Nano-mechanical Test of CNT-Embedded MEMS Structures by AFM

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***Abstract* – This paper focuses on the analysis of mechanical properties of bundled carbon nanotubes (CNTs) embedded in polymer MEMS mechanical structures using an AFM tip. Our goal is to measure the bulk piezoresistive properties of CNTs and use the CNT as a novel material for MEMS sensors. Experimental results from bending a MEMS-fabricated mechanical bridge embedded with CNTs revealed that the results could be used for testing of piezoresistive force sensors. Furthermore, the results also indicate that the Young's modulus of the CNT embedded micro structure and its bending characteristics can also be determined using an AFM tip.**

I. INTRODUCTION

Since the first observation of carbon nanotubes by Iijima in 1991 [1], physical and mechanical properties of carbon nanotubes are undergoing subsequent intensive investigations because of their extreme minute scale and surprisingly-high reported mechanical strengths. Nanotubes are long, carbon fibres consist of graphite, closed at each end with caps which contain precisely six pentagonal rings. Two major types of carbon nanotubes are: single walled carbon nanotube (SWNT) and multi-walled carbon nanotubes (MWNT). The nanotubes range in length from a few tens of nanometers to several micrometers, and in outer diameter from about $2.5\,nm$ to $30\,nm$.

Several tests for the mechanical properties of single carbon nanotubes have been carried out by several research groups [2]–[5]. On account of their outstanding properties, carbon nanotubes could be used, for example, in nanometer-sized electronics or to strengthen the tensile strength and elastic modulus of composite polymer materials. Several experiments verified that embedded carbon nanotubes lead to effective stress transfer [6]–[9]. Researchers have reported elastic properties for nanotubes that exceed $1\,TPa$ and strengths that are many times higher than the strongest steel at a fraction of the weight. Compared to carbon fibers which typically have Young's modulus of 0.1 – 0.8 TPa [10], the elastic modulus of carbon nanotubes are in the range of 1 – 5 TPa [11].

The study of the nano-scale mechanical properties of surfaces to forces has become possible with the atomic force microscope [12] – [14]. The atomic force microscope has the ability to measure differences in response directly. Many techniques have been developed which take full advantage of the nanometer three dimensional resolution offered by the ultra sharp AFM tip. Traditionally, the AFM has been used to measure the topography of surfaces through direct contact between the surface and a probe tip mounted on the end of a cantilever. AFM images are produced by the scanning of a probe across the surface of a sample using piezoelectric scanners. The high resolution imaging capability of AFM allows accurate localization of indentation sites and measurement of indentation depth. The AFM can also be operated under a force mode in order to perform indentation test. During AFM force mode, the probe tip is first lowered into contact with the sample, then indented into the surface, and finally lifted off of the sample surface. Concurrently, a detection system measures the probe tip deflection. A force curve is produced, which is a plot of tip deflection as a function of the vertical motion of the piezoelectric scanner. This curve can be analyzed to provide information on the local mechanical response [15]–[17].

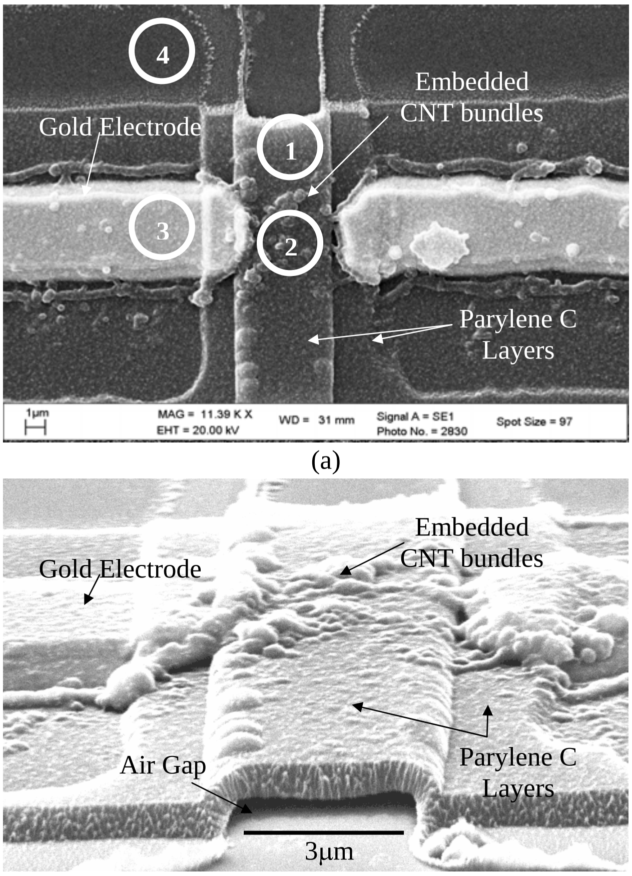
A recent experiment has shown that mechanical deformation can significantly change the electronic behavior of carbon nanotubes [18]. Based on an order of estimate calculation, we project the gauge factor of carbon nanotubes to be about 1000, which is at least an order of magnitude higher than conventional *Si* based strain sensors. Forces and molecules that act upon nanotubes can change their electronic properties, which enable their use as reliable and extremely compact sensors. Motivated by this possibility, our ongoing work is to perform mechanical bending tests of bulk carbon nanotubes embedded in MEMS structures and to examine their bulk mechanical properties.

