Design of an atomic force microscope for topographic studies

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The developments in atomic force microscopy have been briefly reviewed. The atomic force microscope, simple in operation and modular in design has been developed at the Central Scientific Instruments Organisation, Chandigarh. Initial results of topographic studies in contact mode are presented in this paper.

THE development of atomic force microscope (AFM) in 1986 by Binnig and co-workers[1] was a spin off from the scanning tunneling microscope (STM) development which took place in the early 1980. AFM has an important advantage over the STM in that images can be obtained for insulating as well as conducting surfaces.

In the past few years, several scanning probe microscopes (SPMs) have been developed, such as the atomic force microscope, the photon scanning tunneling microscope[2], the scanning ion microscope[3], the thermal profiler microscope[4] and the magnetic force microscope[5]. These new scanning probe microscopes utilize contrast formation mechanisms other than electron tunneling and allow acquisition of high resolution information from insulators. Among these SPMs, the AFM has received greatest attention, as it is thought to be the most suitable for biological research.

In AFM, the sample to be imaged is brought close to a sensitive tip attached to a small cantilever. Interaction forces between sample and tip deflect the cantilever. In the original design, the cantilever displacement was detected by the tunneling tip on the back of cantilever. Subsequently, various optical techniques have been developed for displacement detection. These techniques include diode laser feedback detection[6], interferometry and optical beam deflection[7,8].

AFM was invented to image non-conducting surfaces with high spatial resolution. It is rapidly evolving as an analytical tool for profiling of surfaces from micrometer to nanometer scale. Images of surfaces of this length scale are becoming increasingly important and open up new opportunities in the miniaturization of machines[9].

The interaction force can be measured as a function of the separation between the tip and surface (a 'force curve') by multiplying the cantilever spring constant K_n, by the cantilever deflection, z. The system developed at the Central Scientific Instruments Organisation (CSIO), Chandigarh, uses the optical lever method, the cantilever position can be detected to within ~ 2 nm. For $K_n = 0.01$ N/m, this results in a corresponding force resolution of 0.02 nN.

Here, we present the design of an AFM capable of measuring surface topography. The AFM (Figure 1) is modular, simple in operation and the components can be arranged to calibrate both the z displacements of the piezo and the voltage-to-distance responsivity of the detector. These calibrations are needed in addition to the cantilever spring constant K_n, for accurate force determination between the tip and surface.

Instrumentation details

Mechanical and optical design

All the force microscopes have the following major sub-systems[10]: a sharp tip mounted on a soft cantilever spring; a way of sensing the cantilever's deflection; a feedback system, to monitor and control the deflection (and hence, the interaction forces); a mechanical scanning system (usually piezoelectric), to move the sample with respect to the tip in a raster pattern; a display system, for the display of images of samples surface after transforming the measured data into image. The scanning, feedback and display systems are very similar to those used for scanning tunneling microscopy.

Figure 2 shows a block diagram of the CSIO-AFM set up. The sample approach system consists of a differential micrometer and a piezo electric ceramic tube positioned to move the sample under the tip in X–Y–Z directions. Although the basic elements of all the force microscopes are similar, the details of implementation vary. The original atomic force microscope, for example, used a handmade cantilever spring forged from a piece of gold foil approximately 1 mm long. A small diamond stylus glued to the foil served as the tip. Today, the most advanced AFM cantilevers are microfabricated from silicon, silicon oxide or silicon nitride using photolithographic techniques. Typical lateral dimensions are of the order of 100 μm, with thicknesses of the order

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Figure 1. Atomic force microscope developed at Central Scientific Instruments Organization.

of 1 μm. This geometry gives spring constants in the range of 0.1–1 N/m and resonant frequencies 10–100 kHz. The cantilevers can be fabricated with integrated tips or else small diamond chips can be glued on by hand.

Cantilevers are usually made by lithographic methods using material that can easily be fabricated en masse such as silicon, silicon nitride and silicon oxide. The tip can be made of the same material.

The other critical component of the AFM is the sensor that detects the cantilever's deflection. Ideally, the sensor should have subangstrom sensitivity and should exert negligible force on the cantilever. Electron tunneling, which was the method Binnig used, has the virtue of being extremely sensitive. The tunneling current between two conducting surfaces changes exponentially with distance, typically by a factor of 10 per angstrom of displacement. Although excellent results have been achieved using tunneling detection, it has the disadvantage that its performance can be degraded if the tunneling surfaces become contaminated. For this reason among others, most AFMs being designed use some form of optical detection.

Optical detection schemes are divided into two basic types: interferometry[11] and beam deflection[12,13]. Both of these methods are capable of measuring cantilever deflections of the order of 0.1 Å, with a detection bandwidth of dc to 10 kHz. In a typical beam deflection AFM, light from a laser diode is specularly reflected from a mirror-like cantilever surface. The direction of the reflected light beam is sensed with a position-sensitive (two-element) photodetector.

Interferometry systems used for atomic force microscopy have taken many different forms, and some of the more recent methods are almost as simple to implement as beam deflection[14–16]. One of the main advantages of interferometry is that it can be used with cantilevers that do not have a mirror-like reflecting surface. This is especially important for magnetic and electrostatic imaging, which often use fine wire cantilevers. Some of the early AFM interferometers had rather poor performance at low frequencies due to the large physical path difference between the reference beam and the light reflected from the cantilever. This problem has now been solved by using either a fiberoptic technique that places a reference reflector within microns of the cantilever or by using a two-beam differential technique[17,18].

