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A new method is presented for measurements of friction of microsized particles on surfaces. Specifically in this work, the particles are alumina with diameters between ≈1 and 50 μm and the surfaces are InP, Si, and Cr. Friction is analyzed, its components are determined, and the friction coefficients are estimated from the experimental results. The technique and the specific instrument allow measurements of coefficients of friction for spherical particles with radii as small as 1 μm. For smaller sizes, the instrument needs to be modified by using a more powerful power supply, actuator with extended frequency and amplitude ranges, cooling of the actuator and the power supply, and the related mechanical modifications of the sample holder. © *2009 American Institute of Physics.* [DOI: 10.1063/1.3212672]

I. INTRODUCTION

Organizing micro and nanosized objects on commercially available materials aims at fabricating small devices usable in practical fields as electronics, magnetics, nanobiodevices, and optics. It is a potentially lucrative goal, which needs joint effort of researchers working in various areas. However, some of the aspects of this work are insufficiently addressed in the literature and offer inconsistent and scarce advice on affordable and versatile cleaning of the commercial wafers used in an organizational process. For example, the cleaning recipes for InP (Ref. 1) or Cr (Ref. 2) are too specific and cannot be used on a large scale and in various experimental conditions.

Numerous patterning methods[3] relate their results mostly to the size of the device without sustained regard for virtually important matters such as the impact of the patterning on the feasible physical properties of the fabricated micro or nanostructure. One example of dissonant approach is the question of how a specific patterning method modifies a physical property of a surface, e.g., the mechanical friction or reflectivity of waves. Another not sufficiently elucidated step is the problem of possible incompatibility of the surface-cleaning procedure with the patterning made before that. For example, mechanically grooved Si surfaces may change beyond designed parameters when cleaned with acids used in the advised general cleaning procedure.[4]

Some patterning methods are high vacuum-based[5] and limited at present as mass production affordable tools.

Another practically not discussed issue in the literature is the physical behavior of the particles or wires on the surfaces, on which they have to be organized. Little is known for their mechanical motion on a specific surface in air, the influence of the particle shape on the motion, the nature of the physical contact particle wafer, the acting forces between particles, the collective motion of an ensemble of particles, the impact of the particles' clustering on the organizational process, and the role of externally imposed conditions such as gas pressure, motion of the substrate, or its temperature.

This work addresses a trend in the area of the organizational process: organization of some specific microparticles on commercial or alternative surfaces—InP, Si wafers, and 500 nm Cr sputtered films on Si(100) substrate. The research focuses on the mechanical motion of nonmagnetic particles with sizes between ≈1 and 50 μm to investigate the friction on this scale, its nature, and describe the processes with physical parameters.

II. MEASUREMENT METHOD AND EQUIPMENT

Alumina (Al_2O_3) spherical-shape particles with diameters between 1 and 50 μm were purchased from Alfa Aesar Inc. This kind of particles was chosen to exclude magnetic forces at this step of the investigation as interfering with the basic interactions in the system: particle-particle and particle-surface.

The main components of the experimental setup are purchased from Piezosystem Jena Inc.[6] and the equipment is displayed in Fig. 1.

The equipment consists of an ac frequency generator, voltage amplifier, and a piezoelement, called actuator-type PA 4/12, in which the piezostack elongates or contracts ≈4 μm.[6] The power supply to the electronics is ENT 400, and the voltage amplifier is ENV 800, able to produce voltages on the peizostack in the range of −10 to 150 V.[6] The particles are placed on a substrate and a plate attached to the actuator, which can be adjusted to vibrate horizontally, vertically, or at an angle to the horizontal direction. The recorded physical parameter in these experiments is the frequency, $f_m(r)$, at which a particle of radius r begins moving on the surface.

In an exemplary measurement called load, the results of which are displayed in e.g., Figs. 2(a) and 2(c) with one and the same symbols, few particles are dispersed on precleaned InP, Si, or Cr surfaces. The structure is subjected to mechanical periodic vibrations with amplitude 2.41 μm and frequency-sweep range between 1 Hz and 10 kHz. Experimental plots $f_m(r)$ are shown in Fig. 2. The measured raw

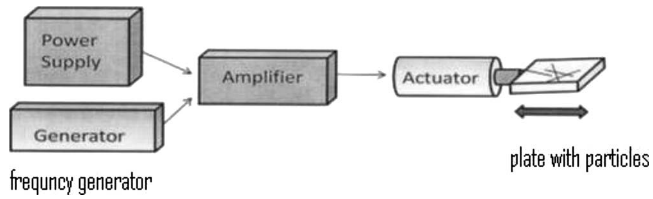

FIG. 1. Experimental setup of the used equipment for measuring the size-dependence of the frequency $f_m(r)$, at which a particle with radius r starts moving on a surface.

data $f(d)$ are normalized, so that every frequency of the measurement corresponds to one and the same mechanical amplitude[7] (d is the particle diameter).

