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Ultra compact multitip scanning tunneling microscope with a diameter of 50 mm

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We present a multitip scanning tunneling microscope (STM) where four independent STM units are integrated on a diameter of 50 mm. The coarse positioning of the tips is done under the control of an optical microscope or scanning electron microscopy in vacuum. The heart of this STM is a new type of piezoelectric coarse approach called KoalaDrive. The compactness of the KoalaDrive allows building a four-tip STM as small as a single-tip STM with a drift of less than 0.2 nm/min at room temperature and lowest resonance frequencies of 2.5 kHz (*xy*) and 5.5 kHz (*z*). We present as examples of the performance of the multitip STM four point measurements of silicide nanowires and graphene. © *2012 American Institute of Physics*. [http://dx.doi.org/10.1063/1.3694990]

INTRODUCTION

The controlled fabrication of self-organized nanostructures with dimensions in the single digit nanometer range is becoming possible.[1,2] However, the measurement of charge transport through such nanostructures is still a challenge. To provide electrical contacts to individual nanostructures is a problem. One approach is to establish such contacts by a multitip STM in order to enable charge transport and scanning potentiometry measurements at self-assembled nanostructures. Key requirements for multitip scanning probe instruments are that (a) all tips are independently positionable from the millimeter range down to the nanometer or atomic scale; (b) optical microscopy imaging or scanning electron microscopy (SEM) imaging is necessary in order to navigate the tips close to each other without unintentional tip-tip contacts and in order to find specific structures on the surface in case of lithographically structured samples; (c) the final electric measurements (e.g., four point measurements) should be performed at one specific position on the nanometer/atomic scale and destruction free, i.e., lowering the tip in a controlled way towards the surface or a nanostructure. In order to meet the last requirement, an instrument with currently unsurpassed stability has to be constructed. The design of single tip instruments has shown over the last decades that the smaller the instrument is, the less drift results and the lower is the vibrational noise.[3–6] In order to miniaturize (multitip) scanning probe microscopy further we developed a new kind of piezoelectrically driven nanopositioner: the KoalaDrive which is described in detail in Ref. 7. This positioner is used for tip-sample coarse approach and allows the miniaturization of the scanning tunneling microscope (STM) design. Specifically, regarding multitip scanning probe microscopy it was possible to develop an ultra-compact multitip scanning probe instrument with a drift of less than 0.2 nm/min at room temperature. The KoalaDrive and the multitip STM are now commercially available. The size of our microscope is considerably smaller than the size of other four tip STM instruments.[8–11] We present the key characteristics of this microscope, such as drift and mechanical resonance frequencies and first four point measurements performed on silicide nanowires and graphene flakes.

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THE KOALADRIVE MULTITIP STM

The advantage of the KoalaDrive is utilized fully in the design of an ultracompact four-tip STM using the KoalaDrive. The modular design consists of four identical units. One of these units is shown in Fig. 1(b). Each unit consists of a KoalaDrive used for the coarse tip-approach towards the sample. The tip is mounted under 45° relative to the vertical direction in order to allow for the positioning of the tip apex to the same region as the ends of the other tips. The KoalaDrive is fixed to a plate which is moved according to the design of the beetle STM.[12] The plate rests on three balls fixed to three tube piezo elements. Saw tooth signals on these piezo elements allow for an inertial motion (coarse motion) of the plate in the *xy*-directions. The *xyz*-scanning of the tip is also performed by these three piezo elements.

Four of these units are integrated inside a housing of 50 mm outer diameter. A photo of this ultracompact four-tip STM is shown in Fig. 1(a). The whole instrument is build ultrahigh vacuum compatible. The tips and the sample can be changed without breaking the vacuum. With the sample holder placed on top of the housing it is closed completely leading to a good electric shielding from outer disturbances. The sample holder can be moved in *xy* directions over several mm in coarse motion by shear piezo elements on top of the housing. The coarse motion of the four tips and the sample can be observed by an optical microscope from below, or in vacuum by a scanning electron microscope. The view onto the four tips brought within couple of μm together on a lithographically structured test sample is shown in Fig. 1(b). The working distance of the optical microscope

