Submitting ICN+T 2008 Proceeding Paper to the Journal of Vacuum Science and Technology
(JVST)

Paper Code: SP-MoP11

Title:

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In this work, we report quantitative investigation of lateral force gradient and lateral force between a tungsten tip and Si (111)-(7×7) surface using a combined non-contact Lateral Force Microscopy and Scanning Tunneling Microscopy (nc-LFM/STM). Simultaneous lateral force gradient and scanning tunneling microscopy images of single and multi atomic step are obtained. In our measurement, tunnel current is used as feedback. The lateral stiffness contrast has been observed to be 2.5 N/m at single atomic step, in contrast to 13 N/m at multi atomic step on Si (111) surface. We also carried out a series of lateral stiffness – distance spectroscopy. We observed lateral stiffness-distance curves exhibit sharp increase in the stiffness as the sample is approached towards the surface. We usually observed positive stiffness and sometimes going into slightly negative region occasionally.

KEWWORDS: Non-contact lateral atomic force microscopy, Small oscillation amplitude, Lateral force gradient – distance spectroscopy

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1. INTRODUCTION

Lateral forces play an important role in variety of phenomena in our daily lives as well as micro and nanoscale systems. Initial approaches to understand the origin of the lateral forces associated with the relative motion of two objects were mainly based on collective mechanical properties of interacting objects1). However, investigation of lateral forces and interaction energy at the molecule and atomic scale need accurate measurement of lateral stiffness of single chemical bonds established between the objects in contact. Lateral Force Microscopy (LFM) has shown its capability to image and identify lateral forces at the atomic scale while a sharp tip scans over a sample surface[2].

In 1987, Mate *et al.*2) imaged lateral forces acting between a sharp tungsten tip and a graphite surface and the stick-slip behavior was observed. Later on, different research groups3,4) detected atomic stick-slip behavior using a UHV- FFM apparatus on ionic crystals and metal surfaces and compared with a theoretically produced lateral force maps based on two dimension Tomlinson model.

Studies of atomic scale origins of lateral forces while sharp tip scanning in low load contact over sample surfaces have already contributed to the understanding of the microscopic origins of friction forces. However, the lateral resolution of force microscopy in contact mode is limited by the contact area of the tip apex, containing many atoms due to adhesion between tip and sample. This problem has been eliminated in non–contact Atomic Force Microscopy. Jarvis *et al.*5) developed a special cantilever for simultaneous control of tip-sample distance and lateral tip oscillations, in order to sense lateral interactions between tip and sample when approaching the surface. Giessibl *et al.*6) presented atomic resolution in lateral force, while using a tuning fork lateral force sensor. Their sensor had a glued tungsten tip at the end of one prong, while the other prong is fixed. The tip was intentionally tilted with respect to the sample by 6°. They showed that the interaction between a single-tip atom that is oscillating with 3 Å slightly cant to the Si(111)–(7×7) surface can be measured in dynamic mode AFM. However, the tilt of the force sensor with respect to the sample might reflect the contribution of normal forces gradient in addition to lateral force component. In another work, Pfeiffer *et al.*7) reported measurement of lateral forces between the tip of a force microscope and the atomic-scale features on the surface in a non-contact mode using 20 Angstrom amplitudes. In their experiment, a rectangular cantilever beam oscillates parallel to the Cu(111) sample surface at its torsional eigenmode, while the tunnel current is kept constant with the feedback loop.

In a recent work, Kawai *et al.*8) also reported atomic resolution in lateral force using few Angstrom amplitude with the frequency modulation dynamic lateral force microscopy. In their experiment, torsional resonance mode of a commercially available rectangular cantilever was used to detect the lateral interaction force gradients caused between the tip and the sample surface.

All of the above attempts have shown that the sub-Angstrom oscillation amplitude lateral force microscopy would reveal more insight into the lateral force interactions between tip and sample, down to single atom. In this report, we present for the first time simultaneous lateral stiffness and STM topography images on Si(111)–(7×7) surface. We also report the direct measurement of lateral forces as we vary the tip- sample separation, similar to our earlier experiments using ultra small amplitude non-contact AFM/STM9,10) in normal forces.