The horizontal vibration mode reveals information on the nature of the particle-substrate friction. The vertical mode additionally informs about possible adhesive forces, which add to the basic forces acting on a particle with mass m: gravitational (G=mg) and normal reaction of the plane. The particles and surfaces were observed with a Nikon optical microscope (1500×) supplied with a video system.

The experiments were made at one and the same humidity of the environment determined by a hygrometer—88%, and the humidity controlled by a dehumidifier.

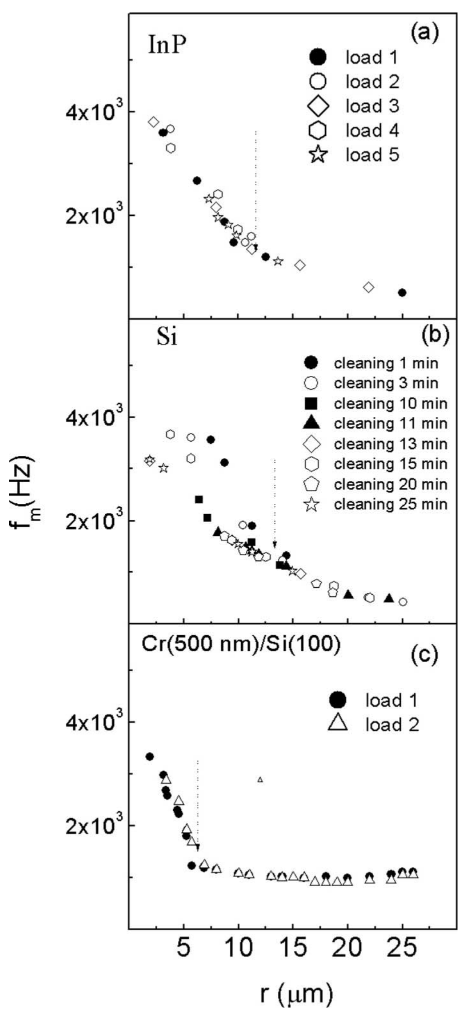

FIG. 2. Frequencies, at which the alumina particles start moving on the specific horizontally vibrating surfaces as a function of their radii: (a) on InP surface, (b) on Si, and (c) on Cr. The dotted arrows show the inflection points, associated with increased contribution of unconventional forces below the indicated sizes at these surfaces.

III. RESULTS AND DISCUSSION

In Fig. 2 are displayed the frequencies, $f_m(r)$, at which the particles start moving on a horizontally vibrating surface as a function of the particle radius. The particles are alumina and the surfaces are InP in Fig. 2(a), Si in Fig. 2(b), and Cr in Fig. 2(c). It is clearly observed in Fig. 2, that these frequencies are size dependent, as well as dependent on the specific contacting materials.

Another observation is related directly to the cleaning procedure of the substrate and its time period of application. In the case, the latter affects $f_m(r)$ and the related friction as seen in Fig. 2(b) for the silicon surface. The dark circles indicate $f_m(r)$ as a result of 1 min of precleaning of the substrate in a mixture of 35% sulfuric acid and hydrogen peroxide (3:1) at 70 °C, the open circles—after 3 min, and the other symbols after 10 min and above, which proves to be the sufficient time for cleaning of the Si surface. The reproducibility of $f_m(r)$ at each load of particles at one and the same size may be challenged by the environmental conditions of the experiment, such as the moisture or the state of the surface as local surface defects, at which the particles may adhere. Our experiments reproduced the $f_m(r)$—plots within 30 Hz, with well observable repeated trend of the changes in $f(r)$.

The interactions between particles and surfaces can be a unique combination of long-range and short-range forces. The long-range forces operate at contact distances from a few to hundreds of Angstroms[8] (Å). The short-range forces act at distances less than 2 Å.[8]

The long-range forces include the van der Waals (VDW) forces, the magnetic, electrostatic, and the capillary forces. Attempts have been made to describe them by mathematical expressions, e.g., the VDW force of attraction between two atoms is described by $1/x$ (Ref. 8) (x is the distance between the two atoms). The VDW force between a particle and a plane is expressed[8] as $F(D)=-H\times r/6D^2$. The parameter r is the particle average radius, D is the distance from the top of the particle to the plane, H is named Hamaker constant. When $D=2r$, $F(r)=-(H/24)\times(1/r)$. (The symbol $*$ in a mathematical expression stands for multiplication in this text.) Thus, the VDW force between a sphere and a plane is similarly expressed to the mechanical rolling friction, as inversely proportional to the size $(1/r)$. The magnetic forces are not relevant in these experiments, and the electrostatic[8] and the capillary forces[9] play more significant roles in microscopy, e.g., between the probing tip and the sample (See details for electrostatic friction in Ref. 8, chapter 3, and Refs. 7–9 therein.)

