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Dynamic enhancement in adhesion forces of truncated and nanosphere tips on substrates

Hongjun Zhou[a, †], Quan Xu[a, †], Shaowei Li[a, †], Yanjun Zheng[a], Xu Wu[a], Chaokang Gu[c], Yusheng Chen[d], Jian Zhong[b, *]

[a] State Key Laboratory of Heavy Oil Processing, China University of Petroleum (Beijing), 102249, China

[b] College of Food Science & Technology, Shanghai Ocean University, Shanghai 201306, China

[c] EnerVault Corporation, 1244 Reamwood avenue, Sunnyvale, California, 94089, USA

[d] Department of Chemistry, University of Akron, Akron 44325, USA

[†] These authors contributed equally to this work.

[*]Corresponding authors: Jian Zhong

Jian Zhong, College of Food Science & Technology, Shanghai Ocean University,

Shanghai 201306, China; E-mail: jzhong@shou.edu.cn, jianzhongpku@hotmail.com.

Abstract: The understanding of the attachment and detachment of micro/nanomaterials to and from a substrate can benefit a wide range of interface scientific fields. Here, we fabricated two types of AFM tips: nanosphere tip with a SiO_2 nanoparticle having a diameter of 980 nm to AFM tip and truncated tip with a triangle surface having a side length of 959 nm at the end of AFM tip. Then we studied the dynamic adhesion enhancement research using AFM. When the retraction velocity increased from 0.02 µm/s to 156 µm/s, the adhesion force of the two tips to four different substrates significantly increased and the highest increase was on sapphire substrate. The order of the adhesion forces of nanoscale samples (nanophere tip and truncated tip) from different substrates was: HOPG > silicon wafer > mica > sapphire, which is reversely consistent with that of Young's modulus of the substrates. In addition, the dynamic forces of truncated tip were larger than those of nanosphere tip. Therefore, the tip shape and substrate had obvious effects on the dynamic adhesion forces. However, the modelling results showed that both the tip shape and substrate type had no obvious effect on the dynamic scaling parameters n and dynamic coefficient parameter C. Our work provide useful information for analyzing the interactions between micro/nanomaterials and their environments. It benefits the understanding of numerous natural phenomena and the development of artificial micro/nanomaterials.

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Key words: Adhesion force; Atomic Force Microscopy; Dynamic Model; SiO_2 particles;

Truncated tip

1. Introduction

Micro/nanomechanics is an important branch of micro/nanoscience investigating and understanding the fundamental mechanical properties of materials at the micro/nanometer scale. The attachment and detachment of micro/nanomaterials to and from a substrate are a fundamental problem relevant to numerous natural phenomena and application process of artificial micro/nanomaterials. The understanding of the dynamic mechanical behaviors of micro/nanomaterials from/to substrates can benefit a wide range of interface scientific fields including interface between microwave and optical photons,[1] surface cleaning,[2] drug delivery systems developments,[3] in vivo implants development,[4] cell adhesion,[5] nanomaterials design,[6] sensors,[7] and molecular recognition.[8-9] Therefore, it is pivotal to understand the dynamic mechanical process in the attachment and detachment of micro/nanomaterials to and from a substrate. To the best of our knowledge, the shape effect of the micro/nanomaterials on the dynamic mechanical process has not been studied.

Atomic force microscopy is an appropriate and pivotal nanotechnology tool for nanomaterials research. It can be used to measure the surface properties such as morphology and mechanical properties of many materials and specimens at nanoscale level.[10-12] Moreover, it can be used to fabricate materials in a precise, controllable and

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reproducible fashion at nanoscale level.[13-15] Especially, AFM is a pivotal tool to obtain

the dynamic interaction force informations by analyzing the tip approaching and

retracting process to/from the substrates (Figure 1). Compared with other dynamic

interaction force measurement methods (i.e. surface force apparatus,[16] optical tweezers,[17]

and magnetic tweezers[18]), atomic force microscopy (AFM) has several advantages such

as nanometer positional accuracy and piconewton force sensitivity.[9] Therefore, AFM can

avoid deformation of the substrates and obtain tiny force response of materials surface

during the force measurements. Such characteristics offer AFM the opportunity to study

the fundamental mechanisms of materials research and to guide the development of novel

materials.

In this work, to study the effects of the sample shapes and substrate types on the

detachment mechanical behaviors of samples with substrates, we fabricate two types of

AFM tips by attaching nanospheres with a diameter of 980 nm to AFM tip or truncating

AFM tip to form triangle surfaces with a side length of 959 nm at the end of AFM tip.

Then, we study the detachment mechanics by AFM dynamic analysis.

2. Materials and Methods

2.1. Preparation of nanosphere and truncated tips

Nanosphere tips were prepared as below. SiO_2 nanospheres were purchased from supplier (Microsphere-nanosphere Co., Inc.,Cold Spring, New York, USA) and deposited in a fresh silicon wafer. The wafer was then maintained in Ar environment for 24 h to decrease the form of water bridge between nanospheres and wafer. To fabricate nanosphere tip, SiO_2 nanospheres with different diameters were selected and picked up by a nanomanipulator installed in scanning electron microscope (SEM-FEI Helios NanoLab, Cleveland, USA), followed by welding at the end of an platinum AFM tip (Probe type: RTESPA-150, Bruker, Inc.). The size of the nanospheres tips was measured by SEM.