2. Experimental Methods

A home-made, high resolution nc-LFM/STM operating in UHV is used in our experiments. The microscope employs a sensitive fiber optic interferometer for high force resolution. Sub-Angstrom oscillation amplitudes can be used for imaging as well as performing direct lateral force – distance spectroscopy. Details of the instrument are described elsewhere[11]. A radio frequency (RF) circuit developed by NanoMagnetics Instruments Ltd.[12] is used to inject RF current into the laser diode to improve the sensitivity. The frequency and the amplitude of the RF current can be adjusted to optimize the noise reduction. A noise level of ~ 1×10^{-4} $Å/(Hz)^{1/2}$ is routinely obtained using this technique. Straight, etched, home-made tungsten levers from tungsten ribbons with typical stiffness of 50 N/m are used in the experiment and by considering the dimensions of the levers, the stiffness is calculated.

A high precision piezo driven 5-axis positioner was used to align the fiber with respect to the cantilever, as shown in Fig. 1. Si(111) sample was cut from 525 µm thick, P-doped, n-type wafers oriented within 0.5° off (111) plane. Both *ex situ* and *in situ* processes are applied to clean the samples. The sample was cleaned with propanol in ultrasonic bath and then rinsed with overflowing deionized water. Samples are then dried with blowing dry nitrogen gas before transferring into the load-lock chamber. The sample is then transferred into the UHV system and degassed and cleaned using an e-beam heater.

Fig. 2 shows the operation of the microscope, the lever is vibrated with sub-Angstrom oscillation amplitudes parallel to the sample surface at a frequency well below its resonance and the changes in the oscillation amplitude are measured using a lock-in amplifier as the sample is scanned across the tip. The microscope is operated with STM feedback and simultaneous scans of STM topography, tunneling current and force gradient can be acquired. The use of very small oscillation amplitudes at frequencies far below resonance allowed us to treat the cantilever motion as a linear spring, and by solving the *linearized* equation of motion13), the lateral force gradient between tip and sample can be deduced using simple expression:

$$k_{lateral} = -\frac{dF_{lateral}}{dx} = k_0\left(\frac{A_0}{A_{\mathrm{int.}}}\cos\phi - 1\right) \qquad (1)$$

where $k_{lateral}$,k_0, A_0 and $A_{int.}$ are interaction stiffness, cantilever stiffness, lateral free oscillation amplitude and the measured lateral amplitude of the cantilever, respectively. ϕ is the phase difference between the drive and the lever. The phase difference ϕ between the driving signal and the actual lever motion gives a measure of losses. In our measurements, we assumed the loss is small enough and it can be neglected.

3. Results and Discussions

In the first series of experiments, we imaged a clean Si(111) surface using a lateral cantilever. The cantilever's resonance frequency was 18.049 kHz and the lever was oscillated parallel to the surface with an oscillation frequency of 7.56 kHz, far below its resonance frequency with an oscillation amplitude of 0.4 $Å_p$. The tunnel current is used for feedback to control the tip-sample distance. The scan speed was set to 40 Å /s and the tip bias voltage and set tunnel current were –1 V and 0.4 nA, respectively. The single atomic and multi atomic steps on Si (111) are resolved in both topography and lateral stiffness channel, as shown in Fig.3. The lateral stiffness quantitatively is measured. At the two upper terraces almost zero lateral force gradient followed by 2.5 N/m at the single atomic step edge are measured. The zero lateral gradient while the tip scans the upper terraces can be attributed to symmetrical force gradient which tip apex senses while it is located at the terraces. However, the lateral stiffness of about 13 N/m is measured at the lower terrace. The difference of the force gradient at different terraces might be due to high density of defects and impurities on lower terrace, which were not resolved in topography image.

