Lecture 2: Surfaces from a manufacturing perspective

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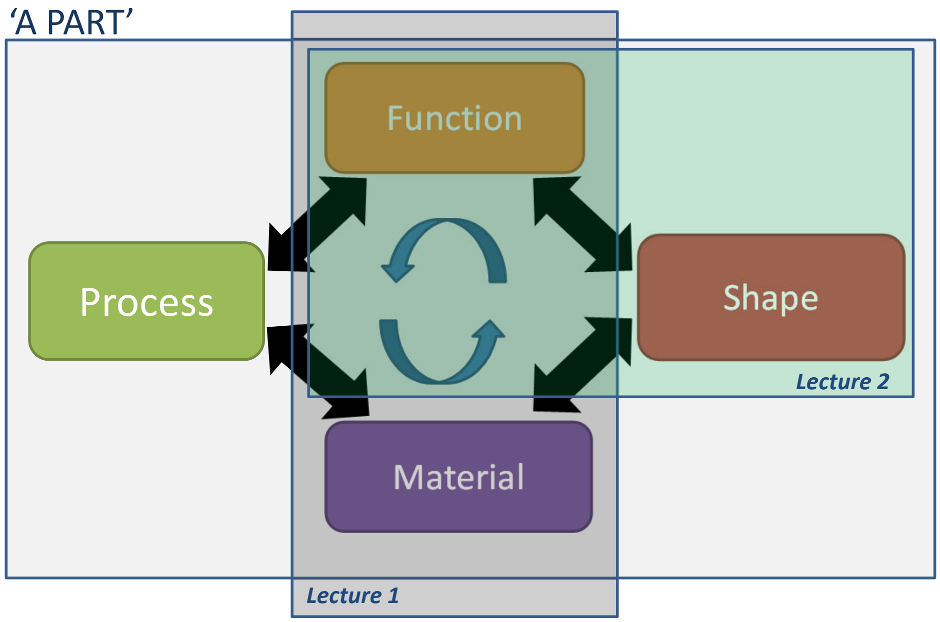
Today's learning objectives

- **Importance** of surfaces in manufacturing
- How do we **define / characterize** a surface?
  - Geometrical parameters, texture
  - Characterizations methods
  - Physical properties



In the previous lecture, we saw that in the process of designing a component, there is a close relation between the material and the designed function. In particular, we saw some methodology for optimizing the choice of a material in connection with a specific task, so that it fulfills a set of requirements.

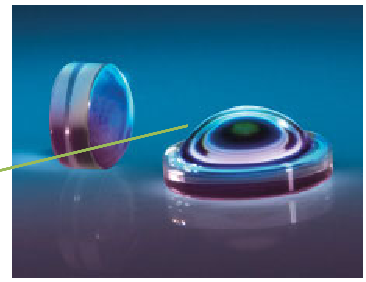
In this lecture, we focus on the shape, and specifically, on the role of surfaces from a functional point of view. The aim is to highlight the importance of surfaces in functional design.

Discussion in class (5 min)
What functions can be associated with a surface?

Surface & functions…

(bizlaunchblog.com)

- Transparent
- Scratch resistant
- Others..



- Transparent
- Specific and precise shape
- 'The shape is the function'

Surfaces can have multiple functions, such as:

- To have optical properties, such as particular reflecting or transparent properties or to convey certain visual effects.

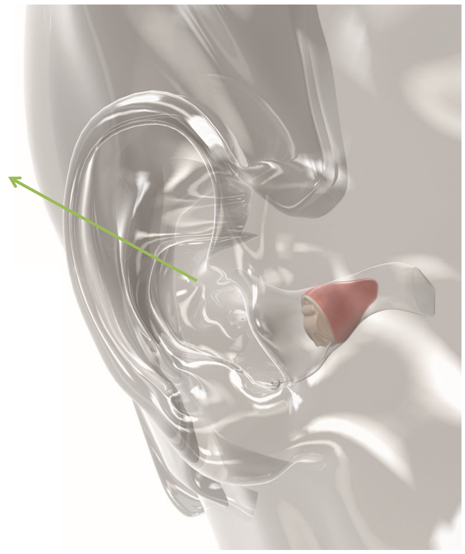
- To be 'scratch resistant' and to have specific hardness. A surface can have significantly different mechanical properties than the bulk of the material.

- To have specific 'wetting' (*in French: 'mouillage'*) properties. Often, and with progresses made in manufacturing, surfaces are often 'engineered and modified' to obtain given wettability properties, like being hydrophobic (i.e. 'dislike water') or the opposite, hydrophilic (i.e. 'like water'). Surfaces are in this context essential for tribology.

- The surface shape itself may define a function by its curvature. This is the case in optics, as the curvature will define how optical rays are bent. In mechanics, specific shapes may be chosen for instance to distribute contact points at specific locations.

Surfaces & functions…

Soft
Skin-friendly (non allergenic)



(Phonak, Nano)

Surfaces may also have specific requirements, like being hypo-allergenic and more generally, to have specific chemical properties when it comes to interacting to a given environment or to biological tissues.

For instance, we may want to obtain a surface that would not be 'bio-friendly' (e.g., to no promote cell growth or bacteria) for hygienic purpose, or the other way around, i.e., bio-friendly, like it is the case, when the surface needs to promote cells adhesion, which is typically the case for medical implants.

The material itself may not have these intrinsic properties, and hence, often it is required to engineer the surface to stimulate some of these functions, not necessarily present in first place.

Surface and functions…

(Girard-Perregaux, Tourbillon bi-axial)

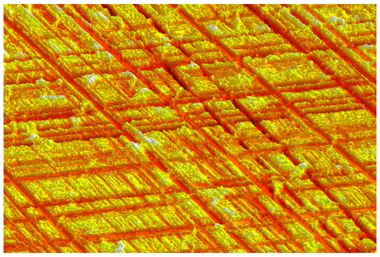
Surfaces can have a purely esthetic function ('mirror polish', textured, etc.), have some enhanced hardness or reduce friction locally.

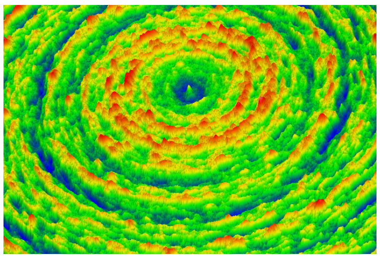
The example above, taken from a high-end watch mechanism, illustrates the variety of functions that might be sought in these types of mechanical components, composing the watch mechanism.

Some specific surface topologies may have no role in the actual mechanical function but are purely for decorative purpose.

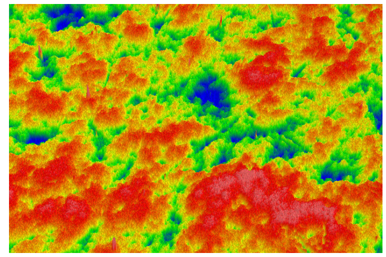
Others may play a key role in the friction behavior (tribology), and hence, be essential for the efficiency of the mechanism. This is typically the case for the stone (ruby, the crystal with a pink color on the watch mechanism) used as a bearing for the oscillating element of the tourbillon. It has to withstand very high pressure (> GPa), while having good wearing and friction properties.

