Nano-scale observation of frictional deformation at Ag single point contact with MEMS-in-TEM setup

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Abstract. Monitoring atomic-level deformation of a frictional interface in real time is crucial for tribology research at micro and nano scale. We have combined nano-scale handling capabilities of MEMS technology with the supreme observation ability of TEM and developed a MEMS-in-TEM experimental setup. We succeeded in observing the frictional deformation of silver interface and measuring its shear force simultaneously.

1. Introduction

Scaling down the dimension of MEMS (Micro Electro Mechanical System) devices gives higher performance devices\(^1\), sensitive sensors\(^2\) and actuators\(^3\). Serious problems such as friction and adhesion, however, have also been arisen because of the increase in the surface-to-volume ratio\(^4,5\). Most of the devices such as MEMS and NEMS (Nano Electro Mechanical System) need contact points to support their body; this results in friction. The frictional phenomenon becomes a serious issue to fabricate micro miniature machines of low energy consumption and high reliability. Therefore, we need further understanding of friction and lubrication.

Conventional liquid lubricants used in macro-scale do not function at micro and nano scale because of the increased capillary force\(^6\). Therefore, solid lubricants such as Ag\(^7,8\), DLC and MoS\(_2\) are good candidates of a next generation lubricant at micro and nano-scale. Since shear deformations of soft metals caused by friction easily occur at contact point, Ag is assumed to be a promising lubricant\(^4,6-8\). However, those phenomena have not been well understood because of the experimental difficulties to observe frictional interface.

Since macroscopic frictional phenomena are attributed to the collectiveness behaviour of microscopic shear deformations, fundamental understanding of friction can be achieved by observing single points contact\(^4,5\). Some groups combined TEM (Transmission Electron Microscopy) with an AFM\(^9-12\) to observe a nano-scale deformation at the frictional interface. Those experimental systems utilize piezo actuation. The piezo actuator can produce nano motion but suffers from poor stability issues. Therefore we utilized a MEMS electrostatic actuator for a stable actuation instead of piezo actuators\(^13\). In this research, we have observed deformation of a lubricant on a frictional interface and measured the shear force simultaneously by using MEMS-combined TEM experimental system. Observing the real area of contact under shear stress gives us a new insight in frictional characteristics and thus, we believe that the proposed experiment is crucial to reveal the lubrication mechanism.
2. Experimental setup

Our experimental setup, combination of TEM with a MEMS device was established by T. Ishida et al. Opposing tips of the MEMS device were actuated at room temperature to provide physical deformation at the frictional interface. The single point contact was formed between two tips and monitored by TEM to collect data for shear force measurements.

2-1 Principle of MEMS actuation

The MEMS device has 4 electrodes and two opposing tips as shown in figure.1. Electrodes provide electrostatic actuation and move the opposing tips in two directions. (1) Contact direction: By applying voltage to electrode 2 in figure.1, Electrode 1 is attracted to the electrode 2. Thus, one tip is approached to the other and ended up with a physical contact. (2) Shear direction: By applying voltage to electrode 3 in figure.1, Electrode 4 is attracted to electrode 3. Thus, tip is actuated in shear direction.

![figure.1 Schematic explanation of the proposed MEMS device](image)

2-2 Fabrication of MEMS device

A pair of suspended silicon opposing tips was made using standard lithography and double side DeepRIE process on an SOI (Silicon On Insulator) substrate. The gap between tips was defined by FIB (Focused Ion Beam) and typically 300nm. Ag was evaporated on the tips of electrodes 1 and 4(figure.1). Average deposition speed was several hundreds pm/s under $10^{-3}$ Pa. 20nm of silver layer was deposited only on the side-wall of the opposing tips using a shadow-mask process.

2-3 TEM setup

TEM (Hitachi, HF-2000UHV) was used in the experiment. The electron beam was accelerated at 200 kV and the vacuum pressure was $4.0 \times 10^{-8}$ Pa. The electric current density in the observation area of the TEM electron beam was $6.2 \times 10^4$ A·m$^{-2}$. With the given electron beam intensity, Joule heating was calculated to be less than 1K.

2-4 Shear force calculation

The shear force between the opposing tips can be calculated by comparing the difference of the displacements of the tips. 1) Displacement before the contact: Before bringing the opposing tips into contact, one tip was actuated in shear direction and we monitored its displacement $x$ (figure.2(a)) which is determined by the balance between the electrostatic force, $F_1$, and the restoring force, $F_2$, of the electrode represented by equation(1). 2) Displacement after the contact: After achieving the contact, the tip was actuated in shear direction and we monitored the displacement $x'$ (figure.2(b)) which is determined by the balance between the electrostatic force, $F_1$, the restoring force, $F_2$, and the shear force, $F_3$, represented by equation(2). The shear force ($F_3$) can be calculated as a function of $x$ and $x'$ by combining equations(1) and(2). Shear force can be measured by the spring stiffness and the
difference of shear displacement monitored from the TEM images. Thus, the resolution of shear force is dependent on errors of the spring constant and the resolution of TEM images. The error of thickness, due to the DeepRIE Si etching process, is smaller than the total thickness of the electrode. Since the constant spring stiffness is proportional to the cube of the thickness, the error of the spring stiffness is negligible small compared to the effect of the resolution of TEM image. The spring constant of proposed MEMS device is 2.2 N/m and the resolution is approximately 1nm, therefore the resolution of shear force can be estimated as 2.2 nN.

