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DETERMINATION OF HARDENING MODULUS OF PLATINUM NANOWIRES

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ABSTRACT

In this study an experimental procedure together with finite element simulation procedure for determining the hardening modulus of Platinum nanowires is described. Platinum (Pt) wires with the nominal diameter of 625 nm are used for this study. The wire with suitable length is taken on a metallic substrate by nano welding just like a micro cantilever. A miniature setup for localized bending of the wire cantilever by using two opposite micro-probes under the continuous observation by a digital microscope is used. In the setup a force sensor, a piezo stage and two nano-manipulators have been accommodated on a single platform. The bending load is applied far from the root of the wire to ensure the failure in the wire across the loading point, and the load-displacement data are recorded. From the load-displacement data the elastic modulus and yield strength of the wire material are directly determined. For estimating the hardening modulus finite element (FE) analysis of the bending problem using the elastic modulus and yield strength with appropriate loading configuration has been performed. The hardening modulus of the Pt wires is successfully determined by fitting the load-displacement relationship obtained in FE analysis to the experimental one.

Keywords: Hardening Modulus, Finite element analysis, Pt nanowire.

1. INTRODUCTION

Knowing the mechanical properties of nanowires is of considerable interest for their uses as nanoscale interconnects and as the active components of electronic and electromechanical devices. An effective technique for quick evaluating the mechanical properties of the nanowires becomes necessary. Determination of elastic-plastic properties especially the hardening modulus is a difficult task. Among the conventional methods, tensile tests are superior in obtaining a stress-strain relation directly. For micro machined samples [1-3] the technique can effectively be used but handling of wire like samples on a nanometer scale is a difficult task, especially for holding with strong adhesive or grips or both, which makes it limited to micro- and millimeter-scale specimens 4-5]. In AFM based tensile tests the nanowire samples can be fixed with AFM tips by using deposition of metal or carbonaceous material [6]. But preparation of sample with such a technique having proper alignment is difficult and during testing most failures occur at the joint due to the combined effect of tension and bending. On the other hand, bending tests are comparatively easier test methods to handle a small size specimen than tensile tests, but there is a big hindrance to induce sufficient bending strain in nanowires. Elastic bending has been reported for various nanowires systems, and involve either positioning the wires across pores on substrates [7-9] or pinning one end of these wires to the substrate. In general, the bending configurations are not visible in these tests as the testing is performed by applying force using AFM cantilever. In case of pinned wires, the flexure lengths are uncertain due to leakage of pinning materials in the shadow-mask process [10]. Atomic force microscope (AFM) and Nanoindentation techniques in evaluating the properties of nanomaterials are well reported. In AFM based tensile or bending tests, the samples are normally fixed on AFM tip or substrate. The roots of the samples on AFM tip or the substrate are very weak under mechanical loading. As a result it is often difficult to establish whether the measured failure is due to the wire itself or the contact between the wire and the AFM tip or the substrate. Moreover, in the AFM based technique, the deformation shape is difficult to be monitored during experiment. In nanoindentation techniques the results are sometimes qualitative and it can hardly predict the elastic-plastic behavior of materials. The methods available at present are not capable of providing reliable measures of nanowire strength or their behavior during plastic deformation and failure. Therefore, a reliable mechanical testing in evaluating their properties becomes indispensible.

In this study a smallscale bending test methodology with a new setup for the mechanical properties of nanowires is presented. The major limitations, i.e., failure at root, wire substrate friction, uncertainty in flexural length due to leakage of pinning material incase of pinned wire, etc., associated in the conventional techniques are removed and the elastic-plastic behavior of Pt nanowires is successfully determined.

2. EXPERIMENTAL DETAILS

2.1 Testing Methodology

Accurate estimation of nanowire mechanical properties by conventional tensile or bending tests is a difficult task. The bending technique is rearranged here in a way to overcome the major limitations. In this technique, a one end fixed beam is considered for small-scaled bending at its free end by closely positioned two opposite probes as shown in Fig. 1. The root A of the sample is considered rigidly fixed. The force *P* at B is considered very close to a fixed support at C. As the loading is considered close to the rigid support and far from the root of the sample, large bending can be obtained across the loading probe inducing a little bending at the root. Therefore, yielding of the wire can be induced across the loading probe without affecting the root by a large stress. The mechanical properties can be determined from the force-deformation relation obtained in this fashion.

