Measuring shear induced adhesion of gecko-inspired fibrillar arrays using scanning probe techniques

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Abstract

The natural ability of geckos and spiders to climb almost all surfaces using the compliant, nano-structured components on their feet provides motivation for making bio-inspired adhesives. The brush-like fibrillar structures have only minimal adhesion upon initial contact. Maximum adhesion is realized after a dragging motion of the gecko's or spider's foot that aligns the fibrillar structures to be almost parallel to the contacting surfaces. The goal of the studies described in this paper is to create an analytical technique to improve our ability to characterize dry adhesives modeled after these biological systems. The technique described herein uses a scanning probe microscope to manipulate a flat test surface in contact with biomimetic fibrillar arrays while monitoring the adhesion forces. Adhesion forces were measured after both normal contact and shear induced contact between the nano-structured fibrils and the test surface. Results confirm that the adhesion forces are higher for bio-inspired adhesives after a shear induced contact. Variations in these forces can be measured across the sample with micron-scale lateral resolution. This method of analysis can be extended to evaluating bio-inspired dry adhesives with realistic mechanisms of attachment utilized in robotic and similar applications of these materials.

Introduction

Following the scientific identification that van der Waals forces contribute remarkably to a gecko's ability to climb [1-3], numerous attempts have been pursued to make artificial adhesives that mimic the fibrillar structures found on a gecko's foot [4-8]. The gecko adhesive does not rely upon fluid secretion and, therefore, is widely referred to in the literature as a "dry adhesive" [1-3,9]. Pressure sensitive adhesive (PSA) tapes, including office and duct tape, form a tacky connection between the tape and the contacting surfaces. Geckos, on the other hand, rely on fibrillar nano-structures to form millions of interactions through weak van der Waals forces with the surfaces [10]. The synergistic effect of millions of weak interactions translates into a collectively strong adhesion force that supports every step of the climbing animal. Fibrils on the skin of the gecko are made from beta-keratin, which is non-tacky and stiff. These materials properties provide wear resistance for the fibrillar structures. The combination of composition and shape of these nano-structures permit the gecko feet to adapt to surfaces of different roughness. To conform to rough surfaces, the gecko adhesive contains a hierarchical structure of branching fibrils of different diameters ranging from nanometers to millimeters. These slender and brush-like structures have a high compliancy to various surface topographies and ensure a maximized number of contact points with the surfaces.

The formation of contact points between the hierarchical fibrillar structures and the contacting surfaces is a dynamic process. A close examination of the climbing motion of the animals reveals that the adhesion mechanism includes a dragging movement of the animal's foot to assist in achieving a high conformation between the fibrils and various surfaces [11-12]. Experiments have been performed that demonstrate this observation for both gecko [13-15] and spider [16-17] seta. In one study, Autumn et al. attached a gecko seta to a cantilever in order to manipulate the seta with control in all three dimensions. This control was essential for their ability to bring the seta into contact with surfaces, to subsequently provide a shear movement across these surfaces, followed by removal of the seta by a pulling in a direction perpendicular to the surfaces. This series of manipulations to the seta is referred as the "load-

drag-pull" (LDP) method [18]. Experimental results indicate that the adhesion force between the seta and various surfaces increases with the additional shear movement after bringing the seta into contact with the test surfaces. Theories and models have been developed to explain the mechanism of increased adhesion resulting from the LDP method. Results of these studies highlight the improvement of fibril alignment during the shear movement applied to the seta. Tian et al. [19] have demonstrated that frictional forces contribute significantly to the adhesion force of gecko setae, but the angle of peeling the seta from surfaces is relatively low. Other models on pre-tension [20] of the adhesive were modeled as a thin film, and analyzed by Kendall's [21] thin film peeling model, which provide further insight into the shear induced adhesion forces utilized by geckos to climb walls and suspend from ceilings. Mathematical modeling of experimental adhesion force measurements for gecko setae by Yamaguchi et al. show a close fit with the LDP experimental results and advances the concept that lateral movement increases adhesion force [22]. Studies by Majidi and Fearing [23] and Cheng et al. [24] also suggest that alignment of the fibrillar arrays is an important aspect of increased adhesion. A mathematical model and supporting experimental results of Filippov et al. [25] suggest that spiders also align their fibrils through a dragging motion of their feet. This study indicated the adhesion force between the fibrils and contacting surfaces is enhanced through an increase in the area of contact. A combination of the hierarchical nano-structures in setae of geckos or spiders and a shear induced alignment of these nano-structures work in conjunction to enhance their adhesion to various surfaces.

