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Measuring interparticle adhesion in cohesive granular media

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Abstract. Understanding the flow of cohesive granular materials plays a crucial role in many industrial processes as well as in large-scale geophysical flows. One of the biggest experimental challenges is controlling and characterising interactions at the particle scale and linking them to macroscopic behaviour. Recently, there has been a growing attempt to synthesise model materials with controlled interparticle adhesive strength. Here, we discuss an experimental technique to precisely measure interparticle adhesion of these model materials based on the deflection of a cantilever, similar to the principle used in Atomic Force Microscopy, but adapted for larger particles with sizes ranging from tens of microns to millimeters.

1 Introduction

The rheology of dry granular materials interacting only by friction and collision has undergone significant progress in the past two decades [1]. These progresses provide the possibility for studying more complex materials, such as cohesive granular materials, where adhesive interactions play a role. These materials are critical in industries handling powders and in large-scale geophysical flows, such as snow avalanches and debris flows. Understanding their behavior is essential for preventing industrial issues (e.g., powder flowability, caking) and mitigating natural hazards.

One of the biggest challenges in understanding the rheology of cohesive materials is how to link interparticle interactions to their collective dynamics. Experimentally, controlling interparticle interactions is extremely challenging and, as a result, the study of cohesive granular flows has been mainly dominated by numerical studies using discrete-element-method simulations [2–5]. Recently, a model cohesive material has been proposed in which silica particles are coated with OH-terminated PDMS and, which enables controlling interparticle adhesion through the thickness of the polymer coating [6]. In order to investigate the role of interparticle adhesion in cohesive flows, one needs to characterise the adhesive strength for different particle sizes. However, for particle sizes less than 1 mm, simple methods based on force sensors are challenging to use because interparticle forces are small. Techniques based on Atomic Force Microscopy (AFM) can measure tiny forces but are difficult to apply to particles larger than a few tens of microns. Here, we present an experimental technique inspired by AFM, based on the deflection of a cantilever, but adapted to measure the adhesive force between two coated particles ranging from a few tens of microns to millimeters in size. The aim is to connect interparticle adhesion to the bulk dynamics of cohesive grains across a wide range of particle sizes, which may shed light on the appropriate dimensionless quantities controlling the rheology of cohesive granular materials.

2 Experimental setup and protocol

The experimental setup is shown in Fig.1(a). The idea is to detach two particles in contact by pulling on one of them, which is glued to a glass fiber acting as a cantilever. The deflection of the cantilever at the moment of detachment provides a measure of the adhesive force. The cantilever fiber, with one particle glued at its end, is fixed, while a Plexiglass plate holding a monolayer of glued beads is positioned below the cantilever on a 3D micro-piezo positioner (Fig.1a). The motion of this plate is controlled by a joystick and computer. Two DMK Imaging Source cameras with Z16-LEICA lenses provide high-resolution imaging from the side and from the top of the zone of interest (Fig.1b). The horizontal camera tracks the deformation of the fiber, while the vertical camera is used to align the beads when creating contact. A background light enhances contrast for precise fiber tracking. The entire setup is mounted on an anti-vibration table to minimize mechanical disturbances. In practice, the experiment operates like an AFM, measuring the detachment forces by tracking the cantilever position.

Each experiment begins with preparing a new fiber. Glue-spray is applied near the tip of a rectangular glass fiber to attach beads, after which the fiber is fixed to the rail. This spray replicates the properties of glue tape, and allow us to stick and remove grains with a brush. Thus, we can use the same fiber for different contact measurements, and change the fiber only once the whole experiment is finished. All fibers used have length $L = 100$ mm, thickness $e = 0.14$ mm, and width $h = 1$ mm. These dimensions determine the fiber stiffness k, calculated using

