Understanding and tuning sliding friction

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This chapter gives a brief description of the experimental and computational techniques that are used for the research presented in Chapters 3 to 6. We discuss the two custom-made tribometers used for the horizontal sliding tests (performed in Chapters 3, 4 and 6) and circular sliding tests (performed in Chapter 5). Before (and after) sliding, the hardness and the microscopic surface topography of the sliding surfaces are quantified; We briefly discuss the methods that were used to do so here. In addition, we introduce the computational techniques that were used to quantify the contact mechanics of a (spherical) slider on a flat surface (used in Chapter 5).
2. Experimental and computational techniques

2.1 Sliding friction

The sliding experiments are performed with two custom-made tribometers: (a) horizontal (linear) sliding, based on a commercially available tensile tester, and (b) circular sliding where a commercially available rheometer is converted to a tribometer.

2.1.1 Horizontal sliding

A typical sliding experiment is performed by sliding an object with a certain load on top of a horizontal flat surface, see Figure 2.1 for a schematic illustration of the experimental setup. This custom-made tribometer is based on a commercially available tensile tester, which consists of a load cell coupled to a stepper motor. We have used the ZwickRoell Z2.5 tensile tester with an HBM Z6FD1 load cell (precision of 5 mN, maximum load of 100 N, sampling rate of 50 Hz), initially built to uniaxially stretch or compress materials to characterise their mechanical properties. The tensile tester is placed on its side and pulls with a coupled rod or stiff cord on the top surface with a preset speed in the range of $1 \mu m/s$ up to 13 mm/s. To pull horizontally, the

![Figure 2.1: Schematic illustration of the experimental setup for horizontal sliding tests. A slider, for example a sledge, is pulled horizontally over a surface with the use of a stepper motor. The load cell allows us to monitor the pulling force $F$ and sliding distance $d$ for an imposed sliding speed $v$ and normal force $N$. The latter is varied with placing dead weights on top of the slider.](image-url)
2.1 Sliding friction

Figure 2.2: Pulling force $F$ as a function of sliding distance $d$ for a sledge sliding on a flat surface. The slider, including certain load placed on top of it, is pulled horizontally with a sliding speed of 1 mm/s over the fixed bottom plate. Both solids are 3D printed plastics with the commercially available resin named Clear (see Section 6.2 for more details). After approximately 5 mm, a stable pulling force is found which represents the dynamic friction force. In black, red, and blue circles the measured pulling force is given for a normal force of, respectively, 0.58 N, 1.07 N and 2.05 N. Inset: The measured friction force $F$ as a function of the imposed normal force $N$. The dashed line represents the fit $F = \mu N$ with $\mu = 0.147 \pm 0.006$.

In Figure 2.2 the pulling force $F$ as a function of sliding distance $d$ is given for a typical horizontal sliding experiment. Here, a plastic-on-plastic (3D printed with the commercial available resin named Clear, see Section 6.2 for more details) sliding experiment is performed at various imposed normal forces $N$. After approximately 5 mm, a stable pulling force is reached corresponding to the dynamic friction force. From these experiments, the friction force as a function of the normal force can be plotted as given in the inset of Figure 2.2. Consequently, the friction coefficient $\mu$, the ratio of the friction force and normal force, can be calculated: $\mu = 0.147 \pm 0.006$. 
2.1.2 Circular sliding

The circular sliding experiments are performed by a commercially available rheometer (Anton Paar, Dynamic Shear Rheometer 502). This rheometer imposes a rotational torque to the measuring system, which is a cylinder with a bottom plate or (truncated) cone that is perfectly aligned with the rotation axis. The rheometer measures the angular displacement and normal force (in the range of 1 mN up to 50 N with a resolution of 0.5 mN) and, with the use of a quick feedback loop, a constant sliding velocity can be imposed. Rheometers are built to perform rheology tests on (complex) fluids. The rheometer can apply, and measure torques from 1 nNm up to 230 mNm and, therefore, are very suitable to use as a custom-made tribometer.