CSIO-AFM is based on an optical beam deflection method. Cantilever holder and optical components are mounted in the same housing as shown in Figure 3. The optical components consist of 670 nm, 4 mW focused laser diode (A) (56DLB302/P2, Melles Griot, Boulder, CO), focusing lens, and a Hammamatsu (S-3932) position-sensitive detector (PSD). These components are mounted on separate translation stages so that the laser spot can be accurately focused on the back of the cantilever, and reflected spot is accurately sensed by PSD.

In the approach mechanism the piezo scanner and sample holder are assembled in an aluminium cylinder as shown in Figure 4. The sample is brought into contact with cantilever tip by operating the differential micrometer. The sample scanner is a five-electrode, 6.7 cm long, 1.27 cm diameter piezo tube with 0.05 cm thick walls. The piezo tube has a maximum Z-displacement of 5 μm and X–Y displacement of 25 μm. The approach mechanism used in CSIO-AFM is simple and reliable when compared to the software-controlled stepper motor driven similar mechanism in the commercially available microscopes.

AFM is a sensitive instrument, care must be taken to ensure that external vibrations, such as from the laboratory building, do not limit the performance. The effect of external vibration is to cause unwanted motion of the tip with respect to the sample and the deflection sensor. The immunity of the AFM to external vibration

depends on the frequency of the vibration relative to the lowest resonance of the mechanical system. The mechanical system includes both cantilever and the rest of the AFM. The amplitude of relative tip motion is attenuated by a factor of in the limit. Thus, if the lowest resonant frequency is greater than 20 kHz, a typical 20 Hz building vibration of amplitude 1 μm will result in relative tip motion of less than 0.01 Å, a nearly harmless level. Because cantilevers can be easily made with high-resonant frequencies, the limiting factor is usually the rest of AFM. Good AFM designers focus on making the mechanical components of AFM rigid and compact, especially in the path from the cantilever to the sample.

Feedback control and other electronics

The feedback electronics is similar to the analogue controller described by Altman et al.[19], with the primary difference being in the detector design. We use 486DX2-100 MHz personal computer with 16 Mb of RAM and DT2821-F-8DI data acquisition board (Data Translation, Marlborn, MA). An analogue feedback loop is used to regulate the applied force by comparing V_N to a reference voltage, V_{ref}. During the scanning, the error signal

$(V_{ref} - V_N)$ is simultaneously recorded and amplified before actuating the Z piezo.

Typical spring constants of cantilevers can range from 0.01 to 100 N/m and the forces measured with them can vary substantially. To accommodate this range of forces and the subsequent range of cantilever deflections, V_N can be externally amplified to a maximum of 20X and internally amplified by 8X. For small input voltages, the amplification is linear to one part per thousand. V_N can also be dc offset by ±15 V.

Present and future applications

The ability of AFM to image both conducting as well as non-conducting surfaces has made it a highly versatile tool. It is already being used in a variety of fields.

Future for atomic force microscopy is easy to predict. Because of its great versatility as a metrology tool, the AFM will find numerous applications in product development. Its use has already started for quality control in the optical, semiconductor and magnetic recording industries. Engineers and scientists will make quantitative measurements of things ranging from surface roughness to magnetic bit shapes and from integrated circuit topography to lubricant thickness. The ability to measure three-dimensional profiles with nanometer resolution should make the AFM an important tool for inspecting optical disk stampers, measuring linewidths on integrated
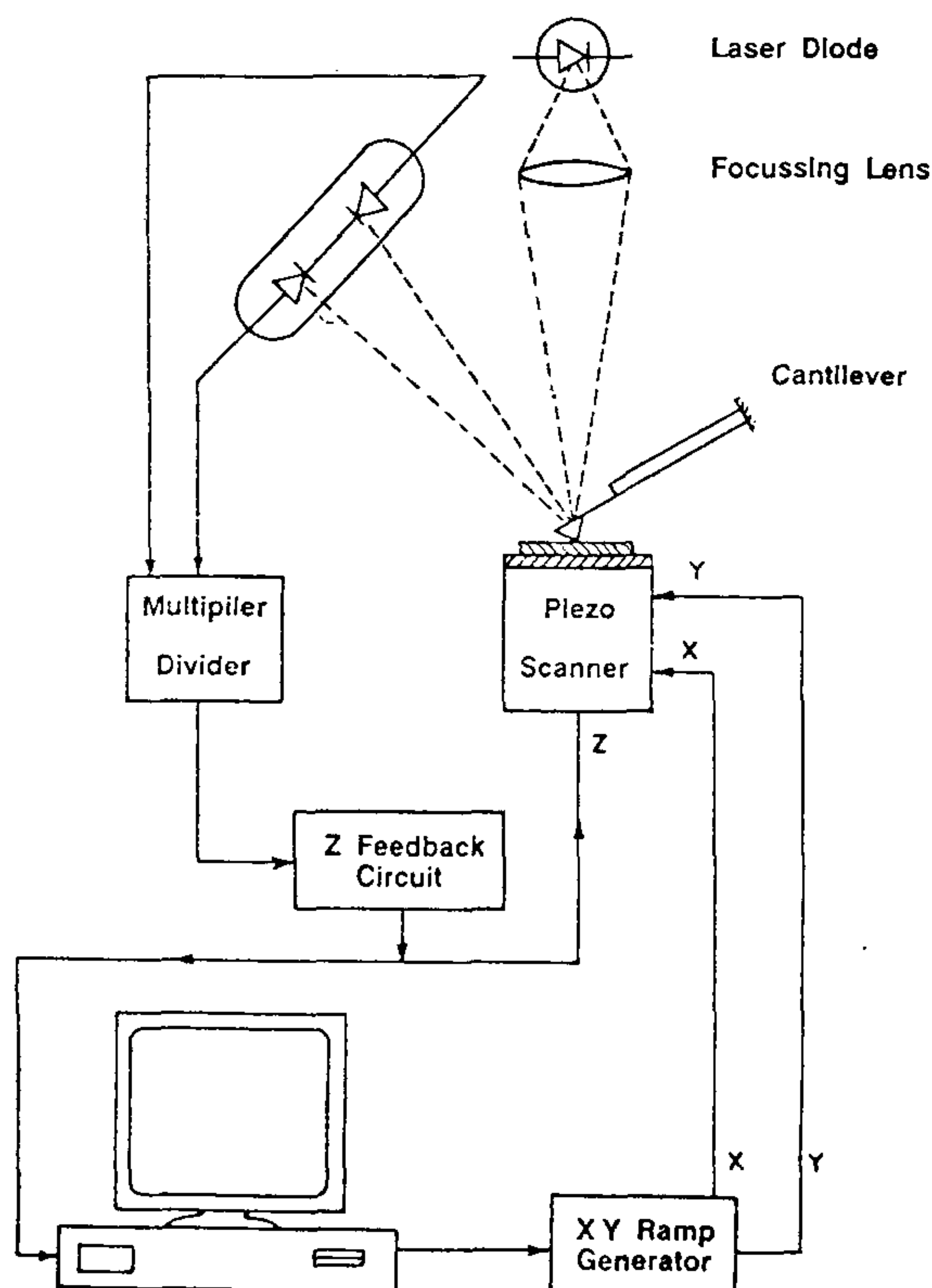