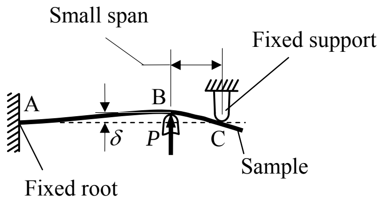

Fig 1. Bending configuration of Nanowire sample.

2.2 Sample Preparation

Nanomaterials prepared by chemical, electromigration or stressmigration processes are normally within the range of a few hundred microns in length. Manipulation of such a small sample in a testing base needs suitable carrier with proper griping. In this study considering the above matter commercially available silver coated Pt nanowire as testing sample and Indium (In) wire as carrier for manipulating the sample are used for establishing a suitable testing methodology. The nominal diameter of Pt nanowire is 625 nm. The coated wire is cut into a small piece and glued on the tip of a small needle. The silver coating from the tip part of the wire is removed by inserting the free end into a nitric acid solution. The In wire with diameter of a few hundred microns is cut into small piece by scissors to have a sharp tip at the end. The In wire and the clean Pt wire are brought in tip to tip contact and joined by Joule-heat welding [11-12]. In the weld joining process properties of Pt are assumed to be unaltered as the melting point of In is very low compared to that of Pt. The Pt wire to form a micro cantilever on In carrier is cut to a desired length (about 200 μm) by Joule-heat cutting [11-12]. A number of samples prepared in this fashion are used for mechanical testing.

2.3 Test Setup

A setup is design and developed for bending the cantilever like micro/nanowire samples by using tiny loading probes in a testing base under a digital microscope. In the setup, a height adjustable rigid stand for carrying support probe and two xyz-manipulators, one for carrying test sample and the other for carrying loading probe, are set on a single platform in similar height level. For measuring the force on the loading probe, it is placed on a force sensor. For computer controlled motion of the loading probe, the force sensor is attached to a piezo stage, which is placed on the xyz-manipulator. The initial setting of the loading probe to any desired xyz position can be done by using the manual manipulator and the precision control of the loading probe motion can be done by the piezo stage. The force sensor is devised as an integrated unit with the combination of a small capacitance sensor, an elastic cantilever and the loading probe as individual and interchangeable tools. The deflection of the cantilever due to forces at the loading tip causes change in gap distance between the cantilever head and the capacitive sensor. This change in gap distance or in other words the force on loading tip is calibrated with the capacitance sensor output.

2.4 Bending Test

The force sensor is set for measuring 0-25 μN force with the rate 1μN/μm by adjusting the pre-calibration length of the cantilever. The test base is taken on an anti-vibration table. The nanowire sample with the In substrate is taken on the carrier and fixed with a small piece of cellulose tape. By the nanomanipulators the sample is first set in contact with the fixed probe with desired length, *L* and then the loading probe is set in contact with the sample with the desired bending spans *a* and *b* as shown in Fig. 2. Note that the rigid stand contains an additional fixed probe, which is not used for bending test but for cleaning the dust (if any) from the sample or other probes. The manual manipulations of the

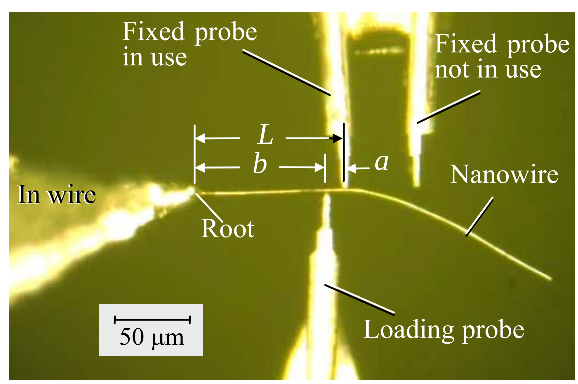

Fig 2. Bending parameters are shown on the microscopic image of the probe-sample assembly.