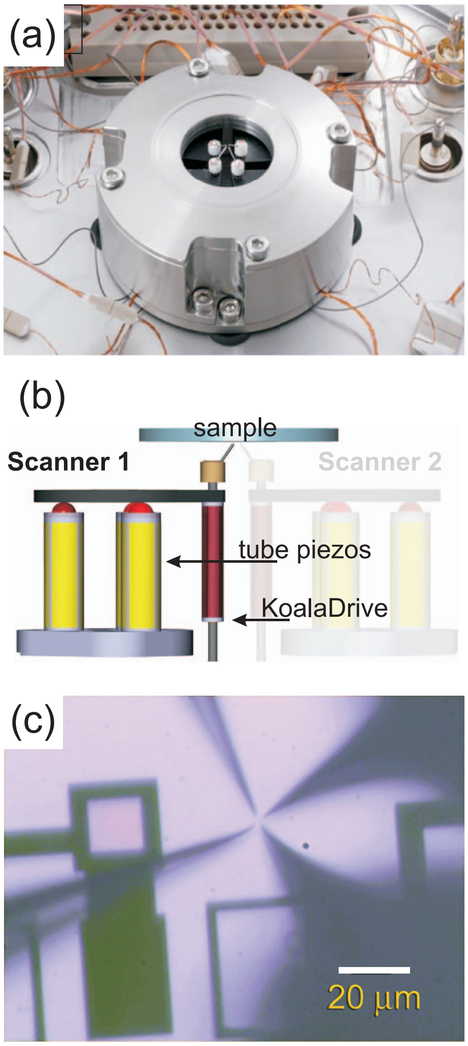

FIG. 1. (a) Photo of an ultracompact four-tip STM with an outer diameter of 50 mm. (b) Sketch of one of the four modular units of the multitip STM. (c) Optical microscope image of the four tips of the ultracompact four-tip STM brought together within a couple of μm. The optical microscope views to the tips and sample from below.

is 50 mm. For optically transparent samples, the optical microscope can be mounted from the top decreasing the working distance to the thickness of the sample. Videos showing tip positioning and sample positioning under the control of the optical microscope can be found in the web under www.fz-juelich.de/pgi/pgi-3/koala.

STM images of a Pt(111) single crystal[13] were taken under ambient conditions with all four tips. An example which shows one atomic layer high steps is shown in Fig. 2(a). A STM image of the Pt(100)-hex-R0.7° reconstruction of the Pt(100) surface taken in air is shown in Fig. 2(b) (raw data without vibration isolation). The Pt(100) surface is known to have a surface reconstruction periodicity of 1.4 nm.[14] We use the stripe structure found in the image and the height of atomic steps to calibrate the scanner. The residual drift of the system was measured by continuously taking many scans over a time of several hours and identifying same features (defects) in those images. The *xy*-drift was measured in this way to less than 0.2 nm/min and the *z*-drift to less than 0.1 nm/min under ambient conditions at room temperature in an ordinary lab which was not specifically temperature stabilized.

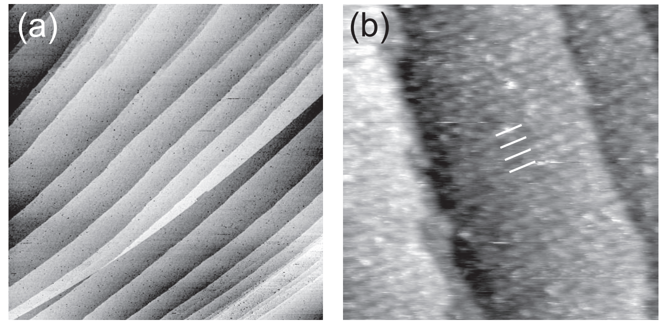

FIG. 2. (a) STM image of atomically high steps on a Pt(111) crystal under ambient conditions (lateral scan size 500 nm × 500 nm). Corresponding images where acquired with all four tips. (b) Image of the surface reconstruction of the Pt(100) surface. The diagonal stripes in the image (highlighted by the white lines) which have a periodicity of 1.4 nm are used to calibrate the STM scanner.

