特　集　7
研　究　速　報

Morphological Changes and Mechanical Properties of Thermally Fabricated Nanometric Oscillators

加熱により作製したナノメートルオーダの振動子の形状変化と機械特性

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Abstract

We are investigating the feasibility of using a three dimensional mechanical oscillator for detecting force and mass. The mechanical oscillator in question consists of a spherical mass supported by a filiform neck. Depending on the fabrication process, the mass can be as small as a few 10 nm to a few 100 nm. In this paper, we focus on the morphological changes observed during its fabrication by means of surface diffusion induced by heating of a sharpened metal tip. Some results on its mechanical properties will also be reported.

1. Introduction

Since the invention of the scanning force microscope[1] (SFM), study of condensed matter and biological samples has been carried out in the subnanometric region. In these studies, silicon microfabricated cantilevers and tips are normally used as the force sensor. However, the size of commercially available cantilevers lies still in the 10 to 100 micron order, limiting the natural frequency and Q factor of the cantilever. Its relatively large mass is one limiting factor of the resolution of detection of mass change induced by emission or adsorption of mass on the scanning tip fixed to the cantilever. Recently, there have been works aimed at redesigning the cantilever to achieve higher natural frequency and/or Q factor.[2, 3, 4]

A nanometric mechanical oscillator has the potential of being used for measurement of force and mass, detected as the frequency shift of the oscillator. Due to its very small mass and the possibility of having high natural frequency and Q factor, the oscillator is expected to be a powerful tool for detecting weak forces, atomic level mass change, and very fast phenomena. There are at least three possible methods of fabrication. One is a process found around 1969 by Drechsler, Vu Thien Binh et al.5) The method consists of inducing surface diffusion at the apex of a sharpened metal tip. The second method was developed by Schiller et al. in 1995, consisting of sputtering a sharpened metal tip with neon. The third method was developed by Hoummady et al.. The method utilizes local etching of a metal tip by means of an electrolyte membrane.[6]

The mechanical property of nanometric oscillators needs to be measured for evaluating their feasibility and sensitivity. A sample stage of a scanning electron microscope (SEM) incorporating a scanning tunneling microscope (STM), and a SFM was developed to monitor morphological changes and to measure mechanical properties of the nanometric oscillator fabricated by the surface diffusion method.

2. Instrumental

Figure 1 depicts the SEM sample stage with an STM/SFM and a heating device for oscillator fabrication. A tip holder of the STM/SFM is pressed against three ruby balls glued on the end of a tube piezo[7] by means of a spring. By applying voltage in the form of a saw tooth to the electrodes of the tube piezo to induce rapid radial motions, the tip holder can be actuated in the two radial directions independently. The configuration was developed independently by Baro and Kaneko, and is based on what is commonly called inertia drive or friction drive.[8–13] Details of the STM/SFM will be discussed in another report of this issue.

3. Experimental

3.1 Morphological changes of the heated tungsten tip

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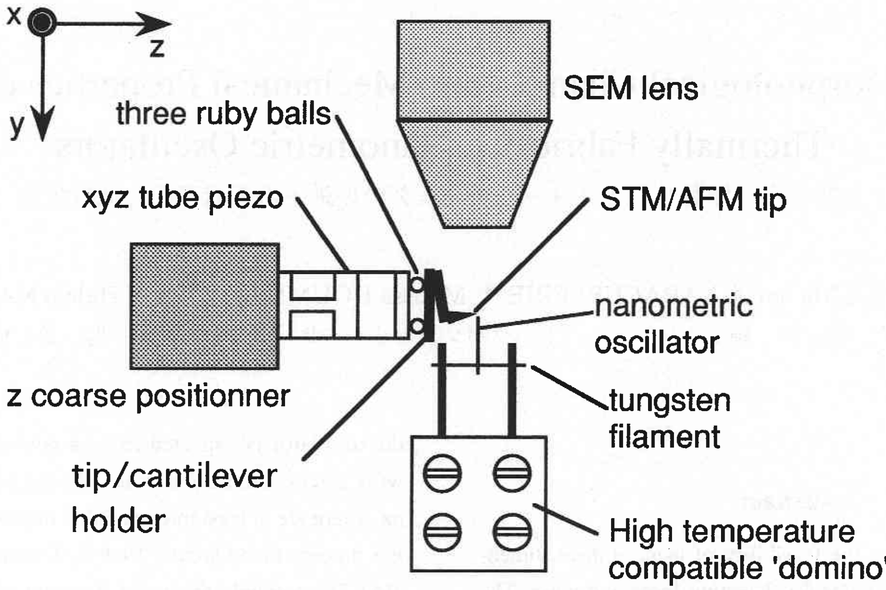

Figure 1　Schematic of the STM/SFM that mounts on a commercial SEM sample stage.

