The 2010 IEEE/RSJ International Conference on
Intelligent Robots and Systems
October 18-22, 2010, Taipei, Taiwan

Tuning Fork based in situ SEM nanorobotic manipulation system for Wide Range mechanical characterization of ultra flexible nanostructures

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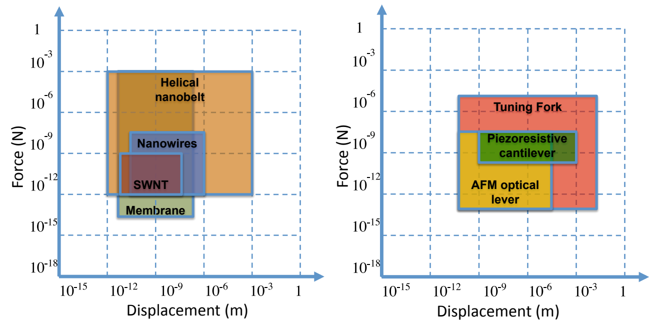
Abstract— **In this article, a nanorobotic manipulation system under Scanning Electron Microscope (SEM) is developed for mechanical property characterization of ultra flexible nanostructures. Frequency modulated quartz tuning fork is proposed as gradient force sensing. Helical Nanobelts (HNB) were used as example to demonstrate the capabilities of the proposed system. The stiffness of HNBs were obtained in full tensile elongation experiments, ranging from 0.009 N/m at rest position to 0.297 N/m at full elongation before breaking with a resolution of 0.0031 N/m. The non-linear behavior of the HNB's measured stiffness is clearly revealed for the first time in full range. Furthermore, the stiffness could be transformed into force measurement that ranges from 14.5 nN to 2.96 μN.**

Index Terms— **Nanomanipulation, tuning fork force sensing, frequency modulation atomic force microscopy, helical nanobelt, Scanning Electron Microscope.**



(a) Ultra flexible nanostructures (b) Force sensors

Fig. 1. Force vs displacement diagram

I. INTRODUCTION

Recently, ultraflexible and elastic micro/nano structures as building blocks to create NanoElectroMechanical Systems (NEMS) are being synthesized, these devices are mainly limited to laboratory prototypes thus not yet commercialized mainly due to the manufacturing challenges and not well-known properties. For both of these problems, precise knowledge on mechanical properties of these nanostructures is inevitable. As an example, three-dimensional (3-D) nanohelixes are inspired from nature to have complex mechanical properties (deoxyribonucleic acid (DNA), protein, cell, or tissue in nature[1]). As inorganic nanohelixes, the electrical and mechanical properties of SiGe/Si/Cr and SiGe/Si Helical Nanobelts (HNBs) were recently characterized separately through experiments and simulations [2]. The fabrication and mechanical characterization of InGaAs/GaAs HNBs have been also described [3]. Their excellent flexibility provides new avenue for fabrication of ultra-small force sensors with high resolution as depicted in Figure 1a. The displacement of these nano helixes was detected by recently developed both visual recognition [4] and piezoresistive smart sensing mechanism [5]. However their mechanical properties were studied only in the limited displacement region mainly due to the lack of proper calibration tools which can study full range mechanics [2][3]. Therefore, mechanical calibration system is necessary.

As conventional force calibration tools, Atomic Force Microscope (AFM) [6], piezoresistive cantilever [7], capacitive force sensor [8] and other MicroElectroMechanical Systems (MEMS) [9] have mostly been utilized. However their sensing resolution and range are also limited as depicted in Figure 1b. They are not sufficient to characterize the full range of HNB mechanics although it is essential to their device applications. Our approach is to use tuning forks, they have been widely used in watch industry. Recently it has also been mainly utilized as force sensors for imaging and manipulating matter under scanning probe microscope [10][11] and to function as force sensor inside SEM from their advantage of simple read-out system by replacing laser optics [12]. However these last approaches were mainly limited to integrating AFM imaging resolution in addition to SEM imaging. Tuning fork based nanomanipulation system for mechanical characterization inside SEM has not been attempted yet.