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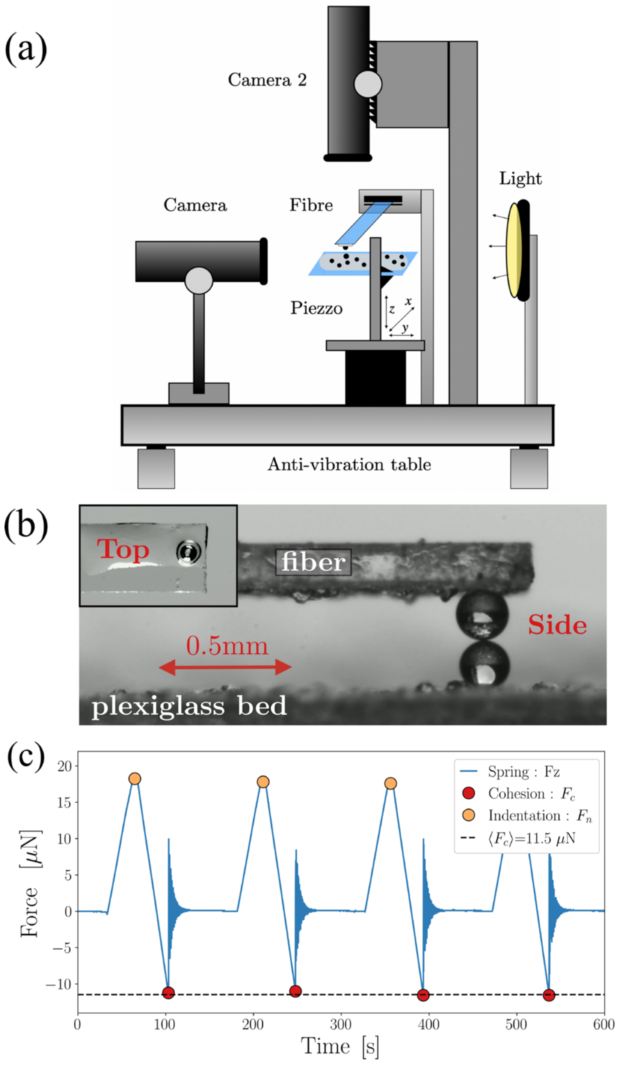

Figure 1. (a) Scheme of the experimental setup used to measure the cohesion between grains. (b) Typical image of the experiment : two beads are put in contact using a glass fiber and a plate of plexiglass. Both beads are glued to their support. (c) Example of a force curve obtained by tracking the displacement of the top of the fiber through time.

Euler–Bernoulli beam theory and given by:

$$k = \frac{Ehe^3}{3L^3}, \tag{1}$$

where $E = 49GPa$ is the Young's modulus of the glass. The average stiffness of our fibers was measured using a translation stage by pressing the tip of the fibres on a high precision scale, to measure the normal force. We found a value around $k = 0.096N/m$, which agrees with the theoretical prediction within 10%.

Once the fiber is ready, a single coated bead is attached to its tip and aligned with another identical bead glued to the bottom Plexiglass bed, as shown in Fig.1(b). When the beads are positioned one directly above the other, the micro-piezo drive lifts the Plexiglass plate to press the two beads together. This action deflects the fiber, generating a restoring force $F_n = k\Delta z$, where Δz, is the fiber deflection (yellow dots in Fig.1c). The plate is then moved down (blue curve in Fig.1c) until the two beads detach. Detachment occurs when the elastic force exerted by the cantilever, F_n, exceeds the adhesive force F_c (red dots in Fig.1c). Then, the fiber oscillates and returns to an equilibrium position. The maximum vertical displacement Δz_{max} at the moment of detachment provides the magnitude of the adhesive force $F_c = k\Delta z_{max}$.

The process is repeated four times per pair of particles (four peaks in Fig 1c) in order to obtain the average adhesive force F_c for a specific contact. To obtain the distribution of adhesive forces, we repeated the process for 50 different pairs of particles, representing a total of 200 measurements of F_c for each coating thickness b and grain size d.

3 Results

Hereafter, we present the experimental results for the adhesion force for different coating thicknesses $b = [0, 15, 34, 65, 100, 200]$ nm and grain diameters $d = [196, 350]$ μm. For each value of b, the data is fitted with a Gaussian distribution to extract the mean adhesion force $\langle F_c(b) \rangle$ and its standard deviation $\sigma(b)$, which represents a measure of the heterogeneities in F_c for each value of b. As shown in Fig.2(a), when b increases, the distribution shifts to higher values of F_c? and tends to narrow, indicating that the force distribution is more homogeneous for thicker coatings (Fig.1b).

