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Scanning tunneling microscopy at the University of Geneva

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(1. II. 1988)

In honor of Martin Peter's 60th birthday.

Introduction

The Scanning Tunneling Microscope (STM), first developed by G. Binnig, H. Rohrer and their collaborators [1] possesses simultaneously very high horizontal and vertical resolution and is able to give three-dimensional and atomically resolved images of surfaces in real space. Imaging can also be performed in very different conditions, at room and low temperature, in ambient atmosphere, in liquids or under ultra high vacuum, and has inspired world wide research activity. There are now abundant examples of surface structure STM images on various materials: metals, semiconductors, superconductors, layered materials, adsorbed materials and biological materials, for some review articles see references [2–5].

The activity in the domain of biomedical applications of Scanning Tunneling Microscopy (STM) has started at the GAP of the University of Geneva in October 1986 under the impulse of Martin Peter. The first idea was to develop a STM to study the interface metallic implant-bone and the phenomenon of biocompatibility which is far from being understood. It was soon realized that it was also necessary to have a good knowledge of the metallic surface.

At the same time the surface physics group at DPMC was planning to extend the field emission studies to include a STM, and the two groups joined their efforts to develop a STM, working under UHV and optimized for the localisation of specific sites on a large scan area with the possibility of zooming to atomic resolution.

After a short introduction to scanning tunneling microscopy we will present these two projects now under development at the University of Geneva.



Figure 1

Schematic of a scanning tunneling microscope (a) and its operation (b) [from Ref. 2.].

Scanning tunneling microscopy

Scanning Tunneling Microscopy, as the name suggests, is based on tunneling of electrons through an insulator, a physical effect known for a long time. However the recent successful development of the STM began with the pioneer work of G. Binnig, H. Rohrer, and their collaborators at IBM Zurich Laboratories, who first succeeded in achieving atomic resolution using a sharp metallic tip as one of the electrodes [1].

(b)

Figure 1 shows a schematic view of one of their designs and the constant current mode operation [2]. If the concept of the STM is quite simple, the practical realization of atomic resolution encounters delicate problems considering that tip and sample have to the kept within a distance of a few angstroms with a stability of a tenth of an angstrom. One of the main problems is the suppression of external vibrations together with those created in the scanning and control process. The general trend now is towards compactness, rigidity and simplicity of the microscope rather than sophisticated vibration isolation systems [5].

The operation of the STM and the interpretation of images as a contour of constant height above the surface are based on the strong distance dependence of the tunnel current I, which is approximately given by [1]:

 $I \alpha f(V) \cdot \exp(-A\phi^{1/2}s)$

where V is the applied voltage, ϕ the average of the two electrodes work functions in eV, s the distance between the electrodes in angstrom and A a constant. For a work function of a few eV, the intensity of the tunnel current I changes by an order of magnitude when the distance s changes by only one angstrom. However a correct interpretation of STM images requires an exact analysis of the tunnel current which involves a complicated convolution of the electronic spectra of surface and tip [6, 7].

STM for surface studies under UHV

We have developed a first STM to be incorporated into the UHV 'Escalab' system which had been earlier modified to include a Scanning Field Emission Microscope [8]. This apparatus gives us the possibility to locate enhanced field emission sites on broad area cathodes of centimetre size and to perform local analysis, including emission I–V characteristics, SEM pictures and Auger spectroscopy. The spatial resolution for site localisation which was limited before to a few micrometers can be improved by several orders of magnitude by the addition of the STM [9]. The vibration isolation has still to be improved to reach atomic resolution in the UHV system.

Figure 2 shows schematically the design of our STM, which is derived from the one built by Besocke [10]. The scanner tube gives the possibility of large area



Figure 2 Schematic design of our STM for UHV system.

scans, with $50 \mu m X$ and Y and $5 \mu m Z$ amplitude. The STM can also move horizontally on its platform support using three feet by steps of about 1000 angstroms to be localised on specific sites. The approach to the tunnel regime is performed using a specifically developed piezo translation device, based on stick-slip movement [11] which allows to raise or lower the STM head by steps of the order of 100 angstroms.

In order to change the tip without breaking vacuum, the scanner assembly can be removed and installed using the standard sample transport system of our apparatus.

Figure 3 shows an STM image of a crossed grid in a carbon film together with an SEM Picture. This type of well-characterized sample allows us to calibrate large amplitude STM scans.

Figure 4 shows STM and SEM images of a high- T_c superconducting YBa₂Cu₃O_{7- δ} thin film of nominally 7500 Å thickness as produced by a research group at the University of Geneva [12, 13]. While the two types of pictures clearly display a similar type of morphology, only the STM image can give quantitative information on topology which is important for the superconducting properties of such a film.

STM for biological studies under ambient atmosphere

The second STM now under development has to be coupled with a high resolution optical microscope and to work under ambient atmosphere. As for the previous system, the main idea is to optimize this STM for the localisation of specific biological objects on very flat metallic surfaces in the micrometer range with the capability of zooming to atomic resolution. We will also have the possibility to compare the STM images with SEM images.

For this STM we have the constraint of coming very near the surface of the sample with the objective lens of a high resolution optical microscope. To solve this problem we have started with a new conception in the way to use the scanning piezo-tube of the STM, this one is now parallel to the surface of the sample instead of being perpendicular as usual. Figure 5 shows a schematic design of a first prototype for this STM. The approach to the tunnel regime is done mechanically using a fine screw and the electronics for monitoring the piezo-tube is analog to the one first developed for the UHV-STM. The maximum scan range for this prototype is 25×5.5 micrometers in XY and 15 micrometers in Z, but we can also achieve atomic resolution as seen in Fig. 6 which represents a STM Image of HOPG under ambient atmosphere. The Pt-Ir tip is positionned on the sample using a binocular microscope.

With this STM we have started to look at different metallic thin films deposited under vacuum on sapphire substrates. Figure 7 shows one of the STM images obtained under ambient atmosphere on a Titanium thin film. The surface observed is the natural Titanium oxyde which is formed on top of Ti-thin film when exposed to air.





(a) $5 \times 5 \,\mu\text{m}$ STM and (b) SEM images of a calibration sample for TEM from Balzers 300 mesh Cu with $d = 0.463 \,\mu\text{m}$. Tip voltage $-4 \,\text{V}$, tunnel current 1 nA.





(a) $5 \times 5 \,\mu\text{m}$ STM and (b) SEM images of a YBa₂Cu₃O_{7- δ} thin superconducting film. Tip voltage $-4 \,\text{V}$, tunnel current 1 nA.



Figure 5

Schematic design of the prototype of our STM for biological application.

Work in progress

The main problem which remains to be improved for the UHV-STM is that of vibration isolation to achieve the atomic resolution. The biological-STM is just a first prototype and has to be improved to allow the possibility of moving the scanning piezo-tube over a large sample and also the coupling with a highresolution optical microscope.



Figure 6

Constant current $15 \text{ Å} \times 15 \text{ Å}$ STM image of HOPG obtained with our STM under ambient atmosphere. Tip voltage 0.1 V, tunnel current 10 nA.



Figure 7 $1600 \text{ Å} \times 1560 \text{ Å}$ STM image of a Ti thin film deposited on sapphire substrate. Tip voltage 0.5 V, tunnel current 5 nA.

With the UHV-STM we are now studying thin films of the new high- T_c supraconductors and we want to continue the study of enhanced emission sites on metallic samples. With the biological-STM we have started the study of adsorbed macro molecules on different metallic substrates to approach the problem of biocompatibility of materials for surgical implants.

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