sample or the probes are performed precisely by the observation of the digital microscope. A computer controlled displacement (constant rate) is applied by the piezo stage and the corresponding capacitance change in potential is recorded. From these measurements the applied force and the corresponding deformation in the sample at loading point are determined. During the experiment the bending configuration of the sample is recorded as video clip by the digital microscope. Since *a* is very small compared to *b* during bending yielding occurs only across the loading probe. After a test a large part of the length b remains unaffected and thus can be used for further bending test with shorter span. Three samples having different diameters have been tested. Several bending tests are performed on each sample with the variation of bending parameters (a, b and L). For any consecutive test the length, L is selected such that the material within it does not yielded in the former test. The option of the use of a single sample for multiple tests minimizes the number of individual samples need to be prepared and many tests can be performed in a short time in similar environmental condition.

3. RESULTS AND DISCUSSION

3.1 Measured P-δ relation and Bending Pattern

A typical force-displacement relationship obtained from experiment for a sample of diameter, d = 640 nm with the flexural lengths, L = 100μm and a = 12μm is shown in Fig. 3. The little zigzags of the force-deformation curves are due to stick and slip for small friction between the probe and sample. As observed very good linearity in small deflection, the force-displacement data clearly depicts that very small forces (up to a few micronewtons) corresponding to the small deformation are precisely measured. Note that in Fig. 3 the horizontal axis indicates the displacement of loading-probe tip. The part AB of the curve shows no change in force but displacement of probe tip. This means for this part the loading probe was not in contact with the sample and contact starts at point B. Therefore, the true displacement of the sample can be determined by subtracting the displacement AB from the probe-tip displacement. The part BC shows linear force-deformation relationship for small deformation of few micrometers. This indicates the elastic bending of the wire. The force with deformation nonlinearly increases in part CD and then decreases in part DE. This confirms that plastic strain is induced in bending the wire.

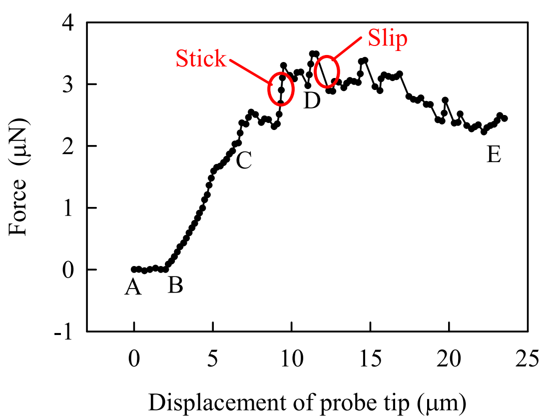

Fig 3. Measured force-displacement relation

3.2 Evaluation of Properties

From the linear part of the force-deformation relation the elastic modulus of the wire material can be determined from the following equation [13].

$$E = \frac{Pb^4(2L+a)^2}{12L^3I\delta} - \frac{Pb^3}{3I\delta} \tag{1}$$

Here, I is the area moment of inertia of the wire cross section, P is the applied force, δ is the corresponding deformation and the other parameters (a, b and L) are lengths as shown in Fig. 2. Using Eq. (1) the evaluated the average Young's modulus for the three samples under different loading spans is found to be 134±22 GPa. The estimated values for tensile modulus of the nanowires show reasonable agreement with their polycrystalline buck property.

The starting point of nonlinearity in force-deformation curve can be considered as yielding of material throughout the full cross section of the wire. As the hardening modulus of pure material is very small compared to Young's modulus, the stress distribution at this section can be considered as constant magnitude of yield stress σ_y. Therefore, for the loading configuration shown in Fig. 2 the yield stress, σ_y can be determined from the following formula [13].

$$\sigma_y = \frac{3P_y ab^2}{L^3 d^3}(2L+a) \tag{2}$$

where, P_y is the yield load corresponding to the starting point of nonlinearity in force-deformation curve and d is the diameter of the wire. Using Eq. (2) the evaluated average yield strength of the three samples for different loading spans is found as 475±37 MPa.