In this paper, we will show that CNT-embedded micro bridges can be bent repeatedly by using the tip of an AFM. The evaluation of mechanical properties of carbon nanotubes is essential for the design of potentially nanometer-scale piezoresistive sensors. We envision our sensors to eventually sense the input force by the change of conductivity due to mechanical bending of the embedded bundled carbon nanotubes, which are sandwiched by 2 layers of polymer (parylene C) thin films. The fabrication process of our CNT-embedded MEMS sensor was recently reported in [19].

II. EXPERIMENTAL PROCEDURE

A. Fabrication of CNT-embedded structure

The CNT-embedded MEMS structure was fabricated by a standard surface micromachining technology. As reported in our pervious work in [20], bundled CNTs were successfully manipulated across a pair of gold microelectrodes by using AC dielectrophoretic manipulation. In this project, gold electrodes with small gap distance (~3μm) were deposited and the same technique in [20] was used to manipulate the bundled CNTs across the gold electrodes. In order to protect CNTs from contamination, a parylene C polymer thin film was used to embed the CNTs to provide a robust protection of the CNTs for a reliable measurement. Parylene C was deposited through an automatic parylene deposition machine (LabCoater® PDS 2010) and the deposition thickness is depended on the weight of the source parylene C powder. For the CNT-embedded structure used in this paper, the thickness of the parylene C layer is around 0.3μm. As mentioned before, the structure will be potentially used as micro piezoresistive sensors, which have the property of changing their resistance under physical pressure or mechanical work, an air gap under the CNT-embedded structure is fabricated for AFM bending experiments. The experimental details for the bending measurement will be presented in the following section. To provide an air gap under the CNT sensor, a sacrificial layer (Aluminium) was deposited on the substrate and then released in the final stage of the fabrication process. The thickness of the air gap is depended on the deposition thickness of Aluminium and it is the maximum allowable bending distance for the AFM experiment. In this work, the height of the air gap is around 0.5μm and the final structure for the CNT-embedded sensor is shown in Fig. 1.



(b)

Fig. 1. Scanning electron microscopic (SEM) image showing (a) the top view of CNT-embedded MEMS polymer bridge structure and (b) tilted at 70degree

B. AFM bending experiments

The experiments were performed with the Digital Instruments Nanoscope III Atomic Force Microscope. A silicon nitride AFM tip is used to apply a nominal force and to measure the resulting deflection of the CNT structure. For a given material, the appropriate spring contact has to be chosen. Cantilevers made of soft material is insensitive to difference among hard materials, the piezo-scanner displacement is similar to the cantilever deflection, as a consequence, a large uncertainty in the surface deformation. On the other hand, if the cantilever is too hard, the plastic onset is reached for small values of cantilever deflection, implying insufficient statistics in the elastic region.

The nanoindentation technique was applied to the bending test of the MEMS structure embedded with bulk carbon nanotubes as shown in Fig. 1. The applied forces, F_n are given by

$$F_n = k \times d \,, \qquad (1)$$

where k is the spring constant of the cantilever and d is the cantilever deflection. The surface deformation, δ , is calculated from $\delta = z - d$, where z is the displacement of the piezoelectric scanner.

