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Instrumentation of STM and AFM combined with transmission electron microscope

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Abstract. A scanning tunneling microscope (STM) combined with a transmission electron microscope (TEM) is a powerful tool for direct investigation of structures, electronic properties, and interactions at the atomic scale. Here, we report on two different designs of such TEM-STM as well as an extension with an atomic force microscope (TEM-AFM). In the first TEM-STM design, a stepper motor, combined with a one-dimensional inertial slider, was used to perform the coarse approach. The advantage of this design was the strong pulling force that enabled notched metallic wires to be broken inside the TEM, which lead to clean sample surfaces. A second design, with a three-dimensional inertial slider, allowed lateral motion inside the TEM, which simplified the adjustment of tip location on the sample. By replacing the STM tip with a standard AFM-cantilever chip, a new combination was demonstrated: TEM-AFM. Here the force was simply measured by direct TEM imaging of the motion of the AFM tip. Some experimental results are included to illustrate the capabilities of TEM-STM and TEM-AFM.

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A scanning tunneling microscope (STM) inside a transmission electron microscope (TEM) is an attractive combined tool for direct investigations of structure, electronic properties, and interactions at the atomic scale.

This method (TEM-STM) has proven useful [1–10], for example, in the creation of atomic sized metallic wires and simultaneous conductance measurements [3], gold contact area observations [7–9], carbon nanotube studies [4], and point contact experiments [10].

One set of experiments suitable for TEM-STM concerns an unknown STM tip as well as an interacting sample, which is a limiting factor in the interpretations of STM data. The same is true for the atomic force microscope (AFM). In the AFM the force is measured by the deflection of a cantilever that supports the tip. The cantilever deflection, however, may not follow exactly the tip displacement. The shape of the tip and sample as well as the actual size and geometry of contact area during the experiment are unknown parameters in routine AFM force measurements.

Several constructions of TEM-STM are presented in the literature. An STM combined with reflection TEM was initially employed [11–17]. Later, operation in transmission mode was demonstrated. Geared electrical motors are usually used for the coarse approach [2, 6, 10]. An exception is a micro-machined TEM-STM [1]. Note also the combination of a scanning electron microscope (SEM) with an STM [18–29] and an AFM [26, 30–32]. In an SEM, however, the resolution is not as high as in a TEM.

Here, we present two designs of TEM-STM and discuss their different advantages. The first design is based on a combined coarse approach system (stepper motor and one-dimensional piezo inertial slider), and the second design is based on a new type of three-dimensional inertial slider.

In addition we show how to modify TEM-STM into a new kind of combination – TEM-AFM – thereby allowing force measurements simultaneous with imaging of tip and sample shape, contact area, elastic or plastic deformation, conductance, etc., to be studied.

1 Experiment

The holders with integrated STM and AFM were inserted into a field emission gun TEM (Philips CM200 Super TWIN FEG microscope). The vacuum inside the microscope was 10^{-5} mbar. The electron irradiation density during the measurements was $2.8 \times 10^{-5}\ \mathrm{A/m^2}$. For the TEM-STM work, the tip and sample were prepared by pulling a notched wire and breaking it mechanically.

For TEM-AFM, the samples were electrochemically etched gold tips, made from a 0.25 mm diameter wire (99.99% Au). The tips were cleaned by argon milling. The AFM cantilever tips were of standard silicon nitride contact mode type with a force constant between 0.03 and 0.37 N/m. The tip and the cantilever were coated with a 5 nm Cr adhesive layer and with a 15 nm Au film. Such a coating increases the force constant by less than 10%, based on the formulae

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in [33] (with a 600 nm-thick cantilever, as in our case). The force constants were as specified by the manufacturer and may have an error of 30%–50%. Inside the TEM, the tip and sample were locally prepared by pressing the tip hard into the sample.

For conductance measurements the current signal was simultaneously monitored on a digital oscilloscope with video TEM images.

2 Design of stepper motor TEM-STM

The highest noise source in SPM is usually external mechanical noise of low frequency. The general method to decrease this noise is to make the device as small as possible, which gives a high resonance frequency. An improvement to our previous TEM-STM [10] would thus be to make a smaller mechanical loop.