It should be noted that the observable residual noise in the measurements are due to the low oscillation amplitude, as well as the relatively short time constant used in the lock-in amplifier.

We have also performed lateral force gradient–distance spectroscopy. The feedback loop is frozen, the sample is first retracted back by a specified distance and re-approached toward the tip, while recording the force gradient as well as the tunnel current to perform the spectroscopy. A threshold current level is used to stop and retract the sample. The lever stiffness is calculated to be 70 N/m and oscillation amplitude was 0.4 $Å_p$. In Fig. 4, the force gradient–distance spectroscopy curve starts to increase earlier than the tunnel current onset and the tunneling barrier height is calculated to be 0.4 eV. This extremely small value for barrier height indicates that the tip or sample has some contamination and to increase current, tip has to be indented in to the surface quit hard. In another series of experiments, as shown in Fig. 5, the tunnel current starts before lateral force gradient onset as expected. The lateral force gradient–tunneling spectroscopy exhibits a sudden change in force gradient, while the tunneling current increasing smoothly in the course of approaching sample to the oscillating cantilever. The maximum repulsive force gradient is measured to be 46 N/m. The barrier height is calculated to be 4.1 eV, which implies that tip and sample are clean. It should be noted that for both experiments we used the same cantilever, but different sites of the sample and the same bias voltage are used for both measurement, $V_{Bias}= -1$ V. Our measurement reveals that there are significant lateral forces gradient acting on the tip during typical STM experiments. The lateral force gradient at 1 nA tunnel currents can be as large as 20–40 N/m. The current levels tested here are typical currents that are used at STM experiments.

In conclusion, a novel fiber optic interferometer based lateral nc-AFM/ STM is used to investigate the lateral stiffness between tungsten tip and the Si(111)–(7×7) surface. The improvement in the resolution of the interferometer allowed us to use very small oscillation amplitudes to oscillate the cantilever parallel to the surface, which overcomes the problems associated with the large amplitude technique that has been widely used. Since we use small amplitudes, we can extract lateral forces with unprecedented sensitivity by measuring the changes in the oscillation amplitude directly, rather than the frequency shift. We have observed single and multi atomic steps in topography and lateral stiffness images. This information allowed us to measure the lateral force direct and quantitatively. Our experiments reveal the first direct measurement of lateral stiffness in dynamic mode. Lateral force gradient–distance spectroscopy experiments have also been carried out, in which we simultaneously measured the force gradient, and tunneling current as the sample is approached towards the tip and retracted back. We obtained lateral force gradient- distance curves exhibiting sharp increase of the force gradient, while the sample is approaching to the surface. We observed positive and slightly negative force gradients. We described the lack of pronounced attractive region in the lateral force gradient could be due to cancellation of long range attractive forces acting on the tip because of the symmetry of the surface structure. We observed for the first time, surprisingly large, lateral stiffness in typical STM operating currents at 1 nA.

Acknowledgments

This project is funded in Turkey by TÜBITAK Grant No TBAG-2329 and the European Union NANOMAN Grant. We would like to thank Mr. Muharrem Demir of NanoMagnetics Instruments Ltd. for the technical support. A.O. thanks Turkish Academy of Sciences (TÜBA) for financial support.

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FIGURE CAPTIONS:

Fig. 1: A picture of the aligned optical fiber at the side of the special cantilever to measure the lever deflections

Fig. 2: a) Schematic view of lateral tip–sample interaction, and b) mass–spring modeling of the tip-sample interaction.

Fig. 3: Simultaneous imaging of Si(111). a) Topography image and lower curve shows the cross section view of the STM topography image b) Lateral force gradient image, and lower curve shows the cross section view of the force gradient image. Image size: 213 Å×80 Å. The lever was oscillated parallel to the surface with an oscillation frequency of 7.56 kHz and oscillation amplitude of 0.4 $Å_p$. Tip bias voltage and set tunnel current were –1 V and 0.4 nA, respectively.

