Colloids and Surfaces A: Physicochem. Eng. Aspects 489 (2016) 86–94

Contents lists available at ScienceDirect

Colloids and Surfaces A: Physicochemical and Engineering Aspects

journal homepage: www.elsevier.com/locate/colsurfa

Calibration of effective spring constants of colloidal probes using reference cantilever method

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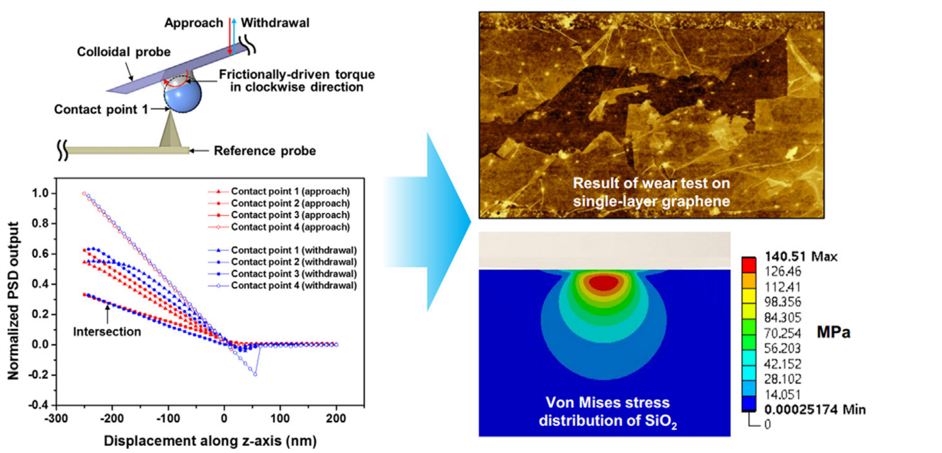
HIGHLIGHTS

- We propose a method of directly calibrating the effective spring constant of a colloidal probe's cantilever by the reference cantilever method.
- The effective spring constants of the colloidal probes by the proposed method were compared to those obtained by the FEA.
- The importance of the colloidal probe's spring constant in mechanical tests was investigated by investigating force distance curves and nanoscale wear test results.
- The von Mises stress distributions for contact between the spherical particle and the sample surface were obtained.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 10 July 2015
Received in revised form 22 October 2015
Accepted 24 October 2015
Available online 29 October 2015

Keywords:
Colloidal probe
Spring constant
Force–distance curve
Tribological test
Contact load
Finite element analysis

ABSTRACT

The precise measurement of the effective spring constant of a colloidal probe's cantilever is very important for the accurate application of a desired contact load to a sample surface since the contact load is determined by multiplying the effective spring constant of the cantilever and its deflection. This paper presents a method of directly measuring the effective spring constant of a colloidal probe by the reference cantilever method. The distinctive feature of the reference cantilever method in this study is that the reference cantilever has a tip. The tip determines the location of the loading point on the reference cantilever and enables detecting the loading point on the colloidal probe's cantilever by monitoring the change in a force distance curve depending on the contact point between a spherical particle and the tip. The effective spring constants of the colloidal probes measured by the proposed method are compared to those determined by finite element analysis (FEA). The importance of calibrating the effective spring constant of the colloidal probe in applied forces and mechanical tests is investigated by comparing force distance curves and nanometer-scale wear test results obtained at two different spring constant settings.

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http://dx.doi.org/10.1016/j.colsurfa.2015.10.043
0927-7757/

1. Introduction

Atomic force microscopy (AFM) has widely been used for acquiring surface images with atomic resolution and studying tribological characteristics of materials [1–3] at the nanometer scales using lateral force microscopy (LFM). In LFM normal loading is controlled in nanonewton load resolution. Such high resolution of measuring contact loads is achieved by the combination of a microfabricated cantilever and an optical detection system with nanometer resolution to track the displacement of the probe tip located at the end of the cantilever. The contact load applied to a sample surface by the tip is determined by multiplying the spring constant of the cantilever and its deflection [4]. Therefore the accuracy of the measurement of the cantilever's spring constant is extremely important for the precise application of a desired contact load and for the improvement of reliable tribological tests. Several calibration methods, such as the Sader method [5], the thermal noise method [6], and the reference cantilever method [7,8], have been developed and widely used to calibrate conventional AFM probes.