Therefore, in the present stepper motor design we introduced a clutch, which decreased the loop. In addition, the pre-adjustment of two STM tips was improved by a ball-clamped mechanism Fig. 1b. To make two tips close to each other, a special tool (7 in Fig. 1b) was placed at the top end of the TEM-SPM. By turning the adjustment screws perpendicular to the main axis, the tips were set next to one another under an optical microscope (with a lateral accuracy of 20 μm a couple of tenths of a mm was left between them). After adjustment, the tool (7) was removed, and TEM-STM was ready to be installed into the TEM.

This construction enables clean tip surfaces to be prepared by breaking wires inside the TEM. The tips were formed from a single wire by fixing it to the rod inside the piezotube (5) and clamping the ball (6). Firstly, a notch on the wire was formed mechanically, leaving about 20–100 μm of material. Secondly, the wire was pulled apart inside the TEM with the help of the stepper motor until it broke at the notch, resulting in two tips at the center of the electron beam. Then the clutch (4) was released by turning the stepper motor back. Thereafter, the tips were approached using the inertial slider, which was formed inside the piezotube (5). A titanium rod slider was shifted against a graphite support at the both ends of the piezotube until the tip-to-tip separation falls in the piezo attenuation range. The final tunneling approach was made by the piezotube (5).

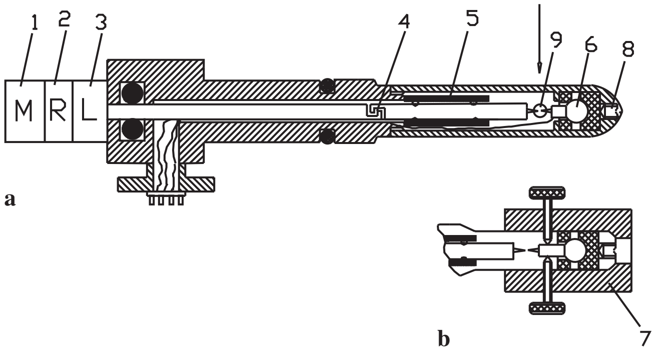

Fig. 1a,b. Stepper motor based TEM-STM (not to scale) **a** 1: stepper motor, 2: reductor, 3: linear translator, 4: clutch, 5: piezotube, 6: adjustment ball, 9: electron beam. **b** 7: preadjustment aid of the tip, 8: stop screw (not to scale)

3 Design of 3D inertial slider TEM-STM

The main problem with a one-dimensional design is the lack of lateral adjustments inside the TEM; the range of the piezo scanner might be smaller than needed to adjust the tips against each other. To solve this problem a new inertial slider Fig. 2 was elaborated. The main idea of this slider is that a tip is placed through the ball (2) so that the center of mass is in the center of the ball.

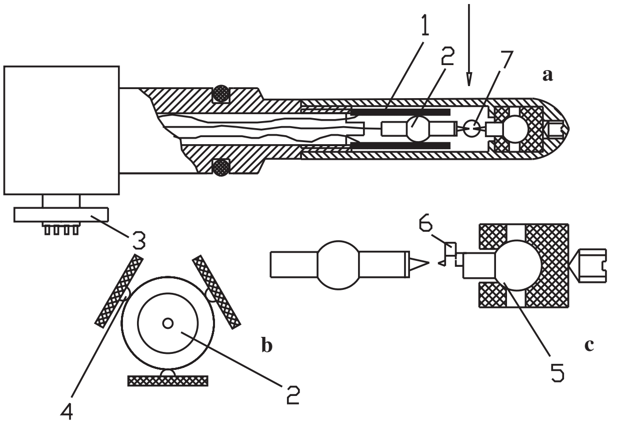

Fig. 2a–c. Three-leg slider based TEM-STM. **a** 1: PTZ plate, 2: shifting and tiling ball, 7: electron beam. **b** Cross-section of the slider; 4: friction rods. **c** AFM chip with the preadjustment ball (increased); 5: preadjustment ball, 6: AFM chip (not to scale)

A saw-tooth-shaped voltage applied simultaneously to three piezo plates shifted the ball (2) along a straight line. For lateral motion of the tip, only one plate in the slider was used, resulting in a tilt of the tip. The approach of the two tips was carried out, step by step, by shifting the ball and by correcting the direction under TEM observation. The main advantage of the three-plate slider TEM-STM was an easy and reliable adjustment of the tips. However, this slider was not strong enough to break a notched wire.

4 Design of TEM-AFM

The standard technique of force detection in AFM involves a thin cantilever with an integrated tip and an optical system used to detect the cantilever deflection. These optical systems are usually large. However, in the TEM, there already exists an electron optical system that could be used for imaging the tip displacement; we used this.