To perform the circular sliding experiments, a slider is clamped at the bottom of the measuring system at a well-defined distance (on the order of 2 \(-\) 5 mm) from the rotation axis and pressed against the countersurface; see Figure 2.3. The imposed rotational speed and measured torque can be converted into a sliding velocity \(v\) and friction force \(F\) where the former can be varied from \(10^{-6}\) up to \(10^{-1}\) m/s. During sliding, the normal force (mN precision) is measured and can be controlled by vertically displacing the rheometer (with sub-\(\mu\)m precision) while holding the

![Figure 2.3: Schematic illustration of the setup to perform circular sliding experiments. A slider, here a sphere, is clamped off-axis at the bottom of the measuring system of the rheometer where, for an imposed rotation speed, the torque \(\tau\) and normal force \(N\) are monitored. The normal force can be adjusted manually by vertically displacing the rheometer (with sub-\(\mu\)m precision). In Chapter 5, the setup is used for ice friction measurements where the setup is thermally isolated and cooled from below.](image-url)
2.1 Sliding friction

![Graph showing friction and normal force as a function of time and normal force.]

**Figure 2.4:** (a) Friction (red) and normal (black) force as a function of the time $t$ when sliding a 2 mm stainless steel sphere over a lead surface. The sphere is rotated with a controlled sliding speed of 0.4 mm/s and is in contact with the lead surface for about 20 seconds due to misalignment between the rotation plane of the sphere and the lead surface. (b) Friction force as a function of the normal force where the black circles are the measured data as presented in (a). The red line is a linear fit of the data to calculate the friction coefficient, here $\mu = 0.248 \pm 0.001$. The red circles represent an average of the measured data for steps of $dN = 0.5$ N in normal force.

For the sliding experiments on ice, as performed in Chapter 5, the setup is thermally isolated and cooled with a coolant liquid (for a temperature between $-15$ °C and $0$ °C) or liquid nitrogen (for a temperature between $-110$ °C and $-15$ °C). The temperature of the ice is measured with an embedded thermocouple close to the surface and controlled with the flow rate of cooling liquid.
2.2 Penetration hardness

The hardness of a material is the resistance to (local) plastic deformation as a result of normal (indentation) or tangential (abrasion) stress. Often the term hardness refers to the yielding compressive pressure of a solid; after (initial) elastic deformation due to the exerted stress, some solid materials deform irreversibly. Note that depending on the material, crack formation in a material and, subsequently, full fracture can already occur directly after elastic deformation. The onset of plastic deformation, including a quantification protocol, is complicated to define as it depends on the specific conditions: the material of the test-sample (viscoelastic behaviour, strain-hardening, purity, heterogeneity), the stress-inducer (geometry, thermal conductivity) and the external conditions (strain rate, geometry-size) [54, 55].

For example, the hardness of a metal largely varies with the characteristic length scale on which it is measured [56]. On the nanoscale, the hardness is determined by the number of intermolecular bonds of the atoms arranged in a crystal lattice. On larger scales, the influence of polycrystallinity is amplified where the hardness is mainly set by the crystallographic defects [57]. A metal consists of crystalline grains in which point and line defects (mainly on the boundary of the grains) can result in movement and formation of dislocations that, consequently, can result in early plastic deformation and creep [58].

The quantification of hardness is therefore strongly dependent on the measurement protocol and should be chosen carefully to match the specific conditions of the application. For example, to test the abrasive hardness of minerals the Mohs scale for hardness can be applied: The hardness is categorised by investigating on which reference minerals — from soft talc up to hard diamond — the test sample leaves a scratch [59]. More quantitative test protocols, mainly in metallurgy, have been developed and performed to define hardness. Commonly used protocols (and corresponding scales) are based on Brinell, Vickers, Shore and Rockwell [54, 60, 61]. In these tests, the specific indenter geometry (pyramidal, spheres, cones), indenter size, strain rate and the loading time are defined.

