JAIST Repository
https://dspace.jaist.ac.jp/

Title	水晶振動子力センサーを組み込んだ透過型電子顕微鏡法による金ナノ接点の機械特性の研究
Author(s)	石塚，慧介
Citation	
Issue Date	2020-03-25
Type	Thesis or Dissertation
Text version	ETD
URL	http://hdl.handle.net/10119/16662
Rights	
Description	Supervisor:大島　義文，先端科学技術研究科，博士

JAPAN ADVANCED INSTITUTE OF SCIENCE AND TECHNOLOGY
Japan Advanced Institute of Science and Technology

Abstract

A study on mechanical properties of gold nano-contact by development of in-situ TEM method using quartz resonator force sensor

Keisuke Ishizuka (Oshima Lab.)

Mechanical properties of nano-contacts (NCs) have attracted much interest, which are expected to exhibit dependence on size, shape and crystal orientation. In particular, the deformation process and Young's modulus of metal NCs would be important issues for not only science but also industry, but they have not been understood fully. Molecular dynamical calculation suggested that some specific structural transformation occurred by stretching the metal NC. Experimentally, it was observed that the shape of metal constriction depended on the stretching process and the long neck could be formed when its crystalline quality was high. Also, Young's modules of silver and zinc oxide nanomaterials have been measured to differ from their bulk values. These experimental results could be supported by the numerical calculation. On the other hand, in gold NCs, experimental results have been rarely obtained, and first-principles calculation has some difficulties due to heavy element. In order to investigate the mechanical properties of nanomaterials, it is beneficial to use an in-situ TEM method combined with a force sensor, called TEM-AFM method, because the shape and size of nanomaterials can be evaluated. In previous TEM-AFM methods, the loading force was evaluated from the bending of a Si cantilever, and the displacement of a sample was measured in the TEM observation. However, it seemed difficult to precisely estimate the displacement, because it was too small (below a few percent of sample length) to precisely be measured in the TEM image. Also, even if increasing the displacement avoid this problem, the large displacement leads to difficulty in TEM observation with atomic-resolution during measurement.

In this study, I developed a TEM holder equipped with a quartz length extension resonator (LER) as a force sensor. The LER with high effective stiffness (e.g., ~ 10^6 N/m) has an advantage in frequency modulation detection mode. It can measure the equivalent spring constant or force gradient of metal NC under strain associated with an oscillation amplitude of the order of several tens pm. Under such small oscillation amplitude, plastic deformation of gold NC or distortion of TEM image can be avoided.

I established an evaluation method of the effective spring constant of the LER. For quantitative measurement, it is critical to estimate the relationship between the LER oscillation amplitude and the electrical signal associated with the oscillation due to piezoelectric effect, called the sensitivity of LER. I found that Fourier transformation pattern of the TEM image shows modulation as if multiplied by the Bessel function corresponding to the oscillation amplitude. In this finding, the sensitivity of LER could be estimated precisely. The effective spring constant could be determined by fitting the thermal oscillation spectrum of LER with the theoretical formula, since the sensitivity has already been determined.

The equivalent spring constant and electrical conductance of the gold NC were measured simultaneously with TEM observation during the thinning process of the gold NC. The shift of the resonance frequency (Δf) could be expressed by $\Delta f = k_{\mathrm{ts}}(Q_0) \times f_0/2k$, assuming that the equivalent spring constant of gold NC ($k_{\mathrm{ts}}(Q)$) around the center of oscillation Q_0 is approximated by a linear function of the displacement, $k_s(Q) = \alpha(Q - Q_0) + k_{\mathrm{ts}}(Q_0)$, where f_0 is the resonance frequency without gold NC, and k is the effective spring constant of LER. The cross section of the gold NC, which cannot be measured directly in the TEM image, could be estimated by the electrical conductance. The experimental results suggested that gold NC (a part of narrow constriction) tends to have almost the same length and cross-sectional area by thinning, which depends on the stretching direction. Since the measured spring constant can be regarded as a series coupling of the springs of the gold NC and the bases supporting the Au NC, it is necessary to obtain the contribution of the gold NC from the measured spring constant for estimating the Young's modulus. I tried to estimate the Young's modulus of the gold NC by assuming that the thinning process of the gold NC can be described by a simple deformation model. In this estimation, the shape was evaluated by the TEM image and electrical conductance value. Young's modulus for gold NC was suggested to depend on the stretching directions. However, Young's modulus of gold NC did not seem to depend on the size of NC. The specificity of Young's modulus of gold NC may depend on the structure of the NC rather than the size.

In conclusion, I developed the newly in-situ TEM method utilizing quartz resonator force sensor which enables us to estimate mechanical properties of single-nanometer-scale materials. Using this method, I could clarify the deformation process and Young's modulus of gold NC.

Keywords: nano-contact, nano-mechanics, in-situ TEM, frequency modulation AFM, length extension resonator