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THE DEVELOPMENT AND APPLICATIONS OF ATOMIC FORCE MICROSCOPE (AFM)

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Atomic-Force Microscope (AFM) developed in recent years is a new instrument for surface analysis. The image in the AFM is generated by laterally scanning the sample under a sharp stylus while simultaneously measuring the separation-dependent ultrasmall interatomic force between the stylus and the surface. This instrument has atomic resolution and allows electrically insulating samples to be imaged directly. In this sense it is complementary to the scanning-tunneling microscope (STM) introduced shortly before the development of AFM. AFM can be used to image surface structures of conductors, semiconductors, as well as insulators, thus showing specific significance in the studies of surface science, materials science, and life sciences. The lateral resolution of the first AFM in the world reported in 1986 was only 30 Å. In 1987, a research group at Stanford University reported that their AFM could reach atomic resolution. Due to technical difficulties, currently only a few laboratories in some developed countries possess such instruments, of which even fewer can reach atomic resolution. We developed the first AFM in China by ourselves, under the support of the Chinese Academy of Sciences, and obtained images with atomic resolution at the end of 1988. It passed the specialists' appraisal in June, 1990 and was considered as "the success in the development of AFM, marked a new progress of our country in this high-tech field."

I. From STM to AFM

In 1982, Dr. Gerd Binnig and Heinrich Rohrer of IBM Company invented the first STM in the world. This new instrument caused much attention in scientific and technological circles. The inventors were awarded the Nobel Prize in Physics in 1986 for their invention.

The principle of STM is based on the quantum tunneling effect. Take an atomically sharp tip and the substance surface being studied as two electrodes. If a small voltage is applied between the sample surface and the tip, electrons will penetrate the insulating layer (or vacuum barrier) between the two electrodes and flow from one electrode to the other one. This phenomenon is called the tunneling effect. The intensity of the resulting separation-dependent tunneling current is very sensitive to the distance between the tip and the sample surface. Keeping the tunneling current constant by an electronic feedback system while the tip is scanning the sample surface, that is, keeping the tunnel gap at a constant value, the moving traces of the tip directly represent the density of states or images of atomic arrangement at sample surfaces.

Since the birth of STM, especially since 1986, the instrument has undergone rapid development, and many important results have been obtained. However, new techniques cannot be fully perfect and free of disadvantages. STM also has its limitations, mainly shown as:

(1) As STM must monitor the tunneling current between the tip and the sample while working, it can only directly observe the surface structure of conductors and semiconductors. For non-conducting materials, their surfaces must be covered with a layer of conducting film. The existence of conducting films often conceals the real structure of surfaces and removes the principal advantage of STM — that it can be used to study surface structures at atomic scale. Many interesting samples are non-conductors.

(2) For STM, information about surface structures is obtained on the basis of the tunneling effect. Strictly speaking, what STM observes is the density of states near the Fermi energy level at the sample surface. For the sample in which different electronic states exist at the surface, what STM obtains is not real surface topography, but a combination of information of surface topography and surface electronic properties.