Illustration of surface textures at the microscale





Textured



Somewhat random

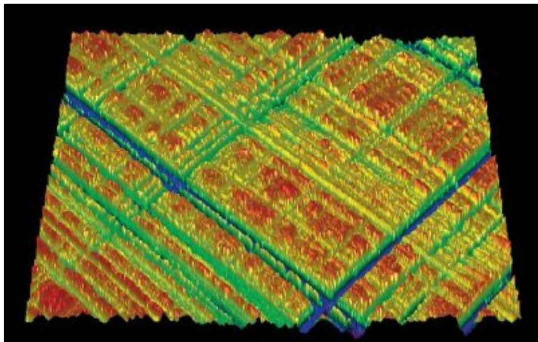
Examples of different surface finish leading to different textures (cross-hatch, turned, grounded)

Surfaces of an object can have a broad variety of textured that may reveal the manufacturing process used to produce it.

For instance, classical machining process, such as turning or milling, will leave characteristics textures on the surface, such as prints left by the tools used to carve the material.

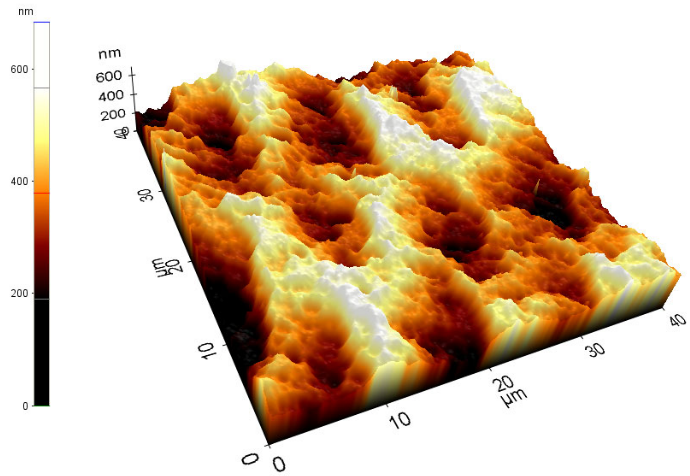
Later in the class and during the reverse engineering project, we will see that these textures provide useful hints about the process that have been used for producing the parts. In quality control, it also provides indication of the tool wear status and possible deviations from in the process operation.

An introduction on statistical methods for production and process control and monitoring will be given in this course (lecture 9).



(azom.com)

A complex 3D problem!



(Exercise with Gwyddion)

Without any consideration for the surface physical properties, let us for now start addressing the surface *topology* itself and how it can be described. The *topology* relates to the surface geometrical properties and is independent to the material itself and the particular physical properties it may have.

Describing a surface uniquely, so that it can be further reproduced or analyze, is a complex problem that requires defining the surface topology in the two directions of the plane. The topology forms a 3D volume that may or may no have symmetry elements.

During the exercise session, you will learn how to measure some of the key characteristics parameters than can be used for describing a surface quantitatively.

Surfaces: key questions

- **How to translate a functional requirement into a physical surface requirement?**
  - Ex. 'optically transparent' = typ. roughness Ra < 10 nm, why is that?
- **What are essential physical properties of a surface?**
  - Ex. hydrophobic = contact angle with water > 90 deg
- **How to characterize a surface? (Metrology)**
  - Ex. define roughness, waviness, etc.

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To engineer surfaces to fulfil a given function, we need to translate *functional* requirements (e.g., to have certain optical reflectance, a given texture for tribology, a certain curvature, etc.) into *measurable* surface characteristics that may themselves be connected to specifical physical properties. The latter is not necessary a simple exercise as it involves a model that connects a given property with an external field.

The necessity of translating *functional* requirements into *metrics* is for enabling engineering methods and models, and most important for defining tangible design objectives.

Properties of surfaces and their characterization

- **Topography characterization**
  - Roughness
  - Waviness
  - Profile

- **Physical properties**
  - Wettability (Hydrophobic, hydrophilic)
  - Mechanical Hardness
  - *Others (chemical, optical, etc.)*

Let us first examine the geometrical characteristics of surfaces, and how it can be defined. Hence, for now, we leave aside the intrinsic physical properties of the surfaces to focus only on the topology, or in other words, the geometrical definition of the surface.

Among the most common terms to define it, are the *roughness*, the *waviness* and the *profile*.

These three terms are independent to the nature of the material considered, but are purely related to quantitative geometrical considerations.

As we will see later on, these terms may not be sufficient to fully define the surface we are considering and to capture the potential complexity of a topography. However, they are first simplest metrics to consider and among the most commonly used as basic information.

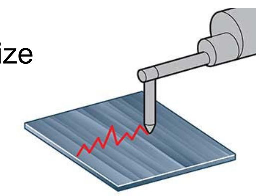
How do we measure surfaces?

Defining a surface with a given set of geometrical parameters implies that one is able to measure these parameters. This is necessary to qualify a surface and to verify that a set of given requirements have been met.

Hence, let us first examining common methods used in surface metrology.

Three main methods

- **Optical-based profilometry techniques** – large surface imaging
  - Confocal microscopy / high-depth of field, limited lateral spatial resolution (diffractionlimit)
  - Phase shift interferometry / high resolution along the optical axis (< a few nm), limited lateral spatial resolution (diffraction limit)
  - Digital Holography Microscopy (DHM), etc.
- **Direct contact methods** (stylus)
  - Resolution depends on the actual stylus size
  - Large surface area can be measured
  - Commonly use in industry
  - Contact method



(Illustration Mittutoyo)

- **Atomic force microscopy** (AFM) – limited surface imaging (typ. 50 x 50 μm^2)
  - Contact and non-contact measurement
  - High resolution (nanoscale down to atomic level in certain conditions)
  - Physical interaction with the specimen

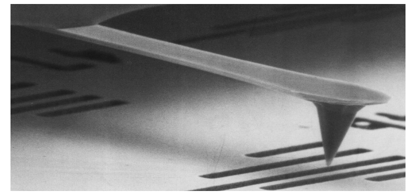
There are several experimental methods available for measuring surface topologies. One can divide them into three main categories:

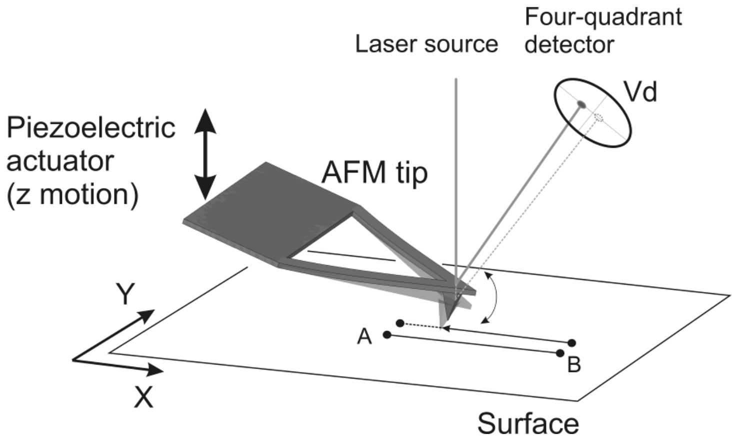
- **Optical-based profilometry techniques** – These methods are based on optical observations and hence, on how light reflects on the surface. It is therefore strongly connected to the optical properties of the surface. It offers multiple advantages, first one being to be a non-contact method, and hence there is no risk of altering the surface. It is a rapid method, allowing for large surface measurements. Limitations are related to the working principle itself. Indeed, as the information related to the surface interaction with light, artefacts and misinterpretation of the signals can lead to an incorrect surface representation that differs from the real physical surface. As it uses microscopy methods for imaging details, the *spatial resolution* (i.e. in the plane) is limited by the numerical aperture of objectives and will typically be in the range of a micron to a few microns. Hence, it cannot resolve spatial details smaller at best smaller than a micron. However, *along the z-axis* (the depth of the profile), the resolution can be in the order of a few nm thanks to interferometric methods.

