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Letter

Weak adhesion between deposited rough films: Relation to dispersion forces

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Although the contact theory between rough surfaces is designed for adhesion energies $\gtrsim 100$ mJ/m^2, microsystems are controlled by much weaker adhesion $\lesssim 100$ μJ/m^2, which is critical for their operation. The weakest adhesion is related to the omnipresent fluctuation-induced dispersion forces. We develop a theory for such a weak adhesion emphasizing that the adhesion energy as a function of the average distance separating the bodies is almost entirely defined by the dispersion interaction. This dependence can be evaluated using the Lifshitz theory, but the effects of contact interaction or plastic deformations give only small contribution to the adhesion. Such a behavior is explained by a specific roughness of the deposited thin films used in microtechnologies. The films deposited on cold substrates have a much larger number of high asperities than is predicted by the Gaussian distribution and the contact occurs over a few asperities with heights much larger than the root-mean-square roughness. Finally, we discuss application of the effect for more precise determination of the distance upon contact, which is crucial for precise measurements of the dispersion forces especially at short separations in the range $5 < h < 50$ nm.

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In contrast with many other forces (electrostatic, capillary, chemical), the dispersion forces (DFs) are omnipresent and cannot be switched off by choosing materials, surface preparation, or external conditions. The DFs originate from quantum fluctuations of the electromagnetic field generated by any material and dissipated via the field absorption channels in accordance with the fluctuation-dissipation theorem. It was recognized first by London [1], who explained the van der Waals (vdW) attraction between gas molecules, then by Casimir [2], who demonstrated that the field fluctuations result in the force between metals (Casimir force), and, finally, the general macroscopic theory of the DFs between arbitrary solids was developed by Lifshitz [3] and co-workers [4].

At very short separations ($h \lesssim 1$ nm) a number of additional effects are involved in the interaction, which are named in general as surface forces [5]. These effects are important for wetting phenomena and colloid science but at larger distances survives only the electrostatic and DFs. At the distances $h \sim 100$ nm the DFs (Casimir forces) have been measured with a precision of 1% in a series of experiments [6–8] (for reviews see [9,10]). The precision of these experiments is essentially determined by our ability to measure the absolute distance h, which can be presented as $h = \Delta h + h_0$. The relative change of the separation Δh can be reliably controlled instrumentally, but the hardest task is the determination of the minimum

distance between the bodies h_0 (called also the distance upon contact or average separation between the bodies in contact), which is restricted by an experimental variance of $\delta h_0 \sim 1$ nm. There are two main problems to measure the forces at shorter distances $5 \lesssim h \lesssim 50$ nm [11]. First, assuming a typical power-law behavior $\sim h^{-\alpha}$ of the force with distance the relative uncertainty in the force increases as $\alpha \delta h / h$ when h decreases, where $\delta h \approx \delta h_0$ and for the sphere-plate configuration $\alpha \approx 2.5$. Second, the pull-in instability makes often impossible measurements at short separations.

The latter effect plays a significant role in modern micro/nanoelectromechanical systems (MEMS/NEMS) because the elements of these systems are separated by rather small distances and have a sufficiently large area for the forces to influence the operation of moving components. Without an application of external load the pull-in instability can lead to unwanted permanent stiction of the elements during fabrication or operation [12–14] and for this reason the DFs have to be carefully controlled. To investigate the effect of stiction, a model system has been proposed [13], which is an adhered cantilever shown schematically in Fig. 1. It is an elastic beam for which one end is firmly fixed at some height and the other end is adhered to the substrate. The adhesion energy per unit area Γ is related to the unadhered length of the cantilever s by a simple relation $\Gamma = 3Et^3H^2/4s^4$ [15,16], where E is the Young modulus of the cantilever and other parameters are defined in Fig. 1. This relation gives a direct method for the experimental determination of Γ [17,18]. The unadhered