Tungsten wires with a diameter of 0.2 to 0.25 mm were etched in KOH solution either by DC or AC current. After obtaining a sharp tip with low cone angle in the range of 1 to 3 degrees, the tip was spot welded to a tungsten filament on the SEM14) sample stage and heated in vacuum of around 10^{-5} Torr. **Figure 2** shows some examples of the changes observed during one heating procedure. Although the heating process was not totally reproducible, what is shown in Figure 2 represent a basic trend. Quite often, the first necking occurred only a few minutes after the start of the heating procedure. The head either dropped off or was reintegrated into the base shank. The formation of the head and the neck and their reintegration is repeated a few times before the structure becomes a blunt tip of about 500 nm in diameter. We do not know at the moment whether the change of shape has stopped because the structure has found a stable form, or whether the surface of the tip was oxidized or contaminated to such an extent that apparent surface diffusion was suppressed. Between Figure 2 (c) and (e), one can see the formation of two heads. The content of the second head from the apex is sucked into the first head.

3.2 Mechanical property of the nanometric oscillator

We need to know the mechanical property of the nanometric oscillator to exploit its potential as a detector. Especially we are interested in the property of the filiform neck since it governs such factors as: (i) the spring constant and the natural frequency, (ii) internal loss and the Q factor, (iii) ductility and robustness, and (iv) electronic properties, such as the current-voltage characteristic across the neck. At the moment, we have only succeeded in tapping the nanometric oscillator with another tip to see if the neck withstands the shock. From experiment, we have found that tapping by another tip as well as a slow rotation of the SEM sample stage can exert enough disturbance to cause decapitation. The force exerted to the neck can be estimated to be a few orders of magnitude smaller than the maximum strength of tungsten. The neck may have been a bridge of contaminant layer, or a grain boundary may have existed at the neck confinement. To clarify the reason of the unexpected fragility of the neck, we need to carry out force measurement of the oscillator during the formation of the nanometric oscillator.

4. Conclusions

A SEM sample stage with a STM/SFM and a heating filament for fabrication of the nanometric oscillator by surface diffusion was developed. The three dimensional positionner functioned well in vacuum and the STM tip could be placed against the oscillator head. Morphological changes of a heated tungsten tip was observed in process in the SEM. A slight tapping on the nanometric oscillator was enough to break its neck.

The neck was much fragile than expected. In the future, we need to carry out measurement of the spring constant of the neck and current-voltage characteristics during the formation of

