International Journal of Adhesion & Adhesives 60 (2015) 109–116

Contents lists available at ScienceDirect

International Journal of Adhesion & Adhesives

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

Effects of system dynamics and applied force on adhesion measurement in colloidal probe technique

Jaeho Lee, Jayendra Maharjan, Muyang He, Chang-Dong Yeo*

Department of Mechanical Engineering,Texas Tech University, Lubbock, TX 79409, USA

ARTICLE INFO

Article history:
Accepted 16 April 2015
Available online 24 April 2015

Keywords:
Dynamic adhesion
Pull-off force
Pull-in force
Colloidal probe technique
Sliding

ABSTRACT

Dynamic pull-in/pull-off forces were quantitatively measured using an AFM colloidal probe technique. Two spherical colloids made of silicon dioxide (SiO_2) and gold (Au) that were attached to an AFM cantilever were approached to and retracted from a silicon wafer specimen, where the speed of tip approaching/retracting (i.e., vertical dynamics) and specimen sliding (i.e., horizontal dynamics) was controlled. First, when the vertical dynamics of colloidal tip was applied on the stationary silicon wafer specimen, it was observed that the slower tip approaching showed higher pull-in force, while the pull-off force was dependent on both the applied force and the retracting speed. For the two colloidal tips, it was found that the higher applied force and the faster tip retraction led to the higher pull-off force. Next, under the constant speed of tip approach and retraction, horizontal dynamics was applied to the silicon wafer specimen. It was observed that the horizontal motion of the specimen made the pull-off force lower, which could be attributed to the breakage of adhesive asperity junctions at the interface. The pull-off force was further decreased at faster horizontal motion of the specimen due to the longer sliding distance. Therefore, from the systematic experiments of dynamic adhesion measurement, it could be known that if a micro/nano-system is under dynamic surface interaction, its adhesive force cannot be fully described by a conventional quasi-static adhesion model but it should include the effects of applied system dynamics in both normal and tangential directions.

1. Introduction

Quantitative analysis of adhesive force enables better understanding of fundamental surface interactions in a variety of fields. In general, the pull-in (during approaching) and the pull-off (during retracting) forces are used to investigate (a) adhesion phenomenon of materials, (b) particle to surface interaction, (c) surface properties, and (d) hydrodynamics and boundary slip [1–5]. In order to get more physical insight into these research topics, it is highly required to improve the reliability and accuracy of adhesive force measurement in instrumentations.

With its well-developed techniques, atomic force microscopy (AFM) is widely used to study adhesion mechanics of materials. Since AFM adhesion force is mainly caused by van der Waals and capillary interactions between a tip and a counteracting surface, the measured force value can be used to characterize the surface free energy of materials [6,7]. In addition, recently many researchers have applied AFM technologies to investigate mechanical behaviors of biomaterials. It has been reported that the measured interactive forces could be used to evaluate (i) the stiffness and elasticity of bacteria, (ii) cell to cell adhesion, and (iii) adhesion between individual ligand–receptor pairs [8–12]. On the other hand, to avoid instrumental limitations in AFM such as errors in calibration and twisting of the cantilever, some researchers have measured adhesive forces using capacitive force sensors or nano-tubes to analyze surface properties of materials [13,14].

Despite those outstanding research works in adhesion, experimental study of surface adhesive force may have critical issues in reproduction or repeatability of the measurements. For example, when pull-in/pull-off forces are measured on a certain material, the measured force value could be very dependent upon the instruments and environments used in experiments [2,15–17]. If environmental conditions are controlled to be the same, the applied dynamic loading/unloading profiles in the instruments could affect the resulting adhesive force values. Yeo et al. [14] investigated the change in adhesive interactive force of solid materials under various approaching and retracting speeds. It was observed that the slower approaching caused earlier jump-in behavior with higher pull-in force, while the slower retracting showed later jump-out behavior with higher pull-off force. This dynamic adhesion behavior could be explained by the attractive surface force coupled with the compliant structure of the capacitive force transducer.

* Corresponding author. Tel.: +1 806 834 5452; fax: +1 806 742 3540.
E-mail address: changdong.yeo@ttu.edu (C.-D. Yeo).

http://dx.doi.org/10.1016/j.ijadhadh.2015.04.004
0143-7496/

In addition, for the convenience of analysis, researchers usually predict adhesive force using quasi-static adhesion models. However, it is frequently observed that the actual adhesive behaviors or experimental adhesion measurements are different from the estimation of those quasi-static models due to unexpected surface properties (e.g., roughness, contaminant layer or particles, etc) of the materials, environmental conditions (e.g., temperature and humidity), and system dynamics of interacting bodies. Even though many surface interactions of engineering devices are carried out under various system dynamics, its effects on the adhesive behavior has not been reported much compared to the related research on surface properties and environments.

In this study, the scientific relationship between the applied system dynamics and the adhesive force was systematically investigated using an AFM colloidal probe technique. In experiment, a piezo stage was installed under an AFM scan head to enable horizontal dynamics (=sliding motion) to a specimen, while the vertical dynamics (=tip approach/retraction) of a colloidal tip was managed through AFM controller. Then, under both vertical and horizontal dynamics of the interacting bodies, dynamic pull-in/pull-off forces were measured with applied surface force. To verify the experimental results, a non-linear system dynamics model was developed and analytical simulations were performed under various dynamic conditions.