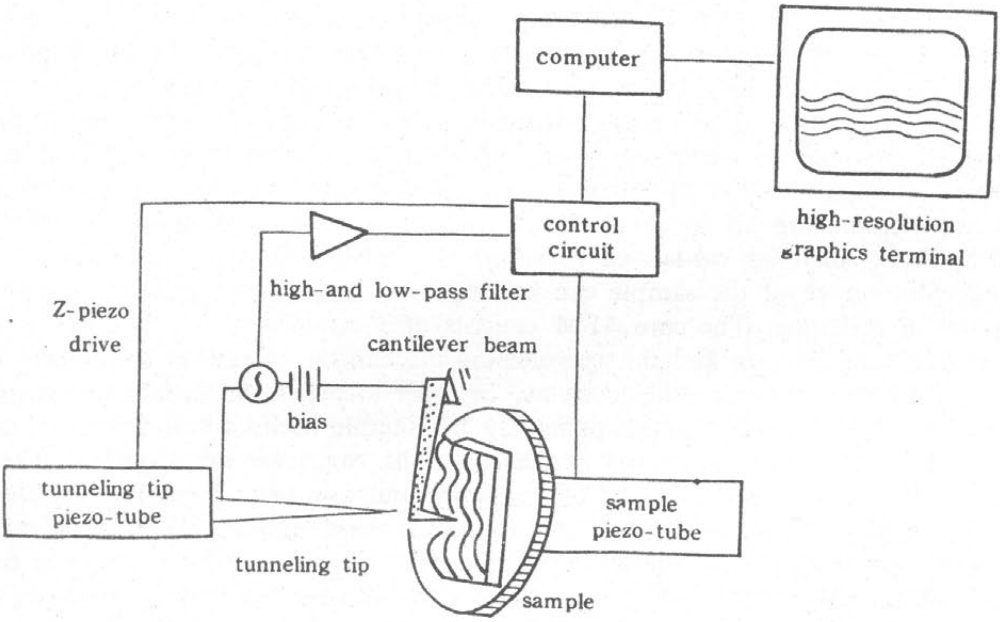

Fig. 1 Schematic diagram of atomic force microscope

In order to make the complementary to STM, Binnig et al. introduced AFM in 1986. The principle of AFM is shown in Fig. 1. The cantilever which is extremely sensitive to weak forces is fixed on one end; the other end has a sharp tip which gently contacts with sample surfaces. When the sample surface is being scanned in a horizontal direction, because of the ultrasmall repulsive force existing between the tip atom and surface atoms of the sample, the cantilever with a sharp tip will move up and down in the direction vertical to sample surfaces, corresponding to the contours of the interaction force between the tip and surface atoms of samples. The structural information of sample surfaces can be obtained by detecting the displacement of the cantilever at each point, using laser beams or other tunneling probes similar to STM. From the principle mentioned above it goes without saying that AFM observes surface topography by monitoring the contours of the interaction force between the tip and surface atoms of samples. Its application, therefore, will not be limited by some properties of samples such as conductivity, periodicity, etc.

II. The Architecture of the Instrument and Key Technical Problems Solved

Although the AFM instrument itself is not an enormous equipment, it is a high-tech product in which precision machinery, electronic components, computer software and hardware are assembled. Any minor fault in designing, processing, assembling and debugging will makes it difficult for the instrument to reach atomic resolution.

Our instrument consists of a computer-control system, image display system, core AFM and vibration-isolation system (Fig. 1). The cantilever, tunneling tip and the sample are mounted in the core AFM. In order to scan sample surfaces precisely and ensure the distance between the cantilever and the tunneling tip to fulfill the condition (<1 nm) by which the tunneling current can occur, the requirements cannot be satisfied by using only mechanism adjustments. More precise approaching and scanning devices are needed. Because piezo-electrical ceramic materials undergo a linear dimensional change in a certain range when an electric field is applied, we used two ceramic tubes in our AFM: one of them was used as a sample scanner controlled by a computer, and the other was used to control the position of tunneling tip by an electronic feedback system. Analog signals are digited by A/D converter cards and put into a computer for data processing. The topographic image of the sample can be displayed on a high resolution graphics terminal in real time. The core AFM consists of a cantilever, a three-dimensional scanner, a tunneling tip and the approaching mechanism. A crucial component for AFM is a force-sensing cantilever stylus. In order to reflect the surface topography of the sample accurately without damaging the sample itself, a low force constant and a high mechanical resonance frequency of the cantilever are required. Therefore the cantilever needed must be of small size and low mass. Expensive cantilever materials and complicated fabrication process are used abroad. We use a cheap tungsten chip fabricated by electro-chemical etching as the cantilever. Its force constant and resonance frequency are 2.3 N/m and 6.8 kHz respectively, with the dimensions of $200\,\mu\text{m} \times 15\,\mu\text{m} \times 3\,\mu\text{m}$. The cantilever fabricated in this way appears a realistic force-sensor. The fabricating process is rather simple and not time-consuming (only several minutes needed), moreover, this cantilever is not expensive and exhibits relative high stability.

Three techniques for measuring the deflection of the cantilever during scanning include: tunneling to the back of the cantilever (employed to measure the deflection amplitude of the cantilever only, with no relation to the sample), optical-fiber interference technique and electric capacity measurement method. The first technique can obtain images with best resolution, and is also what we are using. The measurement of nano-ampere-level weak signals demands decreasing of the system noise as low as possible and shielding the external electric noise and mechanical vibration. All these technical problems were solved in our instrument.