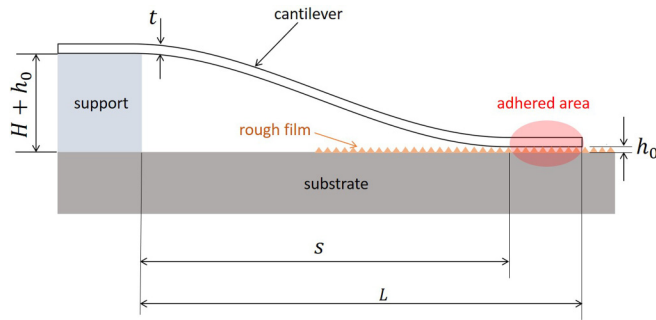*Corresponding author: v.svetovoy@phyche.ac.ru

FIG. 1. Adhered rectangular cantilever. The left end is fixed at a height H with respect to the adhered end. The right end is adhered at the distance h_0 from the average plane of the rough substrate. The total and unadhered lengths are denoted as L and s, respectively, and thickness of the cantilever is t.

length s can be determined interferometrically with a relative precision of $\sim 1\%$.

The standard technology of MEMS/NEMS fabrication uses thin film deposition of different materials, which is often realized by magnetron sputtering, thermal deposition, or related methods. At nanoscale the deposited films are rough with the root-mean-square (rms) roughness depending on the deposition parameters. However, it was noticed [19] that the height distribution of thermally evaporated Au films is not well described by the Gaussian distribution demonstrating a significant number of asperities, which are much higher than the rms value. A recent analysis [20] of magnetron sputtered films of different materials revealed even a more pronounced effect. The excessive number of high asperities has been related to nonequilibrium deposition conditions with the following relaxation of local stresses.

The surface roughness can strongly influence both the DF and adhesion energy when the bodies are separated by the distances comparable with the highest asperities. These asperities are also responsible for the distance upon contact h_0. For the adhesion energy the role of roughness was confirmed experimentally using the adhered cantilevers [21]. On the other hand, the forces measured at the smallest possible separations [22] strongly deviate from those predicted by the Lifshitz theory. The latter was explained [23,24] by the influence of high asperities approaching very close to the opposite surface.

To measure the DFs at a relatively short distances one has to overcome the problem of the pull-in instability. The adhered cantilever has been proposed as a possible solution [25]. Such a cantilever does not suffer from the instability problem in contrast with the elastic suspension systems used for the measurements. The shape of the cantilever is sensitive to the DFs [26] acting between the beam and substrate near the adhered end at $h \to h_0$ and the change of the shape is in the measurable range. One has to note that the information on the dispersion interaction is also contained in the adhesion energy Γ. This energy is easier to measure than the shape of the cantilever, but as an additional contribution it includes the interaction from the area of direct contact between the bodies. The contact contribution is defined by the surface forces and contact mechanics and depends on the parameters, which are not well known.
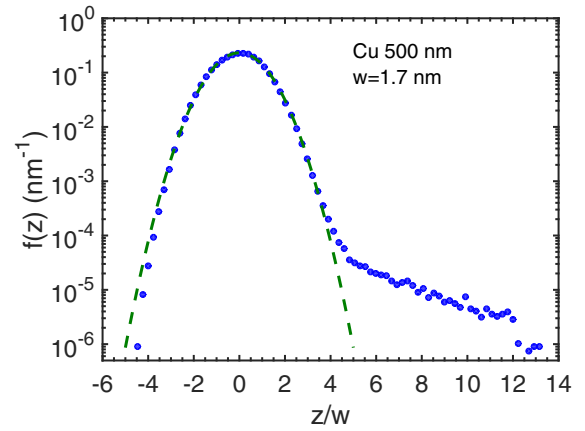

FIG. 2. The density distribution function for Cu (circles) extracted from an AFM image of $20 \times 20 \ \mu\text{m}^2$ with 4096 pixels per line. The height z is counted from the average plane ($z = 0$). The green dashed curve is the Gaussian distribution for the same rms roughness w.

In this letter we demonstrate that the adhesion energy Γ between rough surfaces is nearly completely determined by the dispersion interaction at the average separation between the surfaces in contact h_0. This statement is true for the rough surfaces with an excessive number of high asperities as it is observed for the films deposited on cold substrates. Using this relation one can determine h_0 via the measured Γ with a precision better than $\delta h_0 = 1$ nm.