For FE analysis, the material is assumed linear-hardening elastic-plastic behavioral. The stress-strain relationship of such a material is given by

$$\varepsilon = \frac{\sigma}{E} \quad (\text{for } \sigma \le \sigma_y), \tag{3}$$

and

$$\varepsilon = \frac{\sigma_y}{E} + \frac{\sigma - \sigma_y}{E'} \quad (\text{for } \sigma > \sigma_y), \tag{4}$$

where ε, σ, σ_y, E and E' are strain, stress, yield strength, Young's modulus and hardening modulus respectively. The hardening modulus of wire material is determined from FE analysis by repeatedly doing the FE simulation till the load-displacement relationship obtained by FE analysis is coincident with the experimental one. The procedure is briefly described as follows. Let consider the particular experiment on a sample (diameter = 770nm) with loading parameters, L = 65μm and a = 12μm for FE model. The FE model is prepared with sufficient meshes and with proper boundary conditions that best suit the experimental conditions. At first the elastic FE analysis is performed on the model considering the Young's modulus obtained by Eq. (1) based on experimental data. The experimental data (round symbol) and the elastic FE analysis results (curve I) as force-displacement (P-δ) relationships are plotted for comparison, see Fig. 4. The linear part (up to point B) of the experimental measurement show excellent matching with the elastic FE curve. This confirms that

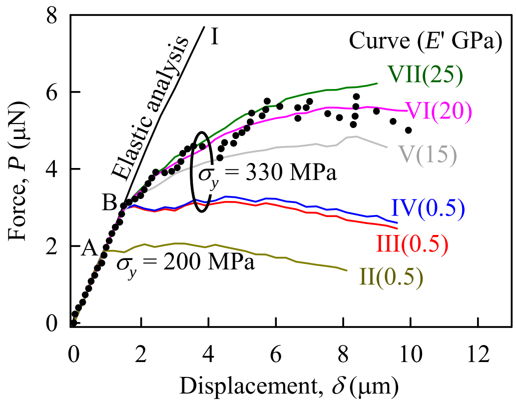

Fig 4. Finite element simulation

the measured force-displacement data are accurate. After that elastic-plastic FE analysis is performed based on the experimentally measured values for E and σ_y, and an assumed value for E'. In the elastic-plastic FE analysis the suitable values for σ_y and E' are looked into for which the best matching between FE and experimental P-δ relations is obtained. At first assuming a small value for E' the value of σ_y is adjusted in FE analysis. The curves II and III are for same E' (0.5 GPa) but for different values of σ_y, which are 200 and 330 MPa, respectively. These two curves clearly show their first-point of nonlinearities, i.e., points A and B. Note that with the increase in σ_y the point of nonlinearity moves from point A to point B; therefore it is easy to find the appropriate value of σ_y in FE simulation by a few trials. The curve III shows very good matching of the first point of nonlinearity with that of experimental measurements. Therefore, the value (σ_y = 330MPa) for curve III is considered appropriate and keeping this σ_y fixed the value of E' is adjusted in the FE simulation (see curves V-VII). Here it is observed that with the increase in E' the FE analysis curves become stiffened having the point of nonlinearity (point B) unchanged. Note that all the curves described above are obtained for a friction coefficient of 0.1 between the probe and sample. The curve IV is obtained for the same properties of curve III but the friction coefficient of 0.2. These two curves are very close therefore the friction coefficient has a little effect on the P-δ relation. In all analysis the friction coefficient is assumed in the range of 0.1 to 0.2 and the value of E' has been determined by matching the P-δ relation for large deformation in elastic-plastic range. The hardening modulus of the Pt wire is found as 18±3 GPa.

4. CONCLUSIONS

The experimental and finite element simulation scheme can successfully produce the elastic as well as the elastic-plastic bending in the nanowire. The deformation pattern ensures that the difficulties associated with the root or holding points are removed completely. Very small force corresponding to the small deformation is effectively measured. Good matching of the FE analysis data with the experimental ones is obtained and eventually, the hardening modulus of 625 nm Pt sample is successfully determined.

5. ACKNOWLEDGEMENTS

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