Truncated tips were prepared as below. Fresh tips (Probe type: OLTESPA, Bruker. Inc.) were precisely truncated by focus ion beam (FIB). The size and shape of the truncated tips were precisely controlled by using a beam of focused ions on the probes. The size and shape were analyzed by SEM.

2.2. Substrate Used in This Work

Four substrate samples (mica, sapphire, silicon wafer (100), highly oriented pyrolytic graphite (HOPG)) were purchased from Bruker Co., Inc. All the substrates were cleaned with deionization water in ultrasonic cleaner (Branson Inc.), dried in nitrogen environment, and then cleaned with oxygen plasma treatment to eliminate surface organic

contamination. Surface roughness was measured by AFM under contact mode (Scan-Icon

AFM, Bruker Co., Inc.). Young's Modulus of these samples were measured under Bruker

Peak Force (PF-QNM) Technology.

2.3. Test procedure

The nanosphere and truncated tips were cleaned with oxygen plasma treatment to

eliminate surface organic contamination. The exact spring constants of the nanosphere

and truncated cantilevers were determined by thermal tune method (Bruker

Dimension-Icon AFM) and re-checked after each experiment. No obvious differences

were observed before and after each experiment (less than 2% error was observed).

Tests were carried out in sealed electrochemical cell (Bruker ECAFM) filled with

nitrogen (99.99% dry nitrogen) at room temperature. The dry nitrogen environment was

established by pumping the dry nitrogen into the sealed system for 12 h to eliminate the

humid effect. The approaching velocity was 1 μm/s. A loading force of 100 nN was used

for the dynamic test except in load force measurement experiments. This applied load

force gave pressure far beyond the yielding strength of the four substrate to exclude the

plastic deformation on the substrate during the test. For truncated tips, the optimal tilt

angle was adjusted before the test to ensure that the surface was perfectly parallel with

the substrates and maximum force was achieved. All adhesion forces were measured at least 10 times to ensure accuracy and consistency.

2.4. Modelling

Previous researches have proved that the dynamic enhancement in adhesion force between micropaticles and substrates is induced by fast crack propagation along the microparticle tip-substrate interface. According to that, the retracting process of a microparticle tip from a substrate can be considered as a crack propagation process along the microparticle tip-substrate interface. In previous researches, a relationship between microparticles and substrate was summarized:[19-20]

$$F = F_0 \left[1 + C \left(\frac{V}{V_0} \right)^n \right] \tag{1}$$

Where F_0 is the adhesion force as V is approaching to zero, V_0 is the reference velocity when $F = F_0[1 + C]$, and n and C are the dynamic parameters depending on the systems. Equation (1) was used to model the dynamic enhancement in adhesion force between nanoscale tips (nanosphere tip and truncated tip) and substrates.

3. Results and Discussion

3.1. Nanoscale AFM Tip Preparation

Previous work showed contact area of microscale samples does not have significant contribution to retraction rate-dependent adhesion.[21] However, it is not clear if contact

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geometry of samples, especially nanoscale samples, has significant contribution to the

dynamic adhesion. Here, in order to analyze the effect of contact geometry of nanoscale

samples on the dynamic adhesion forces from the substrates, nanosphere AFM tips and

truncated AFM tips were used to mimick nanoscale spheres and nanoscale polyhedrons in

this work. Nanosphere tips were prepared by attaching SiO_2 nanospheres onto the tips of

RTESPA AFM probes using a nanomanipulator installed in scanning electron microscope.

Typical nanosphere tip with a diameter of 980 nm was shown in Fig. 2A. Truncated tips

were prepared by truncating the tips of OLTESPA AFM probes by focus ion beam. The

end shape of the truncated tip was a triangle. Typical truncated tip with a side length of

959 nm was shown in Fig. 2B. The XPS survey of the two tips are shown in Figure S1, as

can be seen from the results, they have very similar compositions.

3.2. Substrates Used in This Work

Previous studies unrevealed that surface roughness played important role in

affecting adhesion forces.[22] In order to minimize the effect of surface roughness, four

substrate samples (mica, sapphire, silicon wafer (100), highly oriented pyrolytic graphite

(HOPG)) with low surface roughness (< 1 nm, Table 1) were used in this work. Therefore,

the effects of surface roughness could be ignorable for our analysis. The Young's

Modulus of these samples showed obvious differences from 18 GPa to 345 GPa, as

shown in Table 1.

3.3. Optimization of Experimental Conditions for the Dynamic Measurements

High humidity environment may affect the adhesion forces between samples and

substrates. In humid air, the capillary force can be formed at interface between probes

and substrates and further enhance the dynamic effects.[19, 23] In order to minimize

humidity effects, the AFM tips and substrates were mounted into a sealed

electrochemical cell and dry nitrogenous environment was established by pumping dry

nitrogen (99.99%) into the cell for more than 12 h.

In traditional JKR and DMT model for dynamic interaction,[24] retraction velocity has

not been considered as an important factor. However, recently, more and more studies

revealed that it could be an important factor in affecting the separation process and local

area crack propagation.[19, 21, 25-26] The separation of the microsphere and flat surface from

a substrate can be considered velocity dependent as a crack propagation process along the

sphere-substrate interface.[27-28] The separation between probes and substrates is more like

a peeling process instead of perfect instant separation predicted by JKR and DMT model.