In Chapters 3 to 5, we quantify the hardness for conditions which resemble the parameter ranges exercised in the friction experiments: normal force, length scale, indentation depth, normal- and tangential speed. The hardness can be evaluated from an indentation experiment where a spherical or conical indenter is pressed on the flat test-sample; for increasing loading force $N$ the resulting plastic deformation depth $\delta$ is monitored. Here, the penetration hardness can be calculated based on the
2.2 Penetration hardness

Projected contact area $A_c$ of the indenter on the test-sample and the loading force:

$$P_h = \frac{N}{A_c}. \quad (2.1)$$

See Figure 2.5 for a schematic illustration of the indentation experiment based on a spherical (radius $R$) or conical (apex angle $\alpha$) indenter. The indentation experiments are performed with a tensile tester (see also Section 2.1.1) which pushes the indenter vertically into the sample. The monitored indentation depth $\delta$ can, for the given indenter-geometry, be converted to the contact area $A_c$. For a large range of indentation speeds (1 $\mu$m/s up to 13 mm/s) and indentation forces (maximum of 100 N with a precision of 5 mN), the loading and unloading can be monitored.

When performing an indentation experiment, the stiffness of the measurement system also has to be taken into account. The measurement system, i.e., the tensile tester including the indenter, elastically deforms for increasing load. To correct the measured indentation depth $\delta$ for this elastic behaviour of the system, a reference loading experiment specific for the indentation setup is performed without the sample. The indenter is loaded on the sample stage where normally the test sample is placed, see Figure 2.6 for a typical loading-unloading curve. Here, up to 30 N the loading

![Schematic illustration of the indentation experiment](image.png)

**Figure 2.5:** Schematic illustration of the indentation experiment performed with a spherical (a) or conical (b) indenter on a flat sample. For an increasing loading force $N$ the indentation depth $\delta$ is monitored. The sphere (radius $R$) and cone (apex angle $\alpha$ and maximum radius $R$) indent the flat surface resulting in a projected contact area $A_c$ with radius $r$. 
Figure 2.6: The indentation depth $\delta$ as a function of the loading force $N$ to quantify the stiffness of the measurement system used for hardness tests. In this reference loading-unloading experiment, the indenter is loaded up to 30 N onto the sample stage, i.e., where normally the test-sample is placed. No significant hysteresis can be observed and, consequently, the compliance of the measurement system can be quantified with a linear fit, see the dashed line. In this example, the compliance is $3.676 \pm 0.002 \, \mu m/N$. Subsequently, when a hardness test is performed, the monitored indentation depth is corrected with the use of the measured compliance of the setup.

and unloading is monitored which, as no significant hysteresis is observed, is purely elastic. The measured slope of this reference experiment (red dashed line in Fig. 2.6) reveals the compliance (which is the inverse of the stiffness). In this example, the compliance of the measurement system is $3.676 \pm 0.002 \, \mu m/N$. When an indentation experiment is performed, we correct the monitored indentation depth with the use of the measured compliance of the specific setup.

In Figure 2.7, two typical indentation experiments are given for (a) a sphere indenting a lead surface and (b) a cone indenting a water-sand mixture (water volume fraction of $\phi_w = 5\%$). Both indenters are made from stainless steel which is significantly harder than the samples which are going to be tested. The measured loading-unloading curve for both systems shows a large hysteresis; when the indenter starts
to retract after loading, the measured force immediately drops to zero. This confirms that during loading, the test-sample has been plastically deformed.

Throughout the loading, the projected contact area $A_c$ increases for the spherical and conical indenter. The contact area $A_c = \pi r^2$, with $r$ the radius of the projected circle, can be calculated based on the measured indentation depth $\delta$. For a sphere-on-flat geometry [see Fig. 2.5(a)], we can write

$$r^2 = 2R\delta - \delta^2 = 2R\delta \,.$$  \hspace{1cm} (2.2)

The approximation is valid for $\delta \ll R$. For the conical indenter, the contact radius can be written as

$$r = \tan\left(\frac{1}{2}\alpha\right)\delta \,.$$  \hspace{1cm} (2.3)

Subsequently, the contact area for the specific geometries can be written in terms of