Therefore, we aim to develop 3-D nanomechanical property characterization system with large range and high resolution force sensing. The 3-D characterization based on the developed sensor is achieved by integrating in situ SEM for accurate visual detection, and nanomanipulation system with 3 degrees of freedom and nanometer positioning resolution. Toward this goal, we propose tuning fork based force sensor with large range, high resolution. The performance of the proposed system is proved by full range tensile elongation study of HNB.

The proposed system can be further extended to contribute to mechanical property characterization of other ultra flexible nanostructures (nanowires, carbon nanotubes, helix, graphene membranes etc.). This will bring them closer to their NEMS applications. Furthermore, flexible

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978-1-4244-6676-4/10/$25.00 ©2010 IEEE

and elastic biological nanostructures such as DNA, proteins, cells, tissues are also in the scope by incorporating environmental electron microscopes (ESEM) or fluorescence optics.

This article is divided in five sections. In the section II, the basics of tuning forks force gradient sensing (FM-AFM method) and its application on HNB are explained. Then, in the section III, the preparation of experimental setups to measure the force based on the FM-AFM method are explained. In section IV results are gathered and show that this system clearly reveals the non-linear stiffness behavior of InGaAs/GaAs HNB by full range tensile elongation study. The system is proved and demonstrated to be an useful mechanical property characterization tool for ultra flexible nanostructures. Finally, in section V, conclusion and discussion are presented.

II. Principles of force gradient measurement with tuning fork for HNB characterization

This section is divided in three parts. First, the principles of force gradient measure with tuning fork are presented. Second, HNB model and the mechanical properties measured from previous works are presented. Finally an integrated model for tuning fork and HNB attached is presented.

A. Principle of tuning fork force sensing

Measuring force with a tuning fork can be done with either amplitude and phase modulation (AM/PM) or frequency modulation (FM) AFM techniques. For the first a lock-in amplifier is utilized in order to separate amplitude and phase from the original signal. From these two signals the force can be obtained [13][14]. For the second, an Automated Gain Controller (AGC) and a Phase-Locked Loop (PLL) controller are used to control respectively amplitude and phase, thus frequency shift is obtained. With the frequency shift the gradient of the force can be obtained [15][16]. Choosing AM or FM depends mainly on the measurement environment. In vacuum, the quality factor is higher than in the air, furthermore, AM-AFM regulation highly depends on reaction time $\tau = Q/(\pi \cdot f_0)$ (where f_0 is the resonant frequency and Q the quality factor) of the tuning fork, which will limit the bandwidth analysis if the quality factor Q is very high. FM-AFM removes the time constant dependency [17] of the analysis allowing us to have wide bandwidth with high quality factor which make it our primary selection.
The tuning fork frequency shift can be expressed as [18] :

$$\frac{\Delta f}{f_0} = \frac{1}{A \cdot K_{TF}} \int_0^{1/f_0} F_{int}\left(\omega \cdot t\right) \cdot cos\left(\omega \cdot t\right) dt \qquad (1)$$

Where K_{TF} is the stiffness of the tuning fork, A the tuning fork mechanical oscillation amplitude, F_{int} the interaction force between the tip of the tuning fork and the sample, f_0 the resonant frequency of the tuning fork and Δf the frequency shift. ω is the angular frequency of the tuning fork.

The stiffness of the tuning fork was obtained using a geometrical model. The tuning fork dimensions can be measure either with a microscope or a SEM, thus using a geometric method is feasible (equation 2 where $E = 78.7GPa$ being the Young modulus of the quartz crystal of tuning forks).

$$K_{TF} = \frac{E \cdot w \cdot t^3}{4 \cdot l_1^3}. \qquad (2)$$

In equation 2, w, t and l_1 are the with, height and the length of tuning fork prong (geometrical parameters of tuning fork can be seen on Figure 6). The stiffness of the tuning fork can be assumed to be constant [11].

To demonstrate the performance of the system, a full range tensile elongation study of HNB is proposed. In the following, the modeling of HNB is presented.