According to the Griffith energy balance concept, during fast loading process the kinetic

energy becomes important.[29] The propagation is strongly controlled by the loading type,

loading rate and materials rheology. In this brittle case, we notice the retraction speed

also determines the pull-off speed of the nanoparticles, affecting the crack propagation

rate and the energy release rate, as well as the adhesion forces.[19-20]

   To correctly analyze the dynamic adhesion between nanoscale samples and

substrates, it is necessary to optimize the experimental parameters (load force and contact

time) in AFM dynamic measurements. Here, nanosphere tip with a diameter of 980 nm

(Fig. 2A) and truncated tip with a side length of 959 nm (Fig. 2B) were applied. The

pull-off curves of these tips on four kinds of substrates were obtained. Typical pull-off

curves for these two tips approaching and retracting from mica were shown in Fig. 3. The

adhesion forces at different load force and contact time were measured at a retraction

velocity of 1 μm/s (Fig. 4-5).

   The adhesion force as a function of load force was studied at a contact time of 3 s

and a retraction velocity of 1 μm/s, as shown in Fig. 4. For both nanosphere tip (Fig. 4A)

and truncated tip (Fig. 4B), the adhesion force increased and then almost stabilized with

the increase of load force at a range of 0.1-220 nN. The turning point is 10 nN. The

adhesion force increase at the initial load force phase might be resulted from local shift or

unstable contact at a low load force.[30] It was reasonable to conclude that pure elastic

deformation occurred and no materials transfer occurred between tip and substrate at the

load force range of 10-220 nN.[19,30] In order to ensure pure elastic deformation during the

dynamic measurements, a load force of 100 nN was selected for the below work.

The adhesion force as a function of contact time was investigated at a load force of

100 nN and a retraction velocity of 1 μm/s, as shown in Fig. 5. For both nanosphere tip

(Fig. 5A) and truncated tip (Fig. 5B), the adhesion force increased and then almost

stabilized with the increase of contact time at a range of 0.01-4 s. The turning points for

nanosphere tip and truncated tip were 0.5 s and 1 s, respectively. The adhesion force

increase at the initial contact time phase might be resulted from the effect of the

approaching process on the retraction process.[19,20,31] The turning point difference between

the nanosphere tip and truncated tip indicated that the nanosphere tip had a faster stability.

It was reasonable to conclude that the effect of the approaching process was minimized at

the contact time range from the turning points to 4 s. In order to eliminate the effect

during the dynamic measurements, a contact time of 3 s was selected for the below work.

**3.4. Effects of Tip Shape and Substrate Type on the Dynamic Adhesion Forces
between Nanoscale Tips and Substrates**

To study the effects of tip shape and substrate type on the dynamic adhesion forces

between nanoscale tips and substrates, nanosphere tip with a diameter of 980 nm (Fig.

2A) and truncated tip with a side length of 959 nm (Fig. 2B) were applied. The adhesion

force as a function of retraction velocity was investigated at a load force of 100 nN and a

contact time of 3 s, as shown in Fig. 6. The order of the adhesion forces of nanoscale

samples (nanophere tip and truncated tip) from different substrates was: HOPG > silicon

wafer > mica > sapphire. This order is reversely consistent with that of Young's modulus

of the substrates (Table 1). In addition, the dynamic forces of truncated tip were larger

than those of nanosphere tip, which might be resulted from the different contact geometry.

The truncated tip with a side width of 959 nm had a larger effective contact area than the

nanosphere tip with a diameter of 980 nm did. It clearly revealed that the adhesion force

of nanoscale samples on a substrate was significantly affected by the retraction velocity,

which is consistent with previous work of microsphere or flat surfaces on a substrate.[27-28]

When retraction velocity of increased from 0.02 μm/s to 156 μm/s, the adhesion force of

nanosphere tip significantly increased 40.3% , 47.1%, 49.1% and 62.9% for mica, silicon

wafer, HOPG and sapphire substrates, respectively. The adhesion force of truncated tip

significantly increased 42.5%, 40.1%, 44.3% and 53% for mica, silicon wafer, HOPG

and sapphire substrate, respectively. The biggest adhesion force was achieved on HOPG

substrate and the dynamic force increased most rapidly in the test performed on sapphire

substrate. The different surface energy and the local separation geometry may be the

reasons affecting the dynamic effect of the system. The dynamic effect of nanosphere tip

was larger than the truncated tip, as the crack line of the nanosphere tip (with a shrink

closed-loop crack propagation line) decreased faster than the truncated tip (with a straight

crack propagation line) in mathematic aspects. For a truncated tip contacting a flat

substrate, the crack growth was considerably unstable because the crack front line rapidly

decreased in length from the base line of the triangle towards the vertex of the triangle

during separation. Similarly, for the nanosphere tip contacting a flat substrate, the crack

growth was also unstable as the crack front line (a closed circular) rapidly decreased in

length from the outline of the circle towards the midpoint of the circle during separation.

