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Real-time measurements of sliding friction and elastic properties of ZnO nanowires inside a scanning electron microscope

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ABSTRACT

A real-time nanomanipulation technique inside a scanning electron microscope (SEM) has been used to investigate the elastic and frictional (tribological) properties of zinc oxide nanowires (NWs). A NW was translated over a surface of an oxidised silicon wafer using a nanomanipulator with a glued atomic-force microscopic tip. The shape of the NW elastically deformed during the translation was used to determine the distributed kinetic friction force. The same NW was then positioned half-suspended on edges of trenches cut by a focused ion beam through a silicon wafer. In order to measure Young's modulus, the NW was bent by pushing it at the free end with the tip, and the interaction force corresponding to the visually observed bending angle was measured with a quartz tuning fork force sensor.

1. Introduction

It is well known that the mechanical and electrical properties of single-crystal nanowires (NWs) may be superior in comparison to the corresponding bulk material [1]. Plenty of prototype devices based on NWs have been demonstrated during the last few decades. Individual NWs may be used as sensors, resonance-tunnelling diodes, light-emitting diodes, photodetectors, electromechanical devices and piezoresistors [2–8].

Many methods of investigation of the mechanical properties of either NWs or nanotubes have been developed. Ambient atomic force microscopy (AFM) can be used to vertically load a nanowire suspended over either a hole or a trench to determine Young's modulus and the mechanical strength. This method was applied to Ge NWs and carbon nanotubes (CNTs) [9,10]. The elastic properties and the mechanical strength of SiC NWs and CNTs deposited on a low-friction substrate (MoS_2) and pinned on one end by evaporated metal pads were measured using an AFM lateral force regime [11].

A common way to determine Young's modulus of a NW consists of finding the resonance frequency of a NW fixed from one end and placed inside a scanning electron microscope (SEM) by sweeping the frequency of the external excitation [12]. Another method is based on the lateral bending of the free end of a NW by pushing it with a calibrated contact-mode AFM cantilever, while the NW's second end is fixed on an edge of a rigid substrate. The elastic deformation force is calculated from the visual deformation of the NW and a calibrated AFM cantilever inside the SEM. This method was applied to investigate ZnO NWs [13]. Axial loading or stretching of the NW glued between a rigid substrate and either a calibrated AFM cantilever or between two AFM cantilevers was applied to investigate Si and B NWs, as well as CNTs [14–17]. Analogous axial tensile tests were also performed on ZnO and Si NWs using a MEMS-based nanoscale material testing stage inside a transmission electron microscope (TEM) [18,19]. Real-time force measurement during NW bending was performed by contact-mode AFM inside an SEM to measure Young's modulus of vertically grown arrays of SnO_2 NWs [20].

There are only a few works reporting the measurements of the kinetic friction of a NW on a flat substrate. Manoharan et al. examined the kinetic friction force during the dragging of a ZnO NW parallel to its axis at different loading forces measured by a MEMS-force sensor at ambient conditions [21]. Conache et al. reported the distributed static and kinetic friction of InAs NWs on a Si_3N_4-coated Si wafer based on measuring the curvature of an ultimate NW-bending radius after AFM manipulation in air, where friction was calculated using Young's modulus of a bulk material for calculations [22].

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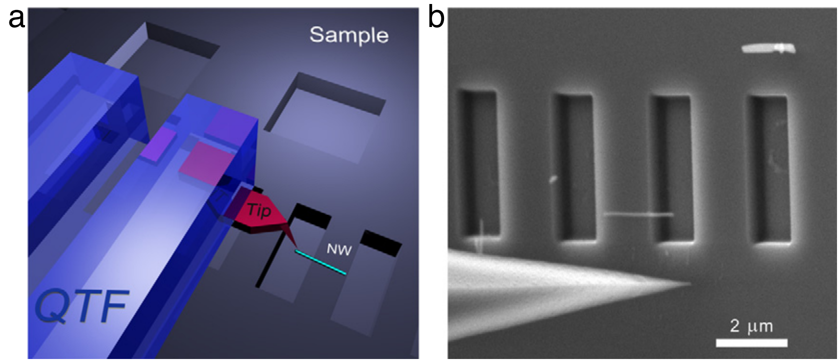

Fig. 1. Schematics of experiment. (a) QTF with the glued AFM tip contacts a NW suspended over a trench on the silicon sample; corresponding SEM image of the AFM tip and FIB cut trenches (b).