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure2.7.png}
\caption{The indentation depth $\delta$ as a function of the loading force $N$ for (a) a spherical indenter (radius $R = 2$ mm) pressed onto lead and (b) a conical indenter (apex angle $\alpha = 60^\circ$) pressed onto a water-sand mixture (water fraction of $\phi_w = 5\%$). The loading-unloading curves show full plastic indentation; when the indenter retracts after loading, the force drops immediately. The red dashed lines show fits based on Eqs. (2.6) and (2.7) that enables us to calculate the penetration hardness: Lead has a penetration hardness of $P_h = 18.9 \pm 0.1$ MPa and the water-sand mixture has a penetration hardness of $P_h = 63.4 \pm 0.3$ kPa.}
\end{figure}
the indentation depth as

\[
\text{Sphere: } A_c = 2\pi R\delta \\
\text{Cone: } A_c = \pi \tan^2\left(\frac{1}{2} \alpha\right)\delta^2.
\]

(2.4)

(2.5)

The penetration hardness can be calculated by fitting the following expressions on the measured loading-curve (red dashed lines in Fig. 2.7):

\[
\text{Sphere: } \delta = \frac{1}{2\pi R \rho_h} N \\
\text{Cone: } \delta = \frac{1}{\sqrt{\pi \tan^2\left(\frac{1}{2} \alpha\right) \rho_h}} \sqrt{N}.
\]

(2.6)

(2.7)

Therefore, the indentation depth increases either linearly or as a square root of the loading force, set by the geometry of the indenter. When the data as presented in Figure 2.7 are fitted, we find a penetration hardness of \( \rho_h = 18.9 \pm 0.1 \text{ MPa} \) for lead and \( \rho_h = 63.4 \pm 0.3 \text{ kPa} \) for the water-sand mixture.

The penetration hardness for a sphere-on-flat geometry [Fig. 2.5(a)] is calculated based on the approximation for the contact radius valid for \( \delta \ll R \) as given in Equation (2.2). For the data presented in Figure 2.7(a), this approximation results in an underestimation of the penetration hardness of 2.6%; precise calculation based on Equation (2.2) results in a penetration hardness of \( \rho_h = 19.4 \pm 0.1 \text{ MPa} \). The sphere-on-flat hardness test performed in Chapter 5 results in a relatively large hardness (\( \rho_h \sim 100 \text{ MPa} \)) and, therefore, has a small indentation depth \( \delta \) compared with the hardness-tests on a lead surface. The approximation \( \delta \ll R \) does therefore not result in a severe error.

Another sphere-on-flat hardness test which is often used is the Brinell hardness method. In contrast to the penetration hardness test with a spherical indenter, where the related area is the projected contact area, the Brinell hardness is calculated based on the curved contact area:

\[
A_c = 2\pi R\delta \\
= 2\pi R \left( R - \sqrt{R^2 - r^2} \right).
\]

(2.8)

Therefore, the Brinell pressure considers the radial stress in the test sample exerted by the spherical indenter. For the data presented in Figure 2.7(a), the Brinell hardness results in \( 18.82 \pm 0.06 \text{ MPa} \), which is 2.9% less than the penetration hardness.
Somewhat surprising, the approximation made in Equation (2.2) makes that the contact areas as computed in the Brinell and penetration hardness calculations are now equivalent. In conclusion, the sphere-on-flat indentation experiments enable us to calculate the hardness where, for the regime $\delta \ll R$, the Brinell and penetration hardness method are equivalent.

