Building a Scanning Probe Microscope Using Open Source Control Software and Systems

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A scanning probe microscope (SPM) operating in air and liquid was constructed using an open-source control software running on Linux, a correspondingly developed commercial SPM controller and a commercial STM head. The results are shown for highly oriented pyrolytic graphite (HOPG) and Au(111) substrate surfaces, which are typical samples for SPM measurements. The cost of building the system was much lower than that of commercial products with comparable performance. The system has great scalability, and can be applied to a various environment, such as ultra-high vacuum systems.

Keywords SPM, STM, FM-AFM, tuning fork, open-source control software, GXSM

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Introduction

Scanning probe microscopy (SPM) has become a common technique in physics, chemistry, biology, materials science, and other fields that deal with surfaces. SPM research began with the invention of the scanning tunneling microscope (STM), which can observe the surface of a clean, atomically smooth, conductive substrate with atomic resolution. It has evolved into atomic force microscopy (AFM), which can observe the surface of non-conductive substrates. The development of AFM from the contact mode to the intermittent contact mode (so-called tapping mode) enabled observation of non-destructive surface topography of soft materials such as organic molecules, polymers and biological materials. Furthermore, with the advent of non-contact AFM (nc-AFM) using frequency modulation (FM) technique, true atomic resolution observations using AFM, which many researchers have been waiting for, have been achieved. The fact that the operating environment of high-sensitivity FM-AFM has expanded from a vacuum to a solution and air is the following reasons; The laser optics, which is the core of the detection part in FM-AFM using cantilevers manufactured by silicon microfabrication, has been significantly reduced in noise; or qPlus sensors using quartz crystal tuning forks with one prong immobilized on the substrate, which does not require a laser system, have been applied to the detection part instead of silicon cantilevers. In the early stage of SPM development, STM measurements were carried out by each laboratory using self-made devices. As the commercial products became more popular, the population of researchers increased and spread to a wide range of research fields. As the commercial products matured, as in the case of other analytical instruments, the price of high-resolution systems increased, and their internal mechanism including the control software became a black box with few parts that could be modified by the researchers. Initially, most of the AFM in ultra-high vacuum (UHV) systems had interferometers or optical detection mechanisms using split diodes and lasers, however, it was reported that atomic resolution observation of Si(111)-(7×7) surfaces became possible using a qPlus sensor in UHV. In research using FM-AFM with qPlus sensors in UHV, the resolution has been surprisingly improved to the extent that even the carbon skeleton of aromatic hydrocarbon compounds and the framework of water molecule clusters adsorbed on a single-crystal metal substrate can be visualized.

As SPM control devices and software become increasingly complete in commercial SPM systems, they have been integrated and closed within the product. This is unavoidable in order to provide complex functions to many researchers in an easy-to-use manner; however, it also prevents the compatibility of devices and the updating and functional improvement of the devices and software as research progresses. On the other hand, attempts have been made to develop SPM image-processing and control software whose source codes are open source. The GXSM (gnome X scanning microscopy) project has been in progress since 1997 by Zahl et al. GXSM is open-source SPM control software based on GNOME/GTK+ library as the user interface running on Linux OS of personal computers. In 2003, a digital signal processor (DSP)-based SPM controller was developed and released, which can be controlled by GXSM. The basic structure, functionality, and scalability of GXSM have already been reported. As of 2021, the project continues to be actively developed by Zahl and the GXSM community. Besides, all of the past discussions on the forum, from the installation to usage of the GXSM software and how to solve problems in use, can be viewed on the website. If we still have questions, we can also ask questions on the forum to receive feedback from the community. Also, a Live DVD that does not require knowledge of Linux installation has been compiled by community volunteers.