Artificial dry adhesives have been prepared to mimic the properties observed in setae of geckos and spiders. A key component of the development of these dry adhesive materials is the verification of their adhesive properties and validation that their adhesion forces are similar to their natural counterparts. In one study, Schubert et al. [26] demonstrated the use of a large spherical probe (contact region ~1x1 mm^2) in conjunction with LDP based adhesion force measurements to evaluate an array of polypropylene nano-fibrils. In an array of nano-scale fibrils with a common directionality (i.e. a specific tilted orientation), Lee et al. [27] evaluated the adhesion force by the LDP method and a "push-pull"

(PP) method. In the later technique, various surfaces are contacted with and separated from fibrils while maintaining a direction of motion parallel to the orientation of the fibrils. The comparative analysis by Lee et al. demonstrated that the adhesion characteristics of the tilted fibrils were similar to those of the gecko adhesive. In contrast, Varenberg and Gorb [28] tested their adhesive composed of an array of micro-scale mushroom shapes with fibril-like stalks by applying a shear force, but the adhesion force decreased due to the smaller area of contact between the test surfaces. A wedge shaped adhesive was similarly evaluated by Parness et al. [29] using the LDP method. In this study, the applied shear forces created a compliant interface between the wedges and the contacting surfaces to increase the area of contact, which also increased the measured adhesion force. In summary, if a shear force is applied between the fibrillar array and the contacting surfaces which results in an increase of contact area, then the adhesion force also increases. The applications on this discovery include construction of climbing robots [30-31] that use gravitational forces to induce a shear loading between their feet and the surfaces of a wall. This demonstration also provides further evidence that a shear assisted contact between the dry adhesive and the contacting surfaces helps to ensure a reliable and strong adhesion interaction.

This study investigates the implementation of the LDP and PP measurement techniques using an atomic force microscope to assess the adhesion forces of an array of artificial fibrils. The benefits of using an atomic force microscope (AFM) for these analyses include micro-scale control over position of the measurements within the samples, fine control over the contacting forces, and precise control of both speed and direction of motion during the force measurements. Previous implementation of AFM techniques to measure adhesion forces of fibrillar arrays includes the use of a colloidal probe [32-33] and a flat probe [34-35]. These measurements implemented PP methods. We extend these demonstrations here to LDP techniques and compare the results with those of the PP method. A benefit of the use of an AFM for administering these adhesion measurements is a fine control over the interactions between the test surfaces and the fibrillar array and the ability to take force measurements from small (e.g., micro-scale) regions of the sample. The latter ability is especially important when

evaluating the adhesion forces of a non-uniformly distributed array of nano-structured fibrils. The results of the study reported herein indicate that the adhesion forces for non-uniform arrays of fibrils increase when using a shear loading (LDP rather than PP methods) with the AFM controlled cantilever. Further results indicated a divergence in the adhesion forces across these samples. These measurements could be useful in determining correlations between structure and function in such non-uniform dry adhesives samples, or for identifying non-uniformities in adhesive forces within well-ordered fibrillar arrays.

Experimental Section

Preparation of fibrillar arrays as dry adhesives. The artificial fibrillar arrays were fabricated using a Reactive Ion Etching (RIE) method we discussed in detail within a previous paper [35-36]. Epoxy based (SU-8 2010, MicroChem) arrays of nano-scale fibrils were prepared upon a regular arrangement of squares measuring 1 mm x 1 mm and with a spacing of 0.5 mm between each pad. The etching time of the RIE process determined the average length of the fibrils within each sample. For the purposes of this study, we present results on samples etched for 10 min, producing fibrils with a nominal length of ~250 nm. Samples were inspected by scanning electron microscopy (Strata DB-235, FEI) to verify the length of the fibrils, monitor for the presence of defects in samples, and to identify changes to the fibrillar arrays after performing the various adhesion tests outlined below in further detail. All samples were also inspected for defects using an optical microscope (Axio Imager M1m, Zeiss) throughout these studies.