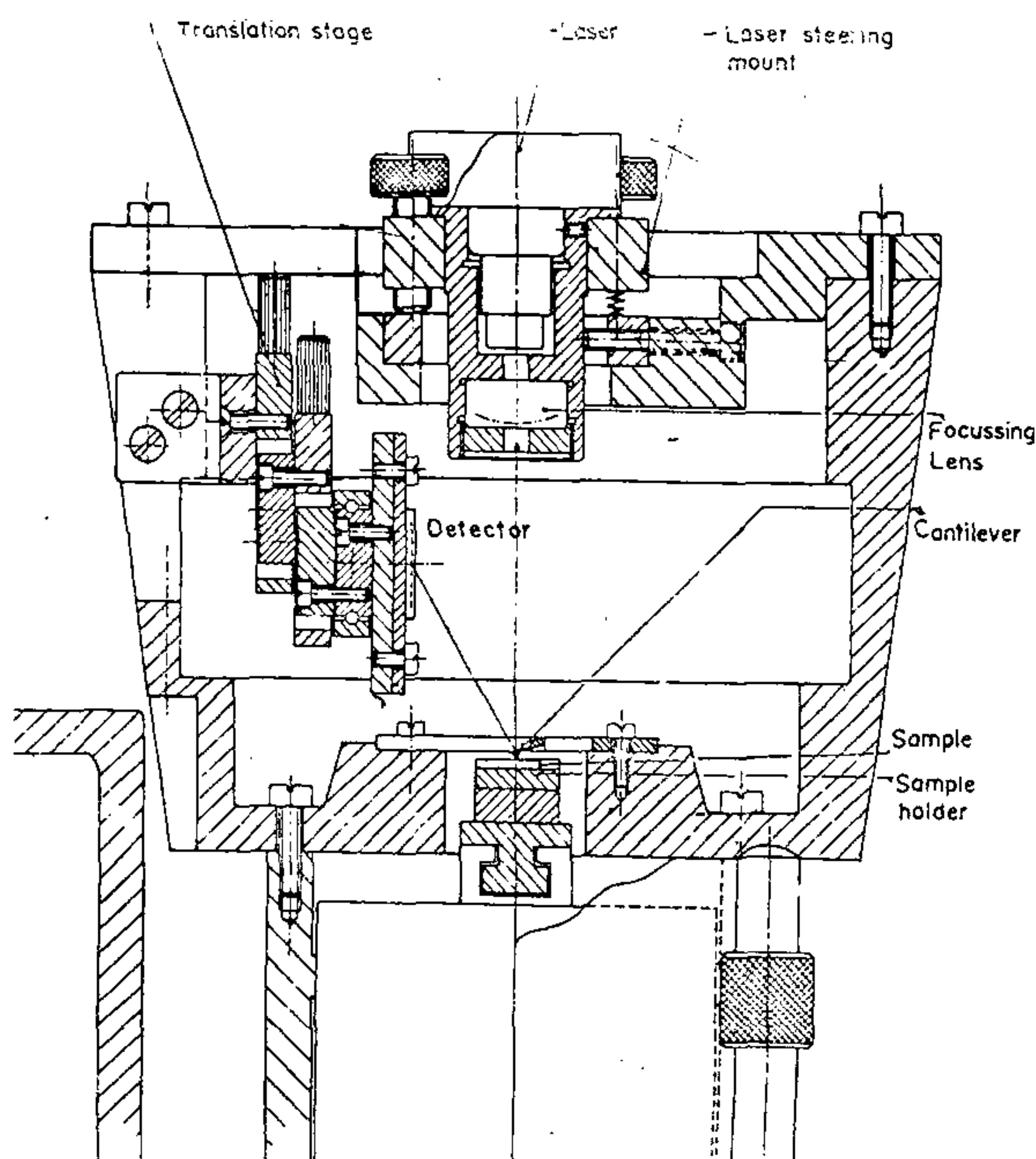Figure 2. Block diagram of atomic force microscope.

