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Measuring wear by combining friction force and dynamic force microscopy

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ABSTRACT

We introduce a technique for measuring wear on the nano-scale by combining friction force and dynamic force microscopy. By measuring the resonance frequency of the cantilever after scratching over a sample surface we are able to detect the increase or decrease of the tip's worn mass down to some picograms. Applying a recently developed technique to attach a small sphere to the upper end of the cantilever's tip we are able to measure the nano-wear of several material combinations with this approach.

1. Introduction

With the invention of the *friction force microscope* (FFM) in 1987 [1] this technique became the standard tool to examine friction on the nano-scale [2–5]. Since friction is closely related to wear the FFM is also frequently used to measure wear from the micro- down to the atomic-scale (for a recent review, see, e.g., Ref. [6]). From the technical point of view, however, some difficulties arise if wear is detected with a friction force microscope. While the wear of the sample surface is easily measured via a change of the topography [7–13], it is not possible to measure the wear of the tip simultaneously in a direct way.

Although, it is well-know by experimentalists from everyday experience that a tip becomes blunt after scanning the sample surface for some time, additional efforts are needed to measure this wear process quantitatively. It is, for example, possible to image the blunt tip after the wear experiment with a *scanning electron microscope* [14]. Unfortunately, this procedure is very time consuming, since the probe (the tip attached to the cantilever) has to be transferred from the FFM to the SEM (and vice versa if wear should be continuously monitored). Another option is the indirect measurement of the tip's shape via a grating sample which consist of an array of sharp tips [15]. Nonetheless, this tip qualification process may also induce wear and this approach relies on the change of the sample in the friction force microscope which is again time consuming. Additionally, both approaches prevent the scanning of the very same area after the retransfer of the probe or sample back to the FFM. Gotsmann and Lantz [16] solved these issues by measuring the wear of their tip indirectly via the detection of the adhesion force between a silicon tip and a polymer sample. This direct link between adhesion and wear, however, was based on the fact that their tip had a perfect flat punch geometry and their polymer sample was extremely flat. An instance which (unfortunately) cannot be generalized to all tip/sample configurations; specially when surface roughness becomes important.

In order to quantify the wear of the tip in friction force microscopy we suggest to combine wear experiments with the mass sensing ability of dynamic force microscopy (see, e.g., Refs. [17–19] and references therein). The basic idea is to measure the mass change of the tip via the change of the resonance frequency of the freely oscillating cantilever. With this approach it is easily possible to detect mass changes down to some picograms using a standard dynamic force microscope or even lower with particularly dedicated set-ups [20].

In this article we report on our wear experiments where we applied this technique to various tip/sample combinations. In order to extend the choice of the tip material from silicon to other materials we prepared colloid probes using a recently introduced approach [21]. After giving the basic technical details of the proposed method in Section 2 we present several measurements with different types of samples and tips (Section 3). Finally, we summarize the results and give an outlook on possible technical improvements and future applications in Section 4.

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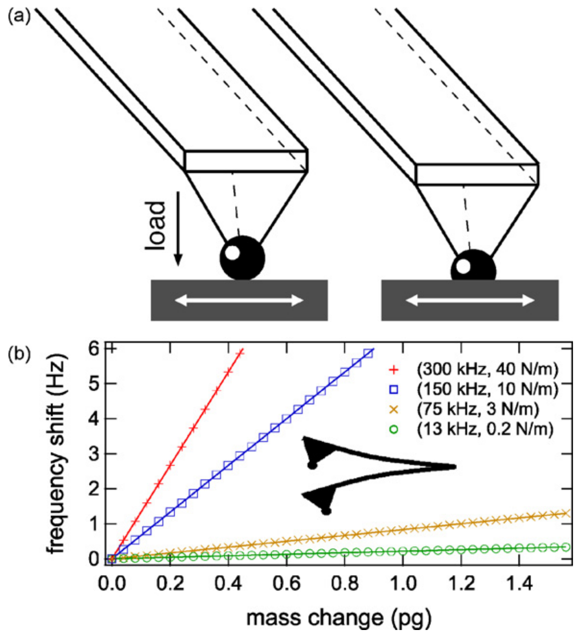

Fig. 1. Basic principle of the introduced wear measurement procedure. (a) If a spherical tip scans over a sample surface, the sphere losses (or picks up) material due to the wear process between sphere and surface. Assuming for example that the material of the tip is considerably softer than the sample, the tip becomes blunt during scratching. (b) This mass change of the tip can be detected as a change of the resonance frequency of the cantilever. The graph shows the linear relationship between the frequency shift and the worn mass for four different types of cantilevers.

