

# SURFACE SCIENCE

Proceedings of the Ninth  
Interdisciplinary Surface Science  
Conference, Southampton  
22-25 April 1991



Edited by  
B E Hayden



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Surface Science

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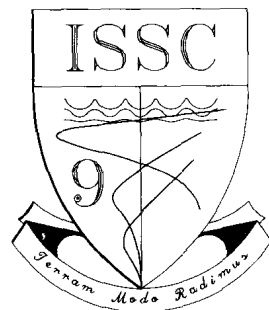
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## Foreword

The *Interdisciplinary Surface Science Conference* held at the University of Southampton from the 22nd to the 24th of April 1991 was the ninth in this series of conferences organized through the Thin Films Group of the Institute of Physics. The main purpose of these biennial meetings is to bring together scientists specializing in all aspects of surface science, and in particular to provide a forum where postgraduate students can present their work and broaden their own scientific perspective. ISSC-9 was enthusiastically attended by about 150 participants from 10 countries, and a third of these were post-graduate students.

The conference organizers decided to schedule five oral sessions each with a major theme highlighted by invited lectures, together with the contributed talks. The subjects chosen were organic molecules on surfaces, dynamics at surfaces, local structure and transitions to long-range order, chemical reactions at semiconductor surfaces and structure at electrochemical interfaces. Besides the eleven invited lectures, thirty four contributions were presented orally. An additional forty six contributions covered a wide area of interests in the field of surface science, and were included as poster presentations at the meeting. The philosophy of concentrating the oral sessions on particular themes was to examine our understanding of particular physical systems, and different aspects (structural, dynamical etc) of these systems, rather than concentrating on any particular experimental surface technique. A result was that specific techniques were nevertheless highlighted as having a significant impact in a wide variety of applications. In particular scanning tunnelling microscopy featured prominently in many of the focussed themes.

These proceedings include over half of the contributed papers presented at the meeting, and broadly reflect the interdisciplinary theme of ISSC-9. The editors would like to thank the authors of the contributions included here, and the referees, for keeping to the rather stringent space and time limitations necessary to facilitate prompt publication.

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## Atomic force microscopy: a tool for surface science

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**Abstract.** The understanding and manipulation of surfaces have been greatly helped by a variety of atomic force microscopes.

### 1. Introduction

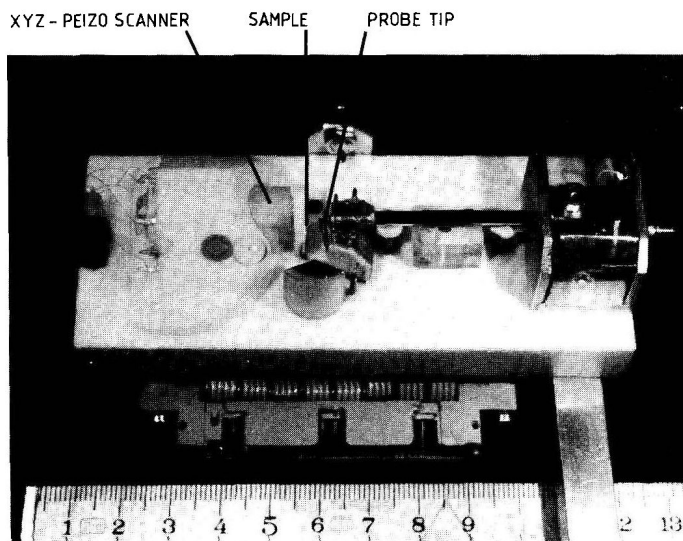
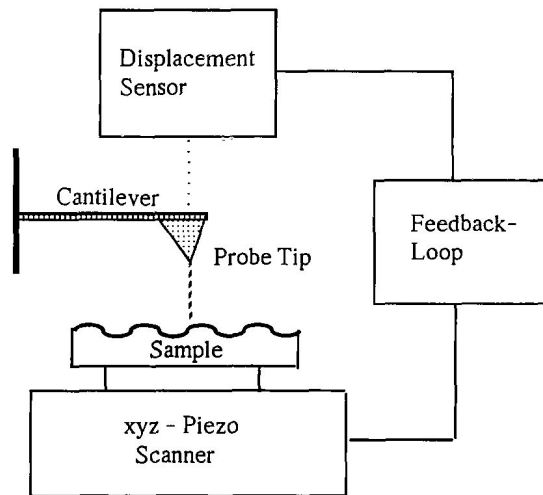
The landscape spreads before you as rolling hills, punctuated by occasional pits and holes, and leads westerly to a precipitous cliff. This scene usually resides in holiday photographs but it is now appearing in the laboratory too in images from atomic force microscopes. The imaged hills are really atomic steps and terraces, the pits are random atomic vacancies, and the cliff is a screw dislocation. What allows us to draw this analogy between the large and small scales of nature? In part, it is the modern techniques of graphics visualisation of data; but increasingly, it is the stunning resolution of atomic and molecular features that this class of microscope can produce. The excitement of studying the physics and chemistry of surfaces with the force microscopes is enhanced by the knowledge that the instruments are still in their infancies.