2.3 Surface characterisation

One of the major parameters that determines friction is the microscopic surface topography of the sliding surfaces. Prior to a sliding test, we quantify the topography of our surfaces with a 3D laser-scanning profilometer (Keyence, VK-X1000). This contactless profilometer scans with an ultraviolet laser (wavelength of 404 nm) and measures the intensity of the reflection for every voxel (volumetric pixel). This

![Figure 2.8: Quantification of the microscopic surface topography of a 2 mm radius stainless steel sphere based on 3D laser-scanning profilometry. (a) The optical image of the sphere for an area of 208 by 208 $\mu$m. (b-c) The height variation before and after subtraction of the macroscopic curvature of the sphere. (d) 3D presentation of the topography including the macroscopic curvature of the sphere. (e-f) The height $Z$ as a function of the lateral direction before and after subtraction of the curvature. The red dashed line in (f) represents the root-mean-square height variation $S_q$.](image)


intensity as a function of the axial direction has a peak where the surface is located. Therefore, by accurate peak detection of the reflected light, the height $Z$ can be quantified as a function of the lateral directions $X$ and $Y$.

The surfaces are scanned over an area of 208 $\mu$m by 208 $\mu$m with a lateral resolution of 138 nm/pixel and 20 nm resolution in the height direction. In Figure 2.8 a typical measured surface topography is plotted where a stainless steel sphere (radius of 2 mm) has been used. In addition to the height variation [Fig. 2.8(b)], a bright field image can be made, as shown in Figure 2.8(a). To quantify the microscopic surface height profile, the macroscopic curvature (if present) is subtracted from the measured height; see Figure 2.8(c). In Figure 2.8(e) and (f) the line profiles that, respectively, include or exclude the surface curvature are plotted. The microscopic surface roughness can be quantified with the root-mean-square (rms) surface height variation $S_q$. For the steel sphere, the calculated surface roughness after curvature subtraction is $S_q = 112$ nm which is represented in Figure 2.8(f) with the red dashed lines.

## 2.4 Contact mechanics calculations

The resistance against sliding of two surfaces over each other can be understood as the resistance to shear the loaded microscopic contact points over each other. This can be quantified based on the stresses on the asperity contact area: the shear stress $\sigma_s$ (tangential stress) and the local contact pressure $P_c$ (normal stress). To understand the loading of the two surfaces in contact, we perform contact mechanics calculations. These calculations are performed in Chapter 5 for a sphere-on-flat (skate on ice) geometry to quantify the real contact area and local contact pressure based on the mechanical properties and measured surface topography. We use a tribology simulator (named TRIS, publicly available at Tribonet [62]) to solve the elastoplastic contact equations through a numerical boundary element model. Based on the mechanical properties and the surface topographies of the solid surfaces, the interfacial gap at each of the in-plane coordinates defined by the topography is calculated for a given normal force. Those locations at which the interfacial gap is zero form the area of real contact where, in addition, the local contact pressure is quantified. This calculation is performed by numerically solving the (elastic-fully plastic) half space model [63–66], which is explained (and experimentally validated) in more detail in References [62, 66, 67].

In Figure 2.9 a typical result of the numerical model is presented. For a sphere-
2.4 Contact mechanics calculations

Figure 2.9: The surface topography and contact area when a smooth ($S_q = 0$ nm), intermediate rough ($S_q = 112$ nm) and very rough ($S_q = 446$ nm) sphere (2 mm radius) are pressed on a smooth flat surface. The contact area (middle row) for the given surface topography (top row) is quantified with the Tribology Simulator [62]. In addition, the lowest row shows the surface topography height $Z$ before contact (black line) and the gap size in-contact (red line) as a function of the width. The calculations are performed with an effective elasticity of $E^* = 0.84$ GPa and a hardness of the sphere and the smooth flat surface of, respectively, 100 MPa and 420 MPa.

For the on-flat contact system, the interfacial gap $\delta$ is calculated for a 2 mm radius sphere with various surface topographies: A perfectly smooth ($S_q = 0$ nm), intermediate rough ($S_q = 112$ nm) and very rough ($S_q = 446$ nm) microscopic surface topography. The intermediate rough surface topography is the measured topography for the steel sphere as presented in Figure 2.8. The perfectly smooth and very rough surface topographies are based on artificially reducing/enhancing the measured stainless steel height variation $Z(x, y)$ with, respectively, a factor of 0 and 4.