2. Experimental

The basic idea of the introduced measurement technique is schematically shown in Fig. 1. A cantilever scans a sample surface in direct mechanical contact which results into a wear process (Fig. 1(a)). In order to have a defined geometry we used colloid probes where small spheres of µm-size are attached to the tip of the cantilever prepared with a recently introduced technique [21]. In this way we can use arbitrary combinations of tip and surface materials. The change of mass of the tip due to wear was measured by detecting the change of the resonance frequency of the cantilever. The correlation between the mass change Δm and the frequency shift Δf is given by

$$\Delta f = f - f_0 = \frac{1}{2\pi}\left(\sqrt{\frac{c_z}{m_{\mathrm{eff}} + \Delta m}} - \sqrt{\frac{c_z}{m_{\mathrm{eff}}}}\right), \quad (1)$$

where f is the actual resonance frequency and f_0 the original eigenfrequency of the cantilever. The effective mass and the spring constant are denoted as m_{eff} and c_z. This equation can be simplified presuming that the eigenfrequency of the oscillating cantilever is much higher than the occurring frequency shift [18]:

$$\Delta f \approx -\frac{2\pi^2 f_0^3}{c_z}\Delta m. \quad (2)$$

A closer look to this equation reveals that the frequency shift increases with larger eigenfrequencies and smaller spring constants. The graph in Fig. 1(b) shows the linear relationship between the frequency shift and the mass change for four different types of commercially available cantilevers. Since this comparison suggests that cantilevers designed for dynamic modes with the nominal parameters 300 kHz and 40 N/m have the highest sensitivity from these four types we used mostly this type of cantilever in our experiments. Finally, we would like to point out that the constant of proportionality is negative, i.e., if mass is removed from the sphere ($\Delta m < 0$) due to wear or other reasons the frequency shift is positive while a pick-up of material ($\Delta m > 0$) leads to a decreasing frequency shift.

For our experiments we used a commercial atomic force and friction force microscope (EnviroScope with Nanoscope IIIa Controller, Veeco Instruments Inc.) where the measurements can be done inside a chamber under a controlled environment. The wear measurements were done in contact mode where the cantilever scratches continuously with a constant load over a rectangular area of the surface. The spring constant of the cantilever is measured with the method of Sader et al. [22]. Afterwards the cantilever is retracted from the sample surface and its resonance frequency is

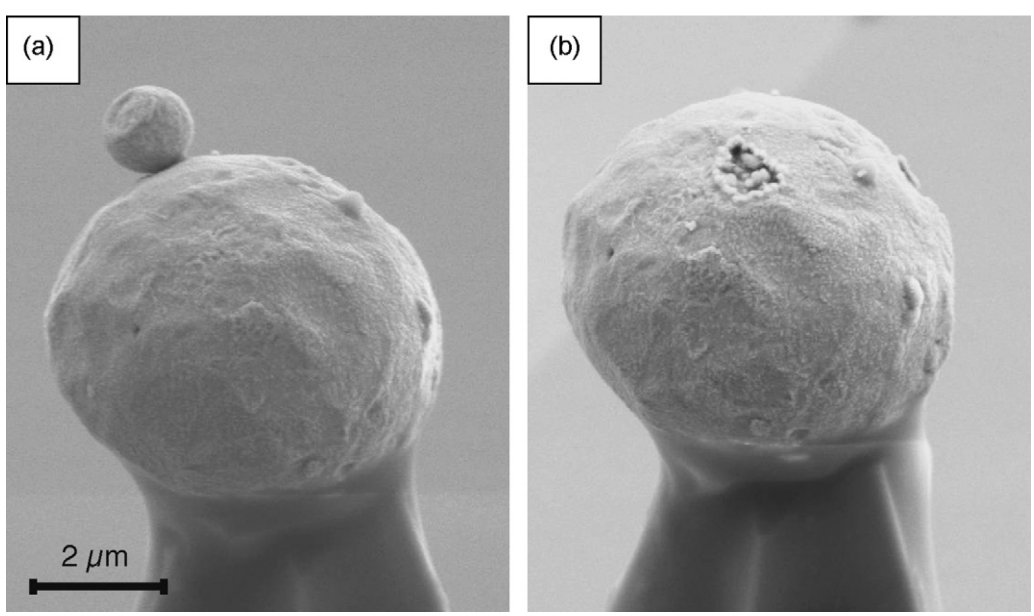

Fig. 2. (a) An SEM image of a copper sphere ($d = 6.2$ µm) before scanning a sample surface. A much smaller copper sphere with a diameter of $d = 1.4 \pm 0.15$ µ m (measured by SEM) is attached to it. Immediately after the first scanning of the sample surface we noticed a large increase of the frequency shift of $\Delta f = +110$ Hz corresponding to a mass loss of $\Delta m = 9.13$ pg leading to a calculated diameter of $d = 1.25$ µm. (b) The SEM image taken after this observation revealed that the smaller sphere was broken off from the larger sphere. Comparing the AFM experiment with the SEM observation we get an agreement which is within the anticipated error of 0.15 µm.