B. Modeling of helical nanobelts

InGaAs/GaAs bilayer HNBs were utilized for the experiments. HNBs were fabricated by the process described in [5]. Finite element method (FEM) simulation was used to estimate the deflection by the applied force onto HNBs, thus, obtain the rest position stiffness. The dimensions of the HNBs used are summarized in Table I. The simulation result has previously been validated with experimental results for similar structures [3]. Simulation was carried out in the linear elastic range (small displacements). Values of the materials properties in the model were taken from [3] with the rule of mixture applied for the InGaAs layer. Both ends of the helix were constrained from rotation around all three axes and one was constrained from all translational movements. From the simulation, longitudinal rest stiffness of the structure is determined to be 0.009 N/m. Furthermore, non-constant behavior of the stiffness for upper elongation range was demonstrated by previous works [3] with atomic force microscope cantilever under SEM. Full range was measurement was not attempted due to the lack of wide range force sensing.

Once the stiffness for rest position of the HNB is obtained, the interaction force F_{int} at the end of the HNB can be expressed with Hooke's law as follows :

$$F_{int} = K_{HNB} \cdot x \qquad (3)$$

Where K_{HNB} is the stiffness of the HNB and x it's elongation. In the following, an unified model including tuning fork and HNB is presented.

TABLE I
HNB specifications

Thickness of InGaAs/GaAs (nm)	11.6/15.6
Length (μm)	25.4
Pitch (μm)	3.9
Number of turns	6.5
Stripe width (μm)	1.5
Diameter (μm)	2
Longitudinal spring (FEM) $K_{long}(N/m)$	0.009

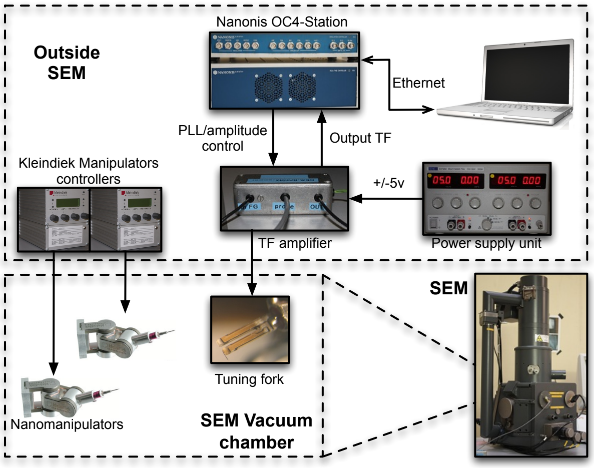


Fig. 2. Experimental setup block diagram.

C. Integrated model for tuning fork and HNB

The elongation of the HNB for the integrated system can be expressed as :

$$x = A \cdot cos\,(\omega \cdot t) + \Delta x \tag{4}$$

Where $A \cdot cos\,(\omega \cdot t)$ is the oscillation amplitude of the tuning fork prong and Δx the linear elongation of the HNB.

In the unified model the tuning fork tip is in contact with the end of the HNB, in consequence, the interaction force F_{int} is the same for both. Replacing model of the HNB (Eq. 3) and Eq. 4 the frequency shift of the tuning fork (Eq. 1) results in Eq. 5 where the stiffness of the HNB can be obtained in terms of the resonant frequency of the tuning fork, the frequency shift and the stiffness of the tuning fork.

$$K_{HNB} = \frac{2 \cdot \Delta f \cdot K_{TF}}{f_0} \tag{5}$$

As A, K_{TF} and f_0 are constant, it is noticeable from previous equation that a frequency shift will only show non-constant and non-linear behaviors of the stiffness of the HNB.

III. Tuning Fork Force Sensor Calibration Setup

A. Hardware configuration

For the oscillation control of the tuning fork and data acquisition, a OC4-Station from SPECS-Nanonis was used. This station has the advantage of having a lock-in amplifier, PLL, AGC, data acquisition hardware and software and real time operating system. The electronic preamplifier for the tuning fork was specially designed for use in SEM imaging conditions. A forthcoming article will describe in details this electronics. A TT*i* EX752M multi-mode Power supply unit was used with fixed +/-5v for the tuning fork electronic preamplifier. The detailed experimental setup block diagram is shown in Figure 2. The main advantage of this setup is that all the electronics for tuning fork and the manipulators are outside the SEM chamber, avoiding influence from electron beam and space occupation. For vacuum and visual feedback a Leica stereoscan 260 of cambridge instruments SEM is used. Two Kleindiek (MM3A-EM) nanomanipulators where used for HNB attachment to tuning fork, this procedure is described in section III-C. Each has 3 degrees of freedom and respectively 5nm, 3.5nm and 0.25nm resolution at the tip in X, Y and Z axis of the inertial Frame. Each axis is actuated with piezo stick-slip principle and is controlled via open loop piezo controller. Configuration of the manipulators and tuning fork inside the SEM can be seen in Figure 3

