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The non-destructive threshold of the graphite surface by STM in the ultra-fast pulse mode*

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In this paper single ultra-fast voltage pulses are introduced to the Pt/Ir tip of a scanning tunnelling microscope (STM), and the non-destructive threshold of the graphite surface is studied systematically in a wide range of pulse durations (from 10^4 to 8 ns). Considering the waveform distortion of the pulses at the tunnelling region, this paper gives the corrected threshold curve of pulse amplitude depending on pulse duration. A new explanation of threshold power has been suggested and fits the experimental results well.

Keywords: non-destructive threshold, voltage pulse, scanning tunnelling microscope
PACC: 0779, 6820

Due to its inherent unmatchable spatial resolution, the scanning tunnelling microscope (STM) is a unique tool to reveal the morphology and electronic structure of surfaces with atomic resolution in real space. Combining its imaging, manipulation, spectroscopic characterization, and chemical modification capabilities, the STM has enabled direct visualization of basic physical and chemical processes and properties related to the surfaces,[1,2] which otherwise would be hidden in the study of an ensemble of atoms and molecules with most of the other spectroscopy. However, STM images are the convolution of the electronic structure of the tip and the surface, which are difficult to interpret even with the sophisticated theories. Its inability to provide elementary information keeps many questions in the surface science society inextricable. A common methodology is urgent to be developed to identify the specific atoms on the surfaces.

If the tip of the STM is treated as a field emission electron gun, general elemental analysis of single atom may be achieved by using a proximity probe which can detect the de-excited electrons from core or inner valence shells. However, in this scheme non-destructive measurement, i.e. neither the sample nor the tip should be destroyed during the measurement, is in conflict with the spatial resolution, because high incident electron energy is necessary for elemental analysis, which requires high tip–sample voltage, but this voltage will surely damage both tip and sample at the tunnelling region. If the tip is retracted to avoid the destruction, then spatial resolution will decrease. Actually there have been some preliminary experiments such as LEED-STM,[3−5] AES-STM,[6,7] EELS-STM[8−10] and x-ray combined with STM,[11,12] but none of them could achieve atomic resolution. In order to figure out the conflict between the spatial resolution and non-destructive measurement, a scheme of STM with pulse bias voltage was proposed.[13] As the first step, the threshold of non-destructive pulse should be investigated.

Voltage pulse is extensively used in STM experiments for the surface modification under various environments,[14−19] but as yet there is still a lack of adequate theory to describe the complex physical process. By introducing single ultra-fast voltage pulses to the Pt/Ir tip of STM, the non-destructive threshold amplitudes of the pulses on the graphite surface in high vacuum have been determined systematically in a wide range of durations from 10^4 ns to 100 ns in our previous work.[20] In this paper, our recent experimental results with the even shorter pulse durations of 8 ns and 50 ns are reported. Meanwhile, the waveform distortion of the pulses is taken into consideration.

Our apparatus consists of a commercial STM sys-

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tem (easyScan, Nanosurf AG) modified to allow application of short voltage pulses between the tip and the substrate.[21] The STM was mounted in a high vacuum chamber. During experiments, the chamber was pumped to base pressure of 5×10^{-5} Pa. Single or a series of pulses in the duration range of 2–200 ns from a commercial rectangular negative voltage pulse generator (AVIR-4-C, AVTech.) were applied to the tip produced by mechanical cutting Pt/Ir wire. The feedback loop was disabled in order to prevent the tip from retracting during the pulses. Clean surfaces of graphite were prepared by cleaving the highly ordered pyrolytic graphite (HOPG) with adhesive tape in air and then transferred to the STM in the high vacuum chamber immediately.

A series of large voltage pulses were applied to the tip to get rid of contamination before approaching to the tunnelling region. Atomic resolved graphite topography image was obtained in constant current mode under the condition of bias voltage 50 mV and tunnelling current 1.00 nA to ensure that the STM tip is stable and sharp enough to carry out the following pulse experiments. The scanning parameters, such as bias voltage, tunnelling current, scan range, etc, are kept unchanged in every experiment so that the tunnelling condition will be almost the same for every applied pulse. Then the tip was moved to a specific spot (usually in the centre of the images) and the feedback was switched off and single pulse with prescribed amplitude and duration were added to the tip. After the pulse, the feedback was restored and the graphite surface was checked by raster scanning the same area again to see whether any destruction occurred (Fig.1). The original defects (such as steps) on the graphite surface or the pulse-induced structures are used as marks to guarantee that the scanning area is not shifted during the pulse-adding process.

