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In situ transmission electron microscopy mechanical deformation and fracture of a silver nanowire

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ABSTRACT

In this paper a fivefold twinning silver nanowire is mechanically tested in real time within a transmission electron microscope using an atomic force microscopy sensor. Our experimental setup allows us to measure, by bending the silver nanowire, the elastic modulus (E), the fracture toughness (K_{IC}) and the stress intensity factor (σ_I) for elastic and plastic deformation regions and finally the fracture of the nanowire. Data of the force applied and the bending of the nanowire was recorded during the deformation and after the point of fracture. The mechanical properties of the nanowire were extracted and compared with nanoindentation using atomic force microscopy.

1. Introduction

In situ transmission electron microscopy (TEM) has opened the possibility to measure physical properties individually in nanostructures. Mechanical properties measured at nanoscale reveal modified regimes compared with their macroscale counterparts. Such is the case of metallic nanoparticles and atomic-layers of transition metal dichalcogenides [1,2]. In addition, at nanoscale gravity forces are not appreciable and the surrounding forces of metallic nanoparticles can be measured with an experimental setup in which an atomic force microscopy (AFM) tip is coupled in a TEM sample holder [3]. Other metallic nanowires are also subject to be studied within a TEM to measure other physical properties such as magnetic behavior and their in situ magnetization by exciting the objective lens [4]. In this way, silver (Ag) nanowires (NWs) are of special interest for measuring physical properties at individual motifs due to their optical properties. For instance Ag-NWs show interesting physical properties [5–7] and are good candidates for a large variety of applications such as plasmonic devices [8,9], plasmonic waveguides [10,11] and biosensors [12,13]. In the literature there are other methods of synthesis in which they have reported a good control of their aspect ratio and how it can change their physical properties [14–16]. Novel techniques on the testing of the mechanical properties of nanostructures have been developed in recent years to perform resonance [17–19], bending [20–23] and tensile [24,25] tests. Previous research on the mechanical properties of the nanowires has not reported the live feedback of the behavior of the material during fracture after plastic deformation. Chen et al. [26] performed nanomechanical bending behavior and theoretical calculation of the elastic modulus of silver nanowires using atomic force microscopy (AFM). However, there is little experimental research done on the failure and fracture of nanowires, for instance Wu et al. [27] performed three point bending on Ag NWs to observe the plastic deformation of hardened silver nanowires in an atomic force microscope. In this work we report the elastic and plastic deformation until failure of an Ag-NW through an AFM holder coupled within a transmission electron microscope to perform a mechanical three point bending test. Video of the test was recorded where the mechanical deformation of NW begins and ends with total fracture. Detailed information of the force exerted by the tip until failure, the stress intensity factor, and the fracture toughness of the Ag NWs were measured from the recorded data. Additionally, the mechanical properties measured in TEM were compared via nanoindentation performed in an atomic force microscope.

2. Experimental methods

Silver nitrate ($AgNO_3$), poly(vinyl pyrrolidone) (PVP, $Mw = 55{,}000$) and ethylene glycol (EG) were supplied by Sigma-Aldrich and used

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without further purification. Silver nanowires were synthetized by using polyol [28]. The synthesis methodology has been performed as follows: 5 ml of ethylene glycol was preheated at 160 °C for 1 h under magnetic stirring in a rounded bottom flask, then 3 mL of $AgNO3$ (0.1 M, in EG) and 3 mL of PVP (0.15 M, in EG) were simultaneously dropped into the hot solution (~0.2 mL/min). The reaction was let to boil for an additional 30 min. The solution changed from yellow to greenish gray indicating the formation of the Ag NWs. Subsequently the product is cooled at room temperature and washed with ethanol three times and two times with acetone followed by centrifugation at 1500 rpm for 15 min. Finally, the precipitate is dispersed in ethanol and a glossy gray solution is observed.

These nanowires characterized by their pentagonal cross section and large aspect ratio are shown in Fig. 1. The changes in the coloration of the colloidal solution serve as a control during the growth of nanowires. In Fig. 1(a) are presented images of the solution at different reaction stages, in which the yellow color of the solution is due to the formation of small particles (seeds). After several minutes, the orange color indicates the formation of larger particles and the anisotropic growth takes place. Finally, the nanowire solution turns gray. Fig. 1(b) shows a magnified SEM image with the inset presenting a geometrical model of the transversal and longitudinal sections of a nanowire. The (100) and (111) facets are shown. These nanowires present a narrow diameter distribution as is noticed. The average diameter of the nanowires was ~60 nm and the length varied from 10 to 20 μm. The Ag nanowires were studied by high resolution transmission electron microscopy (HRTEM) using a JEOL 2010F microscope operated at 200 kV. In Fig. 1(c) the end of an individual Ag NW is presented. The Fast Fourier Transform (FFT) is also displayed in which the growth direction is easily noticed. The darker contrast in the image indicates the twinned nature of this wire. The nanowire growth is along <110 > direction. From the high magnification micrograph shown in Fig. 1(d), the measured lattice distances are 0.23 nm corresponding to the {111} plane of the cubic structure of silver.

