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Lubrication with a thin colloidal layer

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Abstract. Using a surface force apparatus, we observed the lubrication of a colloidal suspension using spherical and planar geometry on a nanometric scale. We have shown that the behaviour of the layer, and in particular the friction instabilities, can be explained in terms of the compaction of adsorbed colloidal particles.

The shear behaviour of colloidal suspensions confined in very small spaces has been recognized as having technological significance in the areas of granular rheology [1], friction [2] and boundary lubrication [3]. For instance, if we consider the macroscopic contact of two solids rubbing together, the real nature of the solid surfaces in contact, and of the lubricant, is very often colloidal. Both the solids and the lubricant consist of assemblies of particles. The size of each isolated particle is larger than that of a common molecule, being greater than 10 \AA . But it is smaller than $1 \mu\text{m}$, which is very small in comparison with the size of the contact area.

We have observed, by mechanical measurement, the consolidation of an adsorbed layer of colloidal particles on solid surfaces during the normal approach of the substrates. From this we have shown that the frictional resistance is strongly dependent upon the consolidation, leading to instabilities for a critical volume fraction of colloid in the interface.

Our experiments were conducted with a surface force apparatus, which has already been described in the literature [4, 5], operating at room temperature ($21.00 \pm 0.01 \text{ }^\circ\text{C}$) and in dry air conditions. Let us consider a highly smooth sphere (radius $R = 4.75 \times 10^{-3} \text{ m}$) in normal approach to a plane. The two surfaces are made of 52100 heat-treated steel, polished with diamond paste to produce a maximum asperity height below 50 \AA [6] and are covered in spherical particles (diameter $c \approx 100 \text{ \AA}$) of calcium dialkybenzene sulphonate containing encapsulated calcium carbonate [7, 8]. The adsorbed material is in equilibrium with a solution of colloid in *n*-dodecane with a volume fraction $\Phi^* = 5 \times 10^{-4}$.

We first measure the interactions of the two surfaces as a function of the surface separation D during their normal approach at a constant speed \dot{D} to which a small perturbation $\Delta D \exp(i\omega t)$ has been added. Sliding is then produced at a given separation D and a constant speed d , again with a small superimposed perturbation $\Delta d \exp(i\omega t)$,

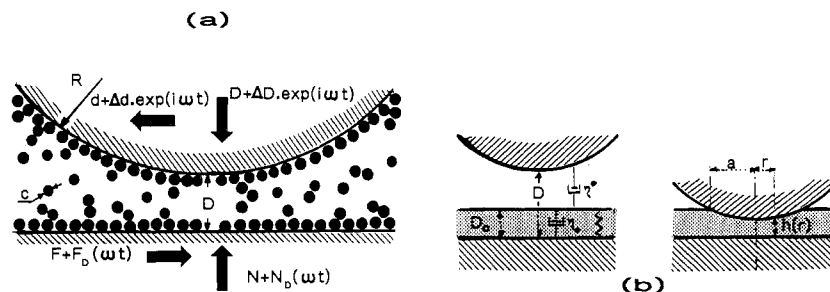


Figure 1. (a) A sphere (radius $R = 4.75$ mm) is covered in small colloidal particles ($c \approx 100$ Å). The interface is lubricated with a dilute solution of colloids. Note that the scale is not respected: $C/R = 2 \times 10^{-6}$; $D/R = 5 \times 10^{-6}$. Squeeze behaviour $N(D) + N_D(D)$ and frictional behaviour $T(d) + T_D(d)$ (d sliding distance) for constant D , are studied. (b) Schematic representation of the interface. All the colloids are on the plane. Mechanically this layer behaves as a viscoelastic solid surrounded by a viscous fluid. The contact radius of the indented layer is a .

with a frequency of 38 Hz (figure 1(a)). Due to the adsorption process and slight flocculation, the surfaces are not covered by a single layer of particles, but by a heterogeneous layer, whose thickness is represented by $D_0/2$. D_0 is greater than $2c$ [5, 9]. In this study, however, from a mechanical point of view, let us consider the two adsorbed layers covering the two surfaces as a single layer of thickness D_0 deposited on the plane. During the squeeze phenomenon, the layer, which has a viscoelastic behaviour, is first covered by a liquid solution (purely viscous), and is then indented by the sphere as shown in figure 1(b). The normal forces are small enough for us to neglect the elastic deformation of the substrates.

