–A isotherm. At some pressure the monomolecular layer loses stability and a volumetric phase begins to form. This pressure, as well as the spreading pressure, is characteristic of a given SAS and is referred to as the pressure of collapse π_c.

At pressures lying in the range

$$\pi_{sp} < \pi < \pi_c \qquad (2)$$

it is possible to receive LB films with uniform molecular thickness. Thus the area of the film is limited only by geometrical parameters of the deposition system.

The distinctive feature of the LB method of deposition is the existence of a large energy barrier of 'step' formation. During the process of immersion of a hydrophobic plate in water, on the surface of which a Langmuir layer with the condition $\pi_{sp} < \pi < \pi_c$ is generated, the surface is deflected. Thus the area of the surface increases and the hydrophobic plate is touched with water. The condition of hydrophoby means that the interface surface energy of the water–substrate system is great. With the presence on the surface of a SAS of some non-zero value of spreading pressure, the process of adsorption of SAS molecules is energy advantageous because it minimizes the value of the interface surface energy. Thus, at plate immersion, a constant adsorption of monomolecular layers of SAS occurs. The adsorption process begins as soon as the meniscus deflects downwards for the value that is appropriate to the angle of wetting of the given water surface.

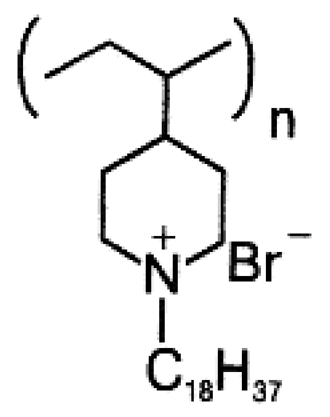
As one knows, the size of meniscus deflection h is equal to (Adamson, 1976)

$$h = a^*(1 - \sin\theta) \qquad (3)$$

where a is the capillary constant and θ is the angle of wetting. $a = \sqrt{[2\gamma/(\boldsymbol{g} \cdot \rho)]}$, where γ and ρ are the surface tension and density of water, respectively, and $\boldsymbol{g}$ is the acceleration of free fall. At room temperature a is of the order of millimeters, and the values of meniscus deflection downwards for $\theta = 100$–$110°$ are equal to $h_{100} = 0{\cdot}05$ mm and $h_{110} = 0{\cdot}23$ mm, respectively.

The meniscus line during LB film formation is a special line. On the air–substrate border the substrate is hydrophobic, and on the water–substrate border the substrate is hydrophilic. There is a jump of free surface energy of substrate on the meniscus line and, hence, on this line there are two equilibrium angles of wetting, θ_{up} and θ_{down}, that correspond to movement of substrate upwards and downwards. At change of direction of movement of the substrate, in fulfilling condition (2), the layer adsorbed already cannot be desorbed backwards onto water. On movement of the substrate upwards, the water contacts with its hydrophilized part and the meniscus deflects upwards to the value that is appropriate to the angle of wetting of the surface that is already hydrophilized. If $\theta_{\mathrm{up}} = 60$–$30°$, the values of meniscus lift are $h_{60} = 0.51$ mm and $h_{30} = 1.9$ mm. We shall see that film deposition does not occur during inversion of the meniscus. Only when the meniscus is inverted is the formation of a new layer possible. The zone of meniscus inversion is a dead zone for deposition of a LB film. We shall name the zone for SASs that satisfy condition (2) as a DZ zone; it is about 1–3 mm.

The diagram of deposition of TG1 SAS is shown in Fig. 1. This is a complex polymer SAS with the structural formula shown below.



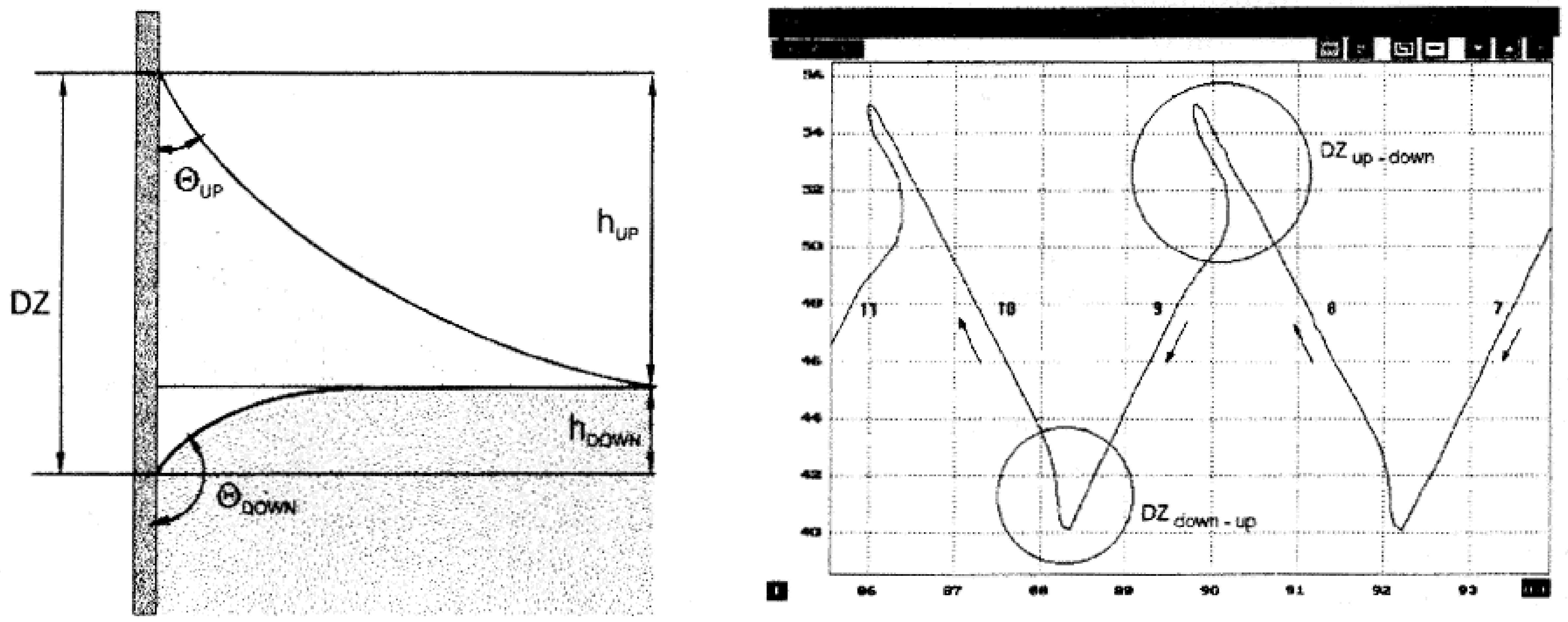
For TG1 DZ = 3 mm, it provides a high quality of films.