\[ F_1 = F_2, \quad F_2 = k\alpha \]  \hspace{1cm} \ldots(1)
\[ F_1 = F_2' + F_3, \quad F_2' = k\alpha' \]  \hspace{1cm} \ldots(2)
\[ F_3 = k(x - x') \]  \hspace{1cm} \ldots(3)

figure.2 Schematic view of the procedure to detect shear force. \( F_1 \) corresponds to the electrostatic force. \( F_2 \) corresponds to the restoring force without the junction. \( F_2' \) corresponds to the restoring force with the junction. \( F_3 \) is the reaction force to the applied shear force to the junction.

2-5. Experimental procedure
The Ag-coated silicon opposing tip (figure.3(a)) was actuated according to the experimental procedure as shown in figure.3. Tips were brought into contact forming a junction at the contact point (figure.3(b)). After obtaining the contact, the one tip was actuated in shear direction with a velocity of 0.5 nm/s while maintaining the contact (figure.3(c)). The junction was finally broken by this actuation (figure.3(d)). We have monitored all steps of the experiment including the resulting shear deformation using TEM.

3. Result and discussion
Changes of shear force are shown in figure.4. The lateral axis corresponds to the shear displacement and the vertical axis corresponds to the shear force. The initial junction without applied force (figure.4(a)) was actuated in the arrow direction shown in figure.5(a). The shear force increased up to 36.8mN (figure.4(d)) and the junction was finally fractured (figure.5(f)). During this shear actuation,
the junction was not deformed until 10nN. The liner characteristic of the graph from 10nN to 36nN showed that the shear force changed in proportional to the shear displacement. The average gradient of the graph was about 2.9 N/m. Furthermore, some abrupt motions were observed in the region. The junction suddenly deformed for 0.6 nm in 1 second around 25nN (figure.4(b) to (c)). After the sudden deformation, the shear force built up with almost no displacement until the next sudden motion happened. Similar behaviors were observed several times before the fracture. This suggests that sudden slipping deformation is a characteristic property of Ag shear deformation.

figure 4 Shear displacement vs. shear force for shear testing of Ag nano junction. The arrows (a)-(f) correspond to the images of figure.5.
The size and the orientation of the junction changed during the actuation as shown in figure.5(a) and figure.5(e). To discuss the deformation quantitatively, we defined the diameter, $d$, of the junction and the plane angle, $\theta$, of the cross sectional area as shown in figure.6.

In figure.7, the lateral axis corresponds to shear displacement and the vertical axis corresponds to the angle of the cross section. The linear characteristic of the graph shows that the plane angle $\theta$ changed in proportional to the shear displacement. The initial plane angle at the beginning of the experiment (figure.7(a),5(a)) was approximately zero. The angle increases gradually with the actuation up to $27^\circ$ (figure.7(e),5(e)) just before the fracture. Thus, the deformation by the shear actuation includes a shear deformation and a deformation in a longitudinal direction.

The change in the diameter, $d$, against shear displacement is shown in figure.8. The initial diameter (figure.8(a),5(a)) was 12nm. With increasing the shear displacement, the cross sectional diameter decreased linearly down to 5.8 nm (figure.8(e),5(e)) before the fracture. It is notable that there are discontinuous deformations in figure.8. The diameter discretely decreased associated with the slipping deformation in figure.4.

The sudden slipping deformation repeated several times by the shear actuation. At the moment, when the diameter of the junction discontinuously became thinner, the orientation of the junction also rotated in association.
In the future we will observe that the dependency of shear force on the diameter of the junction. Furthermore, we will observe the dependency of the diameter on the normal force. Those results gives the relationship between normal force and shear force as Amonton’s law\(^{(4)}\) described. That relationship and the real time observation of shear deformation will be helpful to interpret the mechanism of the Amonton’s law.

4. Conclusion
We have succeeded in observing Ag shear deformation in the nanometer range and measuring the shear force simultaneously for the purpose of revealing the mechanism of lubrication. The Ag junction discretely deformed during the shear actuation; this was associated with discrete decrease in the diameter. The plane angle of the cross-sectional area rotated up to 27 degree. Thus, the deformation by the shear actuation includes a shear deformation and a deformation in a longitudinal direction. We believe that those results provide a new insight of lubrication and it is useful to reveal the mechanism of lubrication.

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