Measurement of adhesion forces in fibrillar arrays. An atomic force microscope (MFP-3D-SA, Asylum Research) was adapted for adhesion force measurements on the fibrillar arrays described above. A specific script was written (with assistance from Jason Bemis, Asylum Research, Santa Barbara, CA) to control the position, movements, and dwell time of a cantilever, including after being brought into

contact with the sample. In addition, the implementation of a "sewing" type of an action enables the cantilever to sequentially measure adhesion forces across the sample by moving from one position on the sample to an adjoining position upon completion of each measurement cycle. Tipless silicon nitride cantilevers (NP-O10, Veeco Instruments) were used for the measurements performed in this study. Each AFM probe contains four cantilevers each with a different spring constant. The spring constant of the selected "C" triangular cantilever is ~320 nN/μm, which is calibrated prior each test by performing a single force curve and determining the resonance frequency (~60 kHz) of this cantilever. The adhesion force normal to the test surface is calculated using the calibrated spring constant and the measured cantilever deformation. The pointed end of the triangular shaped cantilever was directed towards the sample surfaces with an angle between the tipless cantilever and the samples maintained at ~11° when the cantilever was approaching the test samples. The loading force was set to ~10 nN and during the LDP method the contacting cantilever moved a distance of 1 μm during the subsequent shear step at a speed of 20 μm/s. A preload force of 10 nN was chosen for these measurements because it is within the range of adhesion forces (i.e. 0 to 60 nN) that can be measured through the use of this cantilever as determined empirically. From our experience, higher preload values could compress or otherwise significantly deform the fibrillar surfaces as will be discussed below in relation to Figure 3. The vertical retraction speed of the cantilever was set at 1 μm/s. Adhesion force measurements were acquired in a sequence of analyses conducted in a square array of 20 measurements in either direction (x or y). Each of these 400 independent measurements had a corresponding force-distance (FD) curve that was acquired from the beginning to end of the sequences outlined in Figure 1 and discussed in further detail below. Further information on the measurement of adhesion forces by the PP method are described in [35].

Statistical analysis of experimental results. As indicated above, 400 measurements were acquired for each experiment. Results of each experiment were plotted in a histogram for ease of analyzing for trends. Both the median and a 12.5% trimmed mean were calculated for each set of results

as a combined indicator of the average adhesion force and the distribution of adhesion forces for each sample. The median was chosen to indicate the center of the distribution, and the 12.5% trimmed mean to portray the average value of each data set while excluding 100 data points (out of a data set of 400) from either side of the entire population. The portion that is trimmed was determined after a close examination the normal probability plot for each data set (see Supporting Information for further details).

Results and Discussion

A number of approaches are being pursued for making bio-inspired dry adhesives. An essential component of characterizing these materials is to determine the correlation between structure and function on a scale proportional to the dimensions of the components within the adhesives. Often the key features within these materials are micro- to nano-scale fibrils with spacing on an equivalently small scale. We have previously demonstrated the usefulness of measuring the adhesion forces within non-uniform arrays of fibrils through the use of an AFM [35]. In these previous tests, a flat cantilever was brought into contact with an array of vertically oriented fibrils to a predetermined loading force and directly retracted. The direction of movement of the cantilever was maintained parallel to the orientation of these fibrils. The adhesion forces between the fibrillar array and the contacting surfaces (i.e. flat surfaces of the cantilever) were determined from the calibrated deflection of the cantilever. This method has been referred to as a “test without dragging”, “tap test”, or the “push-pull” technique. We refer to this method here as the push-pull or the PP technique. In this manuscript, we extend the PP method with further modifications to mimic the load-drag-pull or LDP method. The LDP method is adapted from previous demonstrations [18] to demonstrate this method in conjunction with use of commonly available AFM hardware. Because the dragging or shear motion of the cantilever is the essential

difference between the PP and LDP methods, we sometimes refer to the latter as the shear induced contact method or the dragging test.