The short-range forces encompass the Pauli repulsion, physisorption and chemisorption, metallic adhesion, and the forces occurring at local elastic and plastic deformations during mechanical friction. The Pauli repulsive forces[8] are described by an expression $\sim$const$/r^n$ with an exponent n greater than 8. Physisorption and chemisorption, relevant for tip microscopy may contribute additionally to any force of attraction. The metallic adhesion occurs at close contact between two metals.

It is regarded as a quantum mechanical effect and its strength is expressed as exponentially dependent on the distance between the contacting media[10] as e^{-D}. When a particle and a surface are in contact, the metallic adhesive force is expressed as $e^{-r/t}$. The particle radius is denoted by r and t is a parameter named depth of penetration, which can be equal or smaller than r, or larger than r, depending on the nature of the contacting media.[10]

Thus, we can conditionally summarize that the long-range forces depend on the particles' radii inversely proportionally, while the short-range forces depend on the particles' sizes exponentially.

Macroscopically, mechanically, and relative to an external reference point, a horizontally vibrating plane with mechanical amplitude A_0 and frequency f will act on a particle with a force $F=ma=mA_0f^2\cos(2\pi \mathrm{ft})$. Additionally, the particle will experience frictional force (F_{fr}) in a direction opposite to the vibration. The force of friction in the conditions of the experiment will oscillate with the same frequency as the vibrating piezostack.

We assume at this point that the adhesive forces are negligible to express the frictional coefficient in a general form as

$$K_{\mathrm{fr}} = K/r^a. \tag{1}$$

Equation (1) covers the pure cases of mechanical friction. For $a=0$, K_{fr} is identified with the coefficient of sliding friction and for $a=1$, K_{fr} describes the rolling friction. (In Eq. (1) r is the radius of the particle in micrometers, K is the size-independent part of the coefficient of friction, and a is the exponent of friction.)

A particle is able to move on a horizontally vibrating surface when it can overcome the force of friction: $|F| \geq |F_{\mathrm{fr}}|$. Therefore, $mA_0f^2\cos(2\pi \mathrm{ft}) \geq mgK/r^a$, which yields for the vibration frequency the expression

$$f_m \geq \sqrt{(K/r^a)} \times \sqrt{(g/A_0)}, \tag{2}$$

where $f \equiv f_m$. Equation (2) expresses the frequency, at which a spherical particle of any mass can start moving on a surface of a vibrating plane. This frequency is square inversely proportional to the mechanical amplitude of vibrations A_0, depends on the radius of the particle and the nature of the contact specified by K, and the exponent a. It is within the objectives of these experiments to analyze and calculate the physical parameters in Eq. (1) for some specific cases of particles moving on substrates. It is worth noting, that the calculated values of K will be the coefficients of initial friction on the plane (K_{start}), therefore larger than the related coefficients during an actual motion (K_{mov}), similarly to the situation when $K_{\mathrm{start}} > K_{\mathrm{mov}}$ in the classical-mechanics friction.

The measured $f_m(d)$ data are presented as $f_m^2 A_0/g$ versus particle radius r in Fig. 3. The experimental data with inserted value of the mechanical amplitude of vibration are fitted to Eq. (2) and the characterizing parameters K and a are calculated.

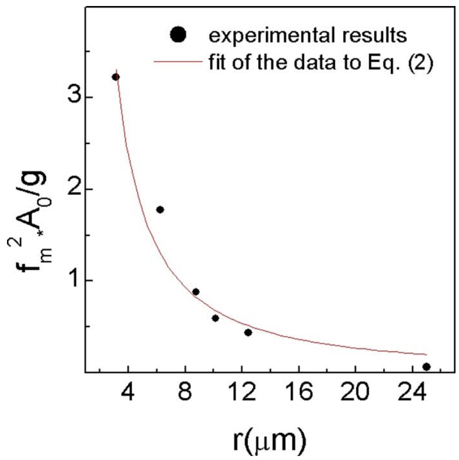

FIG. 3. (Color online) Dependence of $f_m^2 \times A_0/g$ on the particle radius for Al_2O_3 on InP surface: the dark circles represent the experimental results and the dot line is the best fit of the data to the function $y=K/r^a$, for $K=11.52\pm1.52$ and $a=1.19\pm0.10$.