Even if we could obtain a budget to purchase an SPM and to introduce a commercial SPM system, it seems that for the average research groups, SPMs are often only used as routine instruments. This is unfortunate because SPMs, like other analytical instruments, are not inexpensive instruments. However, if we could fully understand the inner mechanisms...
and build an SPM instrument from scratch, we could lower the various barriers, but this is too big of a challenge for researchers with a background in chemistry. Recklessly, although we are researchers in electroanalytical chemistry and have no expertise in surface physics, machining, electronics and software development, etc., we have been trying to build a GXSM-based SPM to apply it to the study of electrode surfaces. In this note, we show an example of building and operating an SPM with GXSM and its DSP-based controller by combining a commercially available STM scan head that is compatible with a UHV environment with commercially available high-voltage (HV) and current-voltage (I–V) amplifiers, etc. By using the appropriate components and optimizing the operating conditions, it is possible to construct an SPM system that is comparable to a high-performance commercial measurement system at a low cost. The results of observing the surface of a typical sample with STM and AFM are indicated. We will prove that the highly scalable SPM system, built around the open-source software GXSM, is extremely effective in the field of chemistry.

Experimental

The setup of the SPM system is shown in Fig. 1. The SPM scan head is a Unisok USM-1100. The probe for STM is mounted downward via a holder in the scan head. The sample holder is located on the 3-axis tube piezo scanner underneath the probe, and the sample holder is coaxially moved in the Z- and X-axes by the shear piezo. The sample is scanned by placing the sample holder on top of the tube scanner. The scan head has a built-in eddy-current damper as a vibration-isolation mechanism and is attached to the ICF203 flange by a fishing spring. The output signals due to the tunneling current in the STM and the excitation of the tuning fork in the FM-AFM is amplified by an I–V amplifier (Turtle Industries IVA-001S, 100 MΩ) directly attached to the ICF203 flange outside. The I–V amplifiers were powered by 6P dry cell batteries or a constant-voltage power supply (Kikusui PAB 25-1TR). The voltage signal output from the I–V amplifier is input to the controller through a band-pass filter (Turtle Industries T-01BPF01C, center frequency 32.8 kHz) in the FM-AFM measurement. On the other hand, the piezo drive signal outputs from the controller are amplified by HV amplifiers (Mess-tek M2679, and M2629B) to drive the tube piezo and share piezo of the scan head.

The measurement software is GXSM 3.11.0/ubuntu 16.04 LTS. Although GXSM can be the latest version based on GNOME3, we use a matured version because of its hardware stability. For the controller, we used SoftdB’s Open Source SPM Controller & PLL. PLL means phase-locked loop for FM-AFM operation. It is based on DSP, and the firmware can be easily rewritten and updated according to the upgrade of GXSM software. If we want to operate only STM measures and not FM-AFM, we can use the Mk2-A810 controller (SoftdB) without the built-in PLL instead. The controller is connected to the Linux PC via USB and has 8 A/D and D/A inputs/outputs, which can be assigned to various input/output functions from the control software, such as analog waveform output for probe approach, fine movement of XYZ axis, bias voltage output for STM and input for Z feedback and so on. SoftdB provides a PLL software for windows OS in the Open Source SPM Controller & PLL, which can be used as a stand-alone PLL in combination with other STM controllers without using the GXSM software. As the probe in STM, Pt/ Ir (90:10) wire (Φ 0.3, 0.2 mm, Nilaco) was used, and the tip was mechanically formed by nippers. The USM-1100 head has an optional folder attachment, fine movement of XYZ axis, bias voltage output for probe approach, and removing the main body. Attaching the probe to the tip of one prong causes an imbalance in the tuning fork and a significant decrease in Q value and resonance frequency, f₀. Therefore, the tip of the prongs on one side was ground with a precision metal file to make it asymmetric (arrow in Fig. 2b), and then the probe was glued with a thermosetting resin (Epo-Tek H74). In the case of FM-AFM only without STM measurement, a tungsten wire (Φ 0.03 mm, Nilaco) was used as a probe, and the tungsten wire was glued to the tuning fork in