Figure 3. Housing containing optical components, cantilever, etc.

circuit masks, and other applications. The AFM is also well-suited for visualizing thin film growth morphology and grain size. Some applications in this area at laboratory level have already been reported. This large base of technological applications will stimulate the development of instruments that are easy to use and that can handle large samples such as whole magnetic disks and silicon wafers. It will also stimulate innovations such as the recent measurement of surface conductance using a contact mode AFM with a conducting tip.

In the physical sciences, the AFM will contribute new knowledge by allowing familiar phenomena such as friction, contact electrification, elasticity and wetting to be studied on a smaller scale than previously possible. It is not yet known whether the AFM will make significant contributions to traditional surface science problems. Only a few microscopes have been operated under ultra-high vacuum conditions with atomically clean surfaces. One interesting application would be to use the AFM to observe surface reconstructions on both insulators and conductors. More work will be necessary, however, to prove that the AFM tips have sufficient resolution and stability to make a significant contribution in this area.
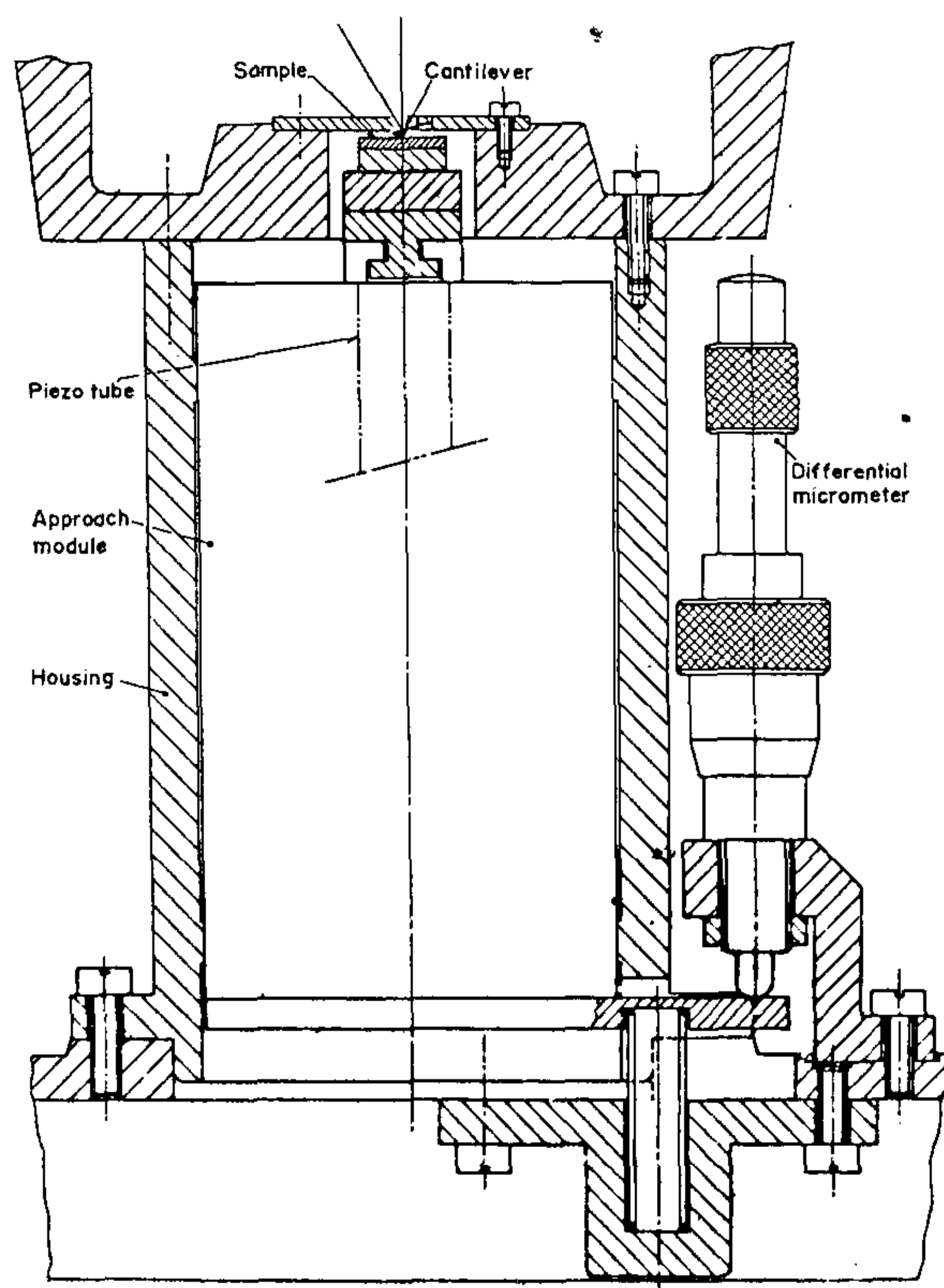
Biological applications of atomic force microscopy, though technically challenging, are destined to be of great importance. Already the AFM has imaged individual biological molecules such as amino acids, biopolymers such as DNA[20,21], macromolecules such as proteins, and even entire cells. In some cases it has even been possible to watch biological processes as they occur, as in the polymerization of the blood clotting protein fibrin. We can expect to see significant benefits if this real time imaging capability can be extended to include processes on cell surfaces. It may be possible, for example, to observe the process by which viral particles, such as the AIDS virus, is attached onto cell membranes. Perhaps the most exciting potential application of the AFM is the sequencing of DNA. Several groups working with AFMs and other scanning probe microscopes are learning how to hold DNA molecules fixed on a substrate and are attempting to distinguish individual bases. Although this is a challenging goal, there is no fundamental reason why the technical problems cannot be solved. When they are solved, it may be possible to reduce the time necessary to sequence DNA, such as that of the human genome, by several orders of magnitude.