Before the bending test, the sample was scanned in a direction orthogonal to the axis of the CNT beam using contact mode AFM, typically at a rate of 1.0 Hz, so that the CNT-embedded micro bridge was located. The sensitivity of the cantilever was calibrated by acquiring curves of the cantilever deflection versus the displacement of the piezoelectric scanner.

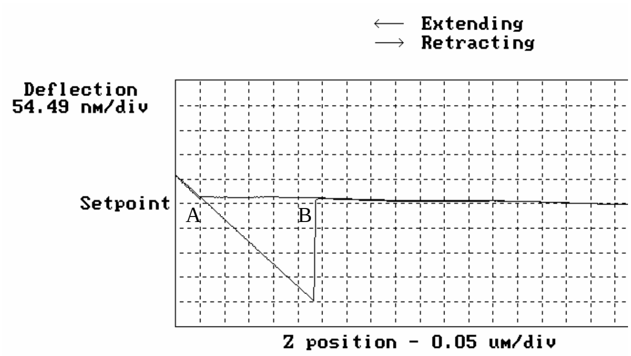


Fig. 2. AFM Force-distance curve

Fig. 2 shows an example of the force-distance curve. The force-distance curves are obtained under the force calibration mode. The horizontal axis shows the travel distance of the piezoelectric scanner and the vertical axis represents the deflection of the tip. As the piezoelectric scanner extends towards the sample surface, the tip deflection voltage remains constant until the probe tip makes contact with the surface due to attractive forces at point A. Just before tip-sample contact is made, the probe tip can be pulled down to the surface by attractive forces, causing a small decrease in the tip deflection voltage. After that, the tip is in contact with the surface and the tip further deflects as the piezoelectric scanner further extends as indicated by the sloped portion of the curve. As the piezoelectric scanner retracts, the tip goes beyond the setpoint and falls into the adhesive regime. Finally, the tip snaps free of the adhesive forces at point B. The horizontal distance between point A and Point B along the retrace line indicates the distance moved by the tip in the adhesive regimen. The adhesive force is obtained by multiplying the distance by the stiffness of the cantilever.

After the scanning, the AFM tip was then positioned above the centre of the micro bridge. The bridge was pushed towards the bottom of the "micro channel" by moving the AFM tip downward. The process was described in the schematic diagram as shown in Fig. 3. There are five load levels used to indent on each particular area of the sample, using the same probe and operating conditions. Each step of AFM tip-movement iteration was $0.139\mu m$ in our experiments.

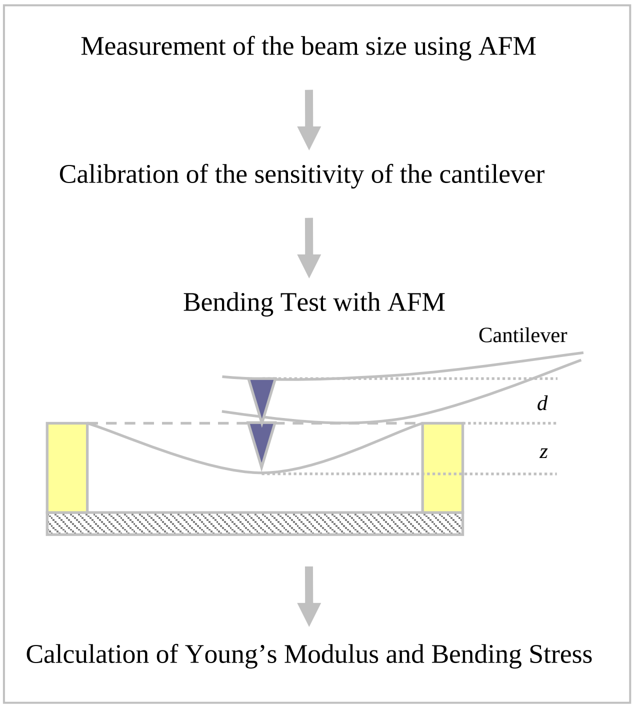


Fig. 3. Schematic diagram of the bending testing of CNT-embedded MEMS structure

The applied force and the maximum vertical displacement of the bridge, z, were calculated from the following equations [21],

$$F_n = ((V_0 + V)/s \times k), \quad (2)$$
$$\delta = z - V/s, \quad (3)$$

where V_0 is the initial different voltage produced by the deflection of the cantilever before the bending test, V is the differential voltage during the test and s is the ratio of the differential voltage obtained from the cantilever's deflection to the displacement of the piezoelectric actuator in z - direction.