An overview is provided in Figure 1 of the implementation of the LDP method using a scanning probe microscope to manipulate a flat cantilever as a probe in order to measure adhesion forces for arrays fibrils. The tipless cantilever is first located above an array of fibrils (Figure 1a). The cantilever is controlled through a series of sequential steps (Figure 1a-1d): i) vertical approach of the cantilever to the test surfaces, bringing the fibrils in contact with the cantilever (load step); ii) lateral movement of the cantilever across a predetermined distance (drag step); iii) cantilever vertically withdraws from the surface and control system records the adhesion force generated from the contacts (pull step); and iv) movement of the cantilever to an adjacent spot to start a new measurement. During each measurement the cantilever approaches the fibrillar array until reaching a predetermined force, referred to as the loading force, of ~10 nN (Figure 1b). After the cantilever has been brought into contact with the array of nano-structured fibrils, its position is controlled to slide laterally over these surfaces (Figure 1c). The direction and distance of this movement can be precisely controlled through a series of piezoelectric stacks that control the position of the stage relative to the position of the cantilever. In these studies, we moved the cantilever in a direction opposite of the pointed end of the triangular shaped cantilever. This direction of motion was chosen to avoid unintentional damage to the surfaces of the dry adhesive (potential damage to the samples following the LDP method is assessed later in this manuscript). Because the average length of artificial fibrils was ~250 nm, the lateral movement of the cantilever during the dragging step was set to 1 μm. This length of movement was chosen in order to travel at least the height of the contacting fibrils in order to align and efficiently contact these fibrils. The cantilever was moved during this shear step (Figure 1c) at a rate of 20 μm/s and subsequently retracted in a vertical direction (Figure 1d) at a speed of 1 μm/s, which were indiscriminately selected as starting points to assess the efficacy of implementing the LDP method using an AFM.

The adhesion forces of the polymeric fibrils under test were measured from the calibrated deflection of the tipless cantilever during retraction from the contacting surfaces. The force to pull the cantilever away from the fibrillar arrays is one aspect of the data contained within the acquired force-distance (or FD) curves (Figure 2). The corresponding force-time response curve is described in detail in the supporting information (Figure S2). The adhesion force for this measurement was ~19.8 nN. There are also many other details within these traces that contain useful information about the fibrillar arrays as they interact with the flat surfaces of the cantilever. This figure depicts the force curve during approach of the cantilever to the fibrillar arrays and during the load step of the LDP method (red trace in Figure 2, read from right to left). The inset depicts the oscillations in the force curve observed during both the dragging movement of the cantilever and the subsequent withdrawal of the cantilever from contact with the fibrils (blue traces in Figure 2, read from left to right). The dragging motion of the cantilever initially induces oscillations that likely correspond to changes in the tilt of the cantilever during alignment of the fibrillar structures. These deflections to the cantilever could be attributed to release of some of the fibrils (considering that the lateral distance traveled is greater than the average height of the fibrils), contact with a non-uniform array of fibrils (essentially these are roughened surfaces), and reorientation of the fibrillar structures during this shear induced alignment process. These interactions reach a steady state during the dragging motion of the cantilever (Figure S2), suggesting alignment of the fibrils in the direction of the moving cantilever. During movement of the cantilever away from the fibrils, the “retraction” trace is a smooth line. The subsequent trace depicts the force necessary to remove the cantilever from these surfaces of aligned fibrils. It may be possible to extend this work in the future with narrower cantilevers to the analysis of adhesion forces between either individual or smaller clusters of fibrils and the cantilever in order to more easily discern these individual processes, but these analyses are beyond the scope of this current study.

