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Design and development of the Scanning Force Microscope for imaging and force measurement with sub-nanonewton resolution

The Scanning Force Microscope (SFM)¹, which started as a derivative of the Scanning Tunnelling Microscope (STM)², has emerged as one of the most powerful tools in the field of surface sciences, study of nano-mechanical properties and nanolithography. The operating principle of the SFM is based on measurement of forces of the order of 10^{-13} to 10^{-4} N between the sharp tip and the surface of the sample. The probing tip is attached to a cantilever and the force acting on the tip causes a small deflection of the cantilever. This deflection is detected and mapped as the tip scans the surface to obtain the image of the surface. Though SFMs are available commercially, it is difficult to improvise on them for experiments other than their basic function, i.e. imaging. In this communication we present the design and fabrication of a SFM with optical deflection detection system and a piezoelectric inertial drive mechanism for coarse approach. This design is easy to machine, assemble and can be modified for different physico-chemical experiments using SFM.

The major components of a force microscope are: a sharp tip mounted on a soft cantilever, the cantilever deflection detection system, feedback to monitor and control the deflection, mechanical

scanning system (piezoelectric tube to raster scan the sample with respect to the tip), tip-sample approach mechanism, and an image display and measurement system via interface. Out of the various deflection detection options available, ranging from tunnelling detection method¹, piezoresistive detection³, optical deflection detection (ODD)⁴ to interferometric detection⁵ using optical fibre⁶, we have implemented the ODD method using a laser diode⁷. This method is used in most of the commercial SFMs, since it offers very high force resolution ($\sim 10^{-12}$ N), compactness and operational simplicity. The choice of ODD system for cantilever deflection detection makes the optical stage the most crucial part of the SFM design and fabrication. The optical stage consists of a laser diode (L; $I = 670$ nm, power = 3 mW, RS components), position-sensitive detector (quadrant photo detector (QPD); RS components, UK), reflecting cantilever ($k \sim 0.2$ – 20 N/m, Olympus Optical Co, Japan) and collimating lenses. The position-sensitive detector is a quadrant photodiode, which gives a signal proportional to the position of the spot of light. For maximum sensitivity, the laser spot on the QPD should move as much as possible for a given cantilever deflection. To maximize the spot movement, we

have used the concept of the optical lever. This has been achieved by adding a reflecting mirror (M; polished silicon wafer) in the optical path from the cantilever to QPD (Figure 1a). Optical lever ratio = (Length of the laser path/Length of the cantilever). For a cantilever of length $100 \mu\text{m}$, we have achieved an optical lever ratio of ~ 500 . Cantilevers have been chosen using two broad guidelines; the resonant frequency of the cantilever should be high (\sim kHz), the minimum requirement for the cantilever resonant frequency is that it should be greater than the data acquisition rate, building and acoustic frequencies of the instrument), the spring constant is chosen depending on contact or non-contact mode.

We have chosen a piezoelectric tube-based inertial drive (ID) mechanism⁸ for the coarse approach. The advantages of using ID are low cost, simplicity in design, controllability via interface with computer and UHV compatibility⁸. The working principle behind the ID is that the driver (D) moves only when the inertial force due to movement of the supporting glass tube (driven by a piezoelectric tube) is larger than the static friction between the driver and the walls of the glass tube (Figure 1a). When a saw-tooth waveform is applied to the

