MEMS-in-TEM for Nano Tribology

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Abstract. We have developed a MEMS-in-TEM experimental system that enables the mechanical testing of nano junctions by MEMS microactuators in TEM (transmission electron microscope) for in situ atomic level observation of dynamic deformation. A nano junction between two sharp opposing tips was formed by bringing them into contact. Applying either tensile or shearing stress, we observed the elongation and changes in shape of the junction at real-time rate while applied force was measured. The experimental system and results of tensile and shear tests are described.

1. Introduction

We have intensively investigated MEMS (micro electro mechanical system) design, fabrication and its application to nano and bio technologies [1]. Our nano scientific research using MEMS devices includes the mechanical testing of nano junctions in TEM (transmission electron microscope) for in situ atomic level observation of dynamic deformation [2]. Test structures in the MEMS are as small as ten nanometers. MEMS electrostatic actuators can provide higher precision at the sub-nm scale, more stable loading control over long time (hours, even days), and better temperature stability than conventional piezoelectric actuators that are used commonly in atomic force microscopes. Furthermore, the integration of thermal or strain sensors or heaters enables multi-functional MEMS devices that can fit in the narrow space of a TEM specimen holder. The tensile testing device has opposing sharp tips; one tip is fixed and another is actuated toward it. By bringing them together, a nano junction is formed. The junction is stretched and separated by retracting the movable tip.

This time, we fabricated a two-DOF (degree-of-freedom) device that had another actuator to apply shear stress to the junction for tribology measurement. The shape and dimension of nano junctions as well as the displacement of the movable tip were observed during the shear testing. We prepared tips of various materials that showed very different behaviors. The applied force can be calculated from the spring constant of the support and the difference in displacement between the cases with and without a junction.

2. MEMS-in-TEM experimental system

As shown in figure 1, a MEMS device driven inside a TEM specimen chamber constitutes a MEMS-in-TEM system. The MEMS device was placed in a special TEM holder with nine feed-throughs and inserted into the specimen chamber of the TEM (Hitachi, HF-2000UHV), with a vacuum level of less than $1 \times 10^{-7}$ Pa. The feed-throughs of the holder enabled the actuation of the MEMS device and
electrical current measurements between tips during in situ TEM observation. The electron beam accelerated at 200 kV irradiated and passed through the gap of the opposing tips. The sample thickness should be less than a few hundreds of nanometers because the electron beam must penetrate the sample for observation with TEM [3]. High-resolution images were projected on a screen with 0.2 nm maximum spatial resolution. The electric current density in the observation area of the TEM electron beam was $6.2 \times 10^4$ A m$^{-2}$. The electron beam of this magnitude causes temperature rise calculated to be less than 1 K [2, 3]. The images were captured by an integrated charge capacitive detection camera (SC200, Gatan, Inc.). High-resolution images could be obtained without scanning the electron beam and therefore allowed real-time observation. A temporal resolution of 1/30 s was achieved with a video recording system.

Our experiments were performed between sharp probe tips of less than 100 nm in radius of curvature. Such sharp tips were formed at the ends of micromachined structures. In order to ensure good alignment between sharp edges for the TEM observation, we designed the device with a symmetrical actuator and supports having proper rigidity. A schematic view of the device is shown in figure 1. The device has a movable electrode, a fixed electrode, a pair of driving electrodes and opposing tips. All the components were integrated within the limited volume, 4 mm x 5.4 mm x 0.7 mm, because the device must be inserted into the objective lens of the TEM. Opposing tips consisted of a movable tip and a fixed tip. Those tips were located at the center of the movable and fixed electrode, respectively. The electrostatic microactuator had two electrodes to drive the movable tip towards the fixed tip. Basic tip material was silicon. They could easily be covered with a thin layer of other materials such as Au, Ag, or DLC [4-6]. If required, the electrical current measurement between the opposing tips was possible when bias voltage was applied between them.

The whole device was micromachined from a silicon-on-insulator (SOI) substrate. Therefore, the thermal expansion occurs uniformly when environmental temperature may change. We also characterized the precision of the electrostatic actuator. The parabolic relationship between driving voltage and displacement was obtained from TEM direct observation. No hysteresis appeared. The displacement around 300 nm, where tips were almost brought into contact, could be approximated with a straight line; its slope was 6.2 nm V$^{-1}$. The driving voltage source had the precision of 0.0001 V (E5263A, Agilent Technologies). Hence, the tip displacement could be controlled within less than sub-nm order accuracy. This driving characteristic was found to be persistent over several experimental runs.

The displacement became smaller when the same characteristic was measured after the formation of a nano junction. The applied force can be determined by multiplying the spring constant of the support and the difference in displacement between the cases with and without a junction.

Figure 1 The schematic illustration of TEM observation system (left). A MEMS device having movable opposing tips (right) was inserted into the TEM specimen chamber. TEM real-time images were obtained with the TV camera.
3. Tensile test of nano junction by MEMS-in-TEM system

The tensile test of a gold nano junction [4, 7] is described to show the operational procedure of nano junction formation between tips and its mechanical testing under TEM observation. Here the tips in the MEMS device were covered with chromium/gold stacking layers of 5 nm/200 nm in thickness.