Fig. 4: Simultaneous lateral force gradient-distance and tunnel current vs. distance spectroscopy. The sample bias voltage was set on –1 V. The cantilever free oscillation amplitude 0.4 $Å_p$.

Fig. 5: Simultaneous lateral force gradient-distance and tunnel current vs. distance spectroscopy. The sample bias voltage was set on –1 V. The cantilever free oscillation amplitude 0.4 $Å_p$. This curve obtained from different site of the sample away from position that the Fig. 5 was acquired.

Fig. 1

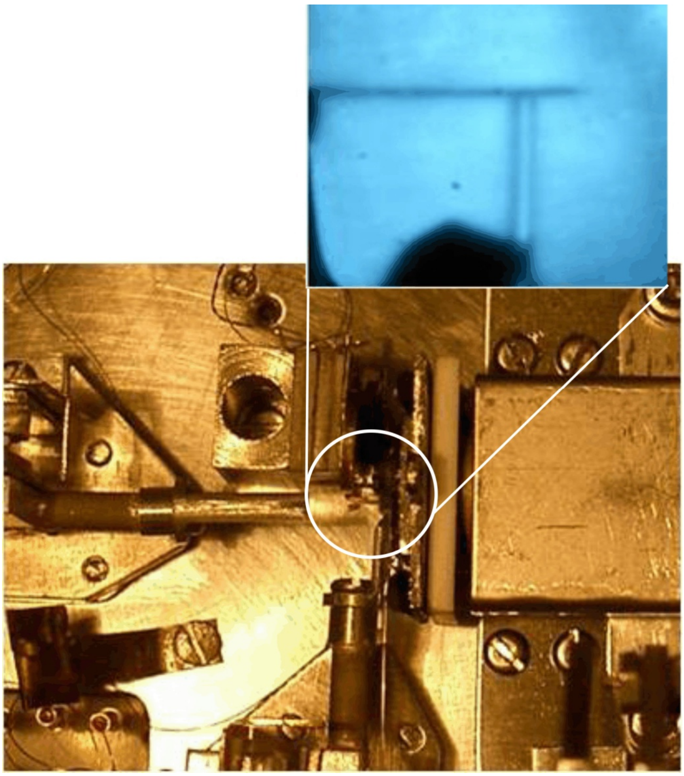

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Fig. 2

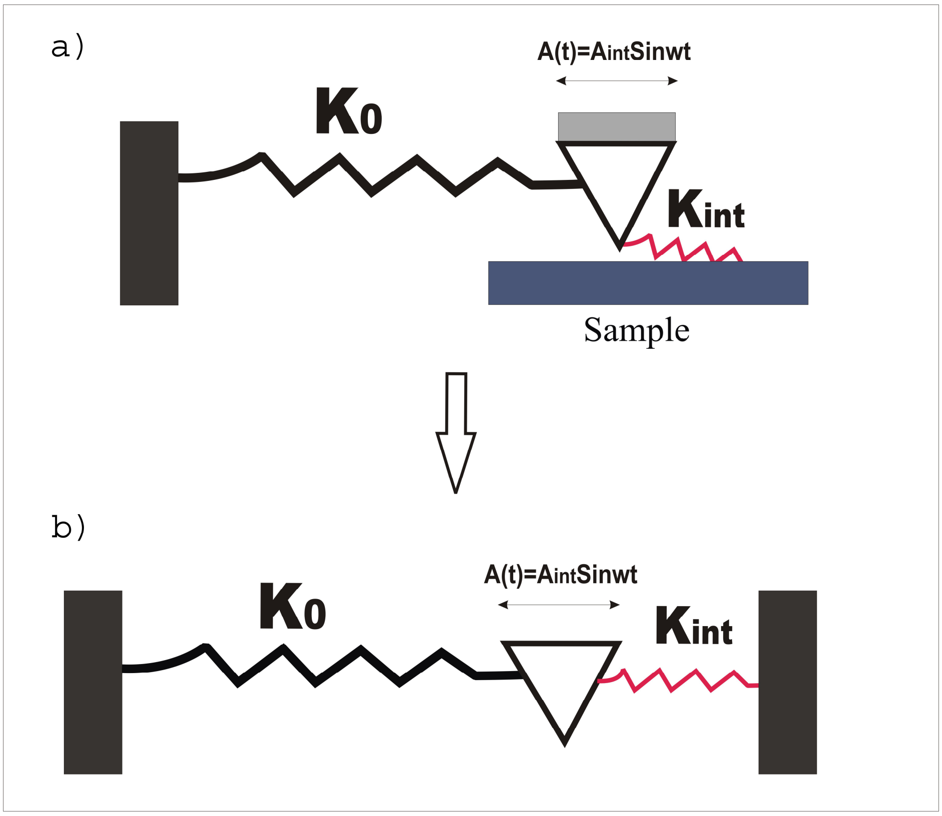

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Fig. 3

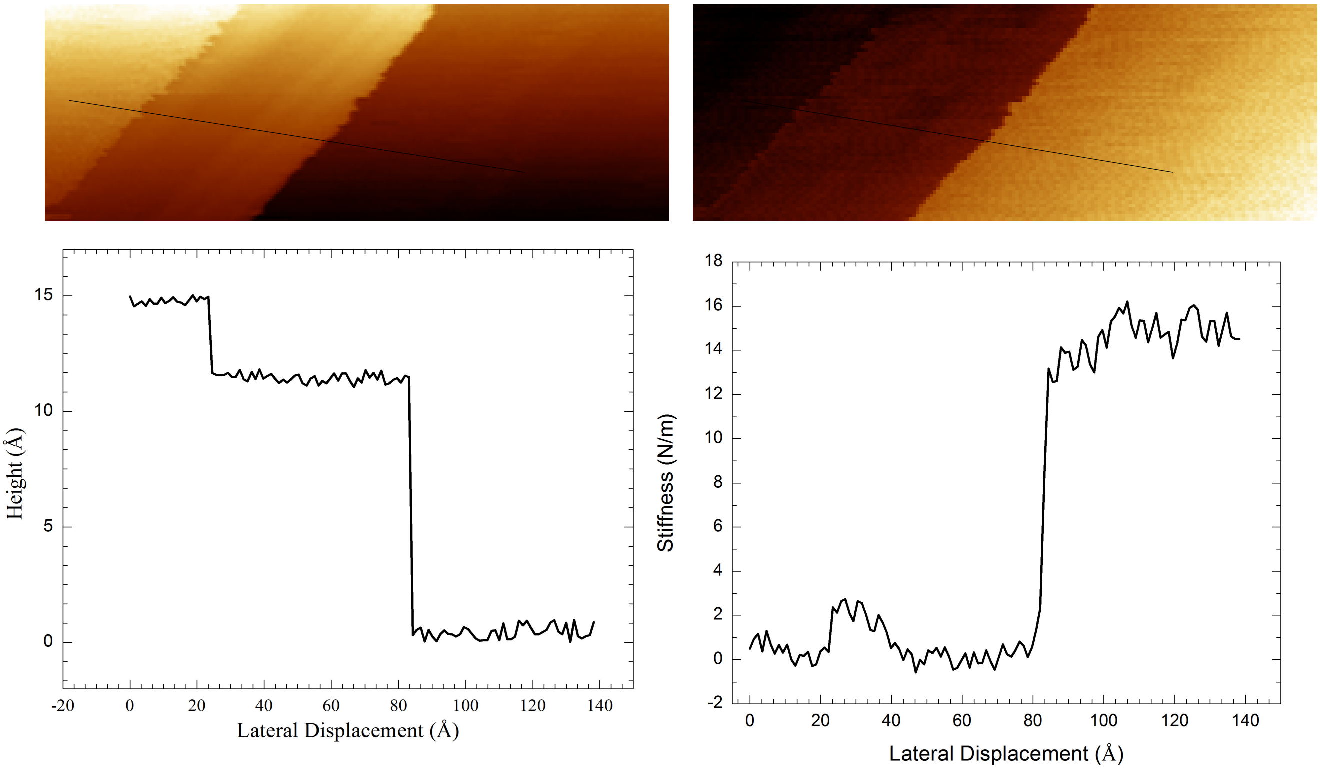