Fig. 1 Picture and connection diagram of SPM system. 1, Controller with PLL; 2, controller for STM alone; 3, HV amplifier for shear piezo; 4, HV amplifier for xyz piezo; 5, band pass filter; 6, DC power source for I-V amplifier; 7, personal computer; 8, SPM scan head; 9, I-V amplifier. for attaching tuning forks instead of a folder for attaching the STM metal probe. For FM-AFM measurements, a tuning fork (DT-38, Daishinku, Le = 2.0 mm, L = 3.5 mm, t = 0.33 mm, f₀ = 32768 Hz, k = 13000 N/m, Fig. 2) was used. The DT-38 is slightly larger and thicker than the tuning fork, which has been used for high-resolution measurements with the qPlus sensor (e.g. Statek TFW-1165, E158), and has the advantage that the body can be easily machined directly and the probe can be attached. The DT-38 was used by opening the metal package and removing the main body. Attaching the probe to the tip of one prong causes an imbalance in the tuning fork and a significant decrease in Q value and resonance frequency, f₀. Therefore, the tip of the prongs on one side was ground with a precision metal file to make it asymmetric (arrow in Fig. 2b), and then the probe was glued with a thermosetting resin (Epo-Tek H74). In the case of FM-AFM only without STM measurement, a tungsten wire (Φ 0.03 mm, Nilaco) was used as a probe, and the tungsten wire was glued to the tuning fork in

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advantage. Then, the tip was sharpened by anodic oxidation using the so-called lamellar drop-off method, in which the tungsten wire was the anode, a 3 mol dm$^3$ KOH (Fujifilm Wako Pure Chemical Co., Ltd.) aqueous solution was the electrolyte, and a platinum ring was the cathode. Au(111) was deposited on a mica substrate by vacuum deposition. The detailed conditions for vacuum deposition have been described previously. For vacuum deposition have been described previously. For vacuum deposition have been described previously.

Fig. 2 Tuning fork DT-38 and solution cell. (a) Diagram of processing; L, length of one beam; L_e, length of the electrodes; t, thickness (b) without metal probe. The arrow indicates the part that was cut away to make it asymmetric before the probe was installed. (c) With metal probe.

Results and Discussion

Surface observation in STM

Figure 3 shows the surface image of HOPG observed in STM in the atmosphere. In Figs. 3a and 3b, the step structure of one layer of graphite is clearly visible. Figure 3c is a surface image of HOPG obtained by scanning a $2.5 \times 2.5$ nm area. Due to the characteristics of the scanner of the STM head and HV amplifier used, it was possible to measure areas of up to $2 \times 2 \mu m$. Although the STM image of a cleaved HOPG surface is expected to be a six-membered ring structure based on the crystal structure, it is actually known to be a hexagonal surface image when measured in air. This is thought to be because the carbon atoms that make up graphite have different functions depending on where they exist. In HOPG crystals, the six-membered ring structure planes are stacked at intervals of 0.335 nm. If we look at the upper layer of carbon, there are two types of sites: one where the carbon atoms of the lower layer exist directly below ($\alpha$ site) and one where they do not ($\beta$ site). The electronic cloud of the carbon atoms in the $\alpha$ site is involved in interlayer bonding, while the carbon atoms in the $\beta$ site are not involved in interlayer bonding. As a result, there is a difference in the electronic structure of the two sites, and only the carbon atoms in the $\beta$ site are emphasized in the STM image, resulting in a hexagonal lattice image with a lattice spacing of 0.25 nm. The image in Fig. 3c illustrates this feature well. Note that the HOPG surface shows a six-membered ring structure when the cleavage is performed in an argon atmosphere, and the STM measurement is also carried out in an argon atmosphere. Therefore, it has been reported that the hexagonal surface image observed in air is due to the adsorption of oxygen and water on the surface.

Figure 4 shows the observed image of the Au(111) surface in STM in the atmosphere. The relatively large area in Fig. 4a ($500 \times 500$ nm) shows the step structure and wide flat area characteristic of Au(111). It can be seen from Fig. 4b that the height of the step corresponds to one atom (about 0.24 nm) of gold. When the terrace of the Au(111) surface is further magnified and the unevenness in the height direction is observed in detail. Figure 4c is an image observed by STM measurement in air for a very short time by searing the Au(111) surface with a $H_2-O_2$ torch just before the STM measurement. The zigzag structure seen in the image is a typical stable structure of the $22 \times \sqrt{3}$ surface rearrangement, which shows a structure called herringbone, in which the two parallel lines (which appear almost together in this image) appear with a repetitive period of about $7 - 8$ nm $\times 14 - 16$ nm. These images show that we can obtain a step structure corresponding to one atomic layer in a relatively large area to an atomic resolution image in a small area.