Results and discussion

Surface topography measurements is a starting point for a new design of AFM. For experiments in a specific area, certain modifications/attachments to the basic AFM may be required.

Some example data is presented here which demonstrates the working of AFM developed at CSIO, Chandigarh.

Force curves

Force curves can be used to measure the pull-off adhesion force and relating this force to adhesion energy. The
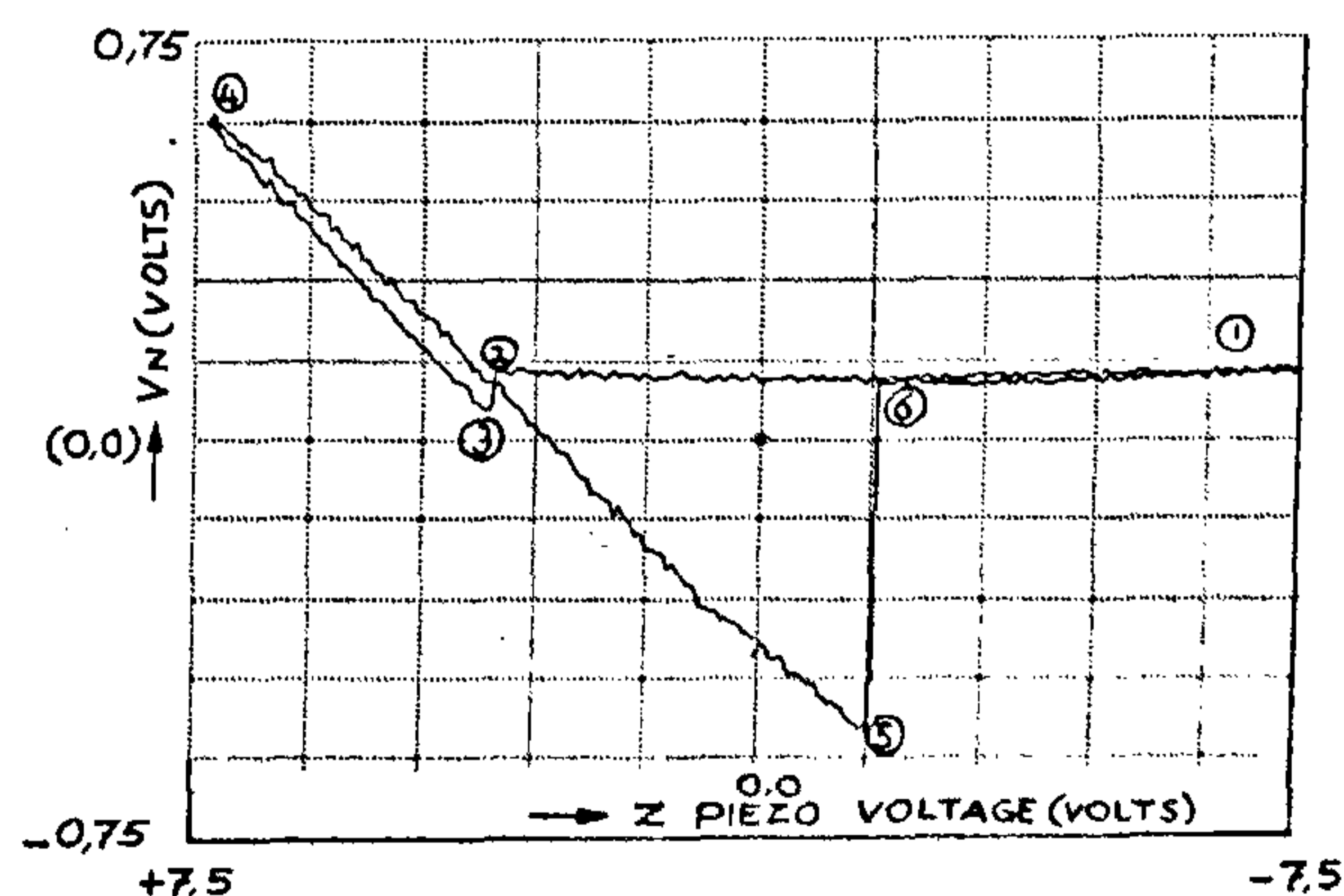
Figure 4. Approach mechanism.

Figure 5. Plot of normal detector voltage V_N as a function of applied voltage.

pull-off force depends on the nature of the binding interaction between the tip and surface during contact. However, relating the pull-off force to the number and strength of bonds broken is difficult because the contact area between the tip and surface depends on the interfacial adhesion and other non-specific forces.

An example force curve measured using a commercial silicon oxy-nitride cantilever with a force constant of $K_N = -0.4$ N/m is shown in Figure 5. The sample surface is a layer of photoresist on glass substrate of holographic grating. In Figure 5, V_N is plotted against the Z piezo voltage, with the numbered labels corresponding to the following reference points: (1) Z piezo fully retracted, (2) pull-on force, (3) maximum attractive force, (4) maximum force which is preset by the computer program, (5) pull-off force (maximum adhesive force), (6) cantilever restored to rest position.

Contact mode imaging

Holographic grating. The method of scanning under constant load (applied force) is frequently used in AFM to image the surfaces. Once the tip contacts the surface and is set to a predetermined load, the X and Y piezo positions are scanned. In Figure 6, gray scale image of a 8300 nm × 8300 nm scan on 1200 lines/mm holographic grating is shown. The images were collected under an applied force of 20 nN.