Consider two rough plates that approach contact. From the mechanical point of view the problem is equivalent to a flat hard plate that is getting into contact with a deformable plate described by a combined roughness (sum of the two rough topographies) [27]. The elastic modulus of the rough plate is given by an effective Young modulus, but plastic properties are defined by the properties of the softest material. The nanoscopic surface roughness is characterized by an atomic force microscope (AFM). This instrument presents the topography of a surface as a set of pixels on the surface with a specific height h_{ij} for each of them. Here the indices i, j enumerate square pixels with a size Δ in the range of a few nanometers. The height of a pixel h_{ij} is determined with a resolution of 0.1 nm. The nominal surface of the rough plate is counted from the average position $h_{\text{av}} = \sum_{i,j} h_{ij}/N$, where N is the number of the pixels in the image. Thus any AFM image can be presented as a matrix $z_{ij} = h_{ij} - h_{\text{av}}$ with the zero average value.

The height density distribution function $f(z)$, which will be used in the following, is defined as the number of pixels with heights in the interval $(z, z + \Delta z)$ with respect to the total number of pixels N and to interval Δz. As an example Fig. 2 shows the function $f(z)$ for a 500 nm thick Cu film deposited by magnetron sputtering [20]. One can see that the probability to find pixels with heights above $4w$ is much larger than that predicted by the normal distribution (dashed green curve).

The contact between a stiff flat and deformable rough plates occurs via the highest asperities as shown in Fig. 3 for a realistic rough profile. Let us stress a significant difference in the vertical and horizontal scales. Local pressure on
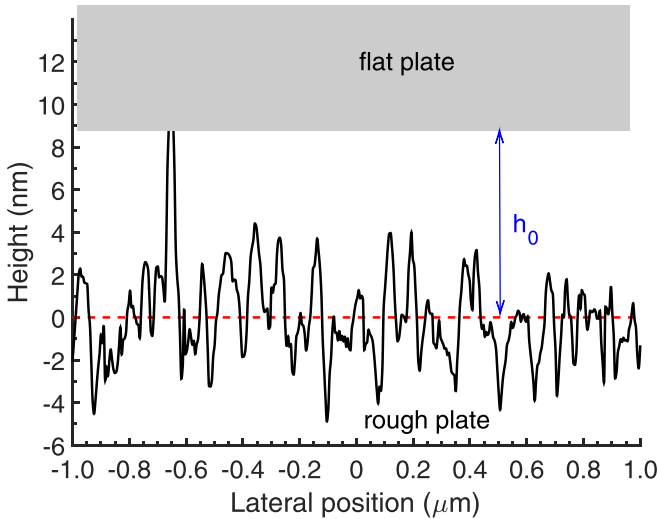
FIG. 3. Contact of a stiff flat and deformable rough plates. The red dashed line shows the zero level of the rough plate (average position). The actual rough profile of Cu film is shown. The highest asperity is deformed plastically.

high asperities can easily overcome the flow stress P_f of the material so that these asperities will be deformed plastically. The deformation of high asperities will stop only when the total repulsive contact force and total attractive force between the plates are equal. This is the equilibrium state which we are looking for. In this state the average distance between the plates is equal to h_0.

Let the force per unit area between flat parallel surfaces separated by the distance h be $P(h)$. It is well known that the DFs are not additive [4] and in general the force between rough plates cannot be calculated via $P(h)$. However, for slowly varying topography one can use Derjaguin's [28] (proximity force) approximation. This approximation can be applied if the lateral size of asperities 2ξ (ξ is the correlation length) is large in comparison with the average distance between the bodies, $4\xi^2/h_0^2 \gg 1$. Since we consider relatively short distances the condition holds true, but even if the ratio $4\xi^2/h_0^2$ is not very large, one can find a correction to the force perturbatively [29–31]. In the frame of Derjaguin's approximation the rough surface is approximated by flat patches, which can be identified with AFM pixels, and the force is calculated as the sum over all patches using the function $P(h)$.