Experiments have been conducted in two separate steps: firstly the squeeze tests and secondly measurements of the effect of sliding. Figures 2(a) and 2(b) show the same surface force apparatus used in two ways: one is designed to measure the squeeze, the second the sliding process at constant thickness D . The metallic sphere is firmly fixed to a piezoelectric crystal P_1 . The flat metallic specimen is supported by a double cantilever spring, whose stiffness is adjusted to 3.6×10^6 N m $^{-1}$. Two capacitive displacement transducers C_1 and C_2 are used. The sliding version is a modified version of the surface force apparatus already described in the literature [4].

The relative sphere–plane displacement (d) is measured by the transducer C_2 and controlled by the piezoelectric crystal P_1 which is also used to add a slight sinusoidal motion. The interface thickness D is measured using the electrical sphere–plane capacitance, and a second piezoelectric crystal P_2 keeps it constant. The transducer C_1 measures the tangential force F . Three types of measurement are carried out simultaneously in the experiments: (i) force against displacement $N(D)$ or $F(d)$; (ii) mechanical transfer functions N_D , F_D ; (iii) sphere–plane electrical capacitance C . The components of the force and of the relative displacement D or d are obtained by measuring the mean value of the two signals given by the transducers: the resolution of the displacement is 1.5×10^{-1} Å and the resolution of the force is 1.6×10^{-8} N. The harmonic components of the two capacitor signals are measured with a ‘two-phase lock-in analyser’. The first derivative of the electrical capacitance C , dC/dD , is obtained by measuring the harmonic component that results from the sinusoidal motion of the displacement. The