Micro-machined surfaces

Silicon surfaces with different features written on it by lithography/etching techniques were also imaged in contact mode under preset applied force. In Figure 7, the gray scale image of a 25,000 nm × 25,000 nm scan is shown. The images were collected under an applied force of 20 nN.

Biological and other samples

Figure 8 is a gray scale image of polycarbonate filter
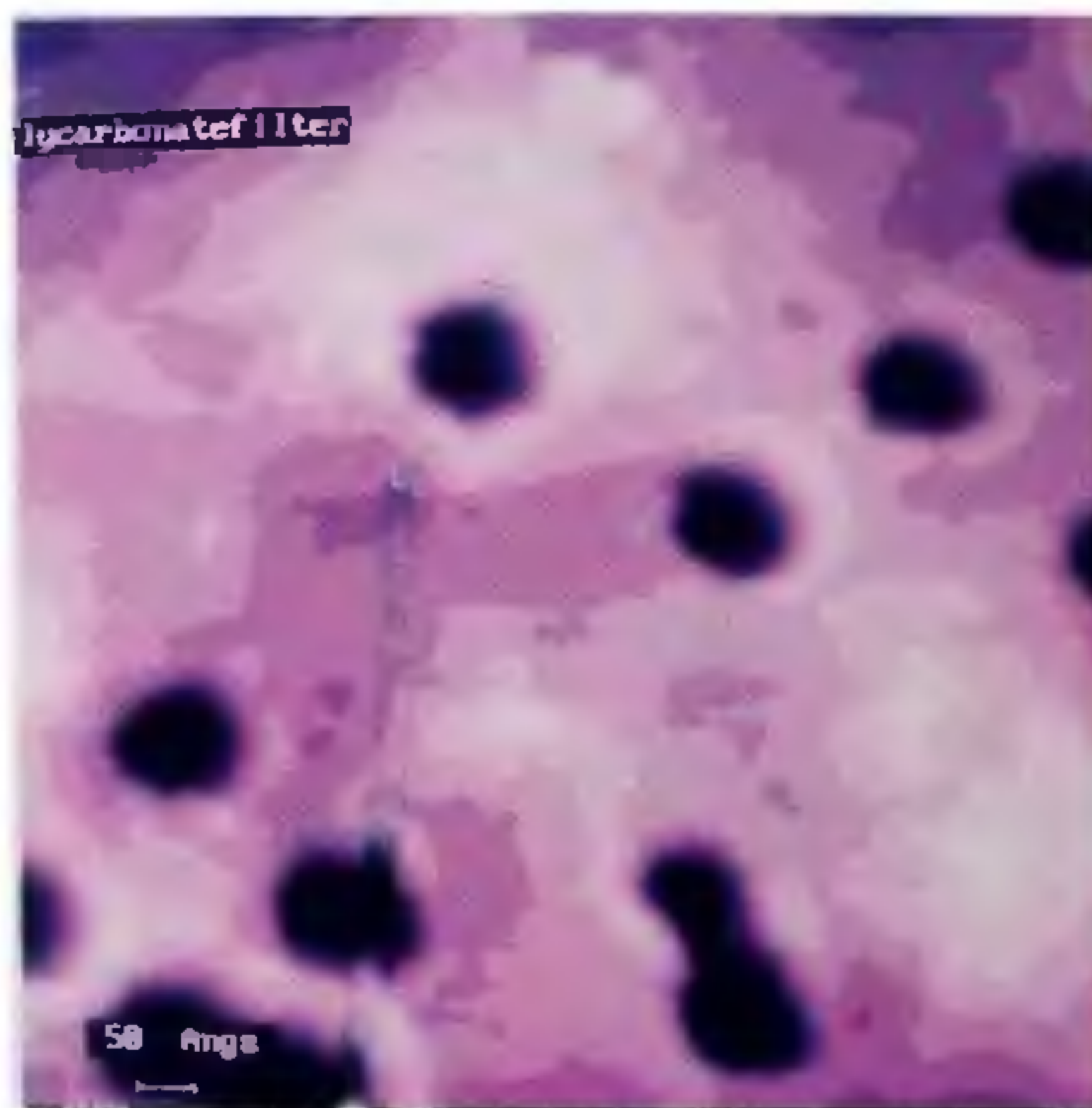