When the plates are in contact in the equilibrium state at $h = h_0$ the total force has to be zero due to balance between the attractive and repulsive forces. If the plates are separated by the distance $h > h_0$, the total force can be written with the use of the distribution function $f(z)$ as follows:

$$P_{\text{tot}}(h) = \int_{-\infty}^{h_{0p}} dz f(z) P(h-z) + P(h-h_{0p}) R(h_0). \quad (1)$$

It consists of two contributions. The first term describes the noncontact contribution where $h - z$ is the local distance between the flat and rough surfaces. The lower integration limit is changed by $-\infty$ since both functions $f(z)$ and $P(h-z)$ decrease fast for deep pits. The upper limit is chosen to take into account all asperities that never have been in contact

with the flat plate. The maximum height of such asperities is $h_{0p} = h_0 - h_{0c}$, where h_{0c} is the distance that separates two flat surfaces in direct contact. This distance is as small as 2–3 Å [32] but not zero and corresponds to the balance of repulsion and attraction between flat plates at a fixed load (Derjaguin's self-consistence approach [33,34]). The second term in Eq. (1) is the "contact" contribution to the force. In the equilibrium state at $h = h_0$ the pressure $P(h - h_{0p}) = P(h_{0c})$ is the contact pressure. In the equilibrium this pressure is such that further plastic deformations are not possible and, therefore, the contact pressure must be on the border separating plastic and elastic deformations so that $P(h_{0c}) = P_f$. This pressure is quite large for nanocrystals $P_f \sim 1$ GPa, but because the plates contact only over high asperities, the relative area of direct contact $R(h_0)$ is small.

The adhesion energy Γ is the work that has to be done against the total force to separate plates from the position at $h = h_0$ to infinity. Integrating Eq. (1) and changing the sign one finds

$$\Gamma = -\int_{-\infty}^{h_{0p}} dz f(z) W(h_0 - z) - W(h_{0c}) R(h_0), \quad (2)$$

where $W(h)$ is the energy between flat parallel plates separated by the distance h,

$$W(h) = \int_{h}^{\infty} dx P(x). \quad (3)$$

In Eq. (2) Γ is just a number that is calculated for the equilibrium distance h_0. This distance is determined from the force balance equation $P_{\text{tot}}(h_0) = 0$, which depends on some not well known parameters. For example, we know the general behavior of $W(h)$ in the distance range $h_{0c} < h < h_s$, where $h_s \sim 1$ nm is the upper limit of the region with important repulsion. This energy is used in molecular dynamics simulations, but we hardly can use this $W(h)$ for precise calculations. In addition, the flow stress P_f depends on the detailed nanostructure of the deposited films, which is not specified. Due to these uncertainties let us consider h_0 in Eq. (2) as a variable and denote it as h_0^* to distinguish from the true equilibrium value h_0. The adhesion energy $\Gamma(h_0^*)$ is given by Eq. (2) after the substitute $h_0 \to h_0^*$.

From the first term in (2) we will separate very short local distances $h_{0c} < h_0^* - z < h_s$, where conventionally it is chosen $h_s = 1$ nm. It separates the range of distances where the forces can deviate from pure DFs due to the presence of other surface forces. Combining this contribution with the second term in (2) we introduce "nearly" contact contribution to Γ:

$$\Gamma_c(h_0^*) = -\int_{h_{0c}}^{h_s} dx f(h_0^* - x) W(x) - W(h_{0c}) R(h_0^*), \quad (4)$$

where a new integration variable $x = h_0^* - z$ has been introduced. Note that due to the uncertainties we can estimate Γ_c, but not to calculate it with a good precision. The rest is the noncontact contribution, which can be calculated for pure dispersion interaction:

$$\Gamma_{nc}(h_0^*) = -\int_{h_s}^{\infty} dx f(h_0^* - x) W(x). \quad (5)$$

In Eq. (5) the function $W(x)$ can be calculated within the Lifshitz theory, but in Eq. (4) this function has to include a short-distance repulsion and possibly some other surface forces. We can estimate Γ_c using the dispersion energy with the short-distances repulsion [35]:

$$W(h) = -\frac{A_H}{12\pi h^2}\left[1 - \frac{1}{4}\left(\frac{h_c}{h}\right)^6\right]. \qquad (6)$$