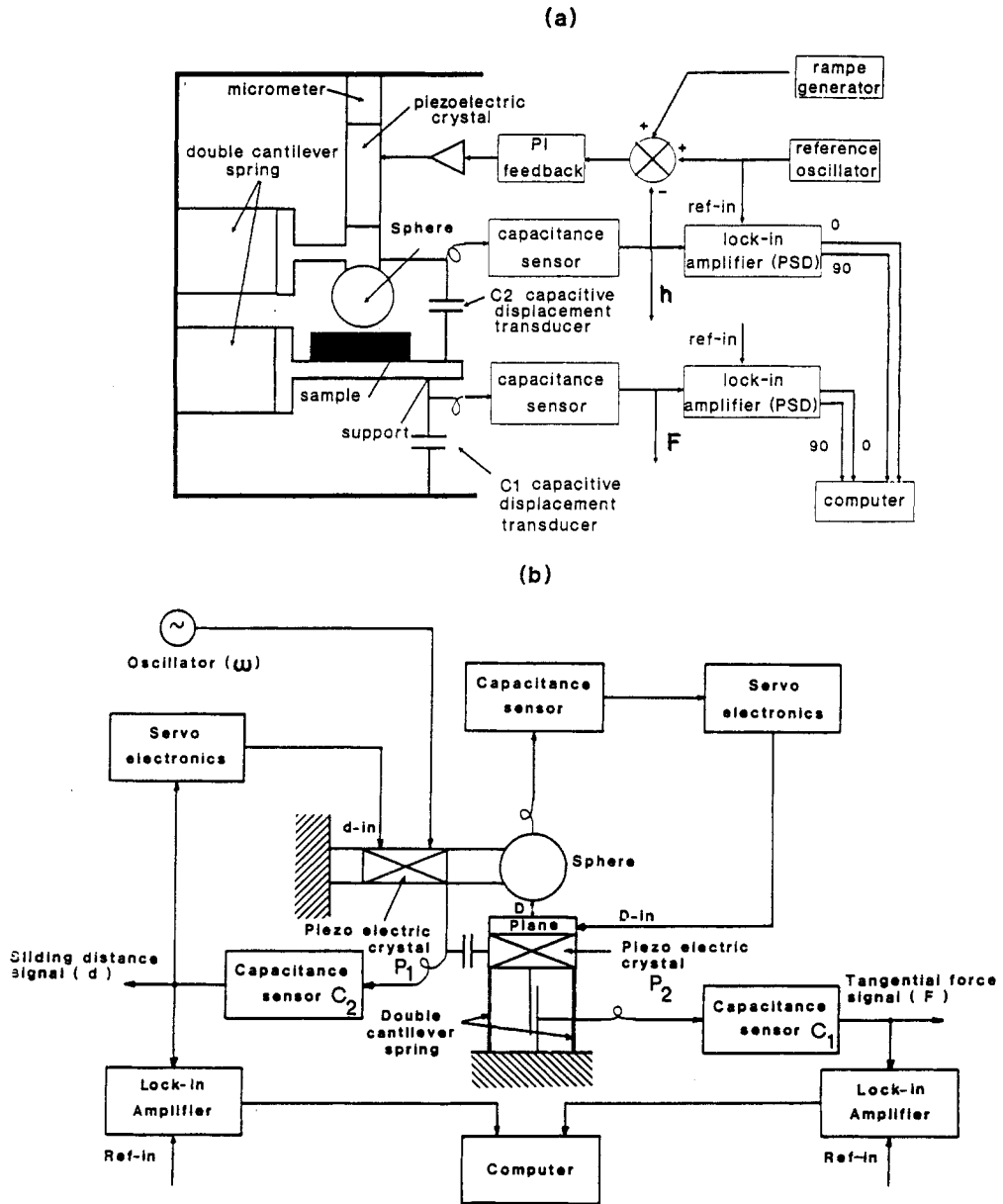


Figure 2. (a) Schematic diagram of the surface force apparatus used to study the squeezed film between the sphere and the plane. (b) Schematic diagram of the nanotribometer. The distance D between the sphere and plane is maintained constant with the help of the piezoelectric crystal P_2 and controlled by measurement of the sphere-plane electrical capacitance.

value of dD/dC is $dD/dC = D/(2\pi\epsilon_0\epsilon_rR)$ where ϵ_0 is the absolute dielectric constant of a vacuum and ϵ_r the relative dielectric constant.

In the squeeze situation, function dD/dC varies linearly with displacement D and therefore the extrapolation $dD/dC = 0$ defines an origin of displacement where the