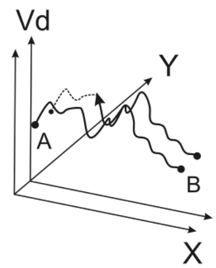
- **Stylus-based methods** – The most intuitive technique, which consists of moving a stylus in direct contact with the surface and to effectively measure the motion of the stylus as it moves across the surface.

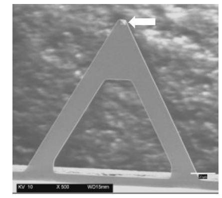
- **Atomic force microscopy (AFM)** – A more set of advanced methods that gives access to the surface topology down to atomic resolution, but that also offers a means to locally measure a set of physical properties of the surface. AFMs have been essential tools in the development of nanotechnologies and are of particular interest in the context of 'micro-engineering'.

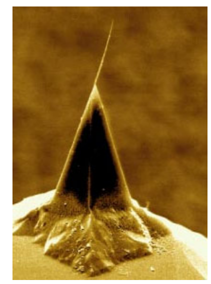
Measuring surface forces at a **nano-scale** level: "Atomic Force Microscope"











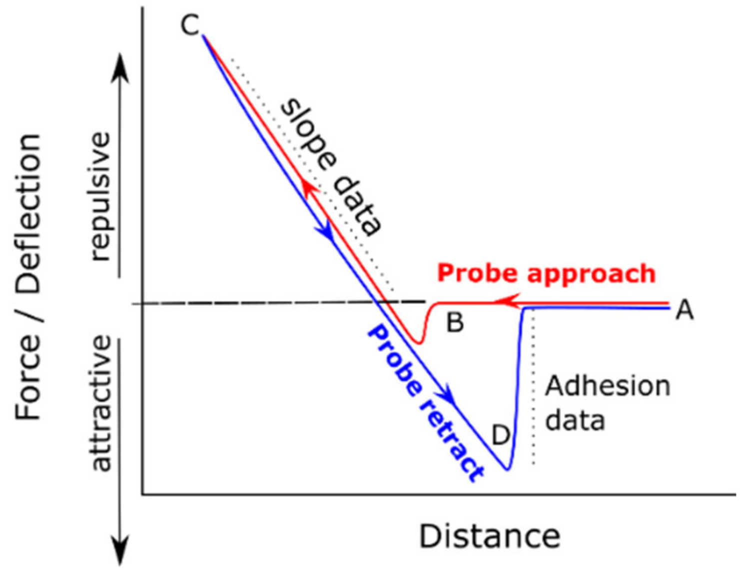
Invented in 1986, Binnig, Gerber, Quate (IBM Zurich Lab)
1986 / (STM) Nobel prize G. Binnig, H. Röhrer (IBM Zurich Lab)

A schematic of an atomic force microscope (AFM). AFM are instruments that are used to *analyze* surfaces, to connect their *topology* to their *local physical properties*. This technology, developed at IBM Zürich Lab was awarded a Nobel Prize in Physics in 1986 (Binnig, Röhrer) in relation with the Scanning Tunneling Microscope.

The fundamental principle is to use a cantilever (in French: 'poutre') (in the example above of triangular shape but most common AFM tips are cantilever beams). These cantilevers are very thin (typically a few microns or less) and produced using cleanroom fabrication techniques. At the tip of the AFM, there is a sharp point (usually with pyramidal shape) that interact with the surface. The tip can be down to a few nm, and occasionnally, just a carbon nanotube as illustrated above.

The AFM cantielver is mounted on a z-scanner (usually composed of piezoelectric actuators to achieve a high dynamics). As the scanner moves the AFM to the vicinity of the surface, it eventually deforms (which happens before it effectively touches the surface as we will see), due to the presence of forces resulting from the proximity of the surface. The cantilever deformation is measured at the tip using a triangulation principle. A laser beam is shone at the tip surface of the cantilever and reflected toward a photodetector that detects when the laser beam is moving due to the bending the cantilever that causes a change of orientation of the tip surface and hence, deflects the laser beam. Since the displacement of the piezoactuators (z-scanner) and the bending of the cantilever are both known, once can retrieve the actual force applied at the tip, and this with very high resolution.

Typical interaction force for an AFM



(source: AFMWorks)

- Force/deflection force provides information about the physics of the surface.
- Specific probes can be used to retrieve rich information, such as thermal transfer, conductivity, etc.
- **An AFM can provide more than just the topography**

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The nature of the interaction forces curve measured as the AFM tip approaches the surface contains a wealth of information about the physical properties of the surface.

This slide illustrates a typical force-adhesion curve observed during an AFM measurement. The cantilever is moved down following the path (ABC) and then up (CDA).

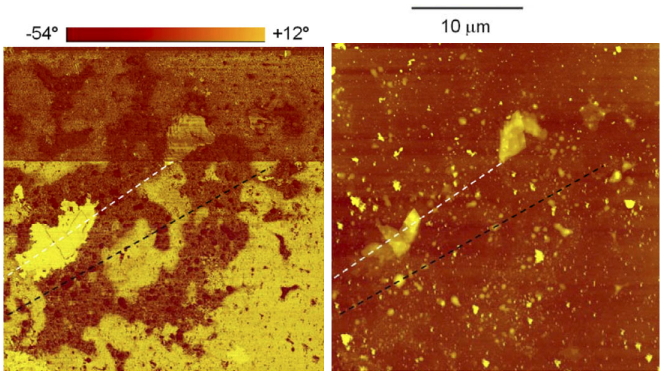
On the approach curve (ABC), a first instability is found at point B. This is called the snaping effect as the cantilevel is suddenly snapped on the surface. On moving the z-scanning up, and the cantielver up, (path CDA), another instability is found at a point D (release point) different than point B. The cantilever behaves like if it was 'sticked' to the surface, untile the force because high enough to overcome the adhesion forces.

This cycle with hysteresis is typical of the presence of adhesion forces, and the shape of it, its amplitude and the occurence of the snaping and release point tells us about the adhesive properties of the surface.

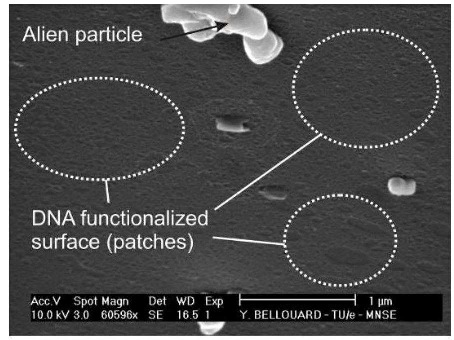
In the additional slides after the conclusion, you will find supplementary on this topic if you wish to dive more into it.

Illustration 1: DNA molecules on a silica fiber

- Atomic Force Microscopy (Phase imaging)



Phase image **Intermittent contact microscopy**



Scanning Electron Microscopy

Source: GOLEM project, TU/e (Madani-Grasset, Bellouard)

Let us now examine a few examples, illustrating the fact that AFMs do see more than just the topography and can be used to also retrieve additional information about the surface.

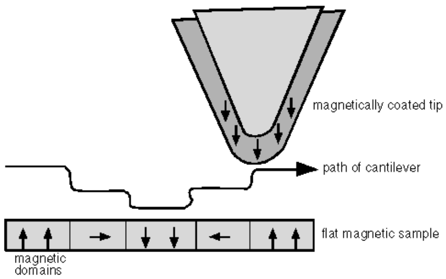
In this first example, we consider patch of DNA molecules attached to the tip of an optical fiber.

The top one shows an image in the electron microscope. Patch of DNA molecules can be seen on the surface.