In contrast to the better-known scanning tunnelling microscope (STM), which detects a current of tunnelling electrons between a scanning tip and a sample, the atomic force microscope (AFM) does not require the surfaces under study to be electrically conductive. Instead, the AFM measures tip–surface interactions due to forces such as van der Waals', electrostatic, frictional, and magnetic forces. Like the STM, the measurements are made on a localised scale, often over areas ångströms in dimension. For studies of larger-scale topographies, the same operating principles are applied with wider-range scanners [1].

Forces of  $10^{-13}$ – $10^{-6}$  N are measured with the AFM. For comparison, typical forces in conventional profilometers are  $10^{-4}$  N over submicron areas. Experimentally, the probing AFM tip is attached to a cantilever spring (figure 1). As the sample is scanned under the tip, the tip/cantilever assembly deflects in response to forces exerted by the sample. A displacement sensor then measures those deflections (from the back of the cantilever), which can be detected down to  $10^{-2}$  Å. The first

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<sup>†</sup> This article originally appeared in the magazine *Physics World* (April 1991, pp46–49).



**Figure 1.** Schematic and operational atomic force microscope (designed by L Howald of Basel)

displacement sensor proposed by the instrument's inventors, Gerd Binnig and Christoph Gerber of IBM Zurich Labs and Calvin Quate of Stanford University, USA, was based on electron tunnelling. Later sensors have been based on optical interferometry, optical beam deflection or capacitance.

There are many types of atomic force microscope. For example, the magnetic force microscope (MFM) incorporates a tip with a magnetic moment so that it can respond to the field of a magnetised sample. In doing so, it has been able to image such features as magnetic bit patterns and Bloch lines [2]. An electrostatic force microscope (EFM) senses isolated charges on surfaces by monitoring the electrostatic interaction between the charged tip and sample. This has been used to map the charge on a polymer surface [3]. In these latter two cases, the detection scheme of the AFM is

modified to detect the longer-range magnetic and electrostatic forces. Instead of measuring single deflections of a cantilever, a vibrating cantilever is used and the change in the resonance frequency of the cantilever is measured as it interacts with the field of the sample. Hybrids of the atomic force microscope have been classified under various names, such as scanning probe microscopes (SPMs or SXMs) – here we refer to them simply as force microscopes.

## 2. Versatility

Currently, the most commonly used force microscope configuration incorporates optical detection methods, due to the ease of alignment with the cantilever, and operates under ambient conditions. More elaborate, customised systems incorporate ultra-high vacuum (UHV), controllable atmospheres, low-temperature capabilities, sophisticated software and electronics, and/or interfaces with other analytical instruments.