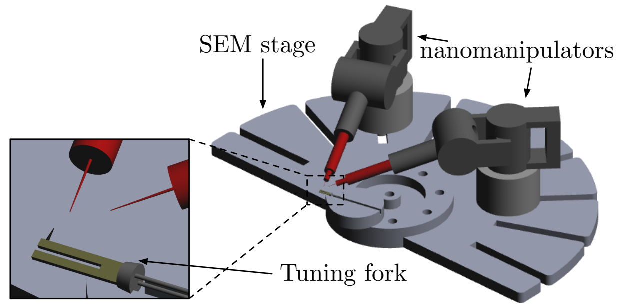


Fig. 3. 3D cad model of experimental setup

The tuning forks used were manufactured by Citizen America - CFS206 32.768KDZB-UB. For manipulation of the cantilever and HNB a tip is attached to the tuning fork, this will amplify the pressure by reducing the contact area of the tip, thus increase the resolution. Tips where glued to the tuning fork with conductive EPOTEK glue for grounding the tip 4. Picoprobes, tungsten tips (GGB industries, T-4-10-1 mm, tip radius : 100 nm) and tips made with platinum iridium Pt90/ir10 wires where used for the nanomanipulator and tuning fork respectively. In the following, the preparation of tuning fork as well as the procedure for attaching tips to the tuning fork is presented.

B. Tuning fork with tip preparation

Several things have to be considered before adding the tip. The quality factor of the tuning fork should remain as high as possible to obtain the best resolution. It is based on the weight balancing between the two prongs. Any weight added on one of the prongs should be compensated in the other one to avoid decreasing the quality factor. As shown in Fig 4, for grounding with prong of the tuning fork, conductive glue is used to attach the tip, thus avoid charging by electron beam inside the SEM. Glue needs to be added for weight compensation on the other prong of the tuning fork, it can be done with both conductive and non conductive glue. Using conductive glue has the advantage charging avoidance by electron beam, however it will increase the risk of short circuiting the electrodes during deposition. Nevertheless, as the electron beam is mainly focused and zoomed to the tip of the tuning fork, the other prong of the tuning fork has reduced risk of charging. The geometry information and the estimated stiffness of TF are summarized in Table II.

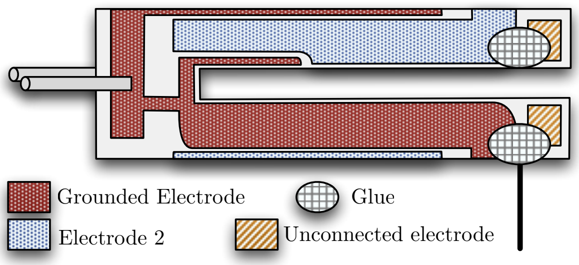
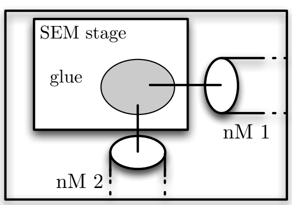


Fig. 4. Schematic of Tuning Fork electrodes with tip and glue

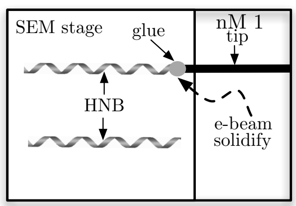
TABLE II
TF SPECIFICATIONS

Resonant frequency $f_0(Hz)$	28325.524
Stiffness $K_{TF}(N/m)$	7936
Quality factor	11145
Prong length $l_1(\mu m)$	3204
Prong heigh $t(\mu m)$	382
Prong width $w(\mu m)$	238