The specifications of the ultracompact four-tip STM are summarized in the following:

- **Coarse tip *xy*-positioning:** type: inertial slider, range: ± 2 mm (each unit).
- **Coarse tip *z*-positioning:** type: KoalaDrive range: ± 5 mm (each unit).
- **Coarse sample *xy*-positioning:** type: inertial slider, range: ± 2 mm.
- **Scanning:** *xy*-range: 6 μm (each unit at RT), *z*-range: 1.5 μm (each unit at RT).
- **Measured lowest resonance frequencies:** *xy*: 2.5 kHz, *z*: 5.5 kHz.

We followed also another independent way to design an ultracompact multitip STM which we call coaxial beetle concept. It is based on the original beetle design.[12] The principle of the design is shown in Fig. 3(b). It consists of four rings coaxially stacked into each other. Each of these planar rings rests on three balls which are attached to the ends of four tube piezo elements. The four rings can be moved laterally according to the beetle principle.[12] The tip-sample approach is performed by a KoalaDrive attached off center to each ring. A photo of the coaxial beetle multitip STM is shown in Fig. 3(a). A SEM column is pointing from the top in order to image tips and sample.

A multitip STM has the disadvantage that only conducting samples can be studied. However, many interesting samples important in nanoelectronics consist of conducting structures on insulating substrates. In order to perform electrical measurements on insulating substrates (e.g., SiO_2) a multitip scanning force microscope (AFM) is required. In the future, we would like to extend our multitip STM to a multitip AFM (atomic force microscope). However, AFM detection method most widely used (the beam deflection detection) is not suitable for this, since four optical systems would have to be adjusted and interference between the four laser beams is likely to occur. For this reason a completely electrical excitation and detection is desirable. This can be achieved by quartz crystal sensors like the tuning fork sensor[15] or the needle sensor. We have chosen the needle sensor, because of its small

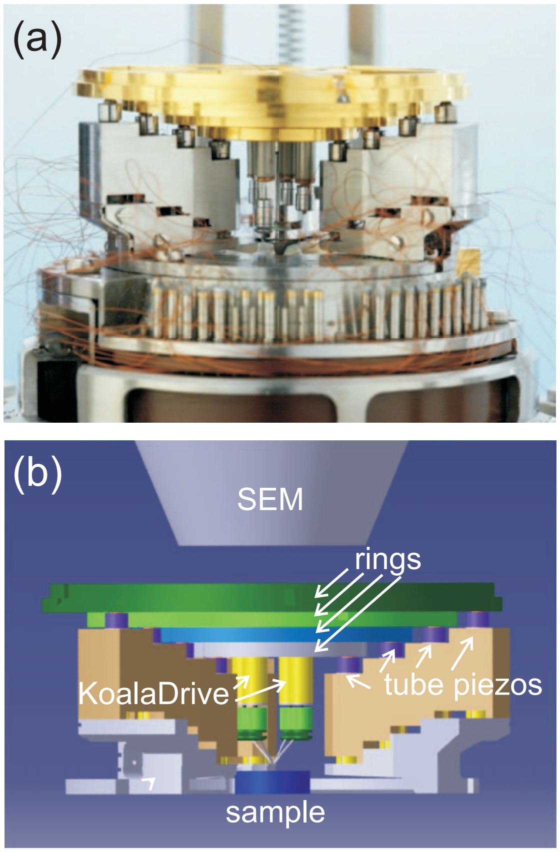

FIG. 3. (a) Photo of the coaxial beetle-type multitip STM. (b) Design principle of the coaxial beetle-type multitip STM with SEM from the top.

footprint which is advantageous in the context of multitip instruments. We have performed tests which showed that the needle sensor can be used to upgrade a STM in an easy way to an AFM.[16,17]

MEASUREMENTS PERFORMED WITH A MULTITIP STM

In the following we report on some measurements performed with the coaxial beetle multitip STM instrument. These results obtained demonstrate a proof of principle for electrical measurements with a four-tip STM.

Making electrical measurements with a four-tip STM is more than to have four tips and to be able to scan with them. Concerted measurements of currents and voltages with all four tips have to be performed on a real-time basis. A typical measurement is performed as follows. Initially, all four tips are scanning in STM mode and positioned to the desired positions at which the electrical measurement will be performed. Then the feedback (e.g., for all four tips) is disabled and the tips are approached towards the sample by a desired distance (or remain in the original position). Subsequently, different I/V ramps are applied between different tips (and/or the sample).