Based on AFM surface imaging, the force curves characterize that different material regions on the device can be identified.

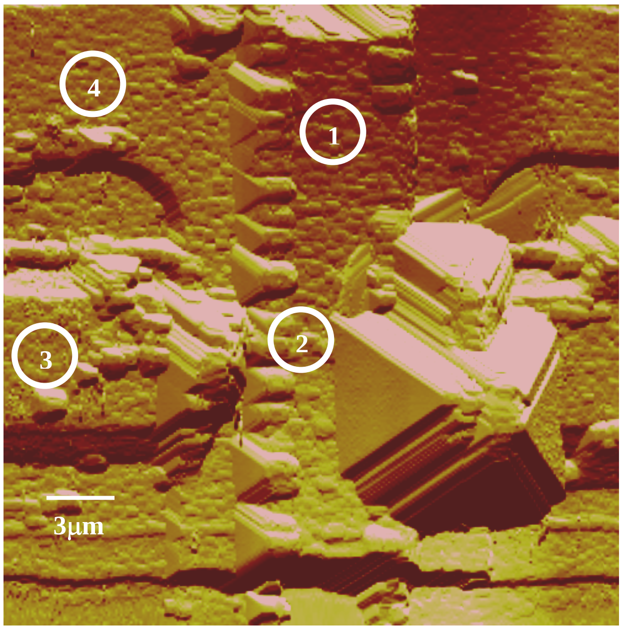


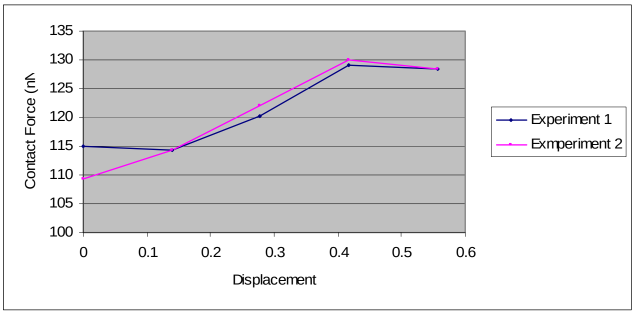
Fig. 4. Specific regions under the nanoindentation test

III. CURRENT RESULTS AND DISCUSSION

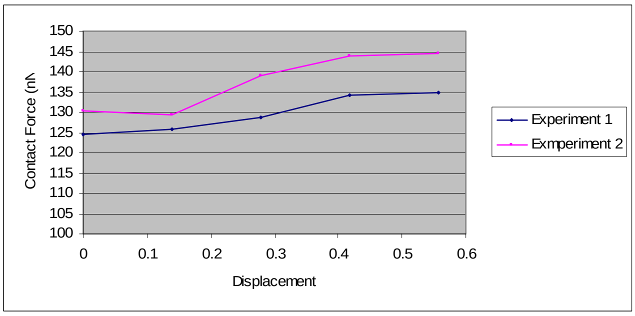
In this experiment, four different regions in the device were being investigated as shown in Fig. 1(a) and Fig. 4. The first region is the two parylene layers, and the second region is two parylene layers with CNTs embedded. Both regions have an air gap underneath. For the third region, it is the parylene layers with gold electrodes on the silicon substrate For the last region, it is only the parylene layer on the silicon substrate.

To test the bending characteristics of the CNT-embedded bridge, the micro bridge was pushed towards the bottom of the trench and then retracted. This experiment was repeated with different downward distance of the AFM tip. Once the bridge made contact with the bottom of the trench, the contact force increased, which allowed us to determine the actual depth of the channel. During the experiment, the AFM cantilever deflection was recorded. The mechanical properties of the micro bridge were calculated by the recorded contact force and the deflection of the bridge. The deflection of the bridge at its centre point was evaluated from the cantilever deflection signal. As the initial tip-surface and the travel range of the AFM tip were controlled, the micro bridge was deflected to various degrees and allowed us to study the effect of mechanical deformation on the electrical properties of the bundled CNT sensing element.