In a mathematic aspect, the crack front line of truncated tip decreased slower than that of

the nanosphere tip. It agrees with our experiment results that the truncated tip shows

lower dynamic effect, as the the crack line decreases slower. This analysis also gives a

reliable method to compare dynamic effect of probes with different shapes separation

from flat substrate.

Finally, Eq. (1) was used to fit the normalized adhesion force (F/F_0) vs retracting

velocity (Fig. 7). Eq. (1) fitted the data well and the dynamic parameters were

summarized in Table 2. Dynamic scaling parameter n was nearly constant (0.38-0.41) for

both nanosphere tip and truncated tip. Dynamic coefficient parameter C was in the range

from 0.013 to 0.018. Therefore, both the tip shape and substrate type had no obvious

effect on the dynamic scaling parameters n and dynamic coefficient parameter C.

4. Conclusions

In this work, we report a dynamically-induced enhancement in adhesion interactions

between two types of contact geometry: contact between nanosphere (nanosphere tip) and

flat substrates (nanosphere-flat contact); and contact between flat surface (truncated AFM

tips) and flat substrates (flat-flat contact). The results showed that the tip shape and

substrate type had obvious effects on the adhesion forces, but had no obvious effects on

the dynamic scaling parameters n and dynamic coefficient parameter C in the modelling

equation. The contact area geometry, crack propagation direction, and contact materials

can affect the dynamic effect of the tips on different substrates, Our work can provide

useful information for analyzing the interactions between micro/nanomaterials and their

environments. The dynamic enhancement is induced by fast crack propagation along the

tip−substrate interface, which is bridged by mechanical interlocks or water. The

attachment and detachment of micro/nanomaterials to and from a substrate are a

fundamental problem relevant to numerous natural phenomena (cell phagocytosis, dust

adhesion, and etc) and application process of artificial micro/nanomaterials (liposome,

drug-loaded polymer microparticles, and etc). There, this benefits the understanding of

numerous natural phenomena and the development of artificial micro/nanomaterials.

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Table 1. Material properties of substrates

Parameter	Mica	Sapphire	Silicon wafer	HOPG
Average roughness R_a(nm)	0.12±0.03	0.52±0.31	0.17±0.24	0.19±0.36
Young's modulus (GPa)	172±14	345±36	160±15	18±3

Table 2. Dynamic scaling parameter n and coeffient C for the nanosphere tip with a diameter of 980 nm on different substrates and for truncated tip with a side length of 959 nm on different substrates. The load force was 100 nN and the contact time was 3 s.

Tip type	Parameter	Mica	Sapphire	Silicon wafer	HOPG
Nanosphere tip with a diameter of 980 nm	c	0.011±0.002	0.017±0.001	0.015±0.002	0.018±0.001
	n	0.4±0.05	0.41±0.0	0.4±0.02	0.38±0.02
Truncated tip with a side length of 959 nm	c	0.013±0.001	0.018±0.002	0.013±0.002	0.013±0.001
	n	0.38±0.01	0.4±0.02	0.38±0.02	0.4±0.03

Figure captions:

Figure 1. Schematic of AFM dynamic force measurement process. (A) Schematic of AFM dynamic force measuring system using a nanosphere tip. (B) and (C) are typical ramping process of z piezo movement vs time and force vs time, respectively. The z piezo movent and force were recalculated from input voltage and output detective voltage, respectively.

Figure 2. SEM images of typical nanosphere tip with a diameter of 980 nm (A) and truncated tip with a side length of 959 nm (B).

Figure 3. Typical pull-off curves for nanosphere tip with a diameter of 980 nm (A) and truncated tip with a side length of 959 nm (B) approaching and retracting from mica. The spring contant of the cantilevers of the nanosphere tip and the truncated tip were 2.73 N/m and 2.56 N/m, respectively.

Figure 4. Adhesion force vs load force analyses for the nanosphere tip with a diameter of 980 nm on different substrates (A) and for truncated tip with a side length of 959 nm on different substrates (B). The contact time was 3 s and the retraction velocity was 1 μm/s.

Figure 5. Adhesion force vs contact time analyses for the nanosphere tip with a diameter of 980 nm on different substrates (A) and for truncated tip with a side length of 959 nm on different substrates (B). The load force was 100 nN and the retraction velocity was 1 μm/s.

Figure 6. Adhesion force vs retracting velocity analyses for the nanosphere tip with a diameter of 980 nm on different substrates (A) and for truncated tip with a side length of 959 nm on different substrates (B). The load force was 100 nN and the contact time was 3 s.