In the simplest case, a current is injected between the two outer tips and a potential difference is measured between the inner tips (classical four point measurement). However, also various kinds of other measurements can be performed, for instance, I/V measurements of every tip to the sample in order to measure the resistance of the contact which has been established by approaching the tip. We usually perform such kind of calibration measurements before and after the actual measurement in order to test the stability of the contacts formed. These different I/V ramps can last altogether 10–20 s and we observe a change in the measured currents of less than 10% for the same measurements performed at the beginning or the end of this time frame. The stability of the electric tip sample contacts established over a measurement period is important in order to obtain reliable results. If all desired voltage ramps are finished the tips are moved back to the original tunneling tip-sample distance and the feedback is resumed. In order to perform such concerted measurements the control electronics of all for tips have to communicate to each other.

Four point measurements at an yttrium silicide nanowire

As a first example, we show a four-tip measurement at an yttrium silicide nanowire. The yttrium silicide nanowires were grown by depositing 0.6 nm yttrium at 1070 K sample temperature. Due to the crystal structure of the Si(110) substrate, the silicide nanowires are aligned along one direction which is vertically in the SEM image shown in Fig. 4. The silicide nanowires have a height of 5–30 nm, a width between 30–50 nm, and a length of several μm. The four tips of the STM are positioned in a line in order to contact one nanowire as shown in Fig. 4.

Unlike in a conventional four point measurement where the two outer probes inject the current and the two inner probes act as voltage probes, we here used only current probes. The principle of the measurement setup is shown in Fig. 5(a) and consists of four current probes which are biased to a certain potential. Technically, they are build by biased STM preamplifiers. The difference between the bias potentials of the outer probes drives a current through the nanowire which is measured by the two (outer) current probes. Before we come to the measurement of the potential by the two inner probes, we consider a possible leakage of the injected current to the substrate.

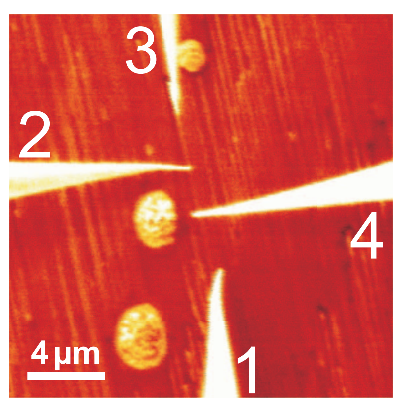

FIG. 4. SEM image of Y-silicide nanowires grown on Si(110). Due to the crystal symmetry of the Si(110) substrate the silicide nanowires are aligned in the vertical direction. The four STM tips are positioned in a line in order to contact one nanowire.

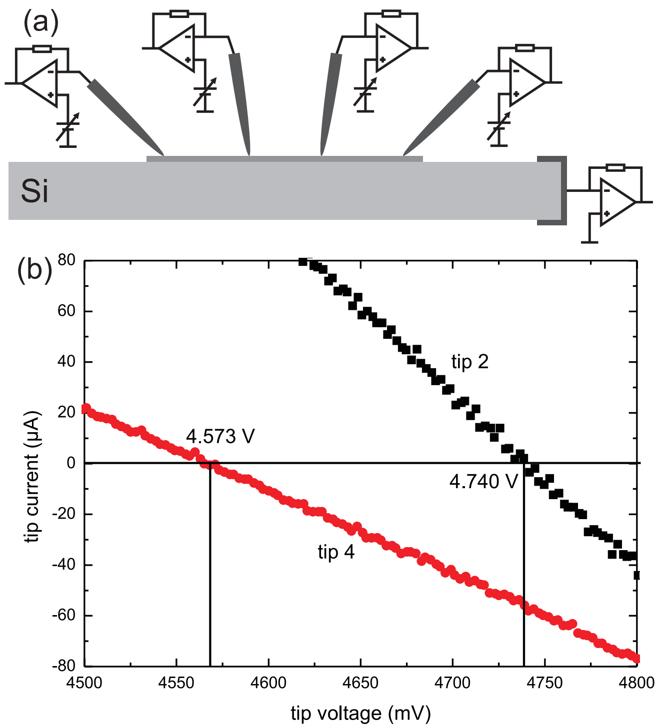

FIG. 5. (a) Principle of a four point measurement using biased preamplifiers as current probes. (b) I/V measurements of tips 2 and 4 in Fig. 4. The voltage at which the current vanishes corresponds to the potential of the tip.