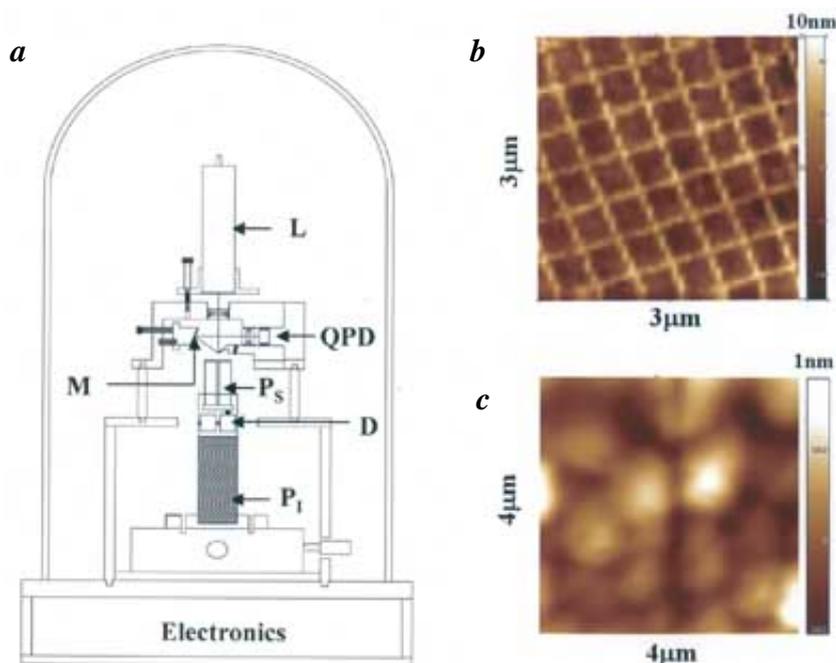


Figure 1. *a*, Schematic of SFM developed in the laboratory; *b*, SFM image of 2D grating replica, and *c*, SFM image of Cu/Si thin film.

piezo tube, the driver follows the glass tube in the slow portion but slides against the glass tube in the fast portion of the wave, resulting in a net upward or downward motion depending on the sign of the waveform⁸. A glass tube glued to the piezo tube P_1 (EBL3, length = 2", $f = 0.75"$, Stavely Sensors, USA) carries the driver. The driver carrying the scanner P_s (EBL3, length = 1", $f = 0.5"$, Stavely Sensors, USA) slides smoothly (~ 1 mm/s) when a sawtooth wave (100 V_{pp}, 1 kHz) is applied. This mechanism gives sub-micron step size. The ID is mounted on an X-Y micrometer stage (Newport Corporation, USA) for coarse-sample positioning. The voltage to the electrodes is generated via a scanning circuit.

In order to eliminate relative motion between the sample, cantilever and detection system we have machined the head of our SFM out of a monolithic non-magnetic block of stainless steel, which also ensures rigidity of the parts. Further, the SFM is placed on vibration isolation system consisting of a mild steel sheet supported on air-inflated tubes (typical resonance frequency ~ 1 Hz) placed on an optical table. The whole system has been designed to give easy access to various parts of the SFM, i.e. the easy changing of cantilever (re-

movable head), laser focusing on the cantilever (spring-loaded laser mount) and focusing of the spot of the laser onto the centre of the QPD (spring-mounted mirror mount) and sample positioning. The main electronic components of the SFM are: low noise preamplifier, differential circuit, scan circuit, feedback and data acquisition system (DAS). The two outputs from the quadrant photodiode have been given to a preamplifier with a gain of 10^7 . The preamplifier outputs are then fed to a differential circuit whose output signal is the measure of the cantilever deflection and is read by the computer. A digital-to-analog converter (DAC) generates the scan ramp, which is given to the scan circuit; and its output is given to the four quadrants of the scan piezo (directly or via high-voltage amplifier; depending on the required scan range). A feedback circuit consisting of a P (proportional) and an I (integrator) is used for controlling the tip and sample surface interaction by keeping the force between the two constant. The DAS consists of a computer, 16-bit ADC/DAC card and software developed in the laboratory.

Calibration of the optical stage for maximum detection sensitivity is very important for the operation of the SFM. A mirror was displaced in the z -direction

by known distance and the differential output of the photodiode was monitored. As the spot moved from one segment of the QPD to the other, the sign of the differential output changed. Near the null position, the large gradient of differential output vs spot-position curve caused the differential voltage to change significantly, even for a small imbalance. So for taking data the laser spot was adjusted to the null position with the help of mirror adjustment screws, so as to get maximum detection sensitivity. Figure 2 (inset) shows the response of the optical stage of the SFM when a mirror was given a displacement in the z -direction. This gives the calibration of the optical stage without the optical lever factor.

Force-distance (f - d) curve is a map of the height dependence of the tip-surface interaction (Figure 2). The measurement starts with the sample far away and the cantilever in its rest position (region a). As the sample is moved in the direction of the cantilever tip, the cantilever bends towards the sample due to attractive surface forces. The maximum forward deflection of the cantilever multiplied by the effective spring constant k of the cantilever is the maximum attractive or pull-on force (point b). Continuing its forward motion the sample pushes the cantilever back through its original rest position until a predetermined load is applied to the sample by the cantilever. The slope of the curve in this region (region c) will be a function of the nanomechanical properties and local geometry of the tip and sample, and will only approach one for perfectly rigid systems. At point d, the sample direction is reversed such that the sample moves away from the cantilever, i.e. the load is removed. The cantilever also changes direction, passing through the position of the maximum adhesive or pull-off force (point e), until the cantilever returns to its rest position when the sample is once again far away (region a). We can choose to image the sample at any point along this force curve. The significant factor is whether the cantilever tip and sample are touching (contact mode). The f - d curves on various samples were obtained. Figure 2 shows a typical f - d curve, obtained using a rectangular cantilever with $k = 20$ N/m on 100 nm gold thin film deposited on Si (100) by evaporation.