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Fig. 4

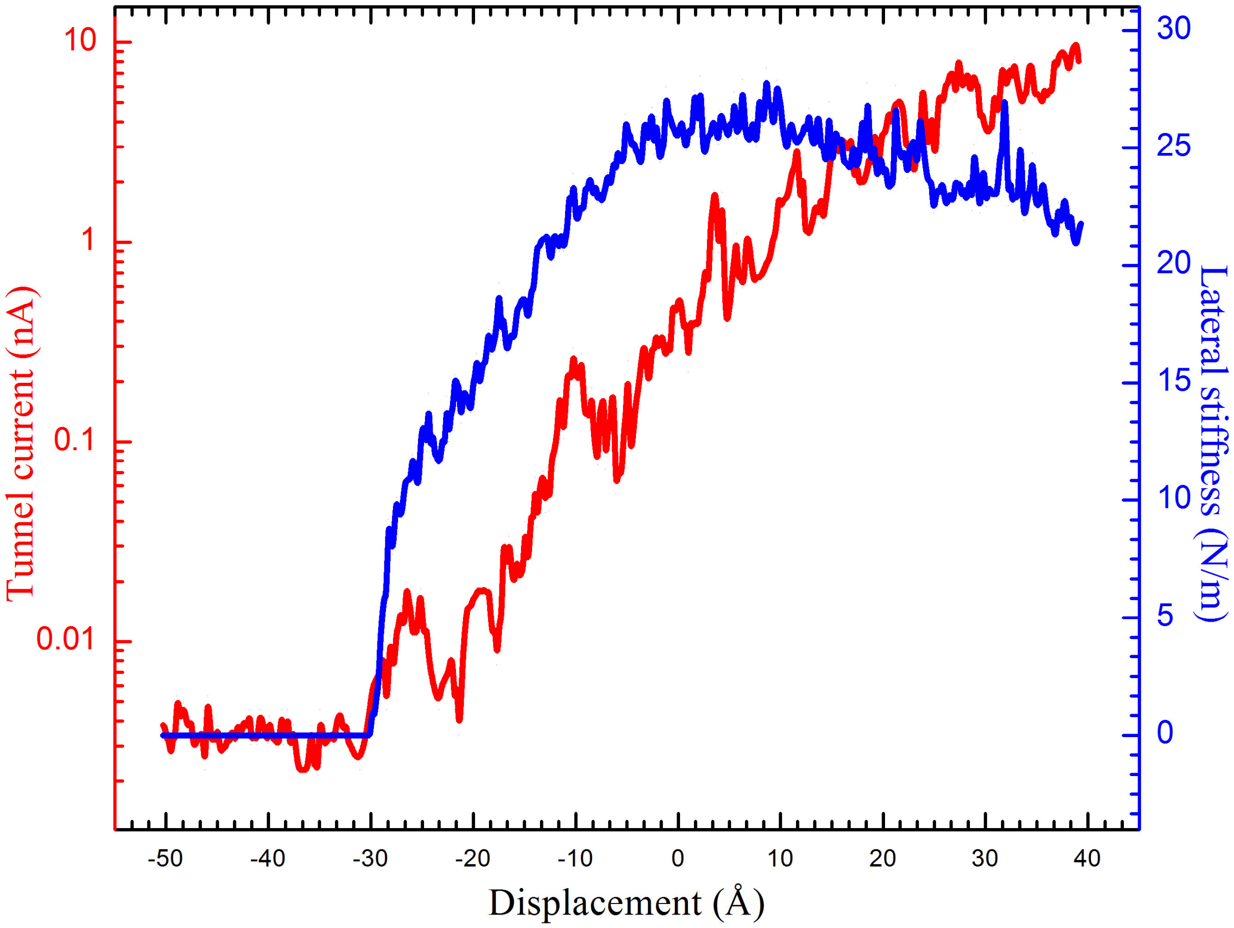

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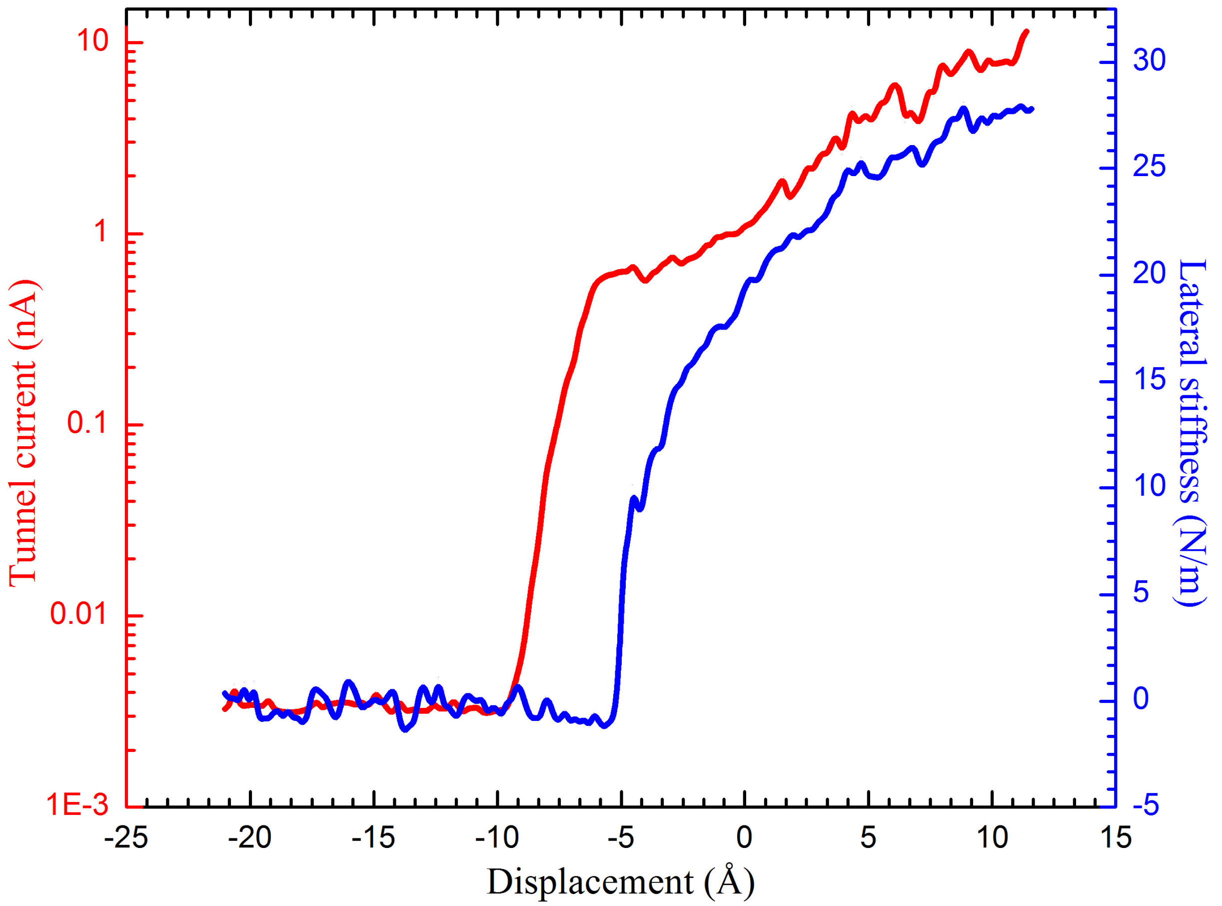
Fig. 5



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