The measurement of adhesion forces for arrays of polymer based fibrils could induce damage during the initial load, shear movement, and retraction of the cantilever. The initial load force could

deform the polymer surfaces. The lateral movement during shear induced alignment of the fibrils could deform, cut or otherwise damage the fibrils, and the retraction of the cantilever from these arrays could stretch or snap the polymer fibrils that have a significant area of contact with the cantilevers. Although the samples had extensive contact with the tipless cantilever, especially during the LDP method, no obvious damage was observed in these our studies. Analysis of the samples by optical microscopy and scanning electron microscopy did not reveal any noticeable changes to the fibrillar arrays. The SEM results in Figure 3 depict regions of the sample as observed before the adhesion force measurements (Figure 3a), after measurements using the LDP method (Figure 3b), and subsequent to using the PP method (Figure 3c). Remarkably, there were no observed collapse of the fibrillar nano-structures after either of these force measurements, and the density of fibrils remains the same as that observed in the as-prepared samples.

The hypothesis that the fibrils have aligned during the shear step of the LDP method is further confirmed by comparing the results of these measurements with those obtained from the PP method on the same samples. An increase of adhesion force is predicted by theoretical studies and has been observed in previous reports [18-24]. Histograms of the adhesion forces measured by the LDP and PP methods for the polymeric fibrils are depicted in Figure 4. The distribution of results in these histograms provides a simple comparison between the LDP and PP methods. The histogram associated with the PP or tap test (Figure 4a) contained adhesion forces ranging from 0 to 26 nN with a standard deviation of 3.0 nN. In comparison, the results of the LDP or drag test (Figure 4b) had a wider distribution of adhesion forces ranging from 0 to 55 nN with a standard deviation of 9.4 nN. Furthermore, the median adhesion force from the PP method was 3.8 nN in comparison to a value of 8.7 nN for the measurements from the LDP method. We also calculated the 12.5% trimmed mean for each study in order to account for potential outliers in these datasets (i.e. due to any random events that may otherwise bias the results). The LDP method yielded a trimmed mean of 9.9 nN in comparison to 3.9 nN for the PP method. Each analysis demonstrates a higher average adhesion force resulting from the drag test.

The adhesion forces of the shear aligned fibrils are more than twice those of the same fibrils under test by direct contact and pull-off of the cantilever. The divergence of these results can be attributed to alignment of the fibrils during the LDP method, and the subsequent increased interactions between the fibrils and the flat surfaces of the cantilever. The shear movement of the cantilever during the "drag step" leads to possible compression, stretching, elongation, and alignment of the fibrils along the direction of cantilever motion. During this process any overlapping and otherwise curling fibrils untangle and are exposed to the flat surfaces of the cantilever. The contacting surfaces with the cantilever can come from many different parts of a single fibril. These points of contact will depend on the shape and stiffness of the fibril and its relative position with respect to the cantilever. These differences in tip-sample interactions vary between each location on the sample and, therefore, between each adhesion force measurement. These variations contribute to the diverse range of adhesion forces observed for the measurements subsequent to shear alignment.

The histograms for the adhesion force measurements indicate a large variation in the results as a function of position across the sample. The variations in adhesion force observed from the LDP method are clearly observed in a force map (Figure 5). This map depicts the adhesion force measured from 400 independent measurements made in a square array across the sample, and the gray scale coloration indicates the strength of these interactions (the lighter the color, or more white, the stronger the adhesion force) in accordance with the scale bar. There is a seemingly random distribution to the measured adhesion forces. In comparison to the spatial arrangement observed for the fibrillar structures (Figure 3), it might be surprising that the adhesion forces vary non-uniformly across the sample. These results are, however, consistent with our previous observations [35] for the spatially correlated variation in adhesion forces measured for a similar array of fibrillar structures using the PP method. The local variations in contact between the tipless cantilever and the fibrils may be larger in accordance with the larger variation in adhesion forces observed for these samples (Figure 4). It is clear that the dragging motion of

the cantilever induces changes within the arrangement of the fibrils and increases the contact points between the contacting surfaces and the fibrils.