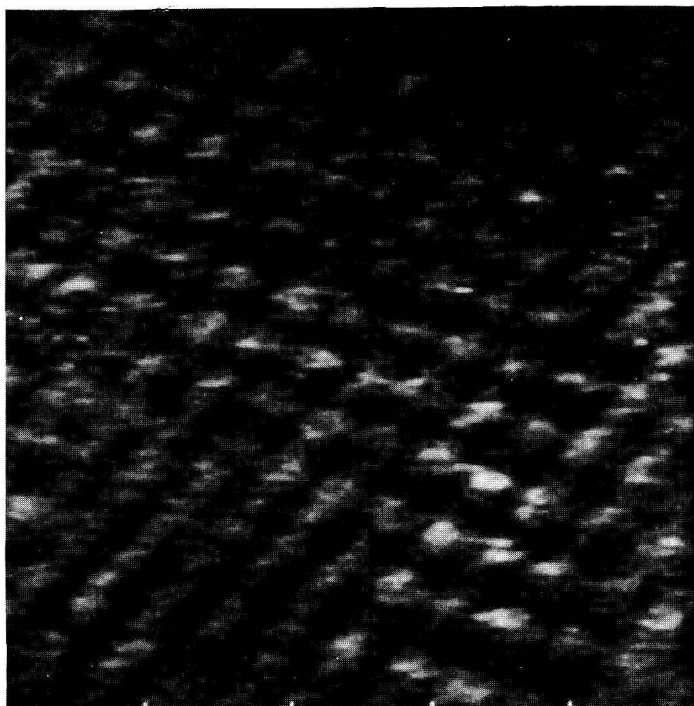
The range of samples seen with the AFM to date is very wide, from blood cells to common salt. Since the instrument can be tailored to sense specific forces [4], it is difficult to rule out a class of materials as unsuitable for the technique – particularly when it is sensitive to even the ubiquitous van der Waals' forces. Force microscopes have been used in a wide variety of environments as well. For example, since the instrument can operate reliably when the tip/sample interface is submersed in liquid, measurements have been made on operating electrodes, hydrated proteins and lubricated discs.

However, we will confine our discussion to several cases in which the force microscope has provided information about surfaces which is not easily obtained from other surface science methods; these examples will demonstrate the value of the microscope in increasing our knowledge of surface properties derived from the atomic scale.

## 3. Organic thin films and crystals

Tailored organic films are being studied with increased frequency, primarily as models for more complex systems. In biology they represent idealised membranes for protein adsorption and selective permeation. In physics, the films provide a molecularly engineered system for 2-D interfacial phenomena, for example nonlinear optics.

Foremost among the tailored films are Langmuir–Blodgett (LB) films, 3-D assemblies of 2-D layers of ordered molecules. These can now be made with high precision and reliability. Low-energy electron diffraction (LEED) [5] and X-ray [6] studies have provided structural information on the lattice structure of the films, confirming the molecules' orientation orthogonal to the substrate plane and the close-packed hexagonal geometries of alkyl tail groups. However, with films of tens of ångströms thickness, difficulties arise in analysis, both from the small amount of material available to give viable signals and from beam damage to the fragile films. Therefore it has been difficult to document the ordering process on those layers close to the interface with the solid support. However, the surface structure of a double bilayer of cadmium arachidate (a cadmium salt of a fatty acid) has been recorded with the AFM at a distance of  $\approx 100$  Å from the amorphous (i.e. unordered) silicon substrate (figure 2) [7].



**Figure 2.** AFM image ( $50 \times 50 \text{ \AA}$ ) of the surface of a Langmuir–Blodgett film comprised of two bilayers of cadmium arachidate on an amorphous silicon substrate. The corrugation height is  $0.3 \text{ \AA}$  [7].

This study revealed that this close to the substrate/adsorbate interface, the molecules are already assembled with a degree of order characteristic of thicker LB films (figure 2). This comes as some surprise since previous LEED [5] studies indicated that substrate order was a prerequisite for adsorbate order close to the interface. Apparently, at this distance from the surface of the amorphous substrate ( $100 \text{ \AA}$ ), the intermolecular driving forces for self-assembly are large enough to overcome the imposition of disorder from interaction with the amorphous substrate. A comparison of this study with other, unsuccessful, attempts to image LB films with an AFM illustrates the importance of close control over the forces applied to the soft films in the imaging process.