In principle, the current injected by one of the outer probes can run not only through the nanowire as desired, but can also leak to the substrate. In this context, it is important to keep in mind that the interface between silicide nanowire to the silicon substrate forms a Schottky barrier. If this Schottky barrier is reverse biased, no current will flow to the substrate. We confirmed this by measuring the current to the substrate by a fifth current preamplifier Fig. 5(a). If the Schottky barrier was reverse biased, only a negligible current was detected proving that the current runs almost only through the silicide nanowire.

After establishing a current through the nanowire by the outer probes (probes 1 and 3 in Fig. 4), the potential of the inner probes was determined by recording successively I/V curves of tips 2 and 4. The potential at which no current flows corresponds to the nanowire potential at the position of tips 2 and 4, respectively. Technically, tips 2 and 4 are contacted one after the other to the nanowire, and the bias voltage of each tip (2 and 4) was ramped and the current flowing through the corresponding preamplifier was recorded. The voltage for which no current flows corresponds to the potential present on the nanowire at the position of the tip. The two I/V curves were recorded for tips 2 and 4, while a current of 200 μA was flowing through the nanowire is shown in Fig. 5(b). As can be seen from this image, the voltage difference between tips 2 and tip 4 is 167 mV, which results in a resistance of 935 Ω. Taking into account the distance between tips 2 and 4 (2.4 μm), as well as the height (∼15 nm), and the average width (∼50 nm) of the nanowire, results in a resistivity of 26 $\mu\Omega$cm. This value can be compared to a resistivity of about 50 $\mu\Omega$cm measured on thin yttrium silicide thin films.[18,19] This value is also comparable to the resistivity measured before on cobalt silicide nanowires using multitip scanning tunneling microscopes.[20,21]

Four point measurements on graphene

In the following, we present four point measurements performed on graphene exfoliated on SiO_2. The fact that the graphene is located on top of an insulating SiO_2 layer without any outer contacts to the graphene flake makes it difficult to contact such graphene flakes by a multitip STM. Using a SEM the tip can be positioned above the graphene flake, but the distance between tip and sample is difficult to estimate from the SEM images.

Here, we present a method to detect the point of contact between tip and graphene flake using SEM images and a biased tip. Figure 6(a) shows a SEM image in which a graphene flake is imaged with dark contrast on the silicon dioxide substrate. The tip approaching the surface is still not in contact with the flake. If the tip is negatively biased at –10 V, the SEM contrast of the graphene flake reverses to a bright contrast if the biased tip comes into contact with the graphene flake, as seen in Fig. 6(b). The negative potential of the graphene flake relative to the sample leads to an enhanced emission of