The macroscopic magnitude of DZ guarantees stability of the LB deposition method against vibrations. For this reason one manages to form similar films with atomic accuracy on large surfaces without special, expensive, means of protection. The cost of equipment for film deposition is sharply reduced because the presence of small vibrations does not result in deterioration of film quality. The great demand on the equipment for LB film formation is the requirement of purity of the used materials from irrelevant SASs. In the case of film formation of compounds that are unstable against oxidizers (LB films of free radicals, strong donors), the requirement of protection of a formed film against convective flows of an oxidizer is added.

One of the modern variants of the LB system, MDT-LB5, which is produced by the Russian company NT-MDT, is shown in Fig. 2. The system consists of a LB trough with measuring gauges and a set of executive gears, an electronic block of signals commutation and a control station on the base of an IBM-compatible computer.

The LB trough structure consists of (Fig. 2):

- Marble base (1) with supporting screws (2) for alignment of the trough level.
- Trough (3), established on four stands (4). The stands are fixed rigidly on the marble base. A trough of Plexiglas or temperature-controlled trough of aluminum alloy covered with Teflon with two demountable thermostats, which enter the complete set (5) from below.
- The system can be supplied with a specialized trough according to customer applications.
- Drive of the right barrier (6) and drive of the left barrier (7). Each drive is fastened rigidly to the base by two screws.
- Two barriers of Teflon, right (8) and left (9).
- Frame (10) for fastening of gauges and



a

b

Fig. 1. The diagram of deposition of Langmuir film (Fig. 1) and the diagram of deposition of SAS TG1 (Fig. 1b). The deposition was carried out with maintenance of constant surface pressure. The barrier coordinate controlled by monitoring system is along x-coordinate, and coordinate of substrate moving upwards and downwards—along y-coordinate. The dissemetry of $DZ_{up\text{-}down}$ and $Dz_{down\text{-}up}$ is caused by unequal stretching of film at deflections of meniscus upwards and downwards. The deposition was carried out by MDT-LB5 system.

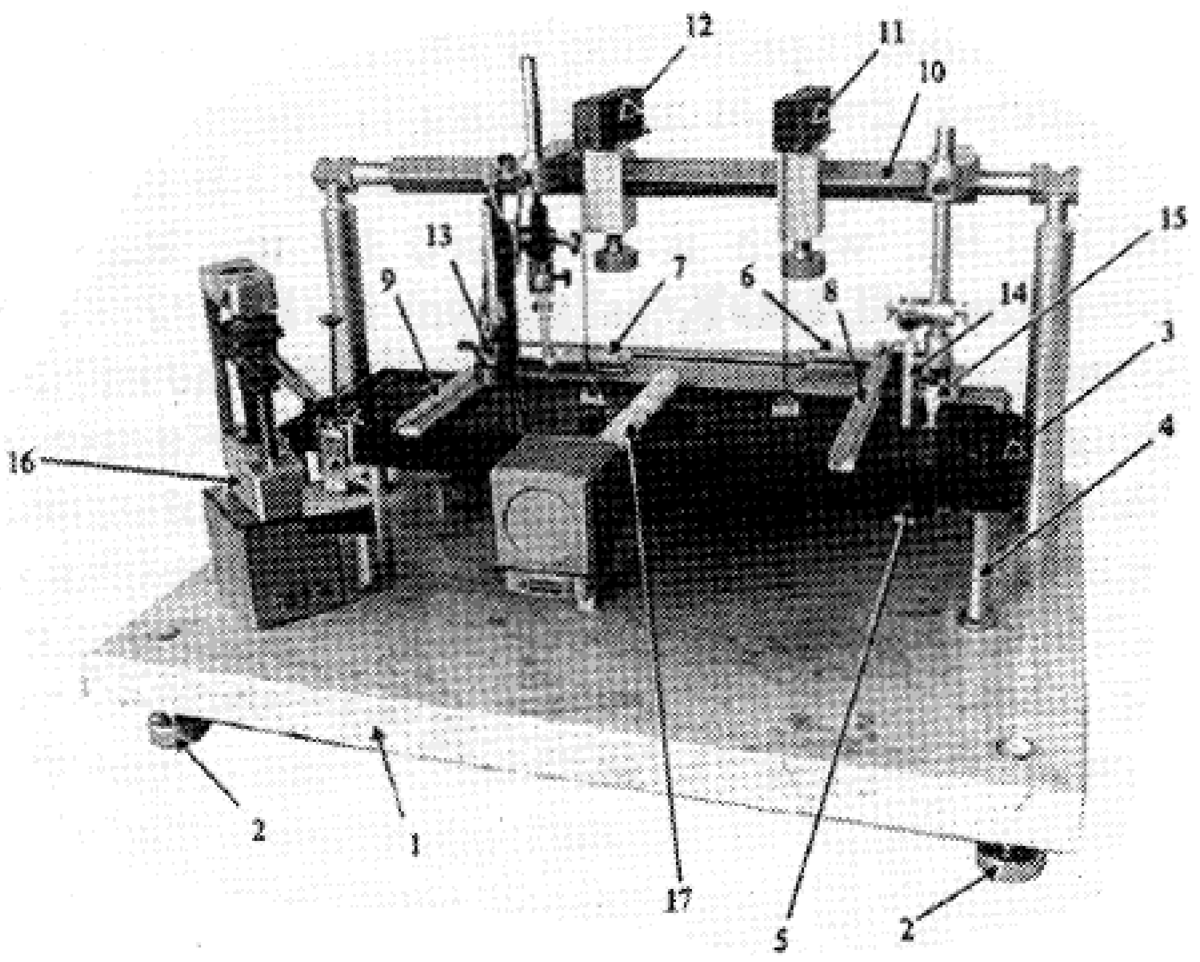


Fig. 2. System of molecular deposition MDT-LB5.

accessories. The frame is rigidly fastened to the base. The right and left electronic balances that measure surface pressure (11, 12), a gauge of electrical potential difference (13), a temperature gauge (14), and mixer (15) are established on the frame.

- A system of monolayer deposition by the LB method (dipper) (16). The dipper is placed in any place on the of marble base.
- A system with the rotating sample holder (rotor) (17) for deposition of complex film structures by the method of alternating layers. The rotor is fixed on the bath by two screws and divides the bath surface in two parts.

Application of such systems provides fast and complex research of SAS properties and the opportunity of forming a wide class of structures.