Conclusion

A lateral or shear movement is essential for climbing animals to efficiently engage the fibrillar structures on their feet to contacting surfaces. The same is true for climbing robots and in other applications of an artificial dry adhesive. We have developed a method to assist in establishing the correlation between structure and function of nano-structured dry adhesives as a result of this shear induced movement. An atomic force microscope was used to manipulate a flat cantilever that could be controllably brought into contact with fibrillar arrays. The distances and directions over which the cantilever moved, as well as the speeds with which it moved could be controlled in order to induce a shear motion of the cantilever across an array of fibrils. A technique was demonstrated that can move the cantilever in a sewing-like manner across the surfaces of a dry adhesive to acquire sufficiently large data sets in order to both assess trends in adhesion forces and spatial variations in these measurements. The shear induced alignment of the fibrils increases the adhesion force measured for polymer-based fibrils, which is consistent with prior studies using macro-scale measurements of adhesion forces after shear alignment. This shear induced alignment of the fibrils increases the number of contact points between the cantilever and the fibrils. This lateral movement between the cantilever and sample also increase the variability in observed interactions and, thus, variations in measured adhesion forces. Results of these studies coincided with predictions from theoretical models and prior research in this field. This new implementation of the shear induced alignment of fibrillar arrays and the subsequent measurement of their adhesion forces expands our ability to understand structure-function correlations in nano-structured dry adhesives. Development of improved methods of making and using these bio-inspired adhesives could benefit from this adaptation of scanning probe microscopy techniques for measuring adhesion forces from normal and shear induced contact.

Acknowledgements

This work was supported in part by the Natural Sciences and Engineering Research Council (NSERC) of Canada, and the Canada Research Chairs Program (B.D. Gates). This work made use of 4D LABS shared facilities supported by the Canada Foundation for Innovation (CFI), British Columbia Knowledge Development Fund (BCKDF), Western Economic Diversification Canada, and Simon Fraser University. We appreciate the assistance of Jason Bemis at Asylum Research for assistance in co-developing the software necessary for measuring adhesion forces resulting from shear induced alignment of fibrils using scanning probe microscopy techniques. We also thank Michael C.P. Wang for his assistance with atomic force microscopy.

Keywords

adhesion forces, dry adhesive, nano-structured fibrils, scanning probe microscopy, shear loading