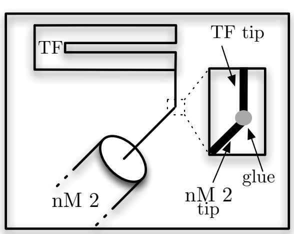
C. Assembly of HNB and tuning fork



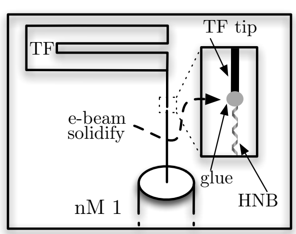
(a) Adding glue to the tips of the two nanomanipulators



(b) Picking HNB from subtract with the nanomanipulator tip and solidifying of glue with e-beam



(c) Adding glue to the tuning fork tip



(d) Attaching and soldering of HNB to tuning fork

Fig. 5. Protocol for HNB attachment between the tuning fork tip and manipulator tip. nM and TF stands for nanoManipulator and Tuning Fork respectively.

Once the tip is attached to the tuning fork, the next step consists in assembling the HNB and the tuning fork. As tuning fork is very sensitive to motion, it was mechanically fixed on top of the SEM, thus the HNB is attached between the tuning fork for force sensing and the nanomanipulator for motion. As seen in Figure 5 the assembly is divided in 4 parts :

1) First, the tips of the 2 nanomanipulators are dipped into EPOTEK glue (Fig. 5.a).
2) Then, the tip of nanomanipulator 1 is approached to contact the end of the desired HNB. The SEM electron beam is focused onto the glue to solidify it (Fig. 5.b).
3) After, glue is added to the tip of the tuning fork with nanomanipulator 2 (Fig. 5.c).
4) Finally, the HNB attached to nanomanipulator 1 is approached to contact the tip of the tuning fork. The SEM electron beam is used to solidify the glue (Fig. 5.d).

IV. EXPERIMENTS AND RESULTS

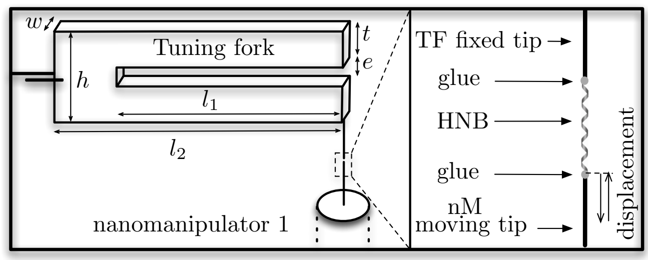
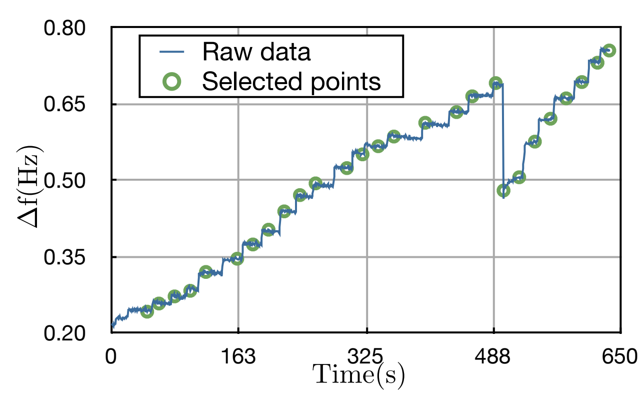


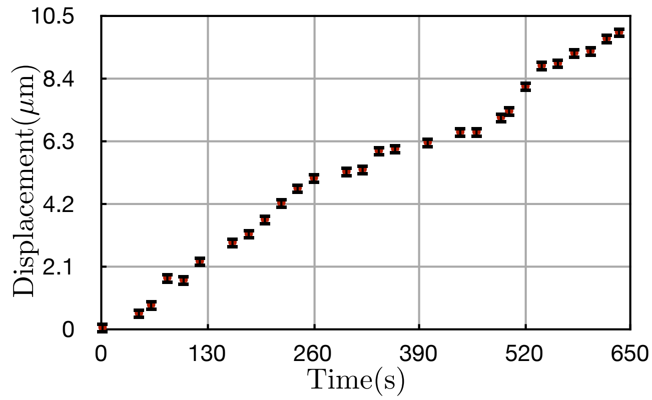
Fig. 6. Longitudinal pulling configuration