Figure 7. Normalized adhesion force (F/F_0) vs retracting velocity analyses for the nanosphere tip with a diameter of 980 nm on different substrates (A) and for truncated tip with a side length of 959 nm on different substrates (B). The load force was 100 nN and the contact time was 3 s.
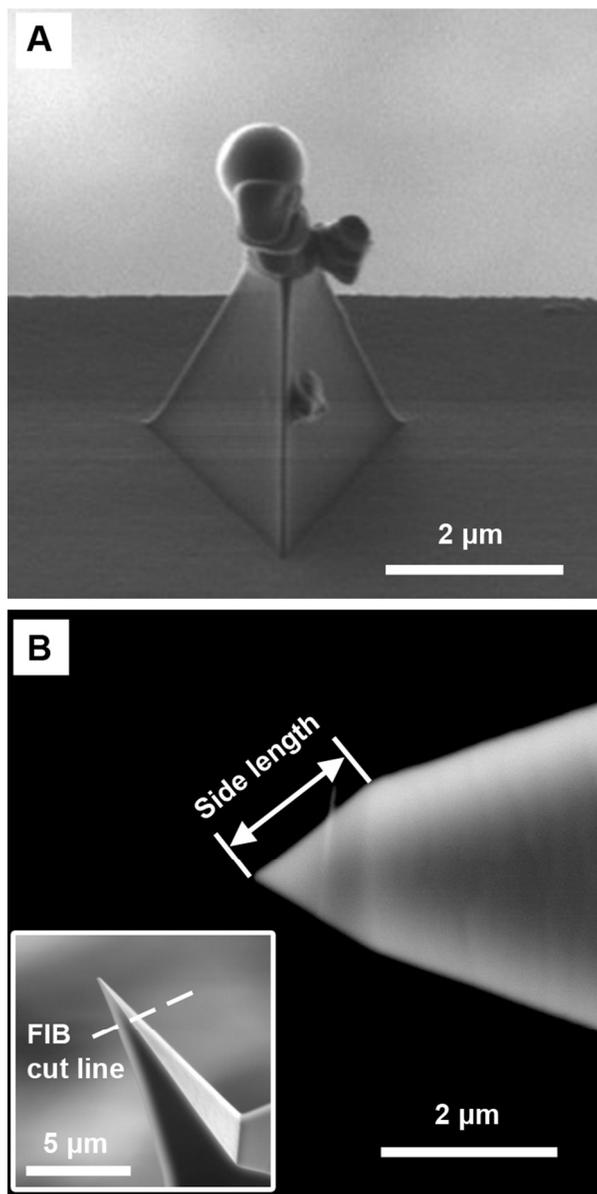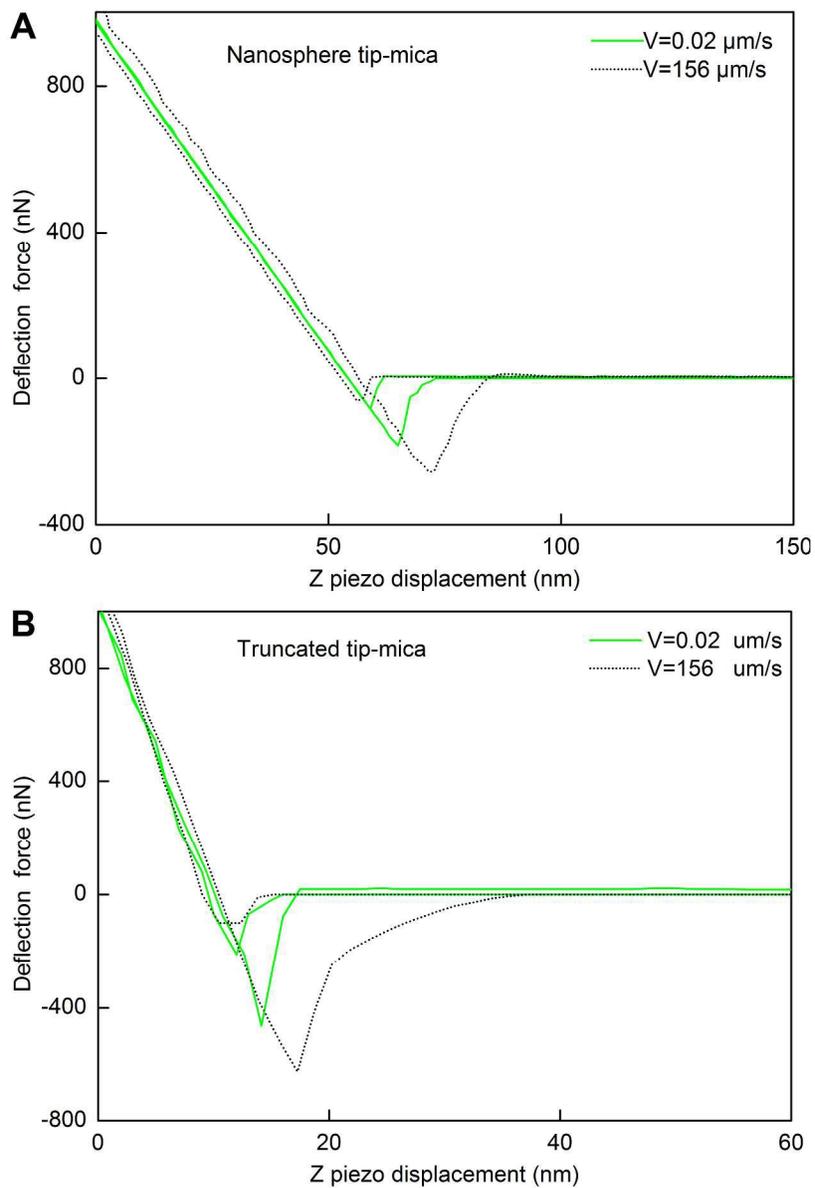
Figure 2. SEM images of typical nanosphere tip with a diameter of 980 nm (A) and truncated tip with a side length of 959 nm (B).
60x119mm (300 x 300 DPI)