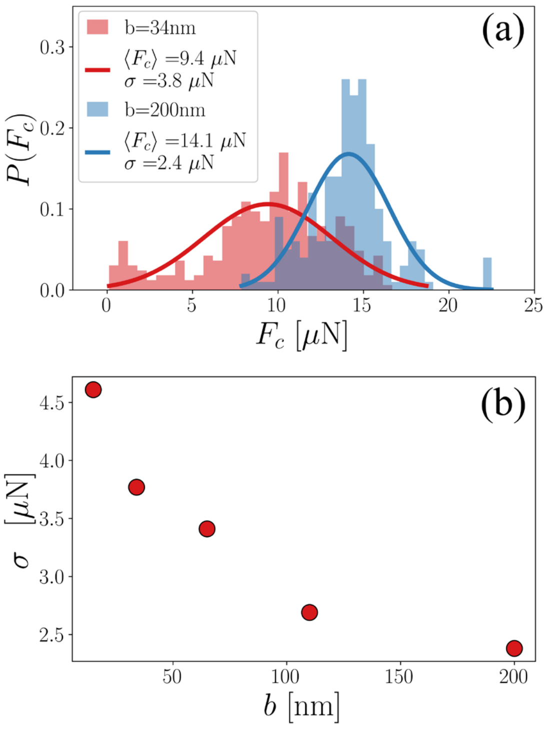

Figure 2. (a) Distribution of adhesion forces for two different coating, on the same type of beads. We can see a clear shift in the average force when the coating thickness increase. (b) Variance of the force measurements compared to the coating b. The higher the coating, and the sharpest the distributions get.

Now, we proceed to investigate the mean adhesion force $\langle F_c(b)\rangle$ as a function of the coating thickness. The average force initially increases with b and then saturates, which is well described by the exponential function:

$$F_c = \frac{3}{2}\pi\gamma d\left[1-\exp\left(-\frac{b}{B}\right)\right], \tag{2}$$

where γ has units of surface tension, which value is close to the surface tension of the PDMS coating, and B is a characteristic length scale of the order of the particle's roughness. This behaviour is similar to the one observed in [6] for larger grains ($d > 800\,\mu$ m). We find that $\gamma = 16$ mN m^{-1} and $B \simeq 80$ nm are the best fits for our data. The value of γ is similar to the surface tension of PDMS and to the one found by [6]. We believe the difference in the value of B found by [6] stems from the roughness of the particles, which is much smaller for our beads than the ones studied in [6]. The idea is that as the coating thickness increases, the PDMS gradually fills the surface asperities, increasing the average adhesive force. When all the asperities are covered all the contacts are made between polymer-polymer, such that increasing further the coating thickness does not increase the average adhesion force. In Fig. 3(b) we plot the adhesion force rescaled by the surface tension and diameter of the grains, and we obtain a good collapse between the different diameters tested.

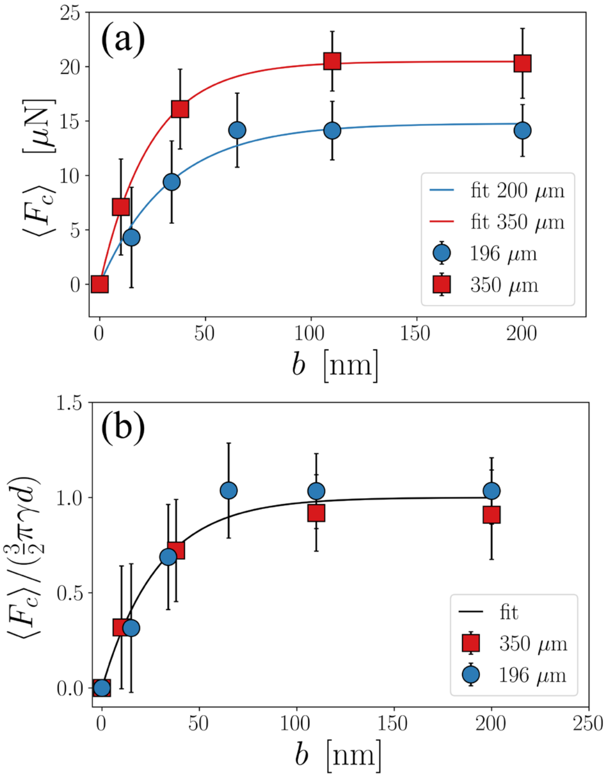

Figure 3. (a) Adhesion force rising with the thickness of the coating b, for two size of beads : $d_1 = 196\mu m$ and $d_2 = 350\mu m$. (b) Normalised adhesion force. The force reaches a maximum around a critical thickness $b_c \simeq 80nm$.

4 Conclusion

In conclusion, we present an experimental setup to study the statistics of interparticle adhesive forces between particles in cohesive-controlled granular materials [6]. Our method for measuring the force required to detach two adhesive particles is based on the deflection of a cantilever, as in atomic force microscopy. However, by tuning the stiffness of the fiber, we have adapted the technique to measure interparticle adhesion for particle sizes that are too large for classic AFM but too small for other simpler techniques, such as those proposed in [6]. Our experimental results will serve in the future to link interparticle adhesion to the bulk rheological properties of cohesive granular flows. Another perspective is to slightly modify the setup to measure the adhesion energy, not just the adhesive force, as this quantity may play a crucial role in the flow behavior of cohesive granular materials.

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