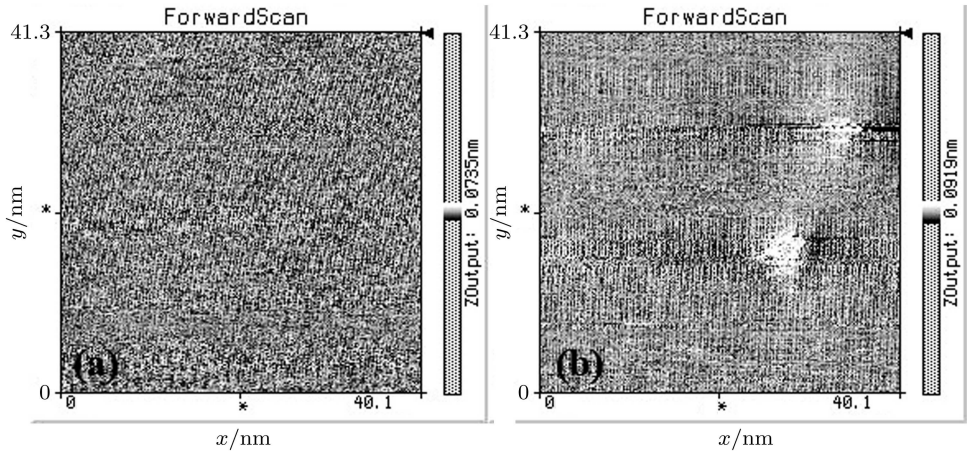


Fig.1. STM images of graphite surface before (a) and after (b) a single 4.25 V, 50 ns pulse applied to the tip, under the condition of bias voltage 50mV and tunnelling current 1.00 nA. Hillocks are induced by the pulse, which indicates that destruction has been done. The scan range is $40 \times 40\,\mathrm{nm}^2$.

Since the destruction is statistical in nature, the above process has to be repeated many times and the destructive probability P at a certain pulse could be calculated according to the equation:

$$P = \frac{n}{N}, \tag{1}$$

where n is the times the destruction happens and N is the total number of the input pulses. By varying the pulse amplitude and repeating the above destructive probability acquiring process while keeping the pulse duration fixed, we could obtain the curve to describe the dependence of destructive probability on pulse amplitude at certain pulse duration. In this paper, the amplitude at destructive probability 50% is defined as the threshold amplitude for the specific duration. Changing the pulse duration and repeating the above procedure, we could finally obtain the variation of the threshold amplitude with the pulse duration.

However, the shape of the pulse at the tunnelling gap will be different from the one generated by the pulse generator because of the I-V converter circuit and the gap load. Since the direct measurement of the waveform of the pulse applied to the tunnelling gap is unavailable for the present apparatus, the amplitude and width of the pulse were determined in the follow-

ing way. First the STM tip was withdrawn out of the tunnelling region and the waveform of the pulse was measured directly at the tip by a 300 MHz-bandwidth oscillograph. Then an equivalent circuit of the *I-V* converter circuit and the gap load was established by circuit emulator software. The gap load was set to be 10 MΩ, which is the input resistance of the oscillograph, to simulate the above-mentioned measurement procedure. It was found that the simulation was in good agreement with the measured result. The gap load was then adjusted to the tunnelling resistance of our apparatus to simulate the real situation of the tunnelling gap. Finally the amplitude and width of the pulse applied to the tunnelling gap were determined.

Figure 2 shows the destructive probability curves acquired at pulse duration of 50 ns and 8 ns after waveform calibration.

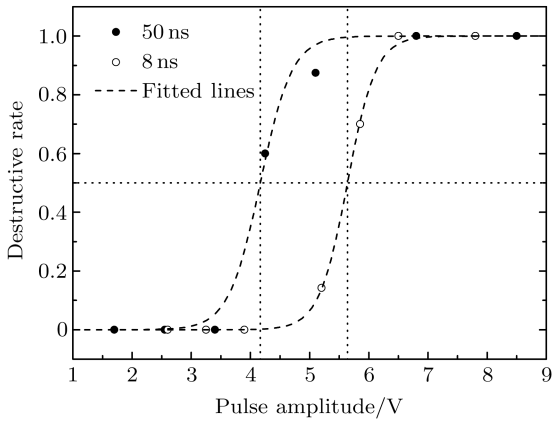


Fig.2. Probability curves of destructive rate versus pulse amplitude obtained at pulse duration 50 ns and 8 ns respectively. The condition of tunnelling junction was fixed by ensuring the scanning parameters (such as bias voltage, tunnelling current, etc) to be the same for every applied pulse. The corresponding amplitudes at destructive rate 50% are defined as the threshold pulse amplitude for these durations.