NANOTECHNOLOGY

During the last 10 years science has taken a new direction which concerns the 'nano' prefix (Bykov *et al.*, 1993; Bykov, 1994), and is developing fast in this field. At present we connect with the 'nanotechnology' term all that permits one to make and study structures of nanometer size, though the second part of the word, 'technology', is usually associated with industry. In the existing context, 'nanotechnology' is a pure scientific technology that enables one to carry out modeling of nanometer elements.

The question of whether there are now ways of making the transition from scientific modeling to industrial nanotechnology which is capable of creating and reproducing functional systems with a number of elements of the order of and more than 10^{12} pieces/cm^2 is quite reasonable. If it is basically clear *how* it is possible it to do so, there is the question of modeling: which elements and how they can work in this range of sizes, which materials should be applied to their manufacturing, which technological processes should be developed, etc. That is, the industrial investments to the scientific development arising in the 'nano' field become justified.

The very idea of penetration to the nanometer area occurred with the invention of scanning probe tools—tunnel and atomic force microscopes (Binnig & Rohrer, 1982; Binnig *et al.*, 1986) (1981–1986). During the past decade these microscopes have been transformed from unique tools to ordinary devices used by all modern material technology laboratories. A probe is a sensitive element in these devices. A rigid conducting tip of metal inert in air (usually of alloys of platinum or tungsten) is a probe in a tunnel microscope. A tip on a flexible beam cantilever is a probe in a force microscope. At present the cantilevers are made by group methods with the application of a microelectronics technological base and micromechanics technologies. The tip materials can be silicon or silicon nitrite. Updating of the tips in order to make them more surface conductive, magneto sensitive and chemical sensitive is possible. Working in the tunnel mode, it is possible to modify a surface by applying short electrical pulses to the tip or by mechanically engraving the surface (Bykov *et al.*, 1994). Besides working with a tunnel probe, it is possible to exert influence on the surface by setting the probe–substrate distance, as well as to measure the electrical properties of a system, which is complex to carry out when working in the force mode. In the force mode it is essentially easier to realize what has taken place as a result of the effect, as well as to work with bad conducting and dielectric surface layers. This is why there is a requirement to combine the STM and AFM modes in one device. In order to implement this idea it is possible to work at the start with two closely situated probes. This idea has already been realized in microscope P4-SPM-MDT. It is P4-SPM-MDT which is used by the author and his colleagues. The STM and AFM probes in this device in essence can be brought together up to several micrometers and they can be used within one scan (the size of a scan in the device is $14 \times 14\ \mu m^2$ in high resolution mode, and in low resolution mode a wide field scanner can be applied with the size of a scan up to $100 \times 100\ \mu m^2$). In order to work with one probe in both the tunnel and force modes it is necessary to know how to change the rigidity of a cantilever. This idea has not yet been technically performed, but in essence it is quite possible to execute.

In Fig. 3(a) the design of an ordinary silicon cantilever is shown, and in Fig. 3(b) is a cantilever with controlled rigidity.

A control structure that consists of piezoceramic layers and electrodes feeding the electrical potential is formed on top of silicon on the spring surface of a cantilever. At voltages applied to polarized piezoceramics it spreads or compresses depending on signal polarity. Thus the spring acts as a bimorphous plate that is capable of pressing against a fixture. If the tip material is alloyed with suitable ions, it becomes conducting and can be operated with such a cantilever in both force and tunneling modes. The appearance of a similar cantilever will occur in the near future. With such a probe it is possible to carry out modeling and a complex study of the geometry and electrical properties of the probe–substrate system. For the realization of detailed research of electrical properties of nanoelements the application of a three-probe measuring system is desirable.

The development of the idea of a probe with controlled rigidity leads to the idea of a 'megaprobe'—a large crystal with a matrix of controlled probes. In Fig. 4(a) the design of such a chip is represented. An elementary executive element of the 'megaprobe' is a triad of probes located within the limits of one scan. At present it is known that the size of one scan can be up to $200 \times 200\ \mu m^2$. The smaller the area where the probes are located, the greater is the possible speed of structure forming. Using already existing piezo or ferroelectric materials, an angular deviation of each probe of more than 2° at a control voltage of ±15 V, can be provided which at a length of cantilever probe of 50 μm can provide a probe deviation of more than $1{\cdot}7\ \mu m$, which is enough for 'megaprobe' operation.

In the device the substrate with the formed structure is established on a scanner. A system of controlled probes—'megaprobe'—is situated over it. Further, the substrate moves under the probes, and the probes controlled by a powerful computer form a desired structure and supervise its properties. The time of such hyperstructure formation is not of any special significance, though it cannot be small and reduced to single

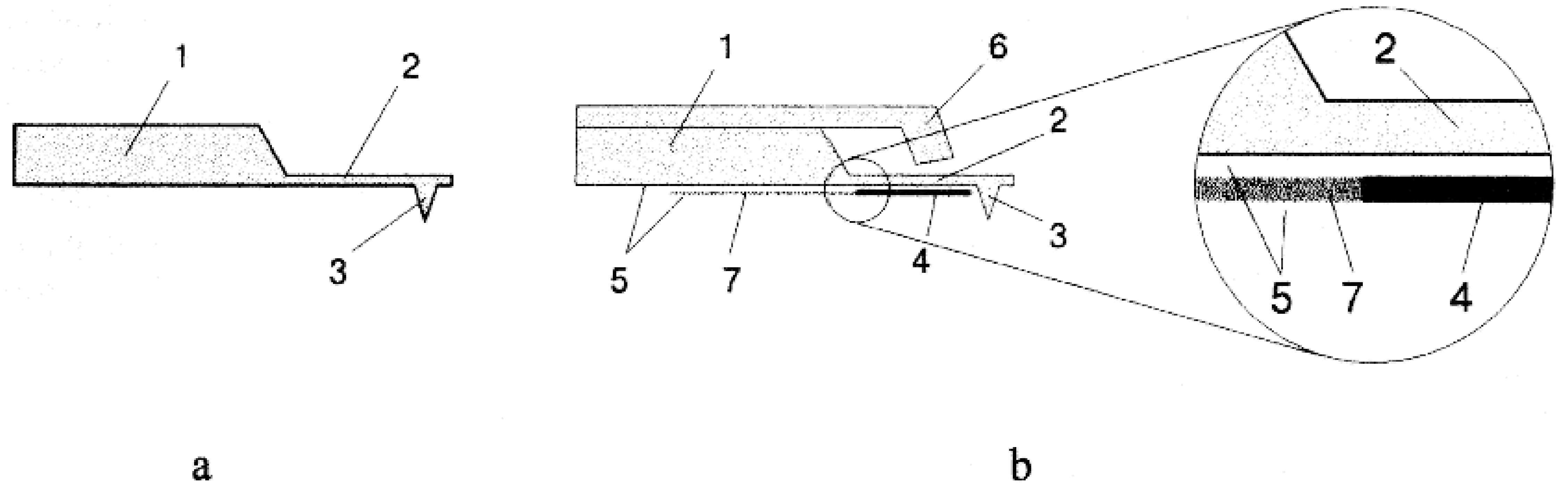


Fig. 3. Design of usual cantilever (a) and cantilever with controlled rigidity (b). 1—cantilever base, 2—deflecting beam, 3—cantilever tip, 4—piezoceramics, 5—conductive layers, 6—damper, 7—dielectric.