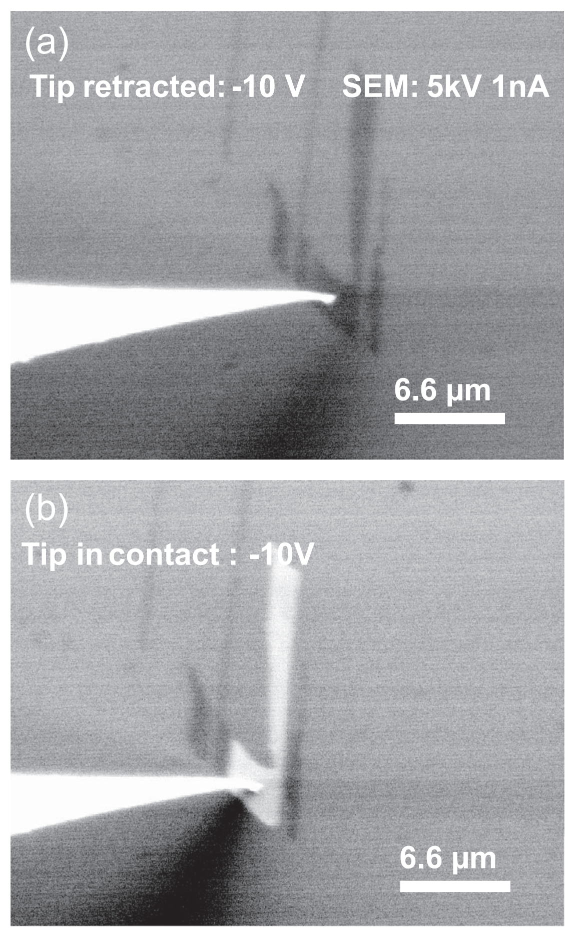

FIG. 6. Contacting of a graphene flake under SEM control using a biased tip. (a) The tip is not in contact with the flake: the flake appears to be darker than substrate. (b) The flake, when contacted with a biased tip, appears to be brighter than the substrate.

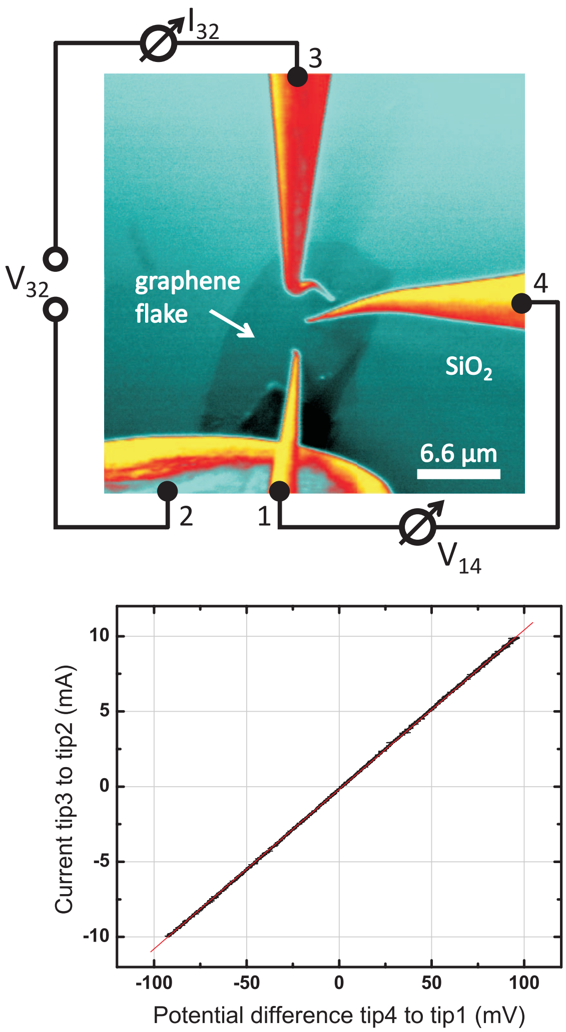

FIG. 7. (a) SEM image of four tips positioned on a multi-layer graphene flake in order to perform a four point measurement. Tip 2 (at the lower left edge of the image) is very much bent. (b) Measured I/V curve during a four point measurement resulting in a sheet conductance of 60 Ω/□.

secondary electrons. Vice versa a positive tip voltage leads to a darker contrast in the SEM image at the point of contact. The method described above allows to contact graphene flakes on an insulating oxide substrate nondestructively by a STM tip.

After the first tip has been brought nondestructively into contact to the graphene flake a bias voltage can be applied to the flake and the other tips can be contacted using the ordinary STM mode approaching the tip to a biased flake. This has been done on several layers thick graphene (graphite) flake shown in the SEM image in Fig. 7(a). Tip 3 and especially tip 2 are bent quite much (from previous experiments) but can be still used to drive a current into the graphene sheet. The potential present at the positions of tips 1 and 4 is measured using these tips as voltage probes, disconnected from the current preamplifiers during the concerted measurement. This conventional four point measurement with the inner tips used as voltage probes results in a I/V curve which is shown in Fig. 7(b). A linear dependence between the measured voltage difference and the injected current is measured. The slope corresponds to a resistance of 9.43 Ω which results in a sheet resistance of 60 Ω/□ taking into account an infinite flake model and the actual distances between the tips. After the measurement of the I/V curve is finished, tips 1 and 4 return to the tunneling position.