From these curves we have obtained the threshold amplitudes for the pulse with duration of 50 ns and 8 ns to be 4.2 V and 5.6 V. By calibrating the pulse waveform, we have modified the threshold amplitudes at pulse duration from 10^4 ns to 100 ns, which were acquired by our previous work.[20] The summed experimental data of the dependence of non-destructive threshold pulse amplitude on pulse duration in the range from 10^4ns to 8ns is shown in Fig.3, which is indicated by solid dots.

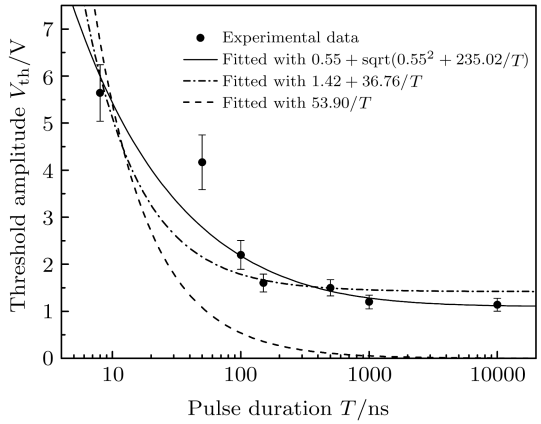


Fig.3. Experimental data of non-destructive threshold pulse amplitude vs. pulse duration and the corresponding fitted curves (see text for details).

An explanation of constant threshold impulse was suggested by Mori *et al* in W-Si system,[13] which showed the inversely proportional dependence of the threshold amplitude $V_{\rm th}$ on the pulse duration $T_{\rm th}$:

$$V_{\rm th} \times T_{\rm th} = C. \tag{2}$$

In our previous work,[20] a constant resistance was taken into account and Eq.(2) was adjusted as:

$$(V_{\rm th} - C_1) \times T_{\rm th} = C_2. \tag{3}$$

C, C_1 and C_2 in Eqs.(2) and (3) are constants. However, with new data of shorter pulse duration added, both Eqs.(2) and (3) are obviously unsuitable for the present experimental results, as denoted by dash line and dash dot line respectively in Fig.3. This indicates that the impulse will no longer dominate the destruction threshold for Pt/Ir-graphite system. If the energy dissipating rate in fracture mechanics[22,23] is taken into consideration, then the power of the electric force should exceed a threshold to do the damage. So we suggest a constant threshold power ($P_{\rm th}$) to explain the present experiment. The expression of the power can be written as:

$$P = C_0 V (V - V_0) T, \tag{4}$$

where V and T are the pulse amplitude and duration respectively, V_0 is a parameter which represents the resistance, and C_0 is a ratio constant. Thus for the threshold pulse, the following equation can be established:

$$P_{\rm th} = C_0 V_{\rm th} (V_{\rm th} - V_0) T_{\rm th}. \tag{5}$$

By solving Eq.(5), the amplitude of the threshold pulse $V_{\rm th}$ as a function of the pulse duration $T_{\rm th}$ is then deduced to be

$$V_{\rm th} = A + \sqrt{A^2 + B/T_{\rm th}}, \tag{6}$$

where A and B are constants which are proportional to the resistance and the threshold power respectively. By least square fitting with experimental data, the parameters are determined to be $A = 0.55$ and $B = 235.02$. The fitted curve according to Eq.(6) is denoted by solid curve in Fig.3, which is agreed with the experimental data much better than the curves fitted with Eq.(2) (dash line) and Eq.(3) (dash dot line) in the duration range from 10^4 ns to 8 ns. In our experiment, most pulse-induced structures are hillocks, while the rest are craters. So we suppose that the destruction is caused by tip deposition, which is much different from Mori *et al*'s experiment.[13] The large difference of the threshold behaviour between their experiment and ours may be due to this, or the difference of the mechanical and electronic properties between W-Si system and Pt/Ir-graphite system, or the pulse duration of Mori *et al*'s experiment is not short enough. Quantitative theory is awaited to describe the complex ultra-fast process in the tunnelling junctions.

In summary, we have reported the non-destructive threshold behaviour of the graphite surface by applying single ultra-fast voltage pulses to Pt/Ir tip in a large duration range from 8 ns to 10 μs. The present experimental data demonstrate that the threshold impulse explanation which deduces the inversely proportional relationship $V_{\rm th}$-$1/T_{\rm th}$ proposed by Mori *et al*[13] could not account for our experiment. A new explanation of threshold power has been suggested and the equation derived fits the experimental results well.

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