For the experiment the nanomanipulator is moved by steps in the longitudinal direction of the HNB (which is aligned with the tuning fork and nanomanipulator tips), the procedure is described in fig. 6. The movement of the tip of the nanomanipulator generates a force in the axial direction of the HNB which elongated it until it breaks. This way full range stiffness characterization can be obtained. Tuning fork frequency shift is recorded during the entire experiment, raw data as selected points for the experiments are gathered in Figure 7.a. This Figure stands the fact that the noise for frequency shift (estimated at $5mHz$) is much lower than the frequency shift due to the smallest motion step. As the manipulators have no position feedback, the displacements are estimated from the SEM recorded video at 33 Hz frame rate. Due to the quality of SEM real time visual feedback automatic detection of nanomanipulator and tuning fork tip position is not accurate, thus the relation between time and HNB elongation (Fig. 7.b) is obtained manually inducing an error of 0.2μm for each measure. Under the assumption of constant stiffness of the tuning fork [11] with equation 5 frequency shift results are transformed into stiffness of the HNB (Fig. 7.c). In addition, with Hooke's law and stiffness result, the force was obtained (Figure 7.d))

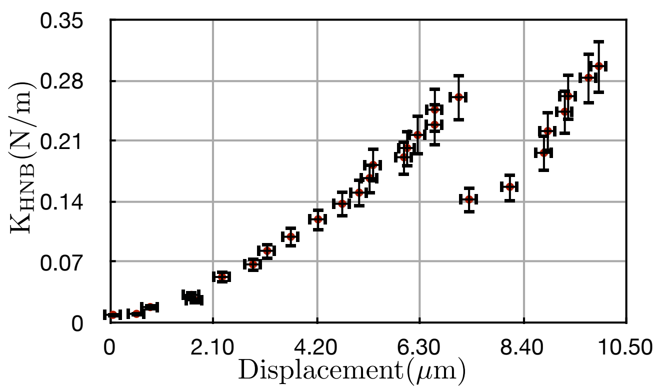
During the motion of the nanomanipulator, different geometrical configurations of the HNB stand out, these are gathered in Fig. 7.e. At the beginning of the experiment, the HNB is in rest position and the pitch looks homogeneous (Fig. 7.e.1). The stiffness of the HNB for this position was obtained with finite element simulation. To obtain the experimental stiffness of the HNB for the initial position, the difference between tuning fork resonant frequency before and after HNB attachment needs to be obtained. However one of the main problems for this measurement was that the vacuum condition of the SEM improved during time making



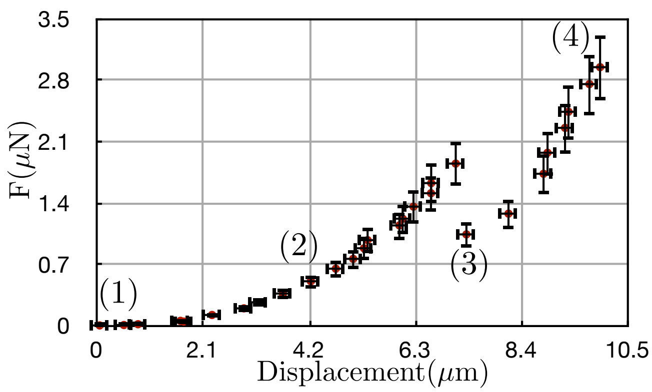
(a) Frequency shift from PLL and amplitude controller



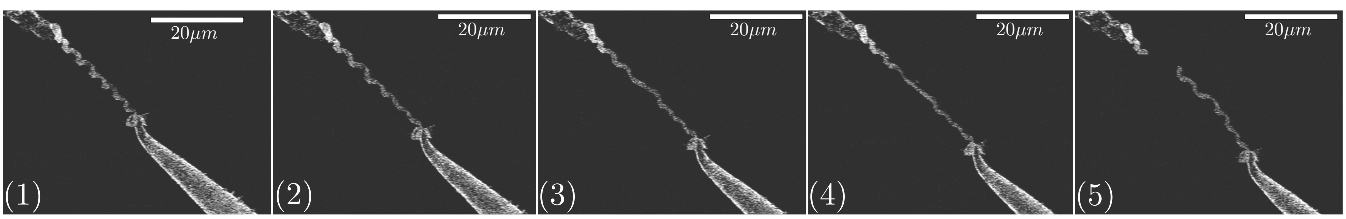
(b) Displacement from SEM visual feedback



(c) HNB longitudinal stiffness.