Figure 6. Holographic grating.
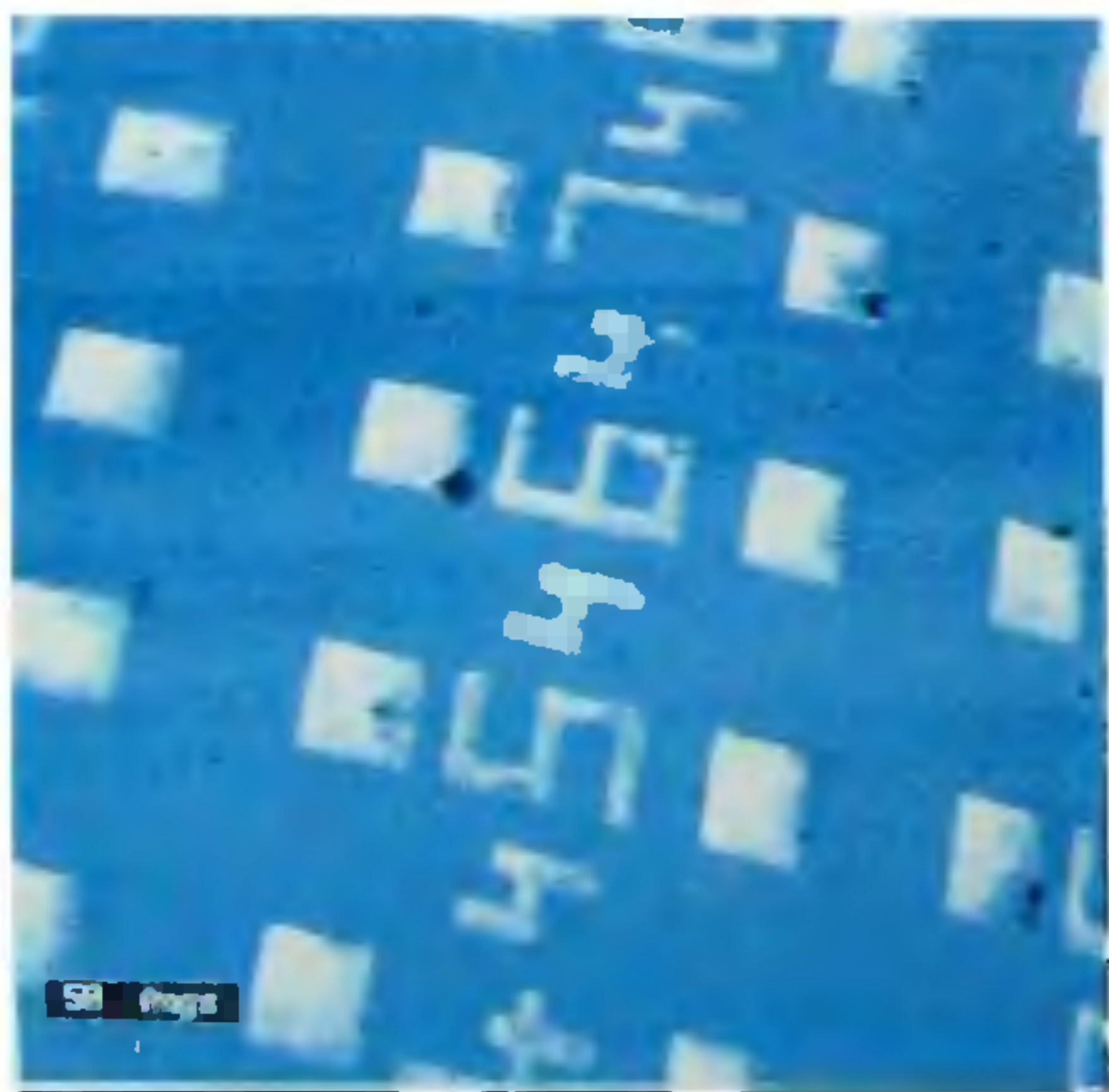

Figure 8. 200 nm polycarbonate filter.
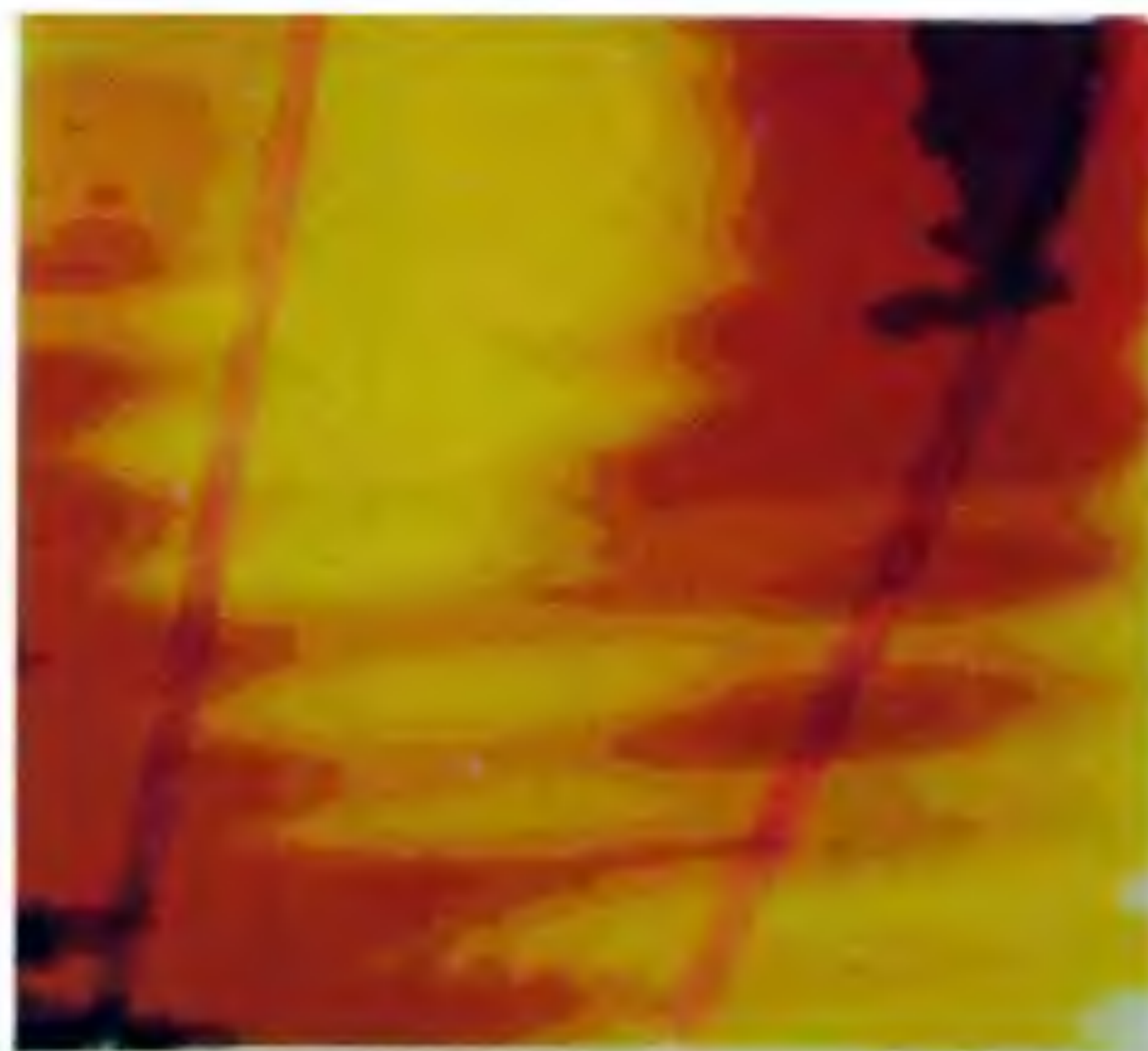

Figure 7. Micro-machined surface.

Figure 9. Surfactant on polymer macromolecules.

with scan size 3.3 µm × 3.3 µm (256 × 256 pixels resolution). The image cleary identifies 200 nm diameter perforations in the filter. Similarly, Figure 9 is a 200 nm × 200 nm (128 × 128 pixels) scan of a polymer macromolecule with Trintoni X as surfactant.

1. Binnig, G., Quate, C. F. and Gerber, Ch., *Phys. Rev. Lett.*, 1986, **56**, 930.
2. Ferrell, T. L., Goundonnet, J. P., Reddick, R. C., Sharp, S. L. and Warmack, R. J., *J. Vac. Sci. Technol.*, 1991, **B9**, 525–530.
3. Hansma, P. K., Drake, B., Marti, O., Gould, S. A. and Prater, C. B., *Science*, 1989, **243**, 641–643.
4. Williams, C. C. and Wickermasinghe, H. K., *Appl. Phys. Lett.*, 1986, **49**, 1587–1589.
5. Martin, Y. and Wickeramasinghe, H. K., *Appl. Phys. Lett.*, 1987, **50**, 1455–1458.
6. Sarid, D., Iams, D., Weissenberger, V. and Bell, L. S., *Opt. Lett.*, 1980, **13**, 1057.
7. Martin, Y., Williams, C. C., Wickramasinghe, H. K., *J. Appl. Phys.*, 1987, **61**, 4723.
8. Meyer, G. and Amer, N. M., *Appl. Phys. Lett.*, 1986, **53**, 2400.
9. Roher, H., *Jpn. J. Appl. Phys.*, 1993, **32**, 1335.
10. Hansma, P. K., *Phys. Today*, October, 1990.
11. McClelland, G. M., Erlandsson, R. and Chiang, S., in *Review of Progress in Quantitative Nondestructive Evaluation* (eds Thompson, D. O. and Chimenti, E. E.), Plenum, New York, 1987, vol. 6B, p. 307.
12. Meyer, G. and Amer, N. M., *Appl. Phys. Lett.*, 1988, **53**, 1045.
13. Alexander, S., Hellenmans, L., Marti, O., Schneir, J., Elings, V., Hansma, P. K., Longmire, M. and Gurley, J., *J. Appl. Phys.*, 1989, **65**, 164.
14. Rugar, D., Mamin, H. J., Erlandsson, R., Stern, J. E., Terris, B. D., *Rev. Sci. Instrum.*, 1988, **59**, 2337.
15. Sarid, D., Iams, D. and Weissenberger, V., *Optics Lett.*, 1989, **13**, 164.
16. Schonenberger, C. and Alvardo, S. F., *Rev. Sci. Instrum.*, 1989, **60**, 3131.