When the spheres are pressed against a smooth flat surface, the real contact area can be quantified as shown in the middle row of Figure 2.9 for a load of 200 mN.
A circular contact area, with contact radius $r = 71 \, \mu m$, is found for the perfectly smooth sphere. In contrast, a very rough sphere results in contact spots based on the asperities of the rough surface topography. The real contact area can then be quantified for increasing load $N$ (see Fig. 2.10) which largely depends on the surface topography.

The calculations for the sphere-on-flat contact mechanics illustrate the influence of the microscopic surface topography and the loading force (often the gravitational force). Several theoretical frameworks are developed, which combine elasticity, plasticity, and adhesion, to capture the contact mechanics. Two models, fully elastic Hertzian contact and fully plastic contact, capture the, respectively, maximum and minimum of the real contact area which can be reached. We will briefly discuss the aforementioned models and compare them to the calculated contact area of the Tribology Simulator for increasing normal load; see Figure 2.10 where the Hertzian

![Figure 2.10](image)

**Figure 2.10:** Real contact area RCA as a function of the load $N$ for a sphere (radius of 2 mm) pressed on a flat surface. The yellow, orange, and red circles represent the numerically calculated contact area for spheres with a surface roughness of, respectively, 0 nm, 112 nm and 446 nm. The continuous and dashed lines represent, respectively, the elastic Hertzian contact area [Eq. (2.10)] and the contact area controlled by pure plasticity [Eq. (2.11) with a hardness of 100 MPa].
model and plastic contact model are represented as, respectively, the red and yellow lines.

**Hertzian contact**

As mentioned in Chapter 1, Hertz developed a theory in the late 19th century to describe the contact formation between smooth and non-adhering elastic bodies [21, 22]. Based upon the classical theory of elasticity and continuum mechanics he describes the elastic, thus reversible, deformation of the solids based on their elastic properties, which enables us to calculate the contact area $RCA$ and contact pressure $P_c$. The Hertzian model for a sphere on a flat surface gives the contact radius $r$ as [21, 22]:

$$r^3 = \frac{3RN}{4E^*}, \quad (2.9)$$

where $R$ is the radius of the sphere and $N$ the imposed loading force. The effective elasticity of the system is given as $\frac{1}{E^*} = \frac{1-\nu_1^2}{E_1} + \frac{1-\nu_2^2}{E_2}$ which is based on the Young’s moduli $E_1$ and $E_2$ and the Poisson’s ratios $\nu_1$ and $\nu_2$ of the two materials. The real contact area for a sphere pressed on a flat surface is

$$RCA = \pi r^2 = \pi \left(\frac{3RN}{4E^*}\right)^{2/3}. \quad (2.10)$$

In Figure 2.10 the real contact area for the same conditions of the performed computational calculations ($E^* = 0.84 \text{ GPa}$) is given in a continuous yellow line for increasing force $N$. As the model matches the calculated RCA for a perfectly smooth spherical slider, it illustrates that the computational technique results in a purely elastic contact. Therefore, the contact area is relatively large and increases sublinearly ($RCA \propto N^{2/3}$) with the normal force.

**Plastic contact**

When two surfaces are pressed onto one another, the highest asperities of the two surface topographies will make contact and, consequently, experience a large contact pressure. As Bowden and Tabor already emphasised (see also Chapter 1), the asperities will irreversibly deform when the contact pressure exceeds the hardness of the material [19, 22]. Plastic deformation of the asperities will continue up to the contact pressure is lowered down to the hardness. This flattens out of the asperities due to plastic deformation result in a contact area described as
2. Experimental and computational techniques

\[ \text{RCA} = \frac{N}{P_h}, \quad (2.11) \]

with \( P_h \) the lowest hardness of the materials in contact. In Figure 2.10 in the red dashed line, the contact area is plotted for a penetration hardness of \( P_h = 100 \, \text{MPa} \), as used in the numerical calculated contact area. It illustrates that the computational calculation for a very rough sphere \( (S_q = 446 \, \text{nm}) \) pressed on a flat surface results in a contact area which is mainly the result of plastic indentation. Consequently, the contact area increases linearly with the normal force.