The peak-to-peak voltage noise in our detection electronics is ~ 3 μ V. To determine noise-limited force resolution we

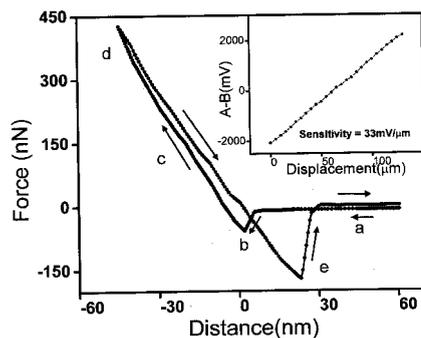


Figure 2. Force–distance curve obtained on gold thin film showing various regions of interaction: *a*, Non-contact region; *b*, Maximum attractive force; *c*, Contact region; *d*, Maximum load, and *e*, Maximum adhesive force. (Inset) Calibration of optical stage without optical lever.

bring the cantilever in contact with the sample and measure the standard deviation of the fluctuation in voltage around this fixed point. For our case the rms fluctuation about the set value is $\sim 10 \mu\text{V}$. The calibration of the optical stage without the optical leverage factor gives a sensitivity of $33 \mu\text{V}/\text{nm}$. Taking into consideration the optical lever factor

of 500 (for the present case, cantilever length = $100 \mu\text{m}$), we get the sensitivity $\sim 17 \text{mV}/\text{nm}$. This gives a displacement resolution of $\sim 0.5 \text{pm}$. This for our case (cantilever with force constant = $20 \text{N}/\text{m}$) gives the noise limited force resolution of $\sim 10 \text{pN}$.

For the calibration of the x – y scan piezo, we have imaged a 2D commercial calibration-grating replica (Pelco International, USA) having $2.16 \text{lines}/\mu\text{m}$ in contact mode (Figure 1*b*). We have obtained a calibration of $40 \text{nm}/\text{V}$ and scan range up to $5 \mu\text{m} \times 5 \mu\text{m}$. The SFM has been used to characterize copper thin films grown using atomic layer chemical vapour deposition technique on Si (100) (Figure 1*c*). The typical scan time was 50 s.

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A halophyte in the Himalaya

The plant *Acanthus ilicifolius* L. (family Acanthaceae) commonly known as sea holly (Figure 1) is found along the sea coasts of India, Sri Lanka, Philippines, Malaysia, Australia and South Africa¹. In India, it is found along the sea coast spanning several states from West Bengal to Orissa, Andhra Pradesh, Tamil Nadu, Kerala, Karnataka and Maharashtra. It is known as ‘Hergoza’ in West



Figure 1. *Acanthus ilicifolius*.

Bengal, ‘Kilichiri’ in Orissa, ‘Etichilla in Andhra Pradesh, ‘Atumulli’ in Tamil Nadu, ‘Payinachulli’ in Kerala, ‘Holechulli’ in Karnataka and ‘Marandi’ in Maharashtra.

So far this plant is known to be an obligate halophyte; however, two bushes of the plant are strangely found to occur in Darjeeling, in the Government College compound just behind the Department of Botany. How this halophytic plant came into existence here and is managing to survive in a temperate climate at an altitude of 2034 m asl, is an open question. The soil conditions in the vicinity of the present habitat of the plant are far from halophytic, with the soil type being acidic in nature (pH 5.8–6.0). The soil is sandy in texture and contains 21% gravel, 33% coarse sand, 40% fine sand and 6% silt and clay. It contains 26% moisture and is abundant in nitrates and chlorides, deficient in sulphates and car-

bonates, while containing traces of phosphate salts². *A. ilicifolius* not only shows some peculiar botanical characters, but also has some important medicinal uses.

The plant is a short (3–4 ft), bushy shrub having a much dissected leaf with sharp spinous teeth on the margins at the endings of the nerves and midrib. The flowers occur in terminal strobilate spikes (10–20 cm long). Sepals are four in number, spiny, and occur as an outer and an inner pair. Corolla forms a short, two-lipped tube, the upper lip of which is obsolete and the lower lip elongated forming a protective covering to the essential whorls of the flower. Anthers four (plus one vestigial) didynamous with one-celled, bearded (hairy) anthers. The base of the filaments forms a hairy cup for trapping the pollen grains for insect pollination. Capsules are compressed with four compressed seeds having a lax, white testa³. Though a shrub, the stems