CONCLUSIONS

We have shown that the development of a new type of piezoelectric motor serves as the basis for ultracompact scanning probe microscopes. The KoalaDrive can tap its full potential for the miniaturization in the case of multitip scanning probe instruments. We constructed an ultracompact multitip STM with an outer diameter of 50 mm with a drift of less than 0.2 nm/min at ambient conditions. This instrument can be combined with an optical microscope or a SEM in order to navigate the positioning of the tips. We demonstrate the capabilities of the coaxial beetle instrument by four point measurements at an yttrium silicide nanowire and on a graphene flake. Here, concerted measurement processes starting and ending with the tips in tunneling conditions are essential in order to perform nondestructive electrical measurements at the nanoscale.

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[1] J.-L. Lin, D. Y. Petrovykh, A. Kirakosian, H. Rauscher, F. J. Himpsel, and P. A. Dowben, Appl. Phys. Lett. **78**, 829 (2001).
[2] M. Kawamura, N. Paul, V. Cherepanov, and B. Voigtländer, Phys. Rev. Lett. **91**, 096102 (2003).
[3] J. W. Lyding, S. Skala, J. S. Hubacek, R. Brockenbrough, and G. Gammie, Rev. Sci. Instrum. **59**, 1897 (1988).
[4] S. H. Pan, E. W. Hudson, and J. C. Davis, Rev. Sci. Instrum. **70**, 1459 (1999).
[5] Th. Michely, M. Kaiser, and M. J. Rost, Rev. Sci. Instrum. **71**, 4461 (2000).
[6] L. Petersen, M. Schunack, B. Schaefer, T. R. Linderoth, P. B. Rasmussen, P. T. Sprunger, E. Laegsgaard, I. Stensgaard, and F. Besenbacher, Rev. Sci. Instrum. **72**, 1438 (2001).
[7] V. Cherepanov, P. Coenen, and B. Voigtländer, Rev. Sci. Instrum. **83**, 023703 (2012).
[8] I. Shiraki, F. Tanabe, R. Hobara, T. Nagao, and S. Hasegawa, Surf. Sci. **493**, 633 (2001).
[9] O. Guise, H. Marbach, J. T. Yates, M.-C. Jung, J. Levy, and J. Ahner, Rev. Sci. Instrum. **76**, 045107 (2005).
[10] R. Hobara, N. Nagamura, S. Hasegawa, I. Matsuda, Y. Yamamoto, Y. Miyatake, and T. Nagamura, Rev. Sci. Instrum. **78**, 053705 (2007).
[11] T. H. Kim, Z. H. Wang, J. F. Wendelken, H. H. Weitering, W. Z. Li, and A. P. Li, Rev. Sci. Instrum. **78**, 123701 (2007).
[12] K. Besocke, Surf. Sci. **181**, 145 (1987).
[13] B. Voigtländer, V. Cherepanov, Ch. Elsaesser, and U. Linke, Rev. Sci. Instrum. **79**, 033911 (2008).
[14] A. Borg, A.-M. Hilmen, and E. Bergene, Surf. Sci. **306**, 10 (1994).
[15] F. J. Giessibl, F. Pielmeier, T. Eguchi, T. An, and Y. Hasegawa, Phys. Rev. B **84**, 125409 (2011).
[16] I. Morawski, and B. Voigtländer, Rev. Sci. Instrum. **81**, 033703 (2010).
[17] I. Morawski, J. Blicharski, and B. Voigtländer, Rev. Sci. Instrum. **82**, 063701 (2011).
[18] M. Gurvitch, A. F. J. Levi, R. T. Tung, and S. Nakahara, Appl. Phys. Lett. **51**, 311 (1987).
[19] M. P. Siegal, F. H. Kaatz, W. R. Graham, J. J. Santiago, and J. Van der Spiegel, J. Appl. Phys. **66**, 2999 (1989).
[20] H. Okino1, I. Matsuda, R. Hobara, Y. Hosomura, S. Hasegawa, and P. A. Bennett, Appl. Phys. Lett. **86**, 233108 (2005).
[21] S. Yoshimoto, Y. Murata, K. Kubo, K. Tomita, K. Motoyoshi, T. Kimura, H. Okino, R. Hobara, I. Matsuda, S. Honda, M. Katayama, and S. Hasegawa, Nano Lett. **7**, 956 (2007).