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118x174mm (600 x 600 DPI)
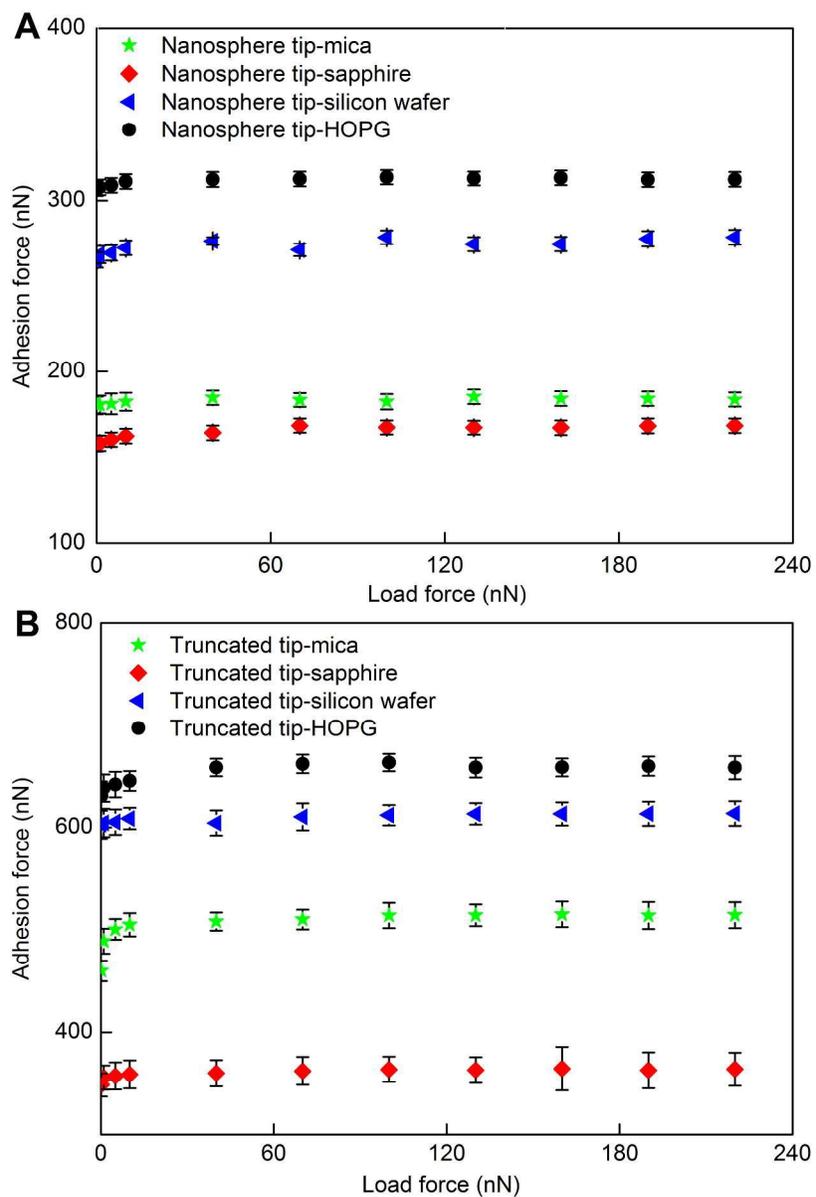
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118x174mm (600 x 600 DPI)