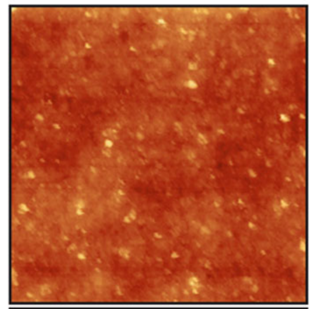
The two bottom images are atomic force microscope images seen under different imaging modes. The left image contains the phase information (i.e., how fast the cantilever deforms back as it is scanned over the surface) and inform us about adhesion forces ('how good the cantilever locally 'stick' to the surface), while the one in the right (intermittent contact microscopy image only) is more informative about the actual texture and surface topology.

These three images that look very different, are nevertheless representations of a same object, but do contain a different set of data.

Illustration 2: Magnetic Force Microscopy

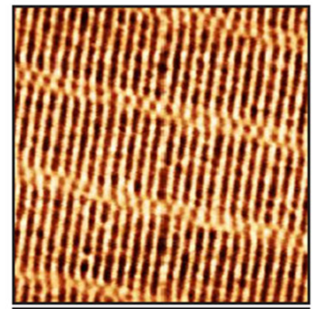


Topography surface information



AFM image, Zip Disk 40 x 40 µm

Functional surface information



MFM image, Zip Disk 40 x 40 µm

Source : AFM workshops (TM)

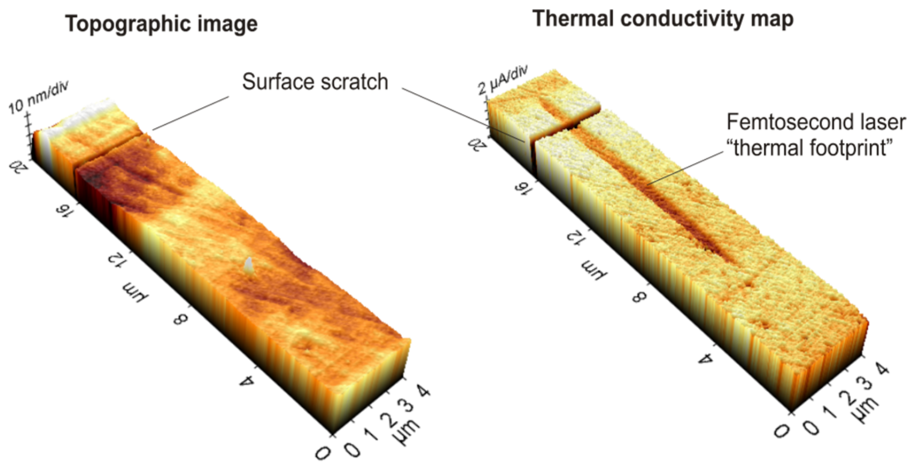
- Ferromagnetic tip: Co, Cr
- Noncontact mode
- van der Waals force: short range force
- Magnetic force: long range force; small force gradient
- Close imaging: topography
- Distant imaging: magnetic properties

This second illustrative example is related to magnetic recording media. It compares a topographic image (AFM image) with an image of the magnetic properties of the surface (MFM image).

The tiny cluster of information that forms 'bits' in the recording media are revealed in the functional surface information image, but not seen in the topography. Likewise, topographic features (such as nanoparticles that appears as bright spot) are not visible on the functional information, hence plays little role on their magnetic properties.

Although representations of a same object, the two images are very different and contains different information, illustrating how the point that AFMs can be used to retrieve not only topological information, but also functional information.

Illustration 3: Waveguide in a glass substrate



Y. Bellouard et al, Appl. Phys. Lett. 89, 161911 (2006).

In this third example, we consider a glass part that was exposed to a femtosecond laser in its volume and then cut in the middle. (Note that we will discuss about laser processing in the next lecture.) The laser exposure led to a localized densification of the material that modifies the thermal conductivity of the glass locally.

The left image shows the topography, while the right one shows the surface from the viewpoint of thermal conductivity. A surface scratch (forming a little ‘canyon’ or fracture) is visible on both images. However, the two images are otherwise quite different.

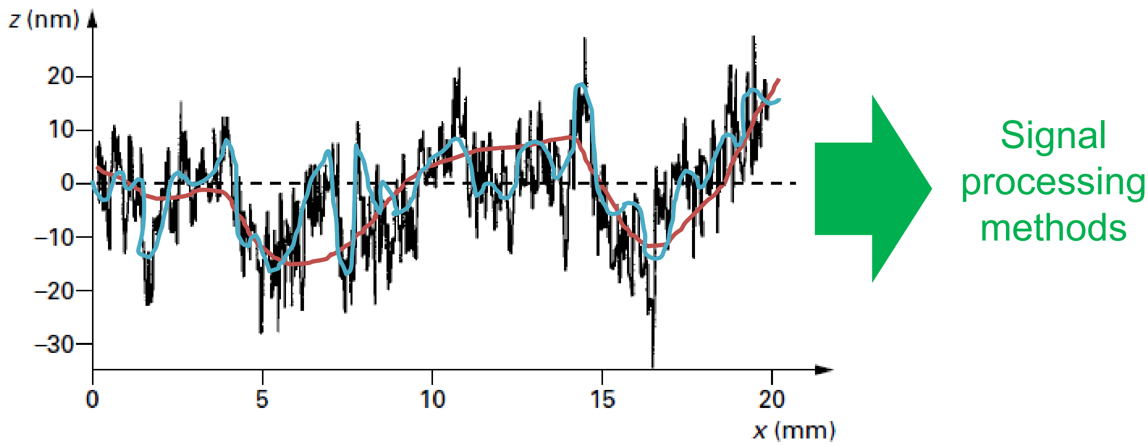
The region where the laser was starts appearing on the thermal conductivity image, although it was invisible on the topography representation. Likewise the smooth topographic features have not much effect on the thermal footprint.

In summary, the key point to remember is that in the context of surface properties characterization, Atomic Force Microscopy (AFM) is a generic method that can be used to extract both topological and physical properties of a surface with nanoscale resolution. It has been a game changer in surface science and nanotechnology in general. Hence, a very important measurement method to be aware of.

‘Geometrical characteristics’ / Topography

Let us know focus only on the topological information related to a surface and let us leave aside materials/physical aspects related to the surface.