The nominal contact force of the tip and sample was calculated and the results showed that forces applied to various materials such as gold, silicon and Parylene polymer, varied with the displacement of the piezoelectric scanner during continuous pushing. Firstly, the contact forces were found to be different for the same material on different substrates. The contact forces are about $110\,nN$ and $125\,nN$ at the end point and the center point of the CNT bridge, where the forces on the surface of parylene on silicon substrate and on gold layer are $165\,nN$ and $240\,nN$, respectively. This clearly showed that the contact forces vary with the structural composition of the structure that the AFM tip intends to 'push'.



(a)



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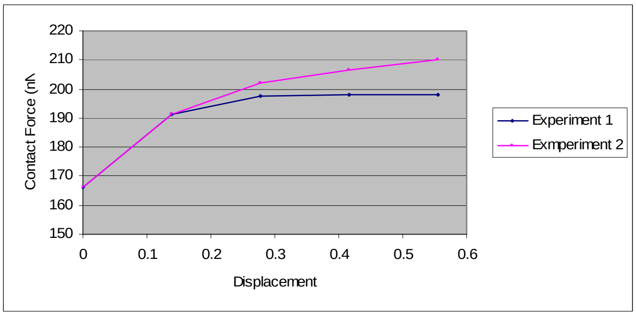

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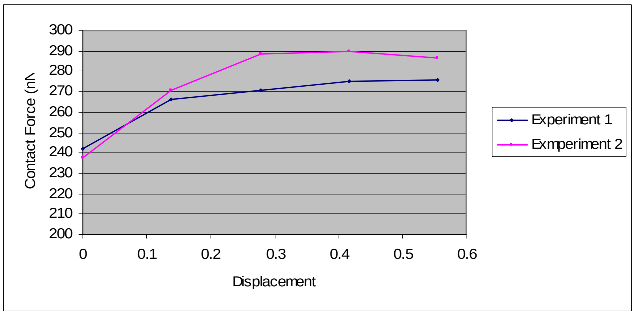

(d)

Fig. 5. AFM contact force data on bending the CNT-embedded MEMS structure with continuous pushing

With the continuous pushing on the sample by the AFM tip towards the surface of the sample, the forces increase with different gradients at different regions. Fig. 5(a) shows that the force increases from 110 nN to 130 nN at the end point of the CNT-embedded bridge. Fig. 5(b) shows that the force increases from 125 nN to 135 nN at the center point of the CNT-embedded bridge. Fig. 5(c) shows that the force increases from 165 nN to 200 nN at the region of parylene on gold surface. Fig. 5(d) illustrates the contact force increases from 240 nN to 280 nN. The variation of the contact force is due to the microhardness of the substrate underneath the top layer of parylene. The contact force increase more significantly on hard surface.

IV. CONCLUSION

AFM nanoindentation technique was evaluated as a viable technique to determine the mechanical properties of polymer thin films. From the results obtained to date, this technique is capable of measuring modulus of thin films down to the submicron levels and gain substantial additional information concerning the true response of a material to indentation at a nanometric scale. The force applied on the surface was controlled by the differential voltage applied to the piezoelectric scanner and the mechanical properties of the cantilever. Changing of the parameters would alter the size and depth of the indent. The nano-scale mechanical properties of the carbon nanotube-embedded structure were investigated and the microhardness of different parts of the device was calculated.

In the next stage, the bending test is applied for further investigation on the Young's modulus and piezoresistivity of the CNT-based sensor. In order to bend the bridge with the AFM tip, a larger scale of nominal force could be applied by the diamond coated cantilever, where the force provided range from 1 μN to 100 μN. Once the bridge made contact with the bottom of the trench, the contact force increased, which allowed us to determine the depth of the channel. During the experiment, the AFM cantilever deflection was recorded. The mechanical properties of the micro bridge were calculated by the recorded contact force and the deflection of the bridge. The deflection of the bridge at its centre point was evaluated from the cantilever deflection signal. As the initial tip-surface and the travel range of the AFM tip were controlled, the micro bridge was deflected to various degrees and allowed us to study the effect of mechanical deformation on the electrical properties of the bundled CNT sensing element.