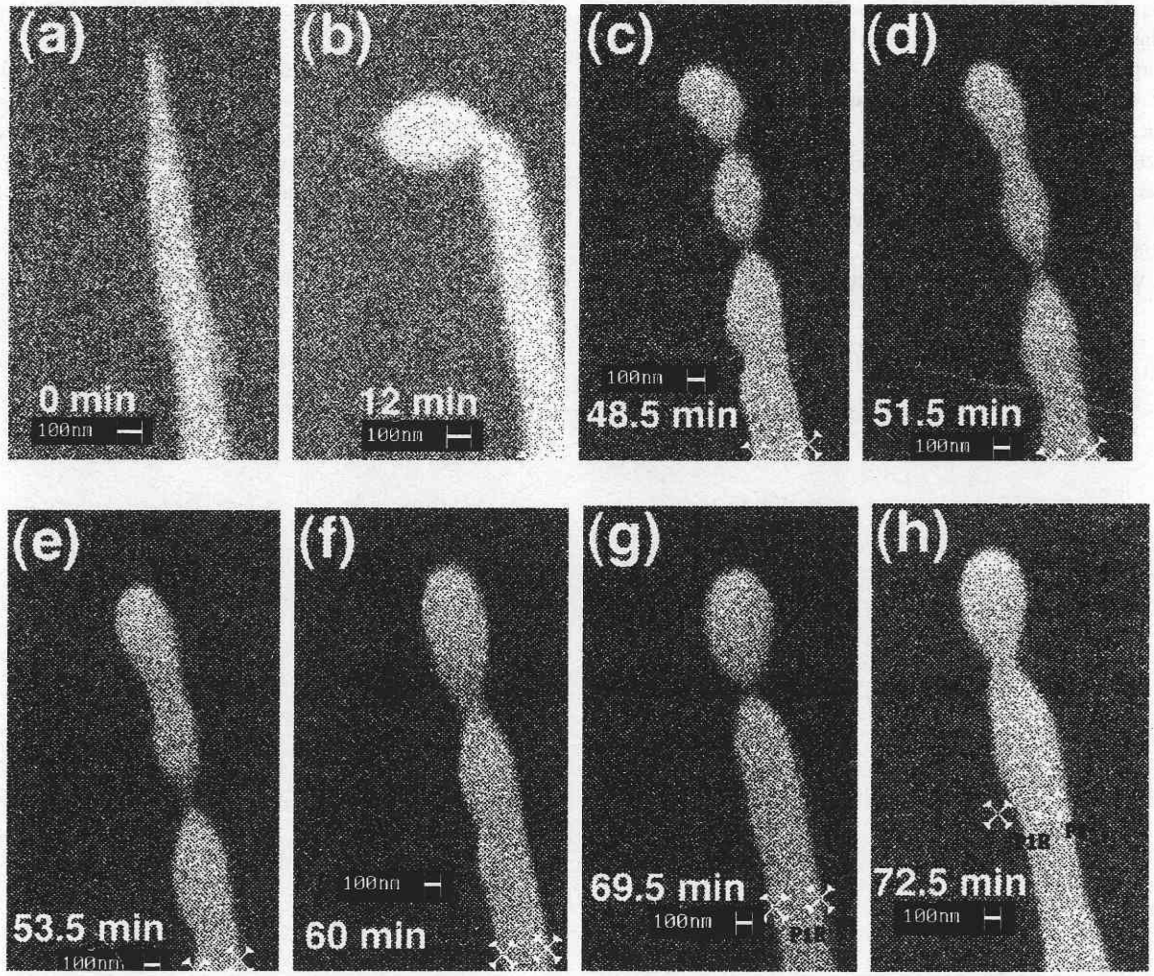

Figure 2　Morphological changes observed when heating a sharpened polycrystalline tungsten tip.

the oscillator to characterize the neck. In order to exploit the advantages of a nanometric mechanical structure as a sensing device, there is a great need to improve and develop a measurement bench of nanometric structures.

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Reference

1) G. Binnig, Ch. Gerber, and C. F. Quate, Phys. Rev. Lett. 56, 930 (1986).
2) K. Wago, O. Züger, R. Kendrick, C. S. Yannoni, and D. Rugar, J. Vac. Sci. Technol., B14, 1197 (1996).
3) D. A. Walters, J. P. Cleaveland, N. H. Thomson, P. K. Hansma, M. A. Wendman, G. Gurley and V. Elings, Rev. Sci. Instrum., 67, 3583 (1996).
4) R. P. Ried, H. J. Mamin, B. D. Terris, L. S. Fan, and D. Rugar, Transducers '97, 447 (1997). J. Brugger, G. Beljakovic, M. Despont, N. F. de Rooij, and P. Vettiger, Microelectronic Engineering 35, 401, (1997).
5) M. Drechsler, A. Piquet, R. Uzan and Vu Thien Binh, Surf. Sci., 14, 457 (1969). M. Drechsler, S. Ramadani, A. Claverie and A.

Maas, Journal de Physique, Colloque C6-209 (1987). Vu Thien Binh, M. Chaudier, J. C. Couturier, R. Uzan and M. Drechsler, Surf. Sci., 57, 184 (1976).
6) M. Hoummady, H. Fujita, H. Kawakatsu, and T. Masuzawa, J. Vac. Sci. Technol., B15, (1997).
7) S. Fukuda, D. Saya, M. de Labachelerie, and H. Kawakatsu, in this issue.
8) H. Kawakatsu and T. Higuchi, J. Vac. Sci. Technol., A8, 319 (1990).
9) D. W. Pohl, Rev. Sci. Instrum., 58, 54 (1987).
10) B. W. Corb, M. Ringger, and H. -J. Gütherodt, J. Appl. Phys., 58, 3947 (1985).
11) Ph. Niedermann, R. Emch, and P. Descouts, Rev. Sci. Instrum., 59, 368 (1988).
12) G. W. Stupian and M. S. Leung, J. Vac. Sci. Technol., A7, 2895 (1989).
13) M. Anders, M. Thaer and C. Heiden, Surf. Sci., 181, 176 (1987).
14) Leica, StereoScan.