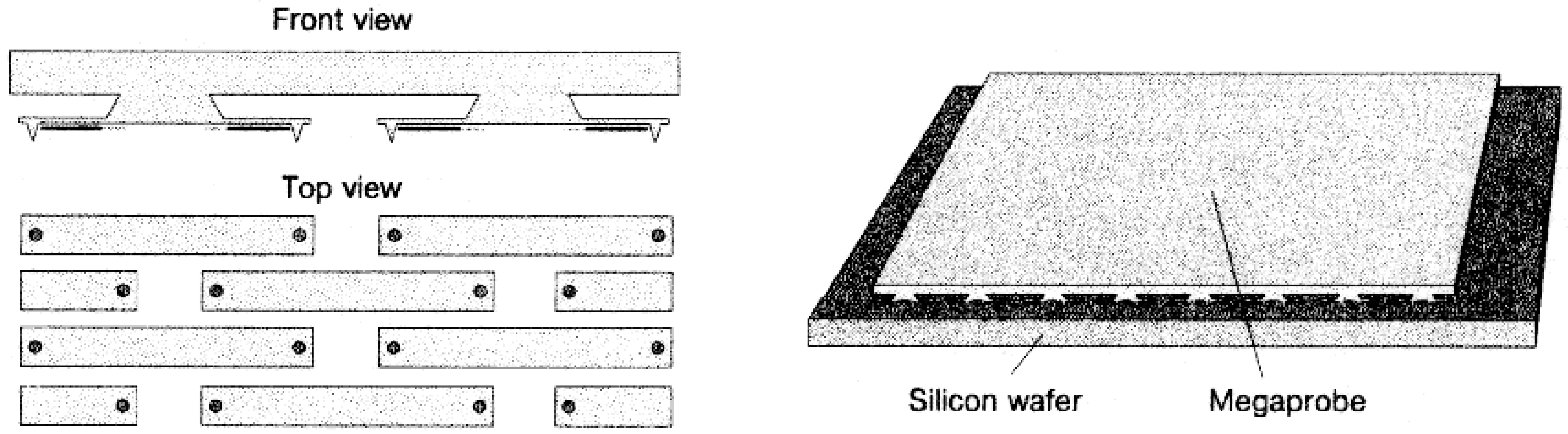


Fig. 4. Schematic image of "megaprobe" and systems of forming of functional structures.

scan processing. We shall evaluate the order of this time: we shall accept the time of processsing of one point of a scan as 10 μs, the number of points in a scan with size $100 \times 100\ \mu m^2$ and step 0·1 nm as 10^{12}, and the time of processsing of such scan will be 10^7 s or 115·7 days. At present, manufacturing a complex chip can exceed a month, and in general a person forms a functional unit over 9 months in the womb and for 20 years in society. So it is possible to say that the transition from the stage of scientific modeling to industrial technology is at least basically seen, which is why the stage of scientific modeling of nanoelements is already economically justified.

At present, application of nanotechnology for solving more simple problems is possible. For example, for the recognition of large organic molecules, viruses and microorganisms. The group of Dr. I. Kurochkin (Institute of Molecular Diagnostics and Treatments, Moscow, Russia) have already carried out this work. They have already proved the possibility of reliable recognition of a number of pathogenic viruses and microorganisms. For film formation they used Langmuir equipment (in particular MDT-LB5 systems). The analysis was performed by probe microscopes P4-SPM-MDT and the recognition made by original software products.

RESULTS AND DISCUSSION

Langmuir layers were formed and deposited by using a computer-controlled LB trough 'MDT-LB5' (produced by MDT Corporation, Russia), which has two independent barriers and an aluminium trough coated with Teflon. Surface pressure measurements were made with an accuracy of 0·02 mN/m.

The investigation of structures and modification of LB films were done by P4-SPM-MDT (produced by MDT Corporation, Russia). This device is shown in Fig. 5. For the modification mode we used $Pt_{0{\cdot}8}Ir_{0{\cdot}2}$ and $Pt_{0{\cdot}8}Ro_{0{\cdot}2}$ tips with and without isolating coating. The quality of the tips was controlled by measurement of $I_t(z)$ dependence. We used tips if $Z(I_t) - Z(I_t/2) <$ 0·5 nm.