Here A_H is the Hamaker constant for the interaction of two materials in vacuum (air) and h_c is the equilibrium distance between flat parallel surfaces, which can be estimated as $h_c = (2/15)^{1/6}\sigma$ using the size parameter σ of the Lennard-Jones model. As an illustrating example we consider Si-Cu interaction, for which h_c is predicted as $h_c = 0.224$ nm. The value of h_{0c} is determined as the equilibrium distance between the flat surfaces with the pressure equal to the flow stress. It is given by a solution of equation $P(h_{0c}) = P_f$, where $P(h)$ at short distances is defined as the derivative of energy (6) with the opposite sign. The flow stress is not a well-defined parameter. For nanostructured Cu with a grain size of about 40 nm it is estimated as $P_f \approx 2$ GPa [36] but significant uncertainties are possible. Note that in any case the value of P_f is much larger than that for the bulk material. For $P_f = 2$ GPa we have found as the solution $h_{0c} = 0.198$ nm. The Hamaker constant $A_H = 25.9 \times 10^{-20}$ J was determined via the short-distance limit of the Lifshitz formula using the optical properties of Cu and Si from the handbook [37]. For $W(h_{0c})$ it was found a value of -84.2 mJ/m^2. For Si-Pt and Si-Au systems similar values of the order of -100 mJ/m^2 were determined.

The relative area of real contact $R(h_0^*)$ can be evaluated from AFM images of interacting surfaces. One can imagine a rough surface as a set of columns with the cross section equal to the pixel size squared. It is assumed that the columns higher than $h_0^* - h_{0c}$ are deformed plastically to the height $h_0^* - h_{0c}$ without significant change of the area. Only these columns contribute to the area of real contact and the result can be presented as

$$R(h_0^*) = \int_{h_{0p}^*}^{\infty} dz f(z), \qquad (7)$$

where $h_{0p}^* = h_0^* - h_{0c}$ is defined similar to h_{0p}. Indeed this is an approximate relation but it can be used as a good estimate.

The softer the material (smaller P_f) the smaller the distance h_0 separating the surfaces in contact. As a lower limit for h_0^* we take the solution of the balance equation for P_f, which is four times smaller than its nominal value. For the Si-Cu system it is 0.5 GPa that gives $h_0^* = 20.6$ nm. The upper limit for h_0^* corresponds to the maximum height that is 23.1 nm for Cu film in Fig. 2. As one can see the plastic deformations can occur in a rather narrow range of heights.

The adhesion energy as a function of separation in the range of interest is shown in Fig. 4. Two curves that correspond to $P_f = 2$ GPa (solid blue) and $P_f = 0.5$ GPa (brown dots) practically coincide. It is important because the value of P_f is known only roughly. Such a weak sensitivity of the adhesion energy to P_f occurs because in $\Gamma(h_0^*)$ the flow stress does not appear explicitly and all the dependence on P_f is realized via h_{0c}. On the other hand, a sharp dependence of
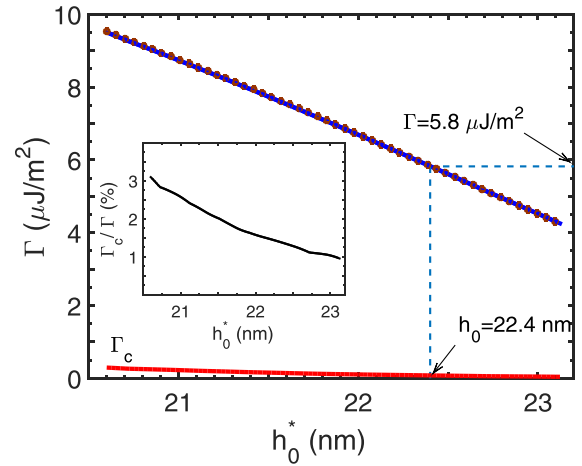

FIG. 4. The adhesion energy for Si-Cu system as a function of separation h_0^* between the flat and rough surfaces in contact. The solid blue line is for $P_f = 2$ GPa and the line marked by the brown dots is for $P_f = 0.5$ GPa. The red curve at the bottom shows the contact contribution to Γ. The inset demonstrates the relative contact contribution in percent.

the repulsion [second term in (6)] on distance results in a very small variation of h_{0c} while P_f changes significantly.