To evaluate tribological characteristics of materials, LFM using conventional AFM probes is dominantly used. However tribological tests employing conventional AFM probes have significant limitations. Extremely high contact pressures are generated on sample surfaces due to nanometer-tip radii of conventional AFM probes (10–30 nm) and therefore they do not properly represent the contact pressures experienced by sample surfaces in practical applications. Furthermore, during tribological tests a tip with a nanometer-tip radius is rapidly worn out due to the high contact load generated on its end and thus the size of the contact between the tip and sample changes, leading to an unwanted variation in contact conditions. In order to overcome these limitations, a tip in a conventional AFM probe is replaced by a micro-sized spherical particle creating what is referred to as a colloidal probe. The micrometer-scale spherical particle of a colloidal probe can generate practical contact pressures because of its relatively large radius. The well-defined contact between a particle and a flat surface enables the interpretation of the contact behavior through contact models such as Hertz [9], Johnson–Kendall–Roberts (JKR) [10], and Derjaguin–Muller–Toporov (DMT) [11] since the indentation depth is much less than the radius of curvature of the micro-sized spherical particle. Owing to these advantages, the colloidal probe has been popularly used to study tribological [12,13], mechanical [14–17], and interfacial [18–20] characteristics of various materials.

Although the advantages of the colloidal probe have been reported by many researchers, studies on calibration methods of the spring constant of the colloidal probe have not been conducted sufficiently. One common approach is measuring the spring constant of a tipless cantilever by one of the existing calibration methods, mentioned in the previous paragraph, before the attachment of a spherical particle [21]. However this approach cannot consider the influence of the spherical particle on the spring constant of the colloidal probe. Only a few studies have been conducted to calibrate the effective spring constant of the colloidal probe after the attachment of a spherical particle. Chung et al. suggested a method of determining the spring constants of conductive colloidal probes by an electrostatic force and the possibility of calibrating them by the thermal noise method [22]. Heim et al. compared the spring constants of the tipless cantilevers and the colloidal probes determined by the thermal noise method and confirmed that the colloidal probes can be directly calibrated by the thermal noise method [21].

In this study, we propose a method of directly measuring the effective spring constant of the colloidal probe after the attachment of a micrometer-scale spherical particle by the reference cantilever method [7,8]. This calibration method requires a reference cantilever and its procedure is simply composed of two steps. First, a force distance curve is obtained by pressing an unknown cantilever against a flat and rigid surface, and the slope of the curve is recorded. Second, a force distance curve is generated by pressing the unknown cantilever against the reference cantilever, and its slope is measured. The two slopes obtained provide the relation between the spring constant of the reference cantilever and that of the unknown cantilever and thereby the spring constant of the unknown cantilever can be calculated. Slattery et al. mentioned that the uncertainty of calibrating the spring constant of a conventional AFM probe by the reference cantilever method mainly depends on three factors: calibrating the spring constant of a reference cantilever, detecting the loading point between a tip and a reference cantilever, and matching the spring constants of a test cantilever and a reference cantilever. They suggested a technique to decrease the calibration uncertainty by precisely finding the loading point between a tip of a conventional AFM probe and a reference cantilever with the positional markers produced by focused ion beam (FIB) milling [23]. However, in the case of calibrating the spring constant of the colloidal probe by the reference cantilever method, one more factor which influences the uncertainty should be taken into account. The factor is detecting the loading point on the colloidal probe's cantilever determined by the location of the spherical particle. The method introduced in this study measures the effective spring constant of a colloidal probe by considering the two loading points: one is the loading point between a reference cantilever and a spherical particle, and the other is the loading point between a colloidal probe's cantilever and the spherical particle. The distinguishing point of this method from the usual reference cantilever method is that the reference cantilever has a tip. The tip determines the loading point on a reference cantilever and enables detecting the loading point on a colloidal probe's cantilever by observing the change in a force distance curve according to the contact point between the spherical particle and the tip. Finite element analysis (FEA) is conducted to obtain the effective spring constants of the colloidal probes in order to assess the validity of the proposed method. The significance of calibrating a colloidal probe's spring constant in mechanical tests is investigated by analyzing force distance curves and nanometer-scale wear test results, which are obtained at two different spring constant settings: one is the effective spring constant of a colloidal probe, and the other is the spring constant of a tipless cantilever commonly used as its alternative.

2. Experimental details

2.1. Calibrating spring constants of colloidal probes by reference cantilever with tip

The effective spring constant of a colloidal probe, k_{EC}, was measured by the reference cantilever method using a reference cantilever with a tip. The use of the reference cantilever with the tip enabled determining the effective loading points on the reference cantilever and the colloidal probe's cantilever. An AFM (SmartSPM 1000, AIST-NT, USA) was used to calibrate k_{EC} and the effective spring constant of the reference cantilever, k_{ER}. Five colloidal probes were calibrated, and the calibration results were compared with those obtained by the FEA.

The end and cross section of the reference cantilever (fpN11TiN, AIST-NT, USA) were rectangular. Its length, L_R, and width, W_R, were measured at 5 different locations by an optical microscope (PSM 1000, Motic, Hong Kong) and its quality factor, Q_R, and resonance frequency, f_R, were measured by the AFM. Finally, the spring constant of the reference cantilever, k_R, was determined by averaging ten calibration results obtained by the Sader method [5], which is suitable for calibrating rectangular cantilevers. Further, k_{ER} was calculated by taking account of the actual loading point determined