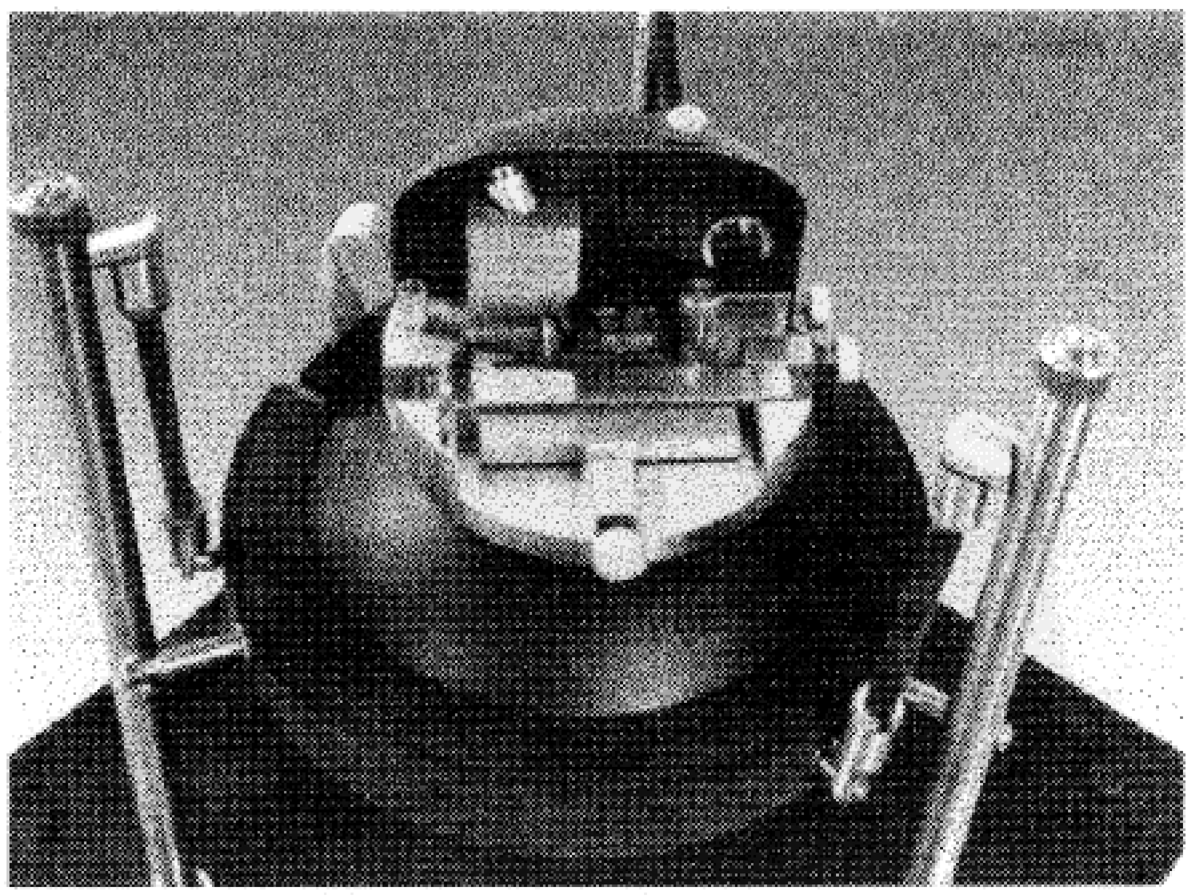


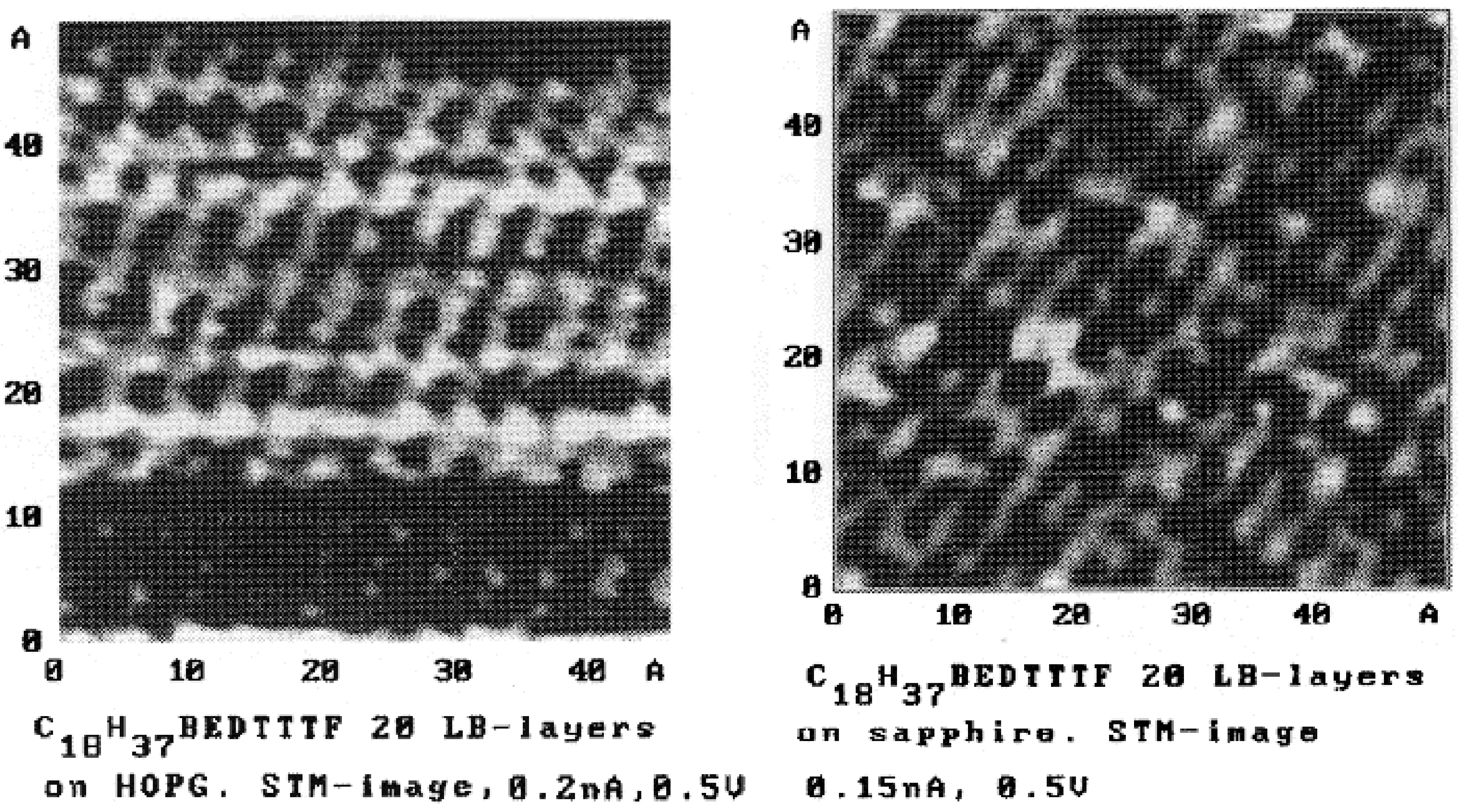
Fig. 5. Scanning probe microscope with lithographic possibilities P4-SPM-MDT.

LB films of $C_{18}H_{37}$BEDTTTF were formed from the water–air surfaces. The concentration of $FeCl_3$ on the water surface was 10^{-4} mol/l. The deposition was done onto HOPG (disorientation angle <30′) and monocrystalline sapphire by the horizontal lift method. After deposition of each layer the substrate was dried by a centrifuge.

The STM structure studies were made at HOPG and sapphire substrates after deposition of 20 LB layers. In Fig. 6 there are STM images of these films. In Fig. 6(a) is the STM image of 20 LB layers of $FeCl_3$-doped $C_{18}H_{37}$BEDTTTF at HOPG. The conditions of STM measurements were : I_t = const. = 0·2 nA, U = 0·5 V. There is the periodical structure of this STM image with a period of ≈0·4 nm. In Fig. 6(b) is the STM image of 20 LB layers of $FeCl_3$-doped $C_{18}H_{37}$BEDTTTF at monocrystal sapphire with a CR spray-coated electrode in the form of contra-direction lines with a distance between lines of 200 mkm (Bykov, 1994). The conductivity of LB films was controlled by a tester. The conductivity was absent before LB film deposition. The resistances of structures were 300 Ω–1·5 kΩ after 20 LB layers deposition. The STM tip was installed between Cr lines by optical microscope control. The conditions of STM measurements were: I_t = const. = 0·15 nA, U = 0·5 V, velocity of scanning = 30 nm/s. There is a periodical structure of this STM image with the same period of ≈0·4 nm! But the quality of the HOPG deposited film is much better than that of sapphire and the sizes of loose defect regions on HOPG are greater.