17. Burnham, N. A. and Colton, R. J., in *Scanning Tunneling Microscopy and Spectroscopy: Technique and Applications* (ed. Bonell, D. A.), VCH, New York, 1993.
18. Burnham, N. A., Colton, R. J. and Pollock, H. M., *Nanotechnology*, 1993, **4**, 64.
19. Altman, E. I., Dilella, D. P., Ibe, J., Lee, K. and Colton, R. J., *Rev. Sci. Instrum.*, May 1993, **64**, 000.
20. Drake, B., Prater, C. B., Weisenhorn, A. L., Gould, S. A. C., Albrecht, T. R., Quate, C. F., Cannell, D. S., Hansma, H. G. and Hansma, P. K., *Science*, 1989, **243**, 1586.
21. Marti, O., Drake, B. and Hansma, P. K., *Appl. Phys. Lett.*, 1987, **51**, 484.

Groundwater quality: Focus on fluoride and fluorosis in Rajasthan

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The problem of high fluoride concentration in groundwater resources has now become one of the most important health-related geo-environmental issues in India. Rajasthan is one state where high fluoride groundwater is distributed in all the 31 districts and is influenced by the regional and local geological setting and hydrological conditions for the fluoride contamination. Studies have shown that nearly three million people are consuming excess fluoride-containing water; as such, the problem of both dental and skeletal fluorosis is widespread, specially in the rural population and in children.

FLUORINE is the most electronegative of all chemical elements and is therefore, never encountered in nature in the elemental form. It is seventeenth in the order of frequency of occurrence of the elements, and represents about 0.06 to 0.09% of the earth's crust[1]. Fluoride is an essential ion for all living beings from the health point of view. It helps in the normal mineralization of bones and formation of dental enamel. Fluoride when consumed in inadequate quantities (less than 0.5 mg/l) causes health problems like dental caries, lack of formation of dental enamel and deficiency of mineralization

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