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Figures and Captions

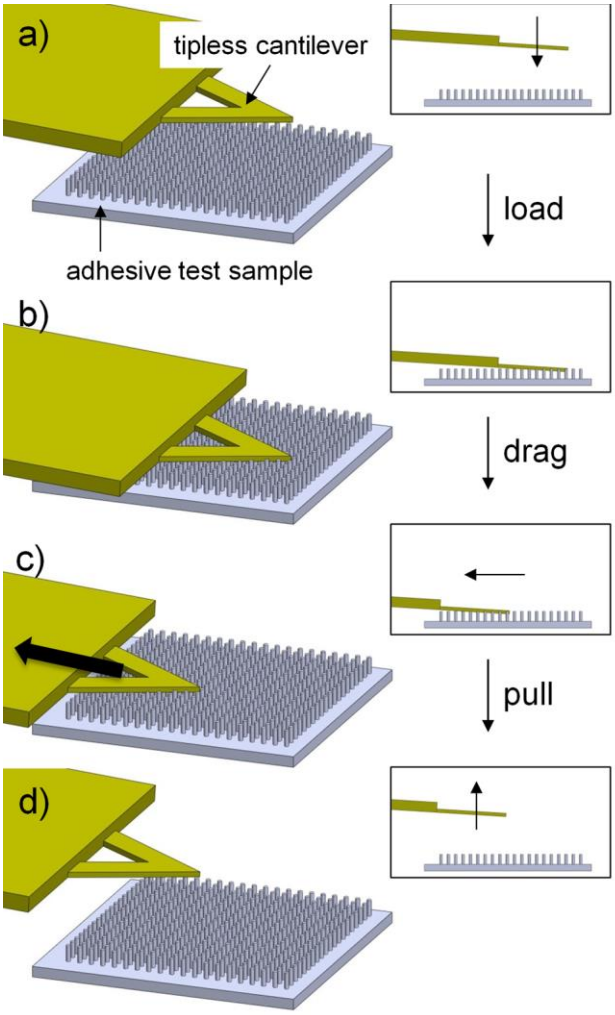


Figure 1. Schematic of the sequential steps implemented during a shear induced alignment of bio-inspired fibrils during adhesion force measurement implemented with a scanning probe microscope. (a) A tip-less cantilever is positioned over the fibrillar array. The cantilever subsequently (b) approaches the fibrillar array in a vertical direction (i.e., load step), and (c) the cantilever is laterally moved across the sample providing a shear force to the initially vertically oriented fibrils (i.e., drag step). (d) The last step of this method is to vertically unload, or pull the cantilever away from the sample. During this entire process deflection of the cantilever is recorded and translated into an adhesion force between the cantilever and fibrillar array.

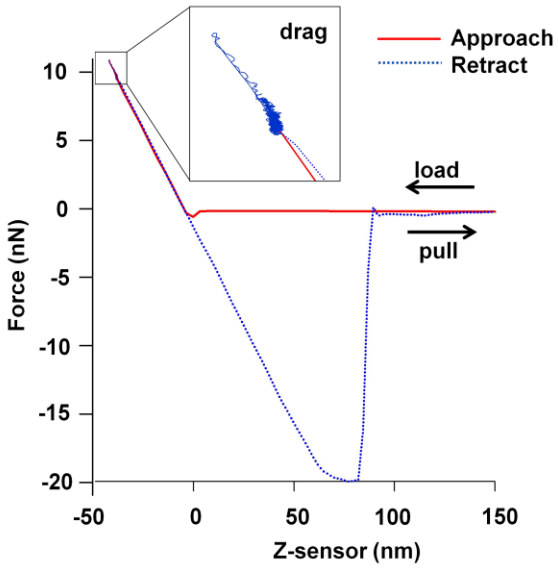


Figure 2. A typical force-distance (FD) curve used to measure the adhesion forces resulting from shear induced contact between a flat cantilever and arrays of nano-structured fibrils. This plot contains traces recorded while the cantilever was approaching (red) and retracting from (blue) these fibrils, indicating the forces of interaction between these surfaces. The inset provides a magnified view of the FD curve during the final load, drag, and initial withdrawal steps of the LDP method.

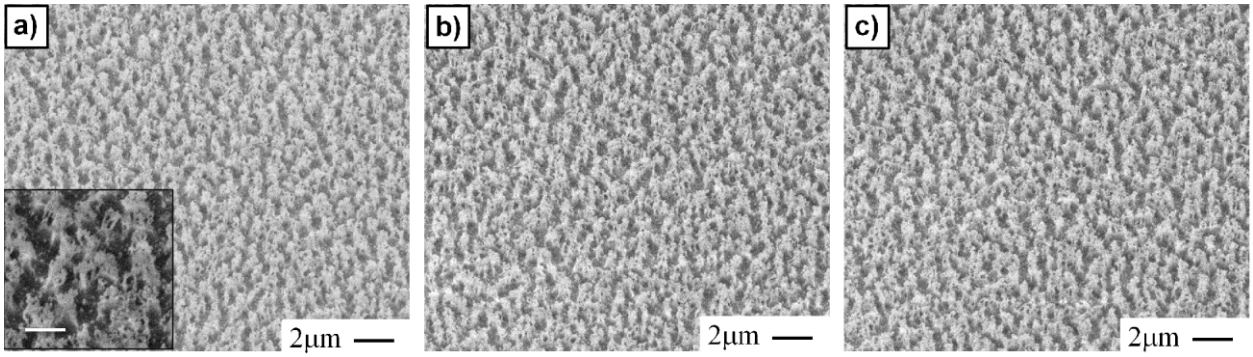


Figure 3. Scanning electron microscope images of polymeric fibril samples (a) before measuring their shear induced adhesion (inset: close-up of the fibrils, scale bar: 1μm), (b) following the drag step of this measurement, and (c) after measuring their adhesion using a normal force loading of the cantilever. All images are taken in 45 degrees tilt. The nominal length of the fibrils is ~250 nm.