Problem statement



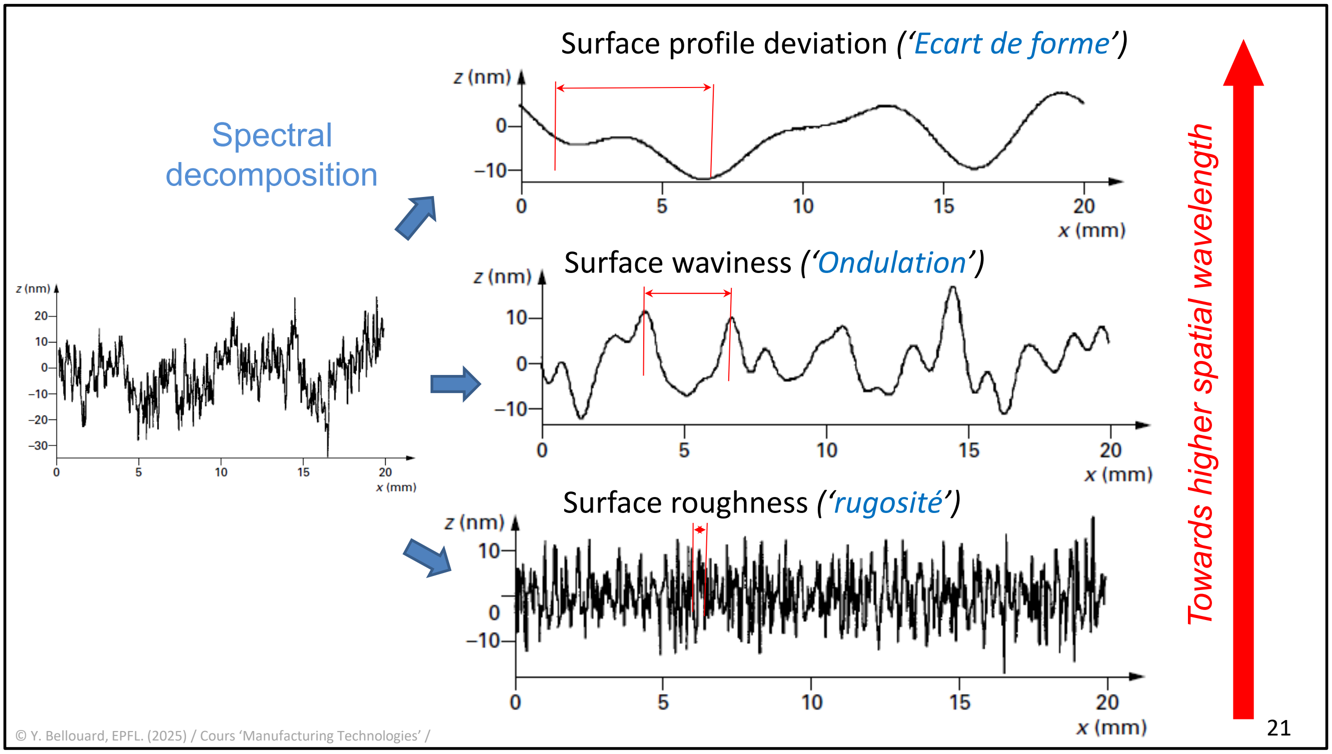
(adapted from P. Bouchareine, Techniques de l'ingénieur) 20

The problem can be stated as follows. Assume we do scan an stylus (or an AFM) on the surface along a line (or assume that we extract a topography line on a 3D profile taken with an optical profilometer).

Whatever the measuring technique used, a typical profile may resemble the black line above. This line seems like a noisy signal at first. But observing it in more details, one can notice the juxtaposition of different 'pseudo-periodic' signals.

Intuitively, one can suspect that signal processing methods are likely to provide useful information about this apparently noisy signal.

If we define a spatial frequency, that is to say the inverse of a distance (1/x). The spatial frequency would be defined in mm^{-1}. If we apply a Fourier transform on the signal above that is expressed in a geometrical space (height of the profile versus actual coordinate along an axis), we should be able to extract information related to the different spatial frequency components.



In essence, our noisy signal (on the left), can be seen as the combination of signals with various spatial frequencies that we can decompose about typical spatial frequencies.

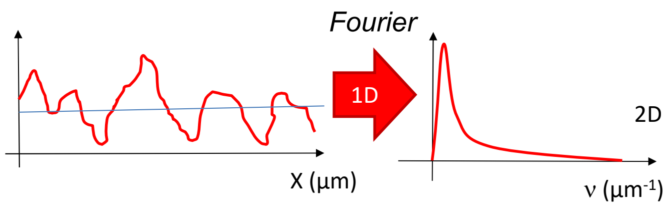
The fast-oscillating signal defines the surface roughness. The slower varying one, the waviness and even slower periodic variation can be defined as a ‘profile error’.

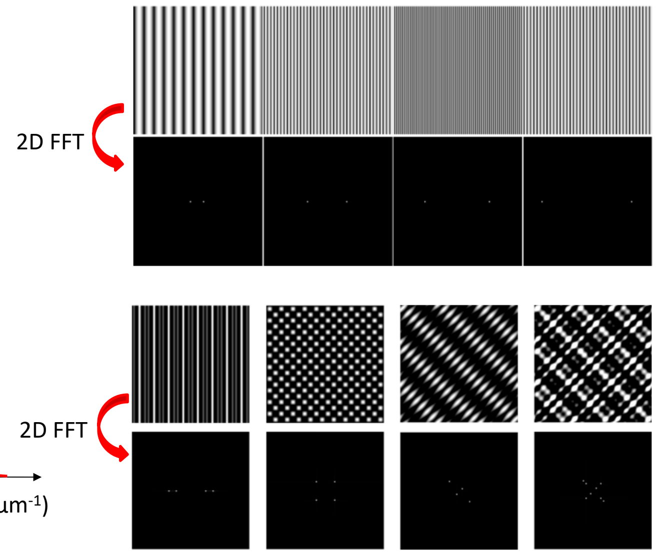
Note that waviness may not be confused with surface profile error. Profile error (‘Ecart de forme’) is more related to deviations between a desired profile and the actual one, which is more related to specific manufacturing errors for which it may not be possible to define a spatial frequency per say on the surface portion that is considered.

Fourier analysis in the spatial domain

- Concept of spatial frequency
- 2D-Fourier transform

$$S(\nu,\omega)=\int_{-\infty}^{+\infty}\int_{-\infty}^{+\infty} s(x,y)\,e^{-2\pi(\nu x+\omega y)}$$





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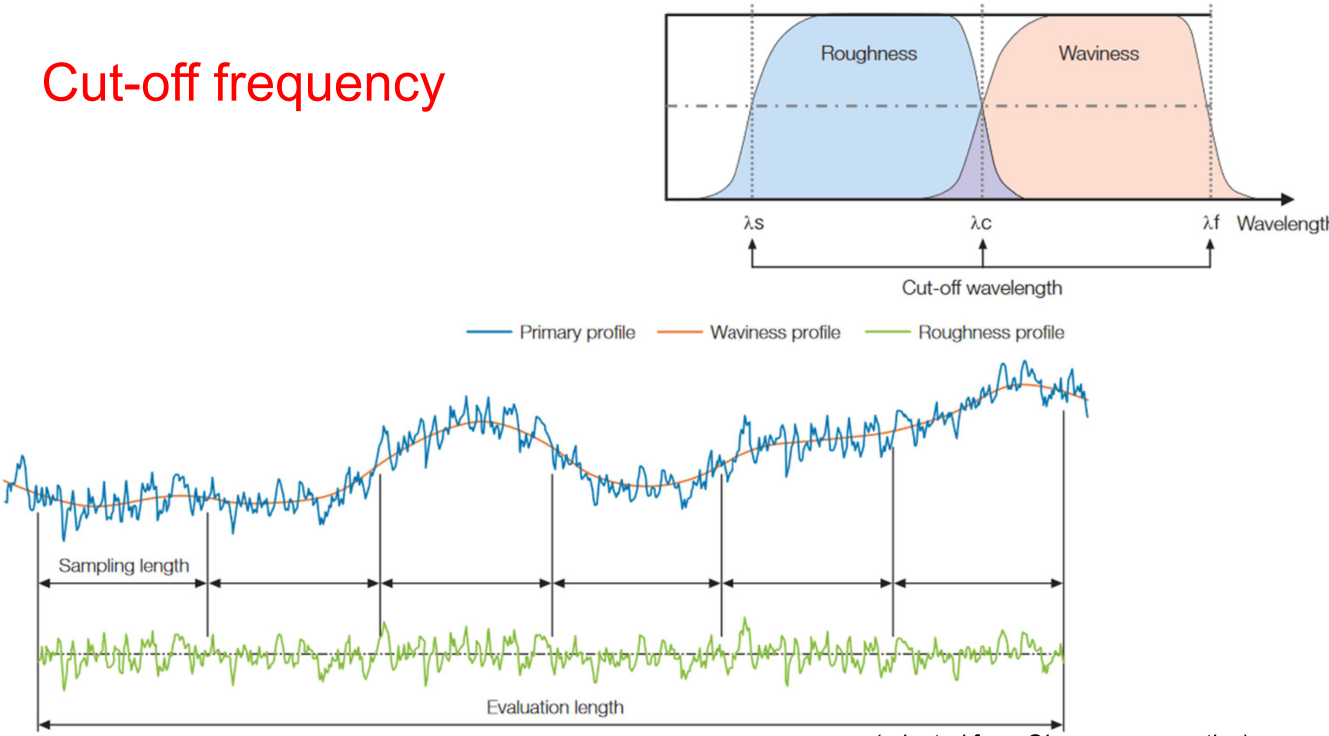
Intuitively, as we are examining periodic features with multiple frequencies, a natural tool that comes in mind is the Fourier analysis, just like use it in other engineering problems, such as image analysis and signal processing in general.