Surface observation in FM-AFM

In FM-AFM using a tuning fork as a sensor, the tuning fork with the probe attached is excited mechanically by external piezo or electrically by inputting AC voltage to the electrodes printed on the tuning fork surface, and the probe is brought close to the sample surface while locked at the resonance frequency by PLL. As the probe approaches close to the sample surface, the resonance frequency shifts due to the interaction (attraction or repulsion) between the surface and the tip of the probe, and the distance between the sample surface and the probe is controlled by the piezoelectric element by converting the amount of frequency shift. As shown in Fig. 2, the tuning fork used in this study was made asymmetric by shaving the prongs for attaching the probe with a metal precision file beforehand to suppress the decrease in the $Q$ value due to the increase in mass caused by the adhesion of the probe. The tip of one prong of the tuning fork was scraped so that the resonant frequency shifts to about 33.2 kHz. It is reported that the tuning fork with the probe attached has a larger $Q$ value as the
The resonance frequency is closer to the value \( f_0 (= 32768 \text{ Hz}) \) before scraping. When a tungsten probe (\( \Phi = 0.03 \text{ mm}, \text{length} \approx 1 \text{ mm} \)) was attached to the asymmetrized DT-38 tuning fork, the resonance frequency decreased to 32780 Hz, and almost no decrease in \( Q \) value was observed. By inputting an AC voltage, the two prongs were excited in anti-phase mode, and a \( Q \) value of around 10000 was obtained even in air. In the intermittent contact mode AFM and FM-AFM, the large decrease in \( Q \) value due to the resistance of the solution has been a problem for measurements in liquids (\( Q \approx 10 \)). Since the SPM scan head used in this experiment is based on the sample scan method with the sample on the bottom side, we prepared a simple solution cell using O-ring and attempted FM-AFM measurements in liquid. Figure 5 shows the FM-AFM image of the Au(111) surface observed in polyethylene glycol. When the probe approaches the sample surface, the \( Q \) value decreases.

Fig. 3  Topographic STM images of HOPG cleavage plane in the atmosphere. \( I, 0.4 \text{ nA}; V, +0.3 \text{ V}; \) scan area, (a) \( 2 \times 2 \mu \text{m} \); (c) \( 2.5 \times 2.5 \text{ nm} \). (b) Line profile between A and B. \( 512 \times 512 \) pixel. (d) Atomic arrangement of graphite surface. 3D view and top view (solid line: first layer, dotted line: second layer).

Fig. 4  Topographic STM images of Au(111) surface in the atmosphere. \( I, 0.5 \text{ nA}; V, +0.2 \text{ V}; \) scan area, (a) \( 500 \times 500 \text{ nm} \); (c) \( 100 \times 100 \text{ nm} \). (b) Line profile between C and D. \( 512 \times 512 \) pixel.
decreases to around 1000, the step structure peculiar to the Au(111) surface can be observed.

High-resolution observations of insulating samples with a microstructure, such as mica, is still in progress, and the challenge is to reduce the amplitude of the tuning fork by reducing the noise of the I-V amplifier.

Conclusions

Open-source SPM control software, GXSM, and a DSP-based controller ware combined with commercially available STM scan head and amplifiers to build an SPM that can operate in air and liquid. It can cover an area from relatively large area for an STM, $2 \times 2 \mu m$ to atomic resolution region. Using the same setup, FM-AFM was performed with a tuning fork that was asymmetrized beforehand to reduce the effect of mass gain due to a metal probe attachment. Even though the probe entered the liquid by approaching the sample surface, the $Q$ value could be maintained at a large value and the step structure of Au(111) could be observed. The scan head could be moved to another environment, such as a UHV system, or it could be used with an electrochemical cell, which is a liquid system, incorporated into the sample holder. The cost of constructing the system was less than 1/10 of that of a commercial product with similar performance, indicating that it is possible to construct a high-performance and scalable system even on a limited budget.

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