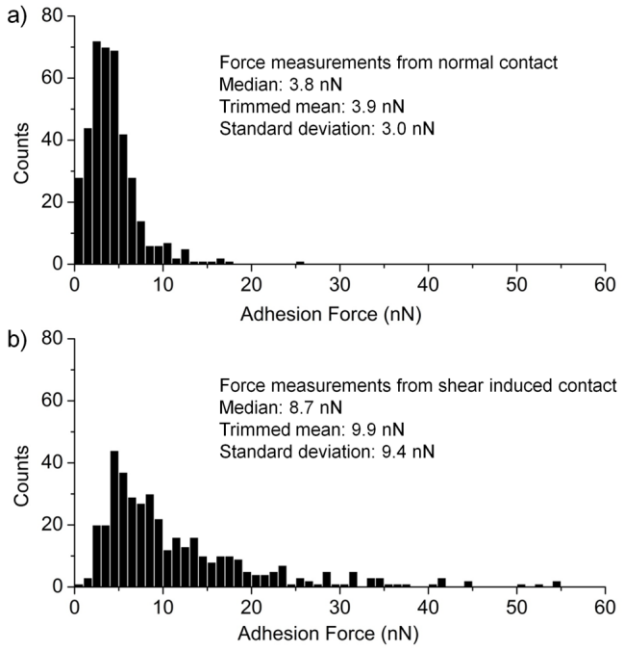


Figure 4. Histograms of adhesion tests acquired from (a) normal contact between the cantilever and the fibrillar array (i.e. non-shear loading of the cantilever in a direction parallel to the orientation of the fibrils), and (b) measurements from shear induced contact between fibrillar arrays and the cantilever by implementing a dragging motion. The total number of measurements for each test was 400. Trimmed mean is calculated by trimming the 12.5% of outliers at the smaller and larger end of the measurements. Standard deviation is calculated from the square root of the variance of the entire population.

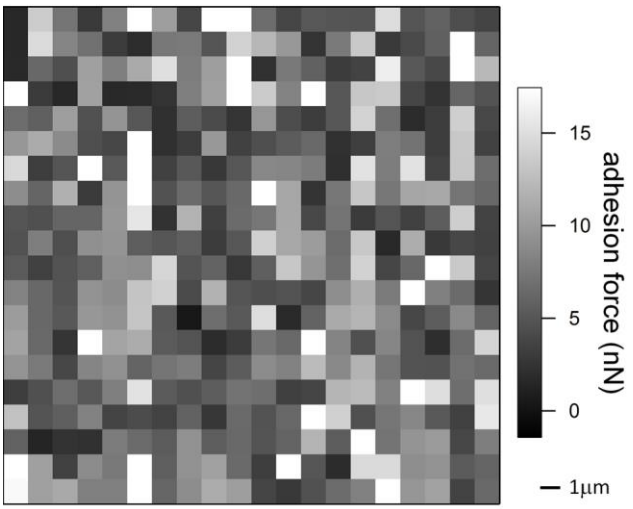
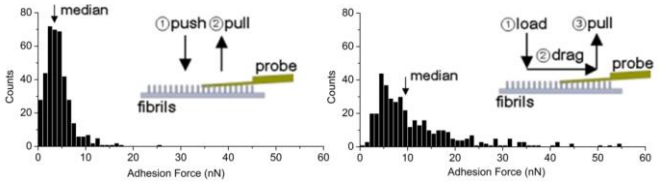


Figure 5. A representative map of the lateral variation in adhesion forces resulting from a series of sequential LDP based measurements performed across an array of ~250-nm tall fibrils. The force map was collected from a total 400 independent measurements, each corresponding to a different lateral position on the sample (varying in x and y by 1 μm movements).

Graphic for "Table of Contents"



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Adhesion forces were measured for an array of nano-scale fibrils using normal and shear induced contact with a scanning probe microscope manipulated cantilever. Hundreds of measurements were obtained with control over directions, distances, and rates of the cantilever movements. This method could provide further insight into nano-scale structure-function relationships for artificial dry adhesives.