The local modifications studies were made as at conductive LB films of $FeCl_3$-doped $C_{18}H_{37}$BEDTTTF (three LB layers on HOPG) and non-conductive LB layers of oligomer molecules of butyl-cyanacrylate (BCA) and chlorbutadiene (CBD) in combination $\{(BCA)_5(CBA)\}_n$ at the HOPG substrates.

The modification of $FeCl_3$-doped



a

b

Fig. 6. STM images of conductive films of 20 LB layers of $FeCl_3$ doped $C_{18}H_{37}$BEDTTTF at HOPG (a) and sapphire (b).

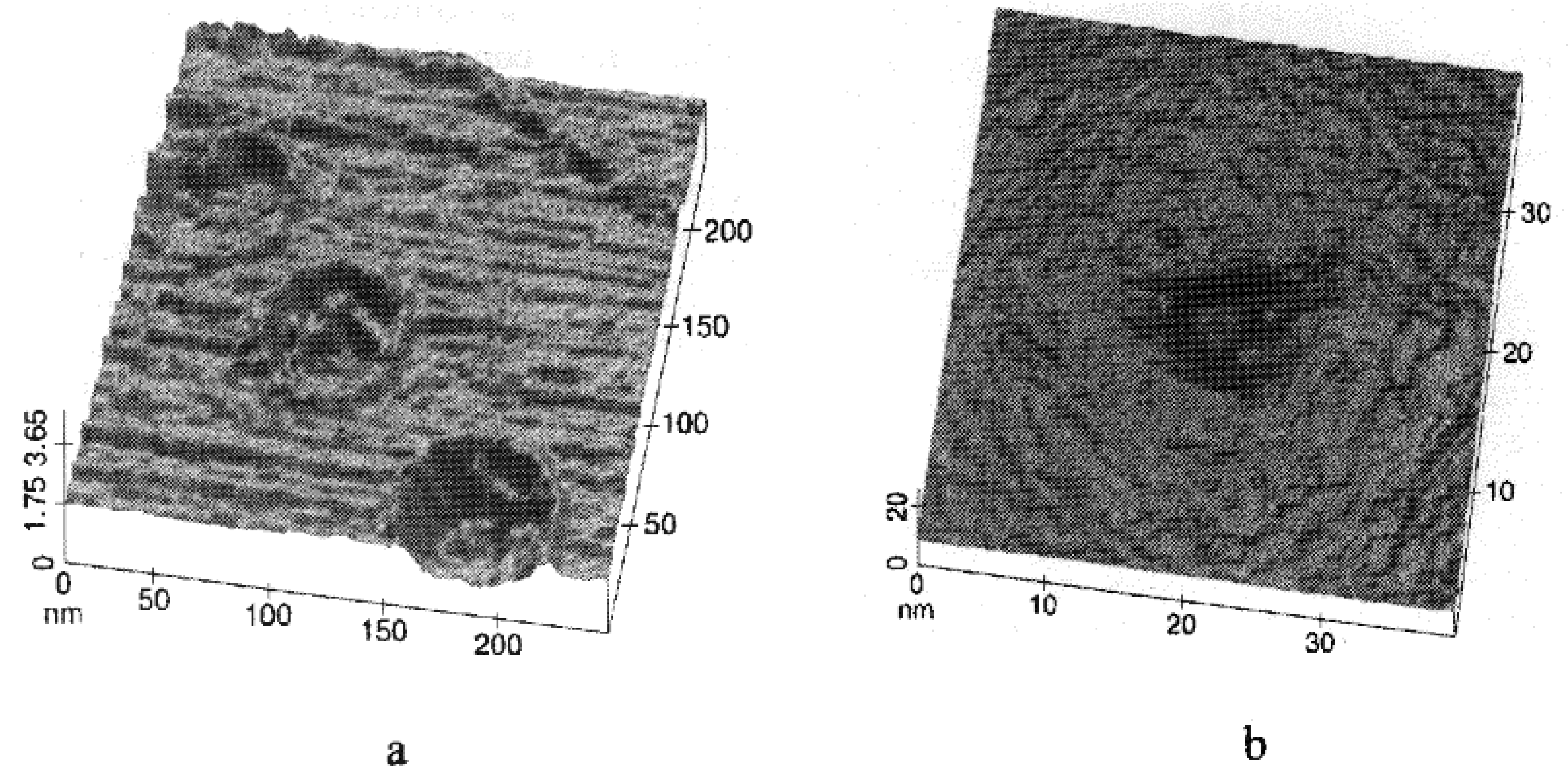
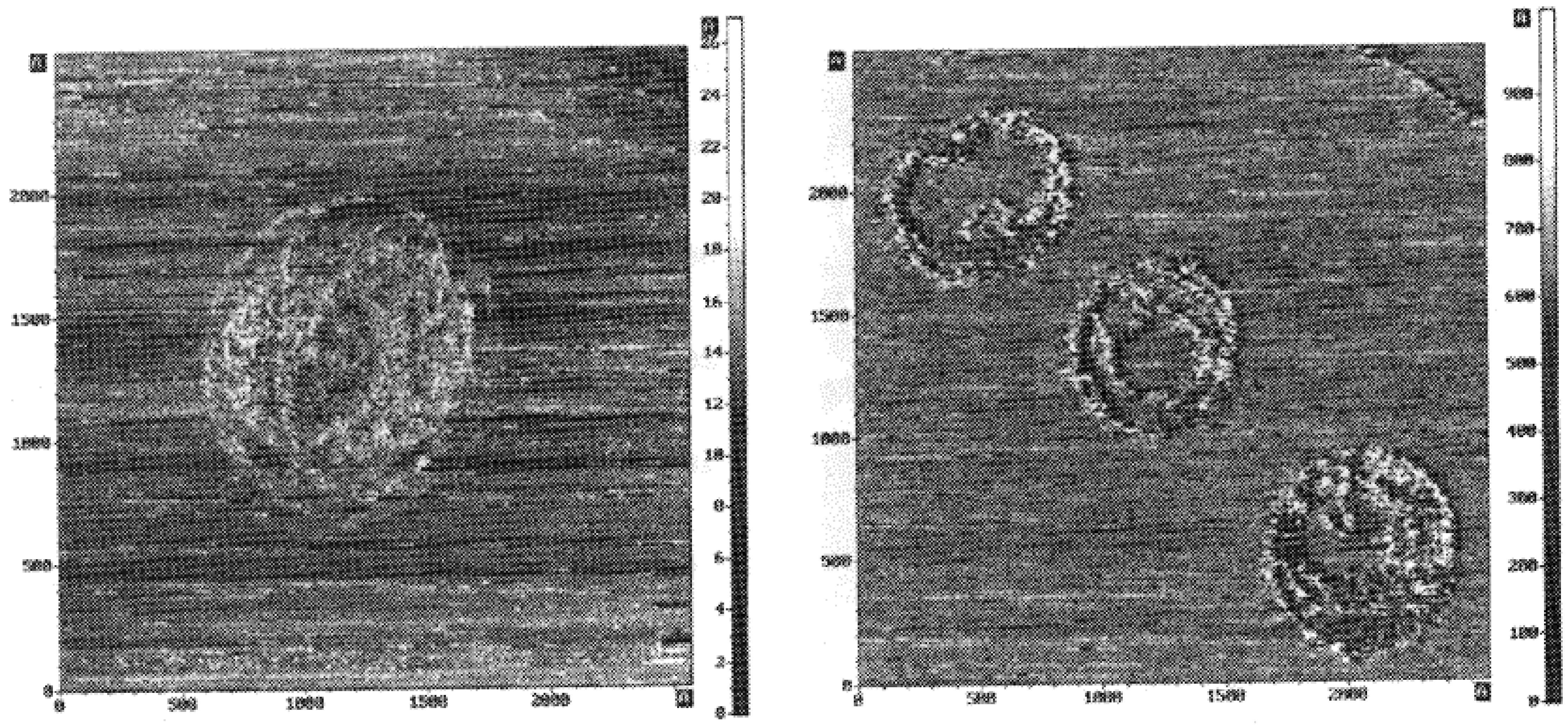