A convenient tool for investigating these geometrical characteristics of surfaces is the two-dimensional Fourier transform, where we consider spatial frequencies.

Apart from that, the principle here is the same than a more classical Fourier analysis in the time/frequency domain. Here, time is replaced by position, and frequency by spatial frequency.

Cut-off frequency



(adapted from Olympus corporation)

Let us consider the profile above in blue. Like the ones examined before, it has a fast oscillating components ('the roughness') and a slow one ('the waviness').

Transposing in the Fourier domain the 'signal measured', i.e. the actual profile of the surface (here for simplicity along a single axis), spatial frequencies form two distinct frequency domains that can be separated with a cut-off frequency.

Low-frequencies (i.e. high wavelengths) correspond to the waviness, while high-frequencies (i.e. low wavelengths) are related to the roughness.

As will be seen in the exercise that uses the software 'Gwyddion', one can separate using low-pass or high-pass filters in the Fourier domain, roughness from waviness, i.e., construct a profile with only the roughness and another one with only the waviness.

Why measuring/defining a surface topology?

- **Surface topography can have a strong impact on the part behavior!**
- Part manufacturing reproducibility / Process control: it tells us about how to the process is evolving / evidence of tool wears, etc.
- Wear state of a surface
- Conformability of a surface with respect to desired functionalities:
  - *Load capacity*
  - *Bearing / lubrication properties*
  - *Optical properties*
  - *Thermal properties, ...*
- It is a signature of a manufacturing process

Although it does not contain information about the physical properties of a surface, topography contains a wealth of other useful information.

For instance, it may contain a signature of the manufacturing process that has been used. As an example, distinct manufacturing processes as illustrated in slides 8 and 9 can lead to distinct textures.

Observing how these textures evolve and their characteristics offer a means to monitor a manufacturing process.

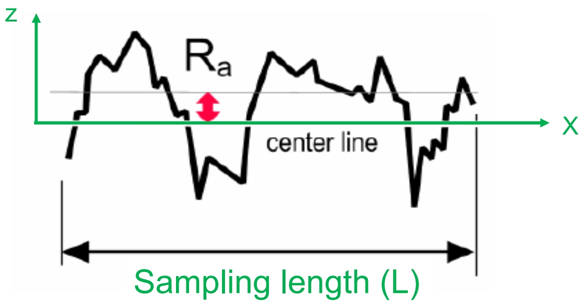
Let us consider a conventional machining process, such as turning as an illustrative example. As the turning machine produces parts, possible early sign of tool wear can be detected by observing how the roughness evolves.

Monitoring the surface topology and how it evolves from one specimen to the others is a method to achieve process control and reproducibility.

Topography measurements provide also a means for verifying the conformability of a surface with specific topology requirements for the surface.

A common surface metric: the arithmetic average (R_a)

- *A statistical parameters: arithmetic average*
- *Very used in industry!*
- *Often the only one you hear about…*



Sampling length

$$R_a = \frac{1}{L}\int_0^L |z(x)|\,dx$$

Profile amplitude

RMS roughness: $R_q = \sqrt{\frac{1}{L}\int_0^L z^2(x)\,dx}$

(illustration adapted from Zygo)

A large number of metrics exist to analyze surface topologies. (A guide about the most common ones is provided in the Moodle, for those interested in exploring the topic further.)

The metrics can be further subdivided into absolute measurements and average ones.

The choice between one metric versus another depends on what characteristics is the most relevant for the applications.

For instance, on a surface where we care more about the roughness across the entire surface (like a surface that should have specific friction properties), averaging metrics such as the Ra will prevail.

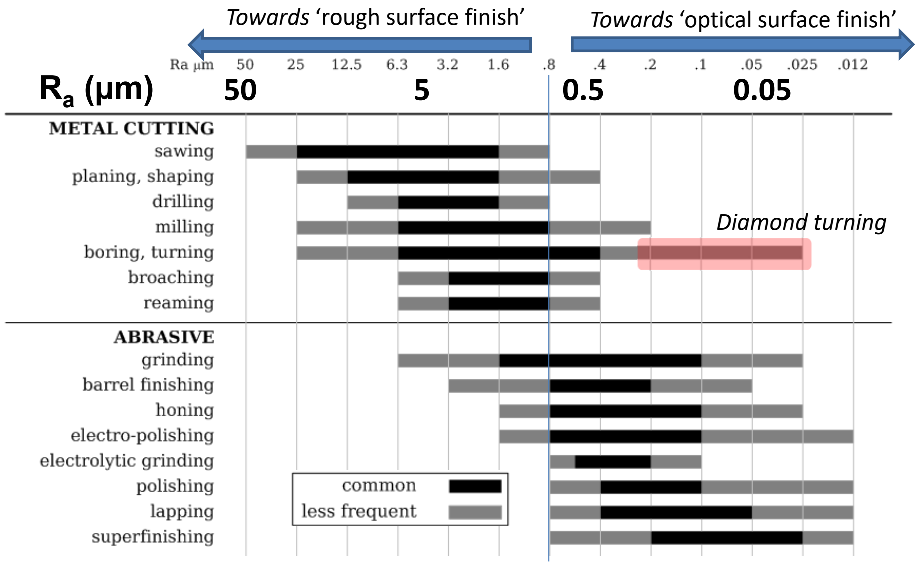
On the contrary surfaces where the size of the largest scratch matters (like in the case where specific visual effect are sought), an absolute measurement (Rz) will be more appropriate.

Whatever metrics is used, a very important aspect to consider when performing surface topography measurement is the *sampling length* that defines the region over which the measurement is performed.

Just like in classical signal theory, to be numerically representative, the sampling length should be carefully chosen so that it is significantly larger than the typical spatial frequency of the variation we are measuring. In other words, it can be seen as requirements over the size of statistical ensemble. If this size is too small, the statistic is biased and becomes non representative (or under-sampled).

As we will see further, the Ra (or Rq) is a commonly used metric. Although useful, it has limited information content and cannot represent alone the full surface topology information.