In our construction an AFM tip can easily replace the STM tip (Figs. 2 and 3). In Fig. 3 a low magnification TEM image is presented which shows the AFM tip on the cantilever and an electrochemically etched Au tip. The motion of the tip was simply imaged directly in the TEM images. With a known force constant for the cantilever, the force was able to be calculated.

5 Examples of measurements

The TEM image in Fig. 4 shows a single-atom-wide gold nanowire between two gold tips observed using TEM-STM. As an example of TEM-AFM other experiment (see TEM

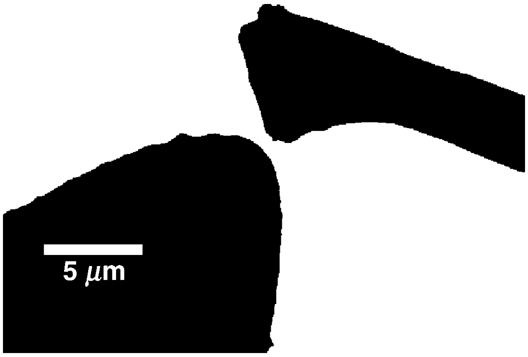

Fig. 3. TEM image of the gold sample and the gold-coated AFM tip and cantilever

image in Fig. 5) a 0.9 nm-diameter gold nanowire was created between the gold sample and the gold-coated AFM tip. The rupture force of this nanowire was determined to be about 9 nN, which allowed us to estimate the approximate force per atom to be about 1 nN. This value is consistent with theoretical [34–36] as well as experimental data [37].

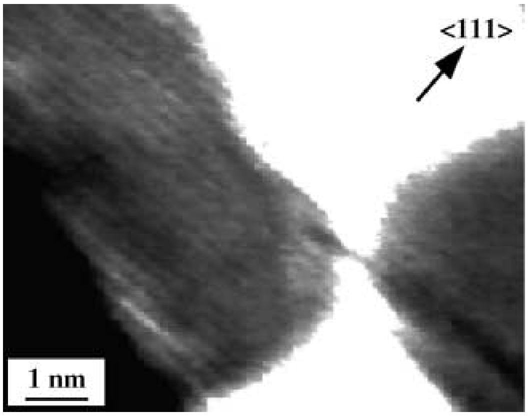

Fig. 4. TEM image of a single-atom-wide wire connecting gold tips (TEM-STM)

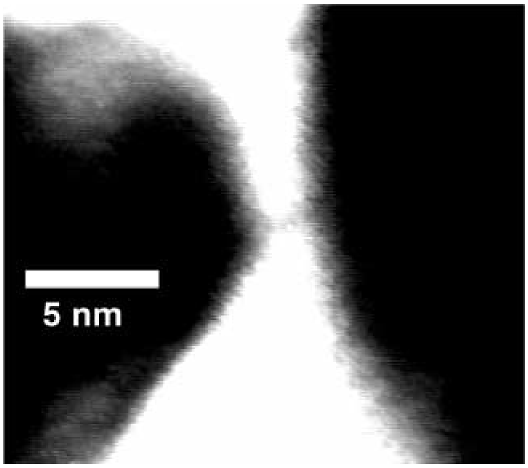

Fig. 5. TEM image of the small gold neck that is formed between the sample and the AFM tip. The attractive force holds them together. Just before breaking, the diameter of the neck is 0.9 nm, corresponding to about 10 atoms

6 Conclusions

We developed two new designs of TEM-STM, as well as an extension to TEM-AFM. With these instruments one can directly visualise the contact between the STM or AFM tip and the sample. However, there is a number of problems not directly related to the STM or AFM methods, such as point contacts or adhesion, that could be addressed by these instruments. The essential advantage of these instruments is that one can dynamically study objects during TEM imaging.

The coarse approach of the tip in our TEM-STM and TEM-AFM was performed by combining a stepper motor and a one-dimensional slider as well as by using a new type of three-dimensional inertial slider. The advantage of the stepper motor design was the ability to break notched metallic wires inside the microscope in vacuum. The three-dimensional slider provided lateral adjustment of contacting tips inside the TEM. Applications of TEM-STM and TEM-AFM for investigation of gold point contacts, interactions, and in situ measurements of force and contact type were demonstrated.

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