In another example of surprising order on organic surfaces, Rene Overney and Lukas Howald of Basel University, Switzerland, have imaged free-standing organic crystals to compare the arrangement of molecules on the surface to that in the bulk [8]. In the case of tetracene (an aromatic compound of four linearly fused benzene rings), the lattice spacings and angles of the surface layer correspond almost exactly with those of the bulk. In this study, the AFM was not only able to record the intermolecular spacings, but also the orientation of molecules relative to each other. However, pyrene, a different four-ring aromatic, shows no correspondence between the surface and bulk structure. In the bulk, pyrene molecules exist in dimer pairs, molecules of a pair separated by  $3.5 \text{ \AA}$ . AFM images of the surface of a pyrene crystal indicate no evidence of dimer pairs; rather, the surface is comprised of regularly spaced monomers at a molecule density similar to that of the bulk crystal. Such a



radical change in molecular spacing is perhaps not surprising when one considers the different dielectric environment experienced by the surface layer. Nonetheless, previous to these AFM studies the unique pyrene surface structure went undetected.

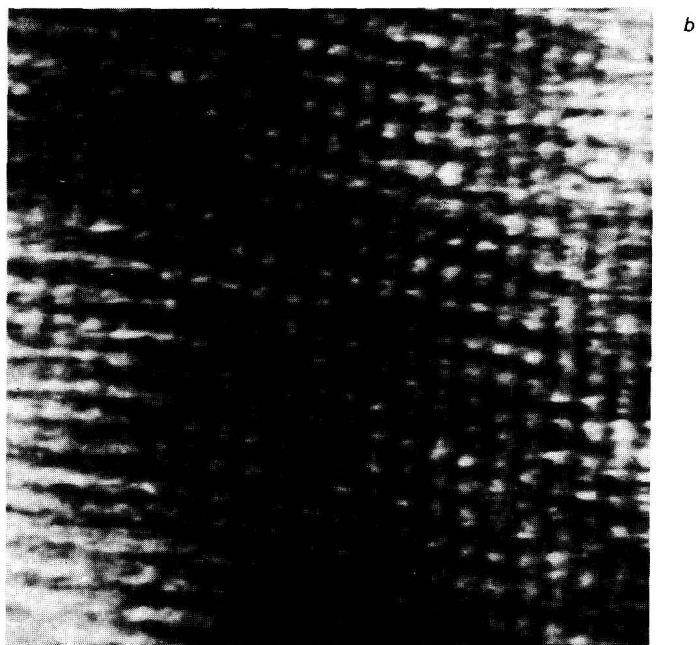
#### 4. Reactive and mobile surfaces

In the last decade the atomic and electronic structure of many semiconductors has been determined and characterised by surface-sensitive techniques. However, techniques such as LEED or RHEED (reflection high-energy electron diffraction) are hindered by charging effects and beam damage. In silver halides, chemical reactivity, in the form of photolysis, further complicates surface analysis. Metal decoration, a technique based on scanning electron microscopy, has previously been used to study silver halide surfaces. However, this method is limited to a resolution of  $\approx 100$  Å whereas AFM can image the atomic and step structure of the AgBr(100) surfaces with a resolution of a few ångströms [9]. In the magnification of figure 3a, monoatomic steps of 2.9 Å which separate terraces and small islands, are observed. The emergence point of a screw dislocation can be seen on the uppermost terrace.

Comparing these AFM results with those from metal decoration confirms some results, such as the orientation of the steps, and verifies many assumptions, such as the height of steps (typically monoatomic) and the emergence point of screw dislocations. At higher magnifications (figure 3b), atomic resolution is achieved and the distance between atomic protrusions is measured as 4.1 Å, which corresponds to the spacing between equally charged ions of 4.09 Å, known from the bulk. Comparison of the ionic radii of  $\text{Ag}^+$  (1.15 Å) and  $\text{Br}^-$  (1.96 Å) suggests that the atomic-scale AFM images are dominated by the bromide ions. Theoretical