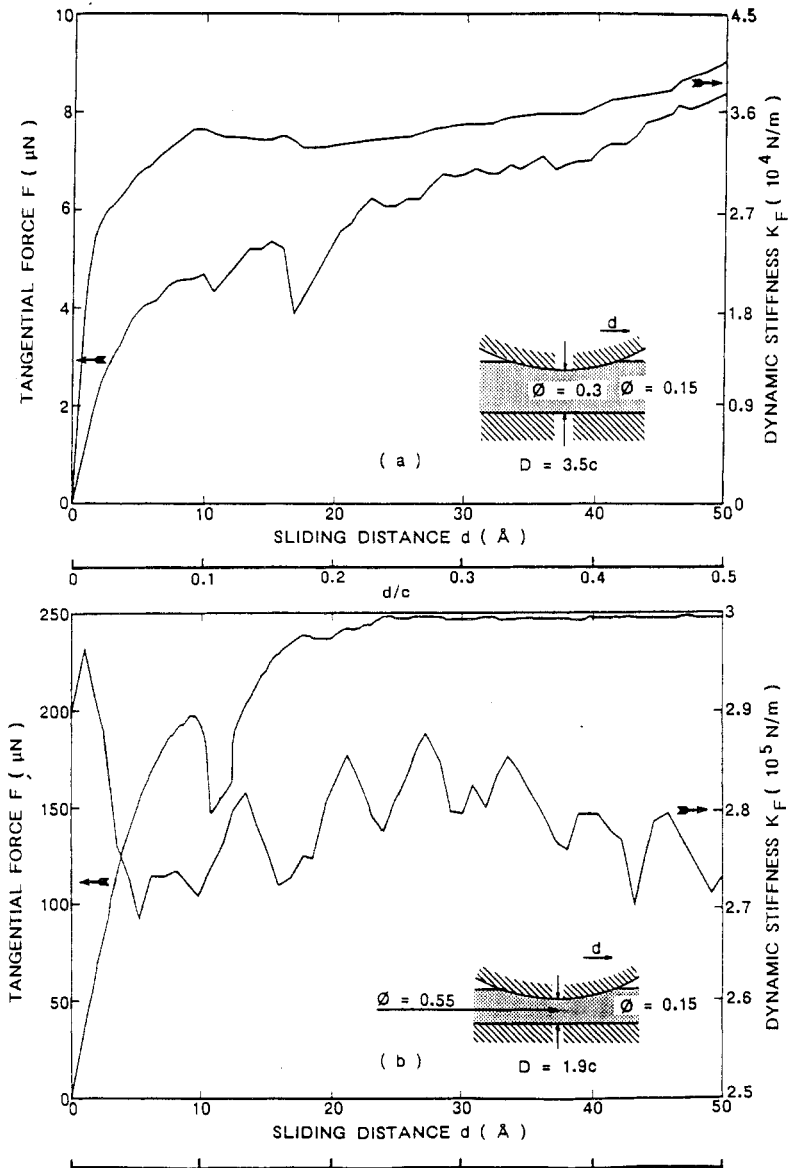


Figure 3. (a) Under-consolidated regime $D \approx 3.5c$ (c is the diameter of the colloid). The volume fraction of colloids Φ in the contact area varies between $0.15 < \Phi < 0.3$. Tangential force T and dynamic stiffness K_T increase as a function of relative sphere–plane displacement. (b) Over-consolidated regime $D \approx 1.9c$. The volume fraction at the centre of the contact is $\Phi \approx 0.55$ (dense solid). The tangential force T after a short sliding distance ($\approx 0.3c$) reaches a limiting value. The dynamic stiffness is unstable.

metallic surfaces are in electric contact. During the indentation of the layer the sphere–plane interface is very narrow and the colloids which are beginning to flocculate cannot escape from the interface, whereas the solvent can. Therefore, the colloid volume

fraction increases during the indentation [5]. Thus, the squeeze consolidates the adsorbed layer by increasing the volume fraction. Figure 3 shows how the frictional force F and the dynamic stiffness K_F vary as a function of the sliding distance d for two separation distances $D = 1.9c$ and $D = 3.5c$. According to the results obtained using the normal approach [10], the volume fractions at the centre of the contact are $\Phi = 0.3$ and $\Phi = 0.55$ for $D = 3.5c$ and $D = 1.9c$, respectively. The speed is almost constant, $\dot{d} = 10 \text{ \AA s}^{-1}$, because the amplitude Δd of the perturbation is negligible ($\Delta d = 10^{-2} \text{ \AA}$).

For the case $D = 3.5c$, F and K_F increase during the displacement d . If after sliding we halt and then temporarily reverse the sliding direction, F decreases rapidly to a negligible value and thus K_F , measured with a small displacement amplitude d , stays constant.

During the sliding, the consolidation of the film in the convergent area leads to an increase in the mechanical properties such as the yield shear stress τ_1 , and the elastic modulus G_1 . The ratio $\tau_1/G_1 \approx T/K_F \bar{D}$ ($\bar{D} = (D + D_0)/2$) has a low value: 5×10^{-3} , which corresponds to a very ductile solid with very mobile defects [11]. The number of particles in the inlet zone is about 3×10^7 , the mean film thickness $\bar{D} \approx 5c = 500 \text{ \AA}$, and so the energy dissipated in flow for a relative displacement $d \approx c$ is $Fd = 10^{-5} \times 10^{-8} = 10^{-13} \text{ J}$. If all the particles participate in the flow, the energy per particle is $3 \times 10^{-21} \text{ J} \approx 1 \text{ kT}$, which corresponds to the Brownian energy. By way of contrast, in the case of $D = 1.9c$, F increases almost instantaneously to a limiting value $F = 2.5 \times 10^{-4} \text{ N}$, for $d \approx 0.3c$. The presence of instabilities on the K_F record suggests, because the value of K_F ($\approx 3 \times 10^5 \text{ N m}^{-1}$) is lower than that of the equipment stiffness ($3.6 \times 10^6 \text{ N m}^{-1}$), that the film is overconsolidated and brittle. The friction coefficient value is near one, and $\tau_1/G_1 = 0.02$. The energy dissipated by one particle corresponds to a value of 10 kT . The particles do not flow, and the shear plane is situated at the layer–steel interface.

In conclusion, we have clearly shown that the consolidation of an adsorbed layer of colloids on a solid surface increases the shear resistance and in the extreme leads to sliding instabilities.

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