To estimate the strength of the adhesive forces, $f_m(r)$ measurements were made with the actuator vibrating in the *vertical direction*. A particle is able to move on a vertically vibrating surface when the acting force overcomes the adhesive force. The results from the measurements for the InP wafer are displayed in Fig. 4, with Fig. 4(a) the standard measurement f_m versus r, and in Fig. 4(b) the dependence $f_m^2 \times A_0/g$ versus r, needed for the estimations of the adhesive coefficients. It is seen from Fig. 4 that these dependencies are nearly linear in the small size-range ($r \leq 11$ μm for InP surface), which is related to the exponential dependence of the adhesive short-range force on the distance between the materials (For small particles radii the exponential dependence of the short-range adhesive force ($e^{-r/t}$) can be presented as linearly dependent on r when in Taylor series, and limited to the first power of r when $r < t$.). The fit of $f_m^2 \times A_0/g$ versus r, presented with the dot and dash lines in Fig. 4(b), allows calculation of the parameter related to the force of adhesion—the depth of penetration, t. Plane isotropy of the adhesive force is assumed. The dash line represents the fitting for small-size particles and the dot line is the fitting for all measured particles. It presents the result for the adhesive depth of penetration more generally and yields overall smaller values for it. In the case for InP, the calculated depth of penetration is 11 μm for small particles, and the fitting yields a value of $\approx$8.5 μm when larger size particles are

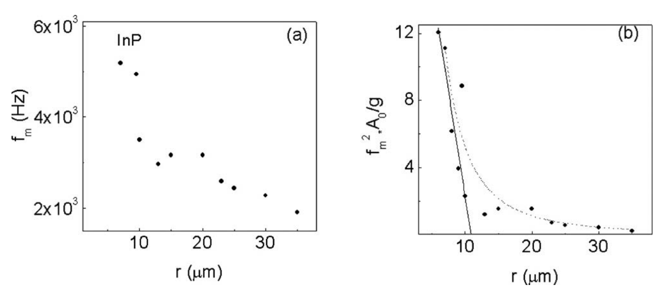

FIG. 4. Vertical vibration of the piezostack: (a) $f_m(r)$; (b) $f_m^2 \times A_0/g$ vs r. The measurements are presented for the InP surface. The solid line in Fig. 4(b) is linear fit of the data for particles with small radii, the dot line is fit of the data to generalized Eq. (2) with $e^{-r/t}$.

TABLE I. Coefficients of friction, K, friction exponent a, and depth of penetration t obtained for the related objects on the microscale.

	Particles		
	Al_2O_3		
	K (μm)	a	t (μm)
Surfaces			
InP	11.52 ± 1.52	1.19 ± 0.10	8.39 ± 0.8
Si	6.21 ± 0.67	0.98 ± 0.07	5.89 ± 0.6
Cr	5.71 ± 0.95	1.03 ± 0.15	3.26 ± 0.5

taken into consideration. This result is reasonable, as smaller particles are known to adhere stronger to metallic and semiconducting surfaces.

Furthermore, the coefficient of friction for a plane vibrating in the horizontal direction is expressed in a generalized form of Eq. (1) with a term $\sim e^{-r/t}$ counting for the adhesion. The depth of penetration t, present in the second term, is determined from the vertical vibrations. Thus, when a horizontal vibration data are recorded and analyzed, t, which is initially calculated from the vertical vibrations of the plane, is further adjusted and the parameters K and a are calculated, using a generalized form of Eq. (2). A user defined function was applied to fit the data in the plots of Fig. 2 to obtain values for the parameters K, a, and t. The estimated parameters for all particles with sizes ≤ 50 μm are enlisted in Table I. While K determines the basic nature of the friction, the exponent a specifies the type of friction as pure rolling, sliding, or more complex. The results displayed in Table I reveal that the friction on these surfaces is complex, comprising of sliding and rollinglike with a portion of adhesive forces, which inhibit the motion of the particles. As seen from Table I the depth of penetration decreases from InP surfaces to Cr. The parameter K descriptive of the sliding portion of the friction also decreases, particularly, two times from the InP plane to the Cr thin-film surface. The exponent a is close to, but different from the value 1, which excludes a pure rollinglike nature of the friction on these surfaces for alumina particles of sizes between 1 and 50 μm.

These values of the exponent a are presumptive of the long-range VDW forces acting between a particle and a plane.

IV. CONCLUSIONS

The motion of a microparticle with size <50 μm on a surface cannot be regarded as simple mechanical sliding and rolling. It is complex and unique for the particle-type and surface. In this work the forces between alumina particles and the surfaces encompass the long-range, size-dependent VDW forces and a good portion of the short-range metallic adhesive forces, especially relevant at sizes below 10 μm. The estimated ratio of the adhesive/VDW forces at low sizes increases more than two times compared to this ratio for larger particle sizes.

The method and the easily assembled equipment allow analyses of the friction on the microscale and its contributing constituents for the specific size, type of the particles, and the surfaces on which they move to be assembled on designed structures.

ACKNOWLEDGMENTS

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