2. Instrumentation and sample preparation

2.1. Surface roughness

When two bodies are in contact or non-contact interaction, the interactive forces can be affected by their topographical parameters. The specimen in this experiment was silicon (SiO_2) wafer which was cut in square 0.7 cm × 0.7 cm and had thickness of 1 mm. Its surface roughness parameters were measured using Asylum MFP-3D Atomic Force Microscope. Based on the scan size of 5 μm × 5 μm with 512 lines, the measured root mean square (RMS) roughness was 2.32 nm.

2.2. Surface energy

The adhesive force between two interacting bodies is directly proportional to their surface energy values [18,19]. In this experiment, the surface energy of silicon wafer specimen was obtained from contact angle measurement [20]. Using a microsyringe assembly and a high resolution camera (magnification of × 150), a small droplet was applied onto the silicon wafer surface and then its contact angle was measured. To determine both dispersive and polar component of surface energy, two reference liquids were applied, i.e., methylene iodide and deionized (DI) water. Methylene iodide interacts only with dispersive force, and its dispersive surface energy is 51.0 mJ/m^2 ($=\gamma_l^d$). On the other hand, DI water is interactive with both dispersive and polar forces, and its corresponding dispersive and polar surface energies are 21.8 mJ/m^2 ($=\gamma_w^d$) and 51.0 mJ/m^2 ($=\gamma_w^p$), respectively. Therefore, if the measured contact angles are put into the following equations (Eqs. (1) and (2)), the dispersive (γ_s^d) and polar (γ_s^p) surface energy of the silicon wafer specimen can be determined as follows [20]:

$$\gamma_s^d = \gamma_l^d (1 + \cos\ \theta_1)^2/4, \tag{1}$$

$$\gamma_s^p = \left[\left(\gamma_w^d + \gamma_w^p\right)(1 + \cos\ \theta_2) - 2\sqrt{\gamma_s^d \gamma_w^d}\right]^2 / 4\gamma_w^p, \tag{2}$$

where θ_1 and θ_2 are the measured contact angle from methylene iodide and DI water, respectively. To check the variation of surface energy over the specimen surface, the contact angles were measured on five different spots. From the experimental measurements, it was found that the dispersive and polar surface energy of the silicon wafer was 18.9 mJ/m^2 and 39.1 mJ/m^2, respectively (i.e., the total surface energy = 58.0 mJ/m^2).

2.3. Dynamic adhesive force measurement using AFM colloidal probe technique

Two commercial AFM colloidal tips (manufactured by NanoAndMore) were used in this dynamic adhesive force measurement. One was the SiO_2 colloidal tip, where a spherical SiO_2 colloid with the diameter of 6.62 μm was attached to an AFM cantilever of the stiffness constant (k) = 2.8 N/m. The other was the Au colloidal tip, where a spherical Au colloid with the diameter of 5.5 μm was attached to an AFM cantilever of the stiffness (k) = 3.0 N/m. Fig. 1 shows the scanning electron microscope (SEM) image of the spherical colloid which was provided by the manufacturer. Using a high resolution AFM (Nanosurf Mobile-S), the two different colloidal tips were approached to and retracted from the silicon wafer surface. As shown in Fig. 2, the vertical dynamics of the colloidal tips (i.e., tip approaching and retracting velocity) was applied through AFM controller, while the horizontal dynamics of the specimen (i.e., sliding velocity) was managed by the

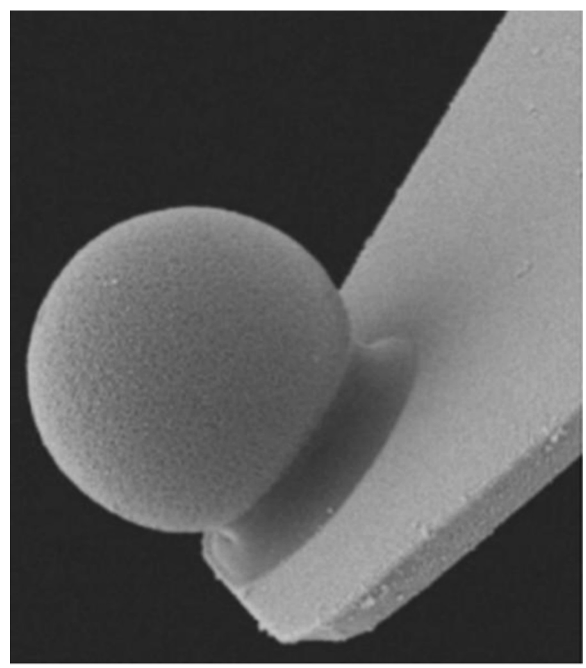

Fig. 1. The SEM image of the spherical colloid (provided by NanoAndMore).

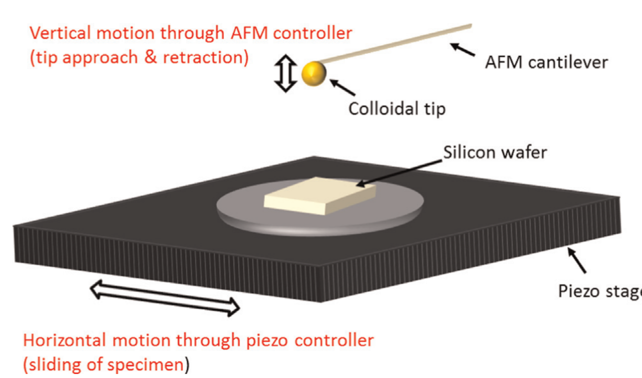

Fig. 2. Schematic of adhesive force measurement in this experiment under vertical and horizontal dynamics.