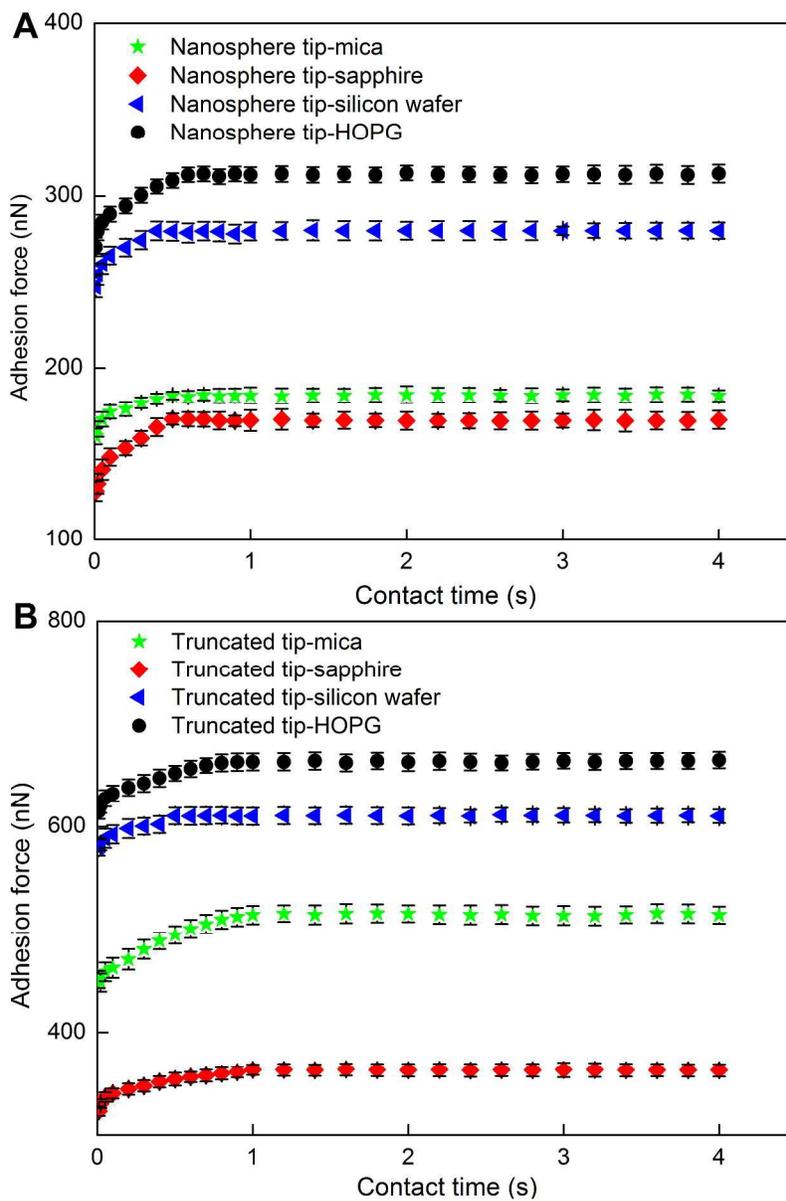
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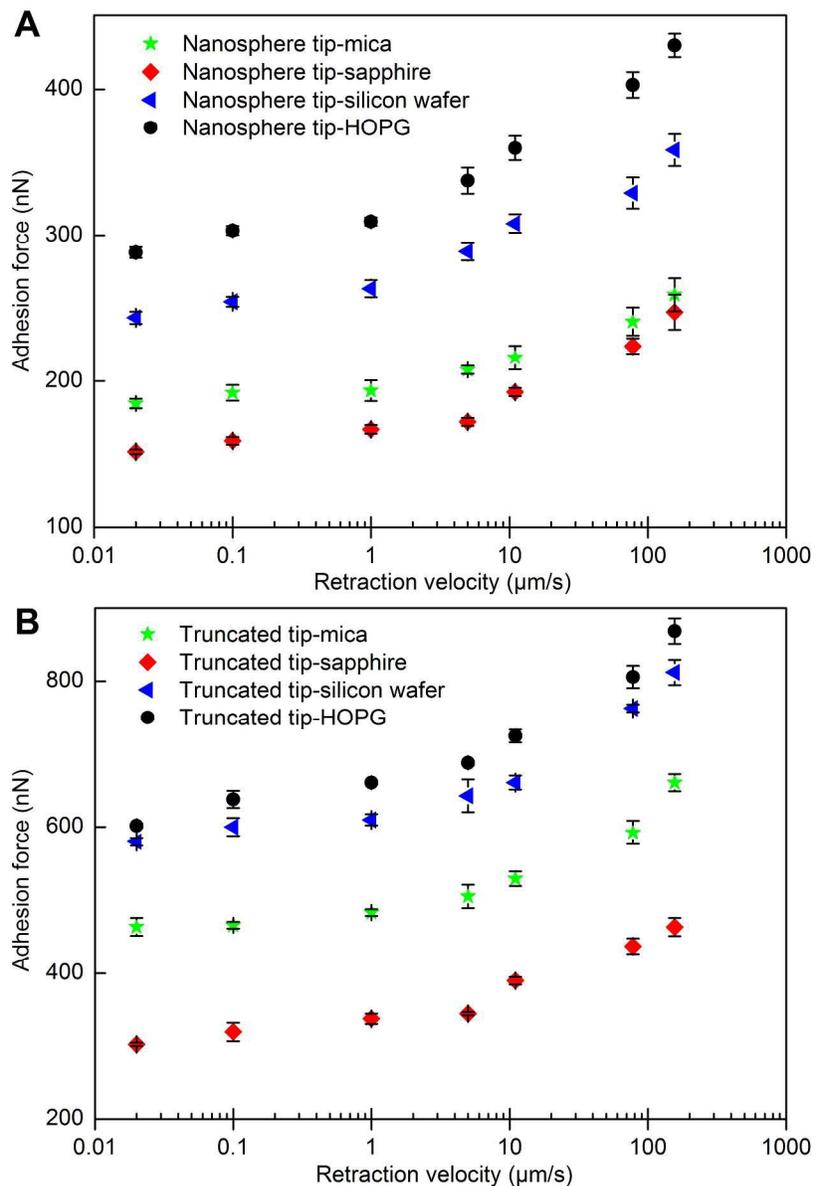120x180mm (600 x 600 DPI)