REFERENCES

[1] S. Iijima, "Helical microtubules of graphitic carbon", *Nature*, vol. 354, pp. 56, 1991.
[2] D. H. Robertson, D. W. Brenner and J. W. Mintmire, "Energetics of nanoscale graphitic tubules", *Phys. Rev. B*, vol. 45, pp. 12592-12595, 1992.
[3] S. Sawada, N. Hamada, "Energetics of carbon nano-tubes", *Solid State Commun.*, vol. 83, pp. 678-680, 1996.
[4] M.R. Falvo, G. Clary, A. Helser, S. Paulson, R.M. Taylor II, V. Chi, F.P. Brooks, Jr., S. Washburn, and R. Superfine, "Nanomanipulation Experiments Exploring Frictional and Mechanical Properties of Carbon Nanotubes", *Microscopy and Microanalysis*, vol 4, 1999, pp. 504-512.
[5] A. Rochefort, Ph. Avouris, F. Lesage and D. R. Salahub, "Electrical and Mechanical Properties of Distorted Carbon Nanotubes", *Physical Review B*, vol. 60, no. 19, November 1999, pp. 13824-13830.
[6] B. I. Yakobson and P Avouris, "Mechanical properties of carbon nanotubes", *Appl. Physi.*, vol. 80, pp. 287 – 327, 20001.
[7] E. T. Thostenson, W. Z. Li, D. Z. Wang, Z. F. Ren, and T. W. Chou, "Carbon nanotube / carbon fiber hybrid multi-scale composites", *J. of Appl. Phys.*, vol. 91, pp. 6034 – 6037, 2002.
[8] D. Hull, *An introduction to composite materials*, 2nd ed. Cambridge University Press, Cambridge, 1996.

[9] C. Bower, R. Rosen, L. Jin, J. Han and O. Zhou, “Deformation of carbon nanotubes in nanotube-polymer composites”, *Appl. Phys. Lett.*, vol. 74, no. 22, pp. 3317 – 3319, 1999.

[10] M. Cadek, B. Le Foulgoc, J. N. Coleman, V. Barron, and W. J. Blau,, “Mechanical and thermal properties of multiwalled carbon nanotube reinforced polymer composites”, *Conference Proceeding of Composite Engineering*, San Diego/CA, U. S. A., 2002.

[11] L. H. Peebles, *Carbon fibers: formation, structure, and properties*, CRC Press, Boca Raton, 1995.

[12] Q. Ma and D. R. Clarke, “Size dependence of the hardness of silver single crystals”, *Journal of Materials Research*, vol. 10, pp. 853-863, 1995.

[13] G.M. Pharr, W.C. Oliver, and F.R. Brotzen, “On the generality of the relationship among contact stiffness, contact area, and elastic modulus during indentation", *Journal of Materials Research*, vol. 7, pp. 613-617, 1992.

[14] G.M. Pharr, "Measurement of mechanical properties by ultra-low load indentation", *Materials Science and Engineering* A, vol. 253, pp. 159, 1998.

[15] N. A. Burnham, S. P. Baker, H. M. Pollock, “Model for mechanical properties nanoprobes”, *Journal of Materials* Research, vol. 15, no. 9, pp. 2006 – 2014, 2000.

[16] M. R. VanLandingham, S.H. McKnight, G.R. Palmese, R.F. Eduljee, J.W. Gillespie, and R. L. McCullough, “Relating elastic modulus to indentation response using atomic force microscopy”, *Journal of Materials Science Letters*, vol. 16, pp. 117-119, 1997.

[17] N. M. Jennett and J. Meneve, “Depth sensing indentation of thin hard films: a study of modulus measurement sensitivity to indentation parameters”, *Proceedings MRS Spring Meeting*, vol. 522, pp. 239 – 244, 1998.

[18] T. W. Tombler, C. Zhou, L. Alexseyev, J. Kong, H. Dai, L. Liu, C. S. Jayanthi, M. Tang and S. Y. Wu, “Reversible electromechanical characteristics of carbon nanotubes under local-probe manipulation”, *Nature*, vol. 405, pp. 769-772, 2000.

[19] C.K. M. Fung and W. J. Li, “Ultra-low-power and hight frequency-response carbon nanotube based MEMS thermal sensors”, *IEEE/RSJ IROS 2003*, invited paper, October 27-31, 2003.

[20] Victor T. S. Wong and Wen J. Li, "Bulk carbon nanotubes as sensing element for temperature and anemometry micro sensing", IEEE MEMS 2003, pp.41 – 44, 2003.

[21] I. Yoshitada, N. Takahiro and T Takeshi, “AFM bending test of nanometric single crystal silicon wire at intermediate temperatures for MEMS”, *IEEE International conference on micro electro mechanical systems*, pp. 135-138, 2001.