(d) Force.



(e) Captures from SEM video. (1) HNB prior to pulling, (2) HNB during pulling, (3) HNB unrolling, (4) HNB before break, (5) HNB after break

Fig. 7. Full range longitudinal pulling

the resonant frequency increase constantly. The order of magnitude of frequency shift due to vacuum improving is the same of frequency shift due to HNB attachment.

After, the HNB begins to elongate (Fig. 7.e.2). This geometrical configuration of the HNB clearly shows that the HNB pitch is not homogeneous, at least 3 different pitches were observed. This implies that the spring start to behave in this range as a composition of at least 3 different springs. In consequence, the gradient of the stiffness will decrease, attenuating the nonlinear behavior of the stiffness of the HNB. This is mainly due to the rotation constraint imposed to the HNB with the gluing

Further elongation increases the pitch differences in the HNB till one part of the HNB unrolls at around 7.3μm displacement at 500 seconds (Fig. 7.e.3). In consequence, there is a release of strain in the HNB that is reflected in a drop of frequency shift, stiffness and force (Figure 7.a, c and d). At this point, one section of the HNB is no longer a spring.

Finally, the HNB is elongated until it's almost completely unrolled and damaged just before breaking (Fig. 7.e.4). After that the HNB breaks (Fig. 7.e.5). The contact between the tuning fork tip and HNB remains after breaking to assure attachment process.

These results confirm the non-constant stiffness behavior of HNB in full range elongation. This behavior was not clearly measured for displacement of less than 10μm in previous works [3] with atomic force microscope cantilevers inside the SEM. Furthermore, the non-homogeneous pitch of this HNB has been revealed by constraining the rotation during elongation. The resulting elongation force shows a highly non linear behavior which goes from 14.5nN for the smallest step done to 2.96μN, showing the wide range sensing of the system.

TABLE III
SYSTEM PERFORMANCE SPECIFICATIONS

Degrees of freedom of manipulator	3
Manipulator resolution in x/y/z (nm)	5/3.5/0.25
Frequency shift resolution (Hz)	0.005
-> Corresponding Stiffness resolution (N/m)	0.0031
HNB rest stiffness FEM (N/m)	0.009
HNB lowest measured stiffness (N/m)	0.018
-> Corresponding Measured elongation (μm)	0.81
-> Corresponding force (nN)	14.5
HNB highest measured stiffness (N/m)	0.297
HNB highest measured elongation (μm)	9.95
-> Breaking force (μN)	2.96

V. CONCLUSIONS

In situ SEM tuning fork based robotic system for dynamic mechanical characterization of ultra flexible nanostructures is presented. The system composed of two nanomanipulators and a fixed tuning fork is used to fix an helical nanobelt between the tips of one manipulator and the tuning fork. The non-constant stiffness behavior of helical nanobelts during their controlled tensile elongation was clearly revealed in full range for the first time. The obtained stiffness of helical nanobelt ranges from 0.009 N/m to 0.297 N/m during full elongation with a resolution of 0.0031 N/m. It was transformed with Hooke's law into forces that ranges from 14.5nN to 2.96μN. The revealed non-linear behavior of the stiffness with SEM visual feedback shows the capability of the proposed system to understand the mechanical properties of the nanostructure due to geometry deformation. Dynamic mechanical characterization of other ultra flexible nanostructures like nanowires, nanotubes and graphene membranes for example are possible in the future with the proposed system. Moreover, the dynamic measurement in addition to the dexterity of the system make it ideal for example for measuring the dynamic oscillation mode of membranes for optical mirror applications.

VI. ACKNOWLEDGMENTS

The authors would like to thank the French Atomic Energy Commission (CEA) Fontenay aux Roses for the SEM and facilities, and the University of Tokyo for allowing us to use the two Kleindiek manipulators with their respective controllers.We thank Guy Blaise for his SEM expertness at INSTN of CEA Saclay. We also thank Jacques Cousty, Christophe Lubin and Laurent Pham Van for the fruitful discussions. This work has been supported by the French National Project NANOROL ANR-07-ROBO-0003.

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