The control circuit of AFM consists of preamplifier, electric feedback, scanning

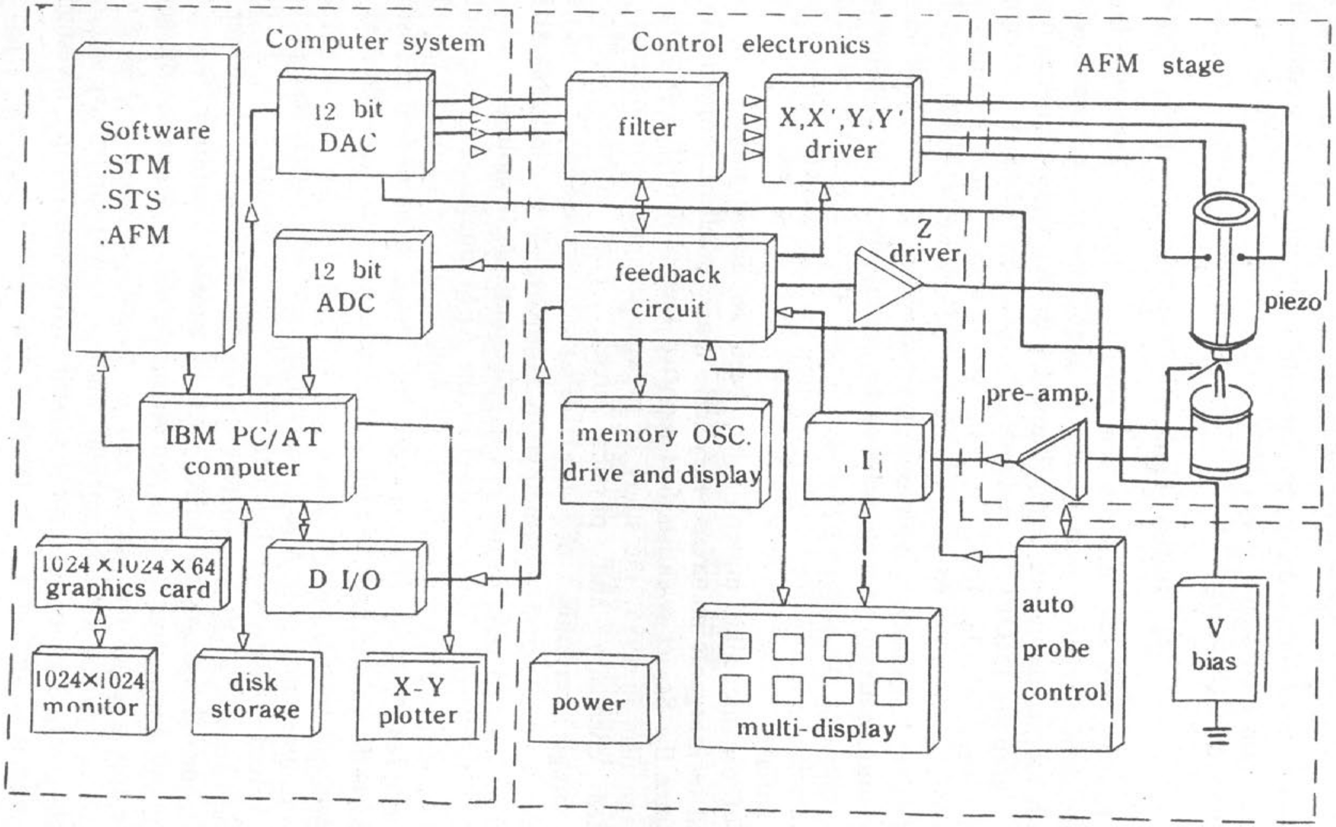

Fig.2 The control system and computer system of AFM

control unit, signal gain and filtering, and multi-display. The overall schematic diagram is shown in Fig. 2.

The hardware of the computer-control system includes the IBM PC/AT computer, high-speed and high-precision A/D and D/A converter cards, a high-resolution graphics card and a graphics terminal.

The software package has two major functions: (1) AFM scanning control, data acquisition and storage, real-time grey-scale display. (2) Analysis and processing of images, including Fourier transformation, image enhancement, cross section analysis, calibration of image distortion, and three-dimensional representation, etc. The AFM-program-user interface has been given careful consideration to ensure that all features of the program are accessible from color menus.

III. Initial Applications

Since AFM has emerged only a short time ago, according to published literatures, applications of AFM are few, especially for high-resolution experimental results. Images with atomic resolution were obtained only for three samples: highly oriented pyrolytic graphite (HOPG), molybdenum disulfide (MoS_2), and highly oriented pyrolytic boron nitride (HOBN). The lateral and vertical resolutions of our AFM reached 1.3 Å and 0.05 Å respectively, by testing with two standard samples of HOPG and ordinary graphite monochromator. High quality images with atomic resolution can be obtained easily and repeatedly by this instrument. We have also investigated the single-crystal sample of TiO_2 which is almost an electric-insulating material, and obtained high-resolution images showing atomic steps on TiO_2 (100) surface.

The study of organic solid materials has been an active subject in recent years. Compared to inorganic materials, they have many excellent properties in various aspects. Since the Soviet scientists first reported their Poly-BIPO organic solid with ferro-magnetism in 1986, the study of organic ferromagnetic materials has greatly interested chemistry and physics circles. The surface studies of nonconductive single crystals of the organic ferromagnetic material, 2-(4-nitrophenyl) 4,4,5,5-tetramethyl-4,5-dihydro-1 H-imidazolyl-1-Oxy 3-Oxide, have been carried out by our AFM. The individual molecules with the dimensions of 4×10 Å^2 are clearly resolved in the AFM images, and the molecular arrangement is in good agreement with the bulk crystal structure obtained by X-ray diffraction method. The AFM images with high resolution demonstrated our instruments' performance and laid down a basis for the extension of our AFM applications afterwards.

As AFM was developed only a few years ago, and some technical problems still need to be solved, scientists in this field have worked primarily on the development and improvement of the instrument during the past years. However, because of the unique advantages of AFM such as atomic resolution, no matter what periodicity and conductivity of the sample is studied, and because the surface structure on the top layer of the sample can be directly observed, AFM will be useful as research equipment for fundamental studies. It will surely have significant effects on revealing subtle structural details resulting from microscopic investigation in some fields, and it is finding a wide range of applications.

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