An additional feature is that the contact contribution Γ_c to the adhesion energy is small as show the red curve in Fig. 4 and the inset. It is important because at short distances some surface forces different from (6) can contribute to Γ_c. The contact contribution is small ($\sim 1\%$, see inset) due to specific roughness of the films deposited in nonequilibrium conditions. These films contain an excessive number of high asperities, which restrict the smallest distance between the bodies by the value of $h_0 \gg w$ that is significantly larger than the rms roughness w. The number of such asperities is small and because of this the area of real contact is also small. Thus the main contribution to the adhesion energy comes from the noncontact term (5), where the energy $W(h)$ is calculated in the Lifshitz theory and well defined.

Since the value of h_0 is large in comparison with w the adhesion energy related to the dispersion interaction is rather small. For the Si-Cu system with $P_f = 2$ GPa it is found $\Gamma = 5.8$ μJ/m^2 and for other metals it stays in the range $\Gamma < 100$ μJ/m^2. Such energies are very small in comparison with the effects usually investigated in the contact theory where the adhesion is $\gtrsim 100$ mJ/m^2 [38]. In the contact theory the Gaussian surface roughness is usually considered. In such a case the surfaces in contact are separated by the distances $(2-3)w$ that gives much stronger adhesion mostly due to a much larger area of real contact. Nevertheless, the weak adhesion considered in this paper is of practical importance especially for microsystems.

For the weak adhesion the function $\Gamma(h_0^*)$ is defined practically only by the dispersion interaction via the average gap between bodies in contact h_0^* and is not sensitive to P_f. It explains the main result of the study [21] where the adhesion energy between rough surfaces SiO$_2$ and Si were measured with adhered microcantilevers. It was convincingly demonstrated experimentally that the weak adhesion (1–10 μJ/m^2)

is due to the dispersion interaction via the average gap separating the surfaces.

In contrast to the function $\Gamma(h_0^*)$, which is nearly universal for a given pair of materials, the equilibrium distance h_0 is sensitive to the value of the flow stress P_f. For example, for $P_f = 2$ GPa we have found $h_0 = 22.4$ nm. This distance gives a point on the curve $\Gamma(h_0^*)$, which corresponds to the equilibrium adhesion energy $\Gamma = 5.8 \; \mu J/m^2$ as shown in Fig. 4 by the dashed lines. For $P_f = 0.5$ GPa the equilibrium distance is $h_0 = 20.6$ nm and the corresponding adhesion is $\Gamma = 9.5 \; \mu J/m^2$. Thus variation of P_f moves the equilibrium point along the universal curve $\Gamma(h_0^*)$, which is calculated using only the Lifshitz theory. It is an important property that relates the adhesion energy and the distance upon contact.

The hardest problem for determination of the DFs at distances $h \sim 10$ nm is a restricted precision for measurement of the distance upon contact h_0, which gives the reference point to count absolute distances. For adhered cantilevers this distance can be determined interferometrically [25] but with a restricted precision not better than ± 1 nm. One can use the universal function $\Gamma(h_0^*)$ to improve this precision. Suppose that we measure the adhesion energy Γ with a precision of 10% by measuring the unadhered length s interferometrically by scanning along the cantilever. Then from the graph

presented in Fig. 4 one can determine h_0 with precision better than 0.5 nm. Optimally Γ can be measured interferometrically with a precision of 2.5% that is on the same level as uncertainty due to the short distance effects. It will reduce the error in h_0 to $\delta h_0 = 0.1$ nm. One can argue that this increase in precision is not a specific property of the Si-Cu system. All the systems that contain an excessive number of high asperities demonstrate similar improvement in the determination of h_0.

In conclusion, we demonstrated that the weak adhesion between rough surfaces is almost entirely defined by the non-contact dispersion interaction across the average gap between the bodies in contact. The contact contribution to Γ, which cannot be calculated precisely, is estimated as a few percent or less. This property is closely related to a specific roughness of films deposited in nonequilibrium conditions. For such films an excessive number of high asperities is observed, which prevent close contact. It is also demonstrated that Γ is not sensitive to such a parameter as the flow stress, which characterize the plastic deformations and its value is not well known. These properties of the function $\Gamma(h_0^*)$ enable us to consider it as a universal function, which is calculated entirely via the Lifshitz theory.

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