The procedure of our experiment, approach-contact-retraction-fracture process, is as follows:

a) The movable tip approaches the fixed tip by increasing the driving voltage.

b) A gold nano-bridge appears between tips when the gap distance is less than 0.5 nm at 1 V bias voltage applied between tips. The bridge enlarges by the atomic rearrangement.

c) The gold nano- junction was retracted. The diameter of the junction is thinned.

d) The junction is finally fractured. Corners on tips are rounded by the atomic rearrangement.

This process was repeated many times. The surface geometry was changed during this process resulting in the change of the contact surface [4]. Retracted shapes of gold nano junctions were different according to the elongation directions (figure 3) [8]. The crystalline orientation was decided from the TEM lattice image.

The gold nano junction was retracted keeping the similar figures when the elongation direction was [011]. The waist of the gold nano junction became thin from 8.8 nm to 2 nm in diameter (figures 3 a-c).

The gold nano junction was transformed into the wire shape when the elongation direction was [011]. The wire diameter was 1.8 nm during the 2.8 nm retraction along the length direction (figure 2 b-1). These gold nano junctions were broken spontaneously in about 10 seconds while the tip was kept at the same position. After breakage the tip became rounded and its final radius curvature was 3 nm (figures 3 d). This fracture was caused due to the atomic rearrangement of gold atoms.

Figure 2 The identification method of the crystallographic facet. TEM images (a1, b1, c1) were schematically illustrated like in the images (a2, b2, c2). The elongation directions were [011], [011], [101], respectively.

Figure 3 The gold nano junction elongation. When the direction of the gold nano junction elongation was [011] at the junction, the junction was thinned, holding the hourglass shape, and finally broken. The tips were rounded by the atom rearrangement.

4. MEMS-in-TEM for nano tribology experiment

4.1. MEMS device

As the extension of tensile-testing devices, we fabricated a two-DOF (degree-of-freedom) device that had another actuator to apply shear stress to the junction for tribology measurement. Its schematic
illustration is shown in figure 4. One tip can move in longitudinal direction by the same actuator as in the tensile-test device. Another tip is driven in lateral direction by the additional actuator (electrode 3 in figure 4) placed on the side of the tip support. We prepared tips of bare Si and those coated with Au, Ag, and DLC (diamond like carbon) films. The shearing force can be calculated by multiplying the spring constant of the support and the difference in displacement between the cases with and without a junction (figure 5).

4.2. Shear deformation and breakage of Ag junction
Here the shear test of a silver nano junction is described [5]. Tips were covered with a 30 nm thick silver layer. The procedure of our experiment, contact-shearing-fracture process with TEM observation, is as follows:

a) Both tips were brought into contact and a junction appeared. (figures 6 a, b)
b) Shear force was applied to the junction by actuating the lateral actuator. (figure 6 c) The loading speed in the shear direction was 0.5 nm s$^{-1}$.
c) The junction was fractured. (figure 6 d)

When two tips met, a junction of approximately 5 nm in width appeared (figure 7 a). Although TEM images were not very clearly reproduced, we could observe lattice fringes sometime. The junction was not a single crystal. The right tip was displaced in the direction shown by the arrow, while other one was kept stationary. The junction slightly moved and tilted in the loading direction but its width stayed almost unchanged (figure 7 b). At the final stage, it became narrower down to 1-2 nm and fractured (figures 7 c, d).

Figure 4  Schematic picture of 2-DOF MEMS device for shear testing.  
Figure 5  Schematic explanation of force measurement method.  
Figure 6  Schematic explanation of the experimental procedure for shear testing of nano junction.
Figure 7  TEM images of silver nano junction at the different stages of deformation under shear load.

Figure 8  Tip displacement vs. applied voltage for shear testing of Ag nano junction. The arrows (a) – (d) correspond to the images of Figure 7.

Figure 8 shows the displacement vs. driving voltage characteristics with and without the junction. The one without the junction is almost linear with the sensitivity of 24 nm V\(^{-1}\). With the junction, the movement was much smaller. At the applied voltage of approximately 0.3 V, the difference between them is 4.6 nm, corresponding to the shearing force of 10 nN. Here, the tip moved suddenly, releasing a part of the stress. The length of the stepwise motion was 1 nm. Two more stepwise motions were observed before fracture. The shearing force at the fracture was 7 nN.

4.3. Material comparison
In addition to silver tips, we also prepared tips of bare Si and those coated with Au and DLC films. The shape and dimension of nano junctions as well as the displacement of the movable tip were observed during the shear testing. The typical dimensions were 2-5 nm in diameter and 3-10 nm in length at the initial formation. Each material showed, however, very different deformation behaviors; Si junction elongated much longer than Ag and Au. The fracture forces for Ag and Au were almost the same. The fracture force for the Si junction was 10 times as large as those for Ag and Au. For the DLC interface, no junction but nano balls appeared between two tip surfaces. The ball rotated when shear load was applied to the interface. Details of those experiments were reported elsewhere [5, 6, 9].

5. Conclusion
We have developed a MEMS-in-TEM experimental system that enables the mechanical testing of nano junctions by MEMS microactuators in TEM for in situ atomic level observation of dynamic deformation. The applied force could also be determined. Tensile and shear tests of nano junctions were conducted for junctions made of various materials; those results showed very different characteristics among materials and from the bulk characteristics. We hope the MEMS-in-TEM system will provide new insights of mechanical properties at nano scale which are valuable to tribology study.

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