Typical surface finish for various manufacturing processes



(adapted from Emok, 2008)

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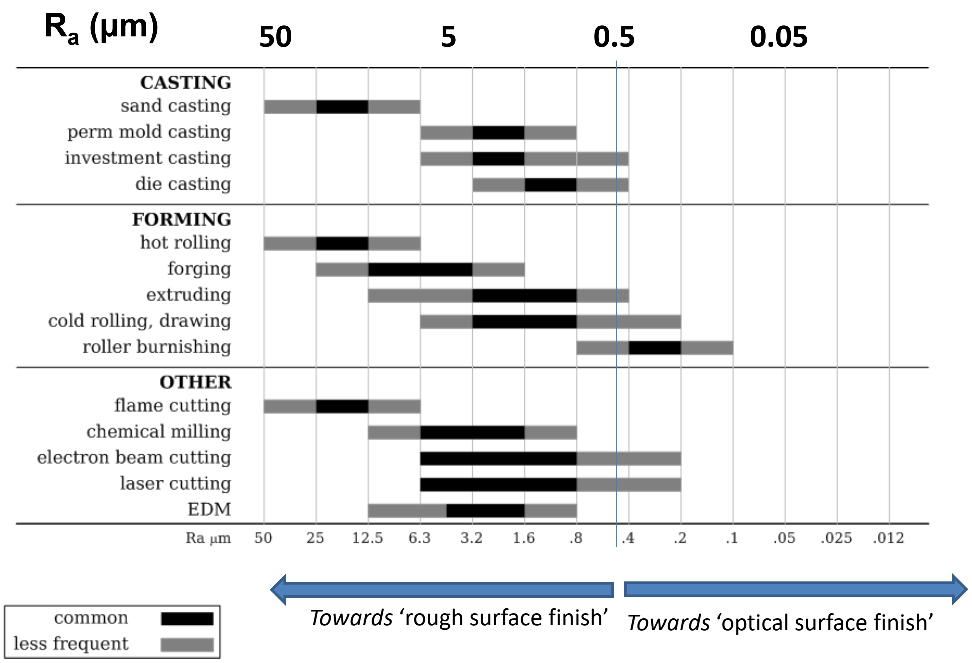
Bearing in mind, the limited information contained in the Ra, we can make a first comparison between a variety of processes, some of which will be examined in the next lectures.

This chart gives an overview of the typical roughness (from the viewpoint of an Ra metric) for an ensemble of manufacturing processes, divided into main process categories.

The roughness scale is logarithmic. Cutting, casting, forming and a few others (see next page) have roughness typically in the micron-range and well above. Only abrasive processes, that include polishing and lapping methods, are capable of reaching nanometer surface resolutions.

There is one notable exception, which is the diamond turning, that is used to achieve some of the lowest roughness on optical elements such as lenses. (In the following pages, there is a link describing the diamond turning process.)



(Adapted from Emok, 2008)

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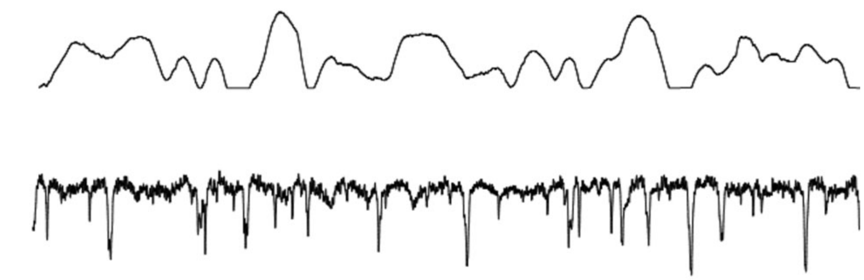
Second part of the table in the previous page but illustrating other family of processes that will be studied in class. It is common to talk about 'optical surface finish', when the roughness starts to be an order of magnitude smaller than the wavelength of light.

Same surfaces?



(Adapted from Mittutoyo, 'surface analysis')

Being a single scalar metric, the Ra only tell us about an absolute average of the surface oscillations and nothing else. Hence, it has limited information content.

To appreciate this important point, let us consider the three different profiles illustrated above. One is a pure regular sinusoidal profile, the second one is a relatively homogeneously distributed but random oscillations with a more or less similar amount of peak and valleys, while the third one has a low amplitude but with occasional high and sharp narrow peaks, appearing randomly.

If ones measure the Ra of these three profiles, strictly applying the definition, they will all yield about the same value, and hence, one could conclude (naively) that the surfaces are identical, which obviously for these examples is a wrong assumption.

This illustrative comparison highlights the limit of the Ra metric and its limited information content. It does not suffice by itself to distinguish the three surfaces above. Hence, the need for additional metrics.

Arithmetic averages (Ra or Rq) contains limited information

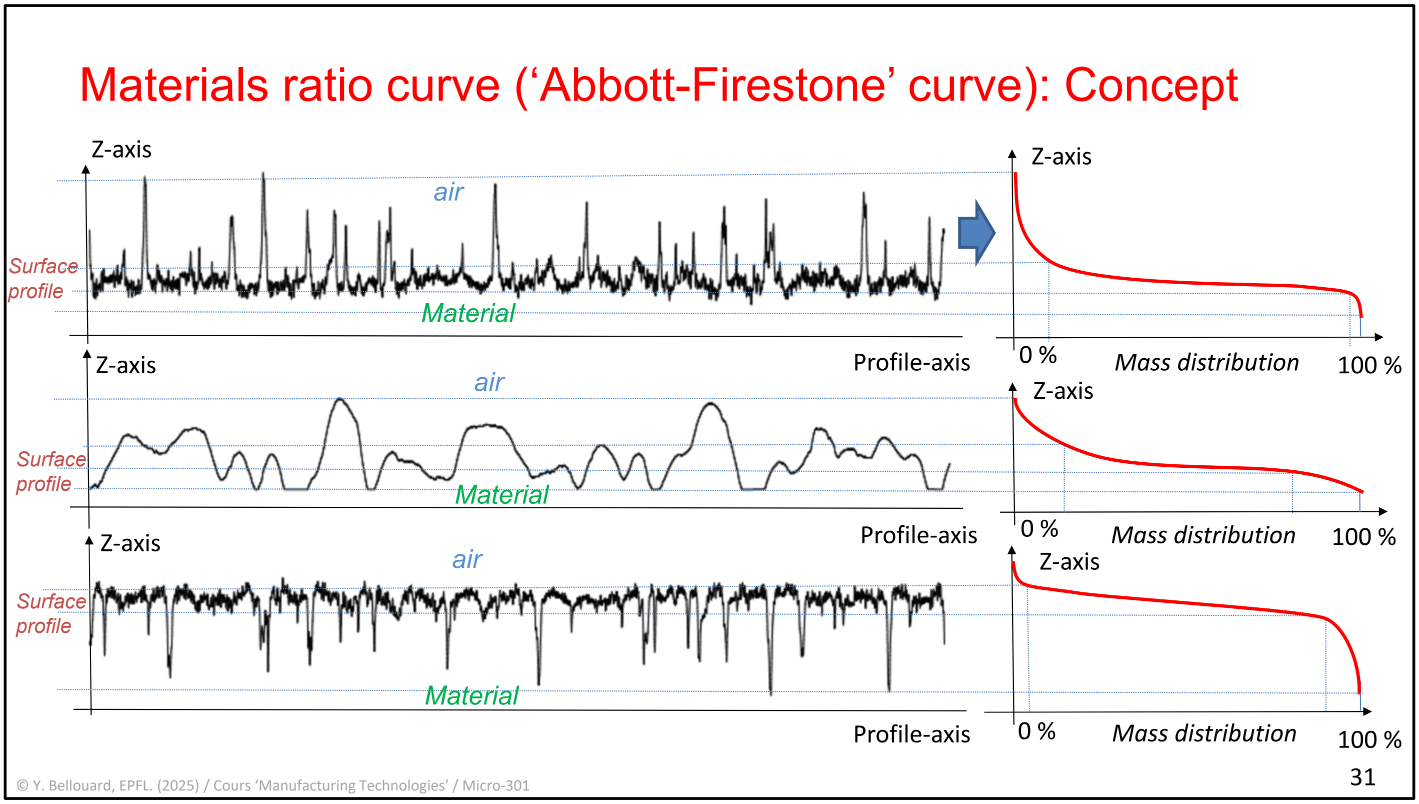
- Ra (Rq) are **informative** not sufficient to fully describe a surface.
  - It does not say much about the density of peaks
  - It does not tell anything about the shape of the peaks
  - Possible textures
- Manufacturing processes (wether by adding or removing materials) produce a typical surface topology that cannot be only characterized by the Ra.
- Additional surface metrics?