The in situ experiment was performed within a transmission electron microscope JEOL ARM200F operated at 200 kV, using an in situ AFM holder (Nanofactory Instruments). This holder has a silicon tip and a reference cantilever to measure the applied load. With this instrument it is possible to observe the AFM tip in contact with the nanowire as it applies a load and creates the fracture as the load is increased. The AFM-TEM holder has a sapphire ball mounted on a piezotube that allows positioning and manipulation in (X, Y, Z) by coarse and fine movements (~1 nm) respectively [1,29,30]. A single Ag NW was supported on the two ends on a gold surface in the three point bending configuration, see Fig. 2. In Fig. 2(a) we can observe the moment in which the AFM sensor creates contact with the NW in a three point bend test set up. Fig. 2(b) is a model of the fivefold nanowire in the same set up.

In order to set the sample of Ag NWs in the AFM-TEM holder, a concentrated solution of pentagonal silver NWs was prepared and suspended in ethanol under constant stirring at room temperature, until the solution color turned beige to assure the presence of NWs. A

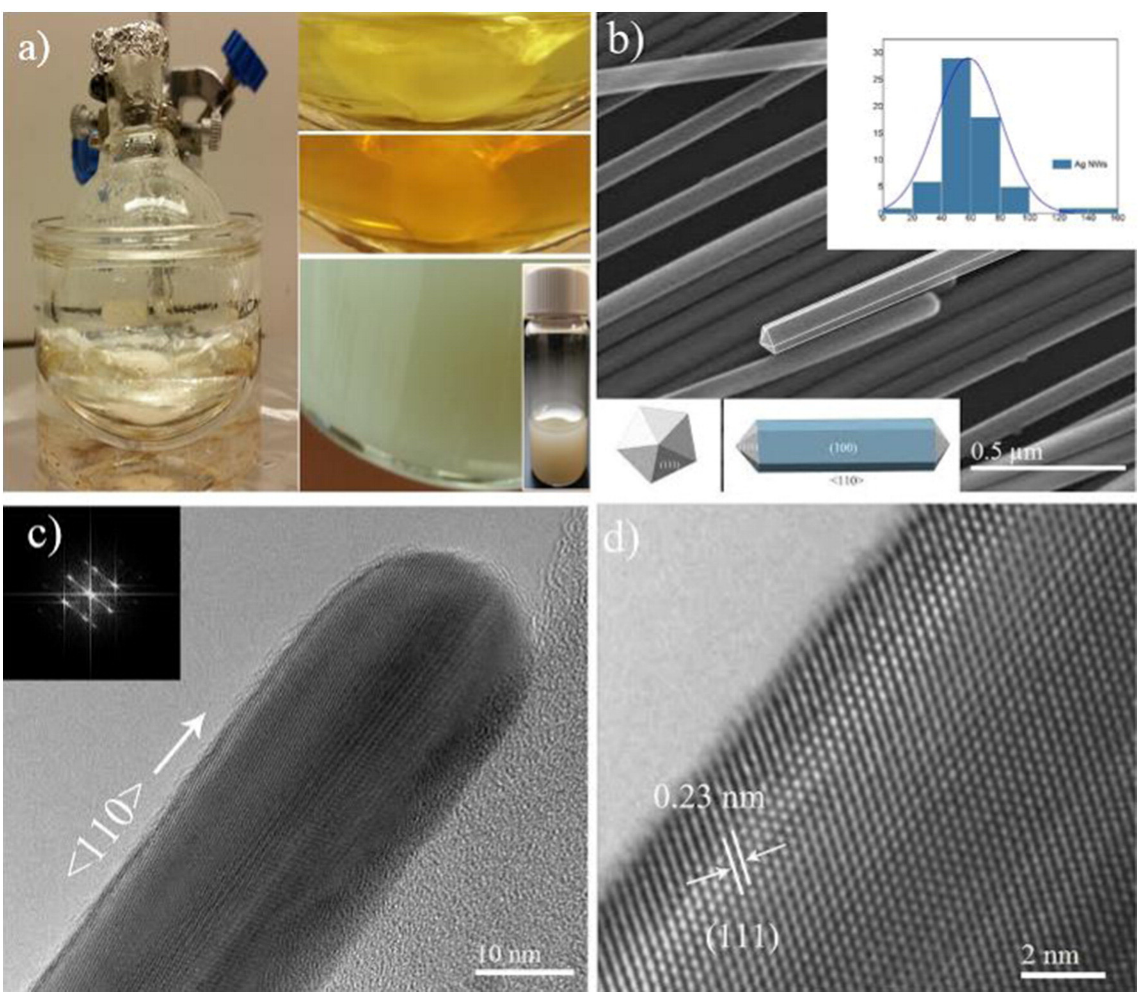

Fig. 1. Micrographs of the synthesized Ag NWs. (a) Coloration changes during the synthesis process. (b) Low magnification SEM image of the Ag NWs with a diameter histogram and simulations of the pentagonal cross sections as insets. (c) TEM micrograph of the tip of one nanowire. The FFT shows the characteristic reflections of a twinned structure. (d) HRTEM image showing the interplanar spacing of the nanowire. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)