Fig. 7. a—VP induced crater defects; b—VP induced deep flop defect.

$C_{18}H_{37}$BEDTTTF takes place if the amplitude of the voltage pulse (VP) is more than 3·5 V (the VP time is ~15 mks). We observed two types of VP-induced modifications: defects that look like a deep flop (Fig. 7(b)) and defects that look like a crater (Figs. 7(a) and 8). Deep flop defects had a triangular form with the side of ~4–5 nm in the plane. The depths of deep flop defects are 6–8 nm, with practically vertical walls. The second type of defect—'crater'—had a diameter of 40–50 nm and a depth of ~2 nm. Around the crater there is a swell with a height of ~2 nm.

The scanning tunneling spectroscopy (STS) studies of crater defect regions (see Figs 8(a,b)) have boundary fluctuations (WF) of work functions and the same WF at the center of the crater and at non-acting surfaces.

In Fig. 8 there are $(\delta I_t/\delta Z)_{I\,=\,\mathrm{const}}(X,Y)$ images of VP, induced crater defects, after the first litho



spectroscopy, 18.5kHz
$C_{18}H_{37}$BEDTTTF, 3 LB-layer on HOPG,
litho 5V, 1nm

a

spectroscopy, 18.5kHz
$C_{18}H_{37}$BEDTTTF, 3 LB-layer on HOPG,
after 3 litho acts

b

Fig. 8. Scanning tunneling spectroscopy.

act and the same film after three litho actions. The actions were done by the voltage with amplitude of 5V, τ = 15 mks.

We assume that deep flop defects and crater defects have quite dissimilar genesis. A deep flop defects is the VP-induced dielectric point on the conductive layer. The depth of it is due to mechanical deformation of the substrate having current during the measurement. The crater defects are due to Coulomb charge destruction of the conductive layer. Only crater defects take place on non-conductive materials.

In Fig. 9 are the STM images of LB film of three monomolecular layers of $\{(BCA)_5(CBA)\}_n$ on HOPG substrate. The conditions of modification were $U_{\text{mod}} = 10$ V, $\tau_{\text{mod}} \approx 10$ mks. The conditions of STM measurements were: I_t = const. = 0·2 nA, U = 50 mV. In Fig. 1(a) is the structure image immediately after action. There is a hill and a hole with the same size. The vertical hole/hill sizes are ≈1·1 nm and correspond to the thickness of one Langmuir layer of this material. In Fig. 9(b) is the image of the same place after five scans; but there is the additional hole in the place of the hill. This the result of mechanics action between the tip and substrate. It should be noted that the hill (Fig. 9(a)) and holes have approximately the same anisotropic sizes. In Fig. 9(b) it is possible to see the regularity of form of the structured inclined rows. The directions of these rows correspond to the hill/hole anisotropy. It is possible to suggest that in Fig. 9(b) we have the molecular resolution of the oligomer LB film and the hill and holes are the separated oligomer molecular clusters.

In Fig. 10 the AFM image of the microorganism rickettsia is shown. The specimens researched were kindly donated by Dr. Kurochkin's group.

CONCLUSION

It is possible to conclude that the transition from scientific modeling to industrial nanotechnology is possible, and consequently intensive scientific search in this direction is justified. LB technology can act as one of the main technologies for the preparation of environments for manufacturing nanoelements.

The combination of LB technology, SPM reading of conditions and computer analysis of results can already give a practical output—bio and chemical SPM sensors.