Figure 6. Adhesion force vs retracting velocity analyses for the nanosphere tip with a diameter of 980 nm on different substrates (A) and for truncated tip with a side length of 959 nm on different substrates (B). The load force was 100 nN and the contact time was 3 s.
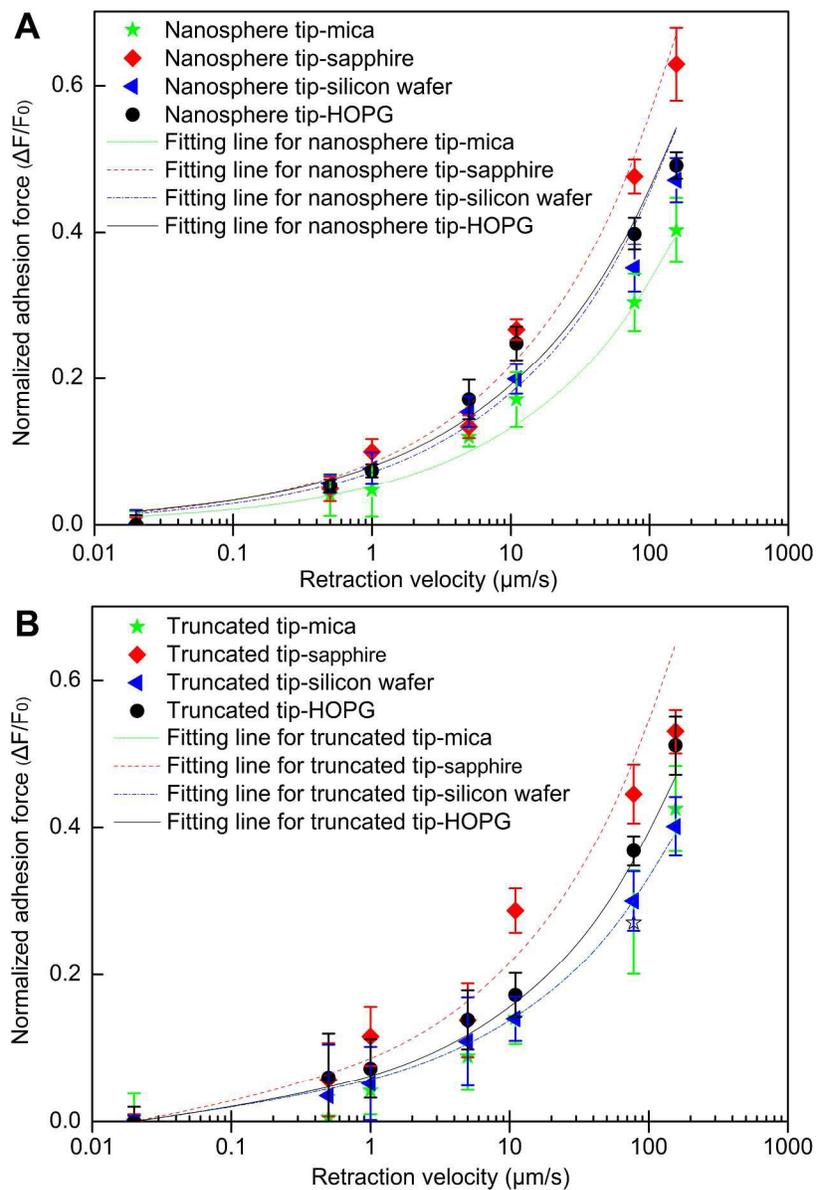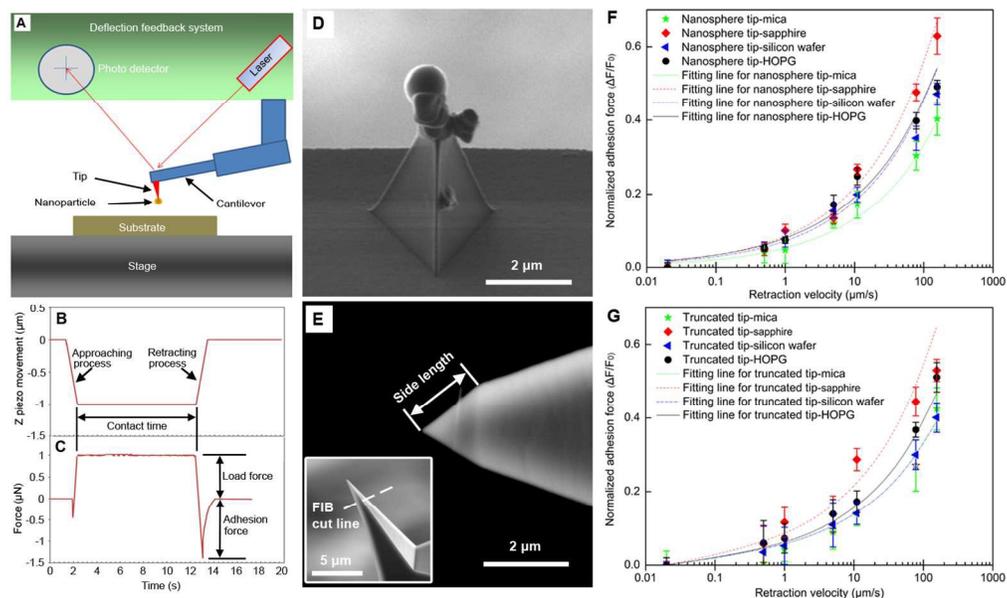118x174mm (600 x 600 DPI)

Figure 7. Normalized adhesion force (F/F0) vs retracting velocity analyses for the nanosphere tip with a diameter of 980 nm on different substrates (A) and for truncated tip with a side length of 959 nm on different substrates (B). The load force was 100 nN and the contact time was 3 s.
118x174mm (600 x 600 DPI)

Graphical Abstract

Both AFM tip shape and substrate have obvious effects on the dynamic adhesion forces of truncated and nanosphere tips on four different substrates (mica, sapphire, silicon wafer, highly oriented pyrolytic graphite).