

Nanomanipulation of 1-D nanostructures using ECS3030 positioners inside an electron microscope

Rodrigo Bernal, Horacio Espinosa
Mechanical Engineering Department, Northwestern University, Evanston IL, USA

The characterization of 1-D nanostructures such as nanowires and nanotubes has recently become a topic of major interest, due to the potential of these nanostructures to be employed in the next generation of advanced materials and electronic devices. Due to their small characteristic size (<100 nm in diameter) and an increased surface to volume ratio, many nanostructures of a given material display significantly enhanced properties compared to the macroscale, bulk material. For example, zinc oxide (ZnO) nanowires, display a greater modulus of elasticity than bulk for diameters less than ~ 80 nm [1]. Similar behavior has also been discovered for other semiconducting nanowires such as gallium nitride (GaN) [2], and metallic nanowires such as silver [3].

The emergence of these size-effects in the properties of 1-D nanostructures has therefore elicited a need for the characterization and unambiguous measurement of these properties, as they are critical for the development, design and robustness of future applications employing nanostructures as their functional elements. However, the small size of the specimens imposes significant challenges for specimen preparation and testing. To overcome these difficulties, Prof. Horacio Espinosa's group at the Mechanical Engineering Department in Northwestern University, USA, has developed a microelectromechanical system (MEMS) that allows uniaxial mechanical testing of nanowires, with nanometer (nm) and nanoNewton (nN) resolution, therefore allowing the accurate measurement of mechanical and failure properties such as the elastic modulus, yield and fracture strengths [4] (see Figure 1). The system can also be employed to carry out simultaneous four-point electrical measurements as the mechanical straining is taking place, therefore allowing measurements of electromechanical properties of nanowires, such as piezoresistivity and piezoelectricity [5].

A critical step in these experiments is the sample nanomanipulation, where a nanowire must be transferred to the testing MEMS. This delicate procedure requires an instrument with capabilities of nm resolution in displacement, smooth movement to avoid any vibrations that may harm the sample,

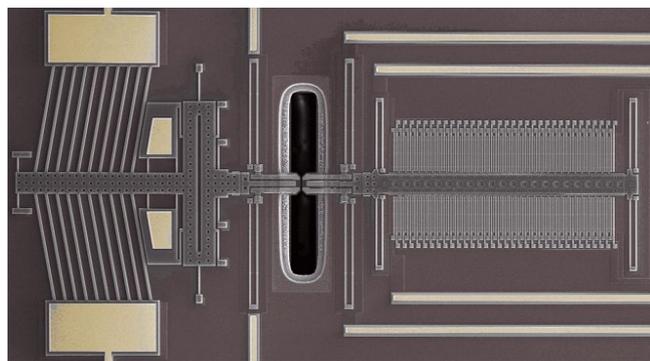


Figure 1: MEMS device for the characterization of electromechanical properties of nanowires.

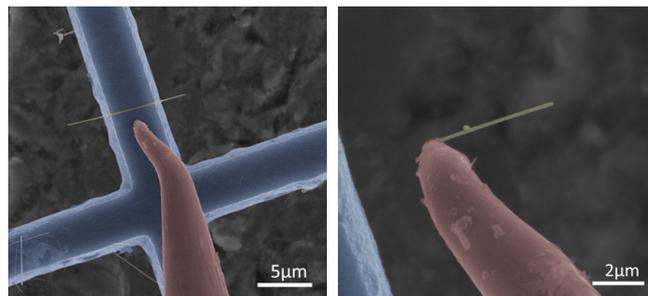


Figure 2: Left side: Silver nanowire (yellow) laying on a copper TEM grid (blue) ready to be manipulated. The tungsten tip (red) attached to the nanomanipulator is used to approach finely to the specimen. Right side: Silver nanowire picked from the grid and ready to be mounted on the device of Figure 1. (Images manually colored for clarity)

and seamless integration with an scanning electron microscope (SEM), to allow observation of the preparation process (an optical microscope lacks the needed resolution). A large range of total movement (> 1 mm) is also convenient to allow flexibility in the setup of the experiment. In recent experiments, the Espinosa group has employed an attocube Nanomanipulator, composed of three stacked ECS3030 positioners, one for each axes of movement, in order to accomplish this task. The nanomanipulator is positioned inside the SEM chamber and interfaced to the ECC100 piezo-controller, located outside the chamber, through vacuum feed-throughs. The controller is connected via USB to a laptop furnished with attocube software for controlling the manipulator.

To perform the nanomanipulation, a sharp tungsten tip is attached to the manipulator (Figure 2, left side). Coarse-steps, of the order of μm , are used to approach the tip to the nanowire. Afterwards, fine steps with magnitudes controllable from sub-100 nm to few nm, are used to establish gentle contact between the tip and nanowire. Once the nanowire is picked (Figure 2 right side), a similar approach is used to position it on top of the MEMS device. Final attachment of the specimen is carried out by electron beam induced deposition of platinum (EBID-Pt) after which the tip is retracted from the area of interest.

References

- [1] R. Agrawal, B. Peng, E.E. Gdoutos, and H.D. Espinosa, *Elasticity size effects in ZnO nanowires - A combined Experimental-Computational approach*. Nano Letters **8**, 3668 (2008).
- [2] R.A. Bernal, R. Agrawal, B. Peng, K.A. Bertness, N.A. Sanford, A.V. Davydov, and H.D. Espinosa, *Effect of Growth Orientation and Diameter on the Elasticity of GaN Nanowires. A Combined in Situ TEM and Atomistic Modeling Investigation*. Nano Letters **11**, 548 (2011).
- [3] T. Filleter, S. Ryu, K. Kang, J. Yin, R. Bernal, K. Sohn, S. Li, J. Huang, W. Cai, and H.D. Espinosa, *Nucleation-Controlled Distributed Plasticity in Penta-Twinned Silver Nanowires*. Small **8**, 2986 (2012).
- [4] H.D. Espinosa, Y. Zhu, and N. Moldovan, *Design and operation of a MEMS-based material testing system for in-situ electron microscopy testing of nanostructures*. Journal of Microelectromechanical Systems **16**, 1219 (2007).
- [5] R.A. Bernal, T. Filleter, J.G. Connell, K. Sohn, J. Huang, L.J. Lauhon, and H.D. Espinosa, *In Situ Electron Microscopy Four-Point Electromechanical Characterization of Freestanding Metallic and Semiconducting Nanowires*. Small **10**, 725 (2014).

H.E. acknowledges funding from US Army Research Office through DURIP award No. W911NF-12-1-0366.