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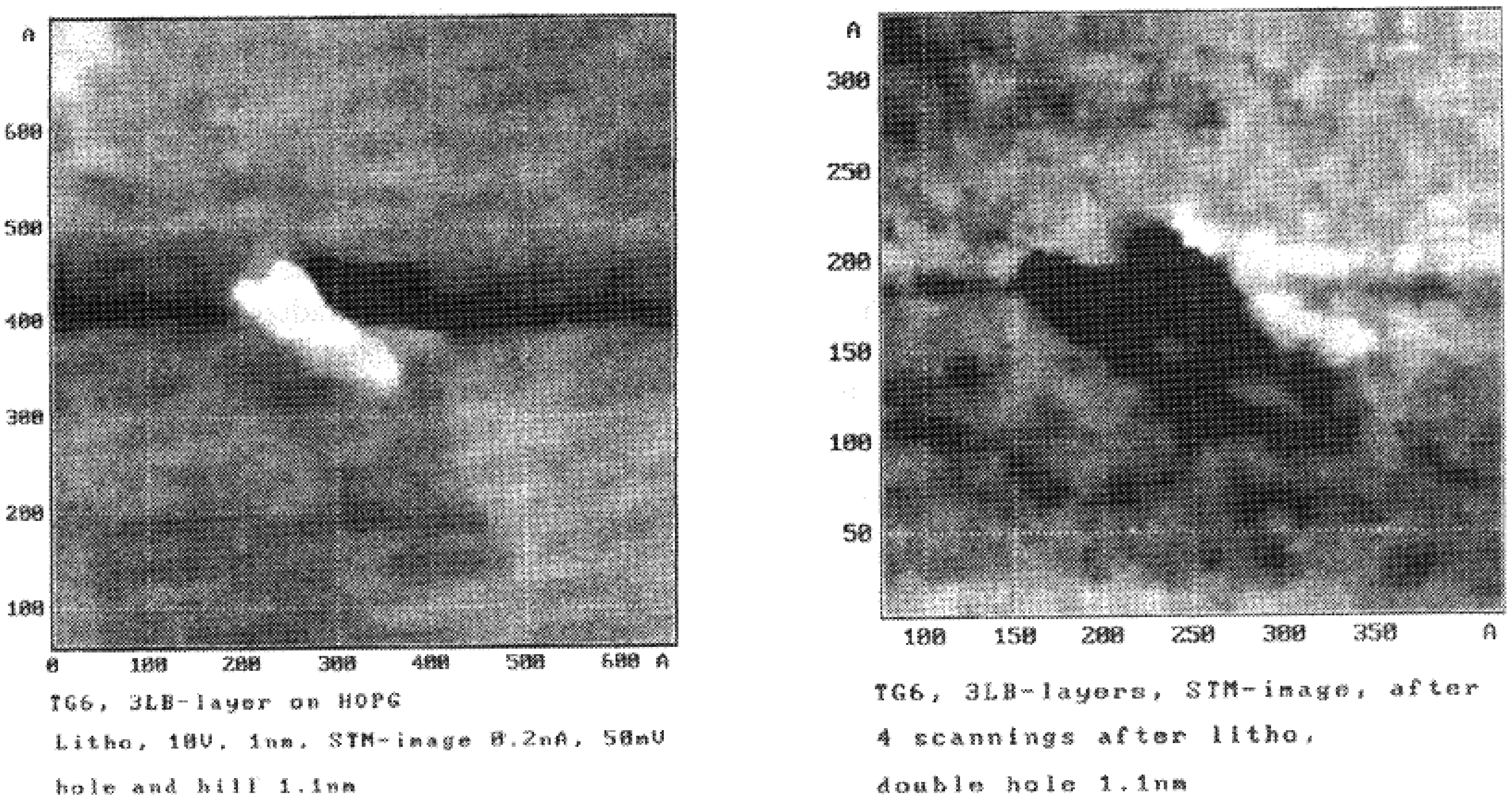

Fig. 9. STM image of three layers of $\{(BCA)_5(CBA)\}_n$ after litho action. a—immediately after action, b—the same place after five scanning.

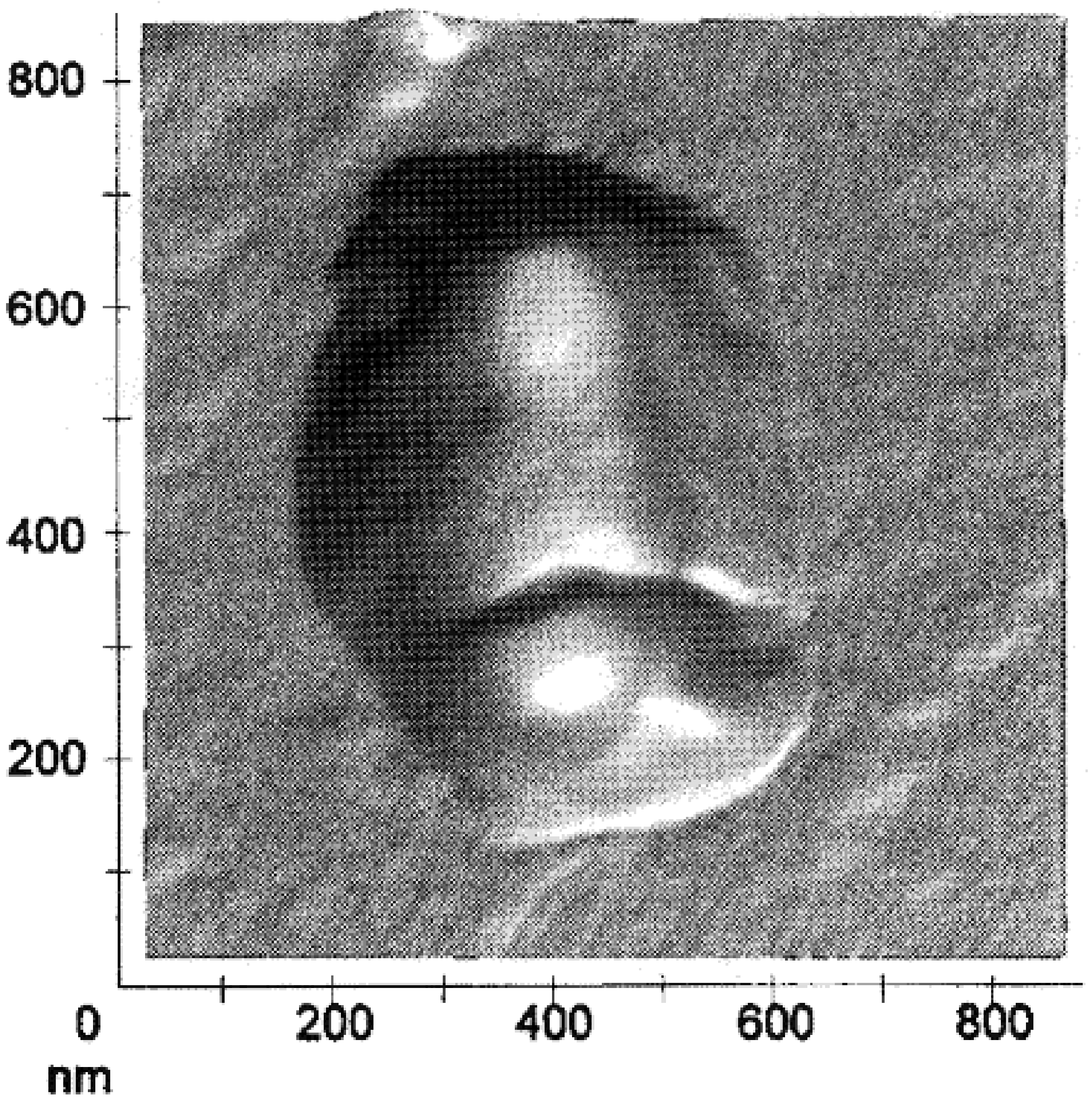

Fig. 10. AFM image of rickettsia. The image is received by microscope P4-SPM-MDT in the mode of scanning with constant force $F = 10^{-8}$N. The scanning was made with cantilever of Si_3N_4 with curvature radius of the tip of 50 nm.

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