(Figure 3b, c and caption overleaf)



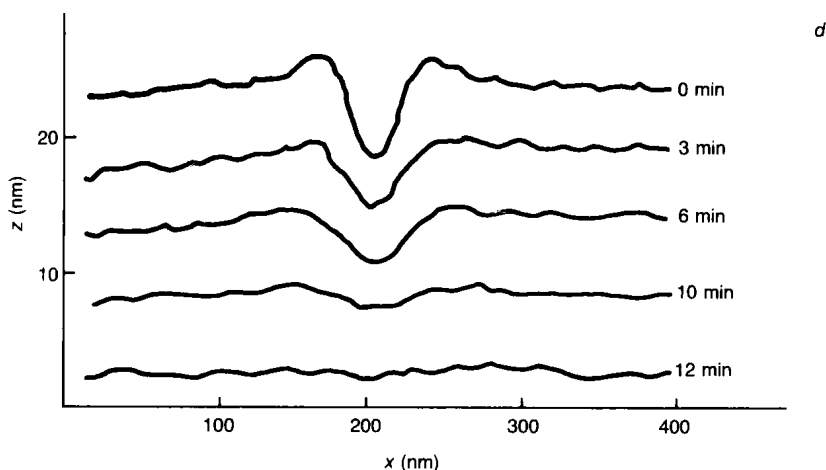
b



c

(Figure 3a on previous page, 3d on following page)

**Figure 3.** (a) ( $5040 \times 5040 \text{ \AA}$ ) AFM image of a AgBr single crystal containing monoatomic ( $2.9 \text{ \AA}$ ) steps and a screw dislocation (centre); (b) high-magnification AFM scan ( $100 \times 100 \text{ \AA}$ ) of AgBr showing crystal lattice, with equally charged ions spaced  $4.1 \text{ \AA}$  apart. The corrugation height is  $1 \text{ \AA}$ . The image is dominated by the larger bromide ions (see text); (c) hole (diameter  $\approx 40 \text{ nm}$ ) made in the AgBr (100) crystal surface by the AFM tip, surrounded by displaced material; (d) line traces track the filling-in process over the course of 12 minutes



calculations [10] by Roger Baetzhold and colleagues at the Kodak labs in Rochester, USA, predict a relaxation of the AgBr(100) surface in which the bromide ions are displaced outwards and the silver ions are displaced inwards. These predictions are in agreement with the corrugation height of 1 Å measured by AFM.

The AFM study of silver bromide provides an example of another feature of the AFM which sets it apart from other surface science tools: localised surface manipulation. Besides imaging surface structure, the AFM can be used as an active device. For example, by increasing the tip's applied force, nanometre-sized indentations are created (figure 3c). The resulting marked surface is then imaged with a smaller, non-invasive force. In the case of the AgBr(100) surface, these 100–1000 Å-wide holes fill in after a few minutes, a process attributed to the rapid surface diffusion of silver and bromide ions. In fact, this property of high  $\text{Ag}^+$  and  $\text{Br}^-$  mobility is essential for the rapid formation of the latent image in the photographic process.

The observation of diffusion demonstrates that the force microscope is capable of imaging not only static structure, but also certain dynamics of surfaces. Gary McClelland and colleagues at IBM Almaden Labs, USA, have dynamically recorded the lateral force acting on the probing tip while scanning a graphite surface [11]. The observed patterns are correlated to the atomic lattice and therefore constitute an example of “atomic-scale friction”. These investigations will undoubtedly lead to a greater presence of the AFM in tribology.

## 5. Liquid or not?

The liquid-like behaviour of organic adsorbates has been investigated widely. In one experiment Greg Blackman and colleagues at IBM Almaden compared the responses of an AFM probe approaching silicon surfaces coated with a physisorbed (by, for example, van der Waals' bonds) fluorocarbon layer (40 Å thick), a chemisorbed (i.e. covalent bonds) fluorocarbon layer (20 Å), and self-assembled fatty acid mono- and multilayers (28–140 Å) [12]. The movement of the tip through the adsorbate layer