In this article, we describe a real-time manipulation technique inside a SEM chamber, which enables the measurement of both the elastic and frictional properties of the same NW on a flat substrate. ZnO NW was translated over a surface of an oxidised silicon wafer using a nanomanipulator equipped with a force sensor composed of a quartz tuning fork (QTF) with a glued AFM tip. The elastic deformation of a translated NW was used to determine the distributed kinetic friction force. The same NW was then positioned half-suspended on edges of trenches cut by a focused ion beam (FIB) on a silicon wafer. To measure the Young's modulus, the free end of the NW was pushed laterally by the AFM tip, and the interaction force corresponding to the visually detected NW bending was measured by a QTF force sensor. No gluing or welding of the NW was performed in these experiments, enabling us to preserve an unchanged NW and making the NW usable for other experiments. Our approach enables us to reduce uncertainties in the measured friction caused by either the use of the bulk value of Young's modulus or the averaged value of the Young's modulus measured on a set of NWs, providing that there is a Young's modulus measured for each particular NW.

2. Experimental

ZnO NWs were grown by a vapour transport method using Au nanoparticles (*BBI international*, 60 nm) as catalysts [23]. NWs were grown on silicon substrates by heating a 1:4 mixture of ZnO and graphite powder to 800–900 °C in an open-end quartz tube for 30 min.

An array of 1 μm-deep trenches sized 3 × 3 μm and 1 × 3 μm was cut by FIB (*FEI* Helios NanoLab) on an Si wafer (50 nm of thermal SiO_2, *University wafers*) (Fig. 1). The wafers were cleaned with RCA-1 solution ("standard clean-1"), followed by 12% HCl, rinsed with distilled water and then blown with nitrogen. The NWs were transferred from the original substrate onto an FIB-patterned wafer surface using a piece of cleanroom paper (Fig. 1(b)).

The tip of the *AdvancedTEC* AFM probes (*Nanosensor* ATEC-CONT cantilevers $C = 0.2$ N/m) used in the experiments was tilted about 15° relative to the cantilever, providing tip visibility from the top. The cantilever was glued with conductive silver epoxy (*Agar Scientific*) to one prong of the QTF (*Elfa*, nominal resonance frequency at 32.768 kHz) forming a force sensor working in the shear oscillating regime (the tip oscillates parallel to the sample surface). To make the QTF response faster, the Q-factor was reasonably decreased by putting a small drop of epoxy resin (Ecobond 286, *Emerson & Cuming*) onto the opposite prong of the QTF. The force constant of cantilevers glued on the QTF was estimated using the Cleveland formula to be 10–20 N/m [24].

The signal from the QTF was amplified by lock-in (SR830, *Stanford Research Systems*) and recorded through the ADC-DAC card (*National Instruments*). The typical values of the driving voltage were 10–30 mV. The force sensitivity of the QTF was calibrated on precalibrated cantilevers (FCL, *AppNano* and CSG11 $C = 0.03$–0.1 N/m, *NT-MDT*) inside the SEM similar to the procedures described in [25,26].

The QTF force sensor was mounted on a 3D nanomanipulator (SLC-1720-S, *SmarAct*) and installed inside the SEM (Vega-II SBU, *TESCAN*) with a typical chamber vacuum of 3×10^{-4} bar. The nanomanipulator enables two types of movement; in the scan regime, the movements are made by either the gradual expansion or contraction of the piezo-nanomanipulator, allowing the force sensor to move smoothly. In the step regime, movements are made in a series of gradual expansions of the piezo-nanomanipulator, followed by abrupt slips achieved via a sawtooth signal sent to the piezo-positioner. Special software was developed to control the nanomanipulator and record simultaneous signals from the nanomanipulator's position sensors and signals from the lock-in amplifier.

We would like to stress some of the advantages of using a QTF with a glued AFM tip as a probe and force sensor compared to the application of a soft AFM cantilever to investigate NW mechanical and frictional properties inside the SEM [13–17]. The QTF provides the real-time data flow of NW-tip interaction with high time and force resolution. Moreover, QTF force sensitivity can be tuned in a wide range by the variation of the applied driving voltage. A high force constant (10–20 N/m) of the AFM cantilever glued to the QTF force sensor enables the easy manipulation of either highly adhered NWs or nanoparticles on the substrate surface [27], which may be problematic if soft AFM cantilevers are used as probes [28].

3. Results and discussion

NWs of suitable lengths (in the order of a few μm) and situated in the proximity of the patterned area were chosen and moved by the AFM tip toward trenches cut by the FIB. To increase loading during the NW translation and to ensure that the tip would not slide over the NW, the force sensor was lowered another 1–2 μm after the tip came into contact with the substrate surface. The oscillation amplitude dropped to zero due to the high repulsive force, and no force measurement was performed during the NW translation.

When the NW is pushed at its midpoint and has travelled over a few microns, it bends into an arc due to the distributed kinetic friction force acting along the NW's length (Fig. 2). The NW's characteristic shape remains constant during the translation due to the fact that the total kinetic friction force acting on the NW is balanced with the external force applied by the tip. The determination of the distributed kinetic friction for too-short NWs was problematic due to the large radius of the curvature during the translation. The minimal length suitable for the determination of kinetic friction depends on the NW's diameter, and was usually at least 2 μm in our experiments.