Fortunately, there are numerous other surface metrics that have been defined to provide a more complete quantitative description of the surface profiles.

Tribology – Lubrication/Hermetic sealing

Depending on the functional need for the surface, it is useful to look at other metrics than the Ra.

This is notably the case once looking at lubrication and/or hermetic sealing properties of a surface. Let us examine this example as a case-study.

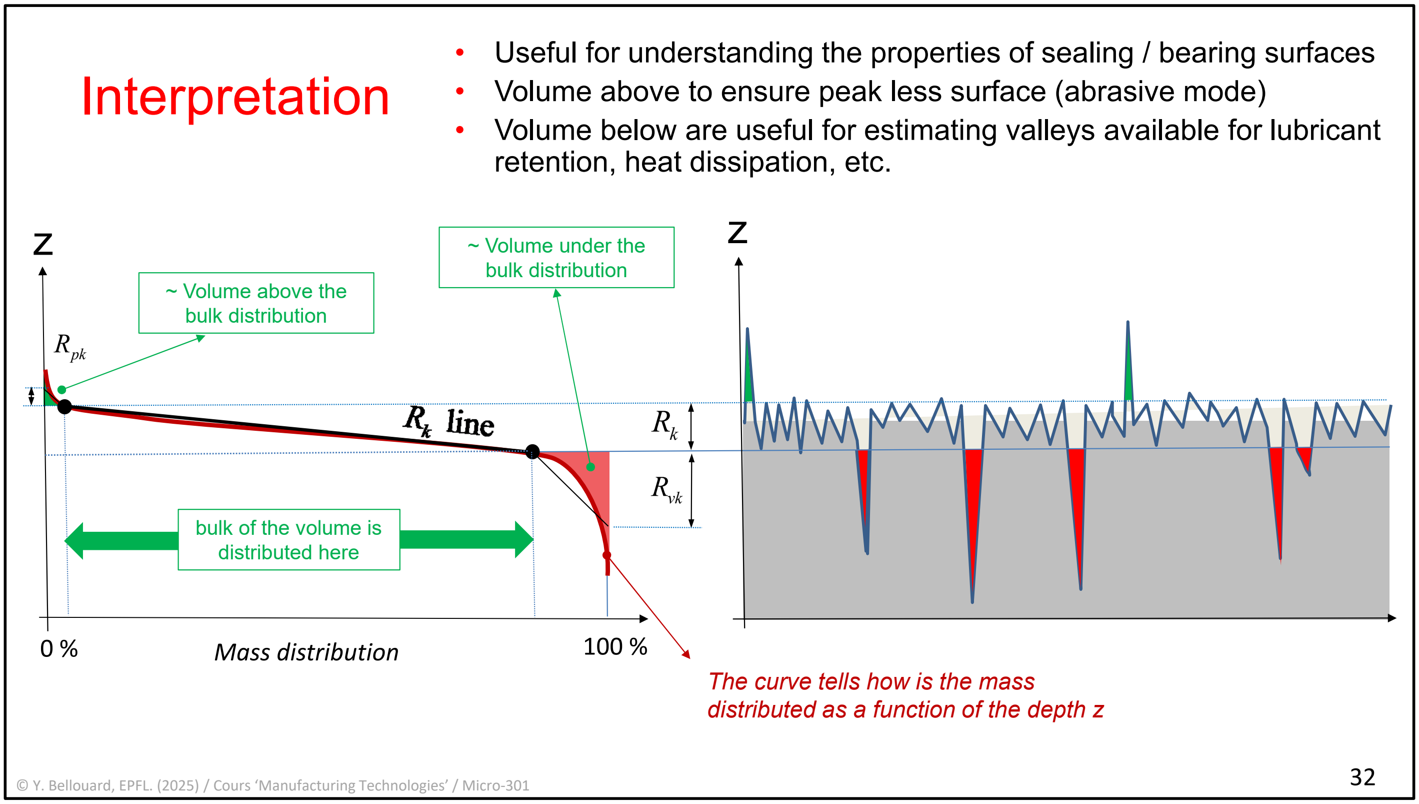


To this end, an intuitive concept to characterize surfaces in the context of lubrication/sealing that may otherwise have the same Ra is the concept of 'Material-ratio curve', also called the *Abbott-Firestone* curves.

This curve, imagined in 1933 by Ernest Abbott and Floyd Firestone, consists of plotting a histogram of the material distribution as one moves across the surface, moving from outside the material to inside along the z-axis. (On the illustrations above, the materials is below the curve that represents the surface topology, while there is no material above.)

Surfaces with spikes will yield a histogram with a flat tail for the first few percent of material and sharp increase of material distribution thereafter.

A surface with a homogeneous material distribution will define a smoother and spread profile across the surface thickness, while a surface characterized with sharp pits, will appear as a histogram with a steep increase of material distribution first, and a flat and stretched tip for the remaining percents of materials.



To further analyze the Abbott-Firestone curve, a few metrics are defined.

The Rk defines the spread in z for the majority of the matter and is defined as the z difference of the two extreme points that *best* define a linear approximation of the material distribution.

The volumes defined at the two extremities, i.e., where the curve departs from a linear approximation, define the volumes above and below the bulk distribution, respectively.

The knowledge of these volumes is useful for predicting lubricant retention for instance, or heat dissipation as well as sealing properties.

For instance, if the goal is to have a high lubricating retention, one will try to maximize the volume under the bulk distribution.

How surface topology parameters correlate with surface functionalities?

From the previous discussion, a key question is now to relate metrics with functional properties. In other words, what are the necessary metrics that are relevant for a specific functional property.

Illustration 1: surface roughness and mechanical strength

Influence of roughness on mechanical properties: example

- **Intuitive reasoning:** Lower roughness = more contact points, higher surface strength?

$$\sigma^2 = \frac{1}{L}\int_0^L Z^2 \, dx$$

Contact radius → β; Contact points → N; 'Plasticity index' (<1, 'elastic behavior'; >1, 'plastic behavior') → Ψ

	σ (µm)	β (µm)	σ/β	N	$N/(\sigma/\beta)^{1/2}$	Ψ A	Ψ B
Fine Polishing	0,028	2 400	10^{-5}	9 000	0,073	0,16	0,027
Grinding	0,13	800	$1{,}6 \times 10^{-4}$	6 000	0,061	0,66	0,11
Turning	–	60	0,05	3 400	0,045	11,7	1,9

A: Steel / Stainless steel

B: Steel / Nitrured steel (surface treatment)

Examples taken from G. Béranger, H. Mazille, Techniques de l'Ingénieur

It is important to note that roughness (and therefore, surface finish) can have a direct impact on surface physical properties. This example highlights how the surface roughness affects the mechanical properties, with here, the plasticity of the surface, through a metric called 'the plasticity index'.

A plasticity index below 1 indicates and elastic behavior, while a value above points towards a plastic behavior. The higher the value, the more plasticity is observed.

Case A describes a stainless steel with no specific surface treatment. As can be seen, the quality of the surface dramatically affects the plasticity index. The less rough, the lower the plasticity index. An intuitive interpretation of this phenomenon is that the lower the roughness, the more numerous are the contact points and therefore the highest the strength of the surface.

Case B is similar than **case A** with this time a stainless steel on which a coating (i.e. a think layer) has been applied to it. It is quite remarkable to see the impact on the surface performance, although such coating are typically very thin (a few microns or less). Although the core material is the same, the presence of the coating has dramatically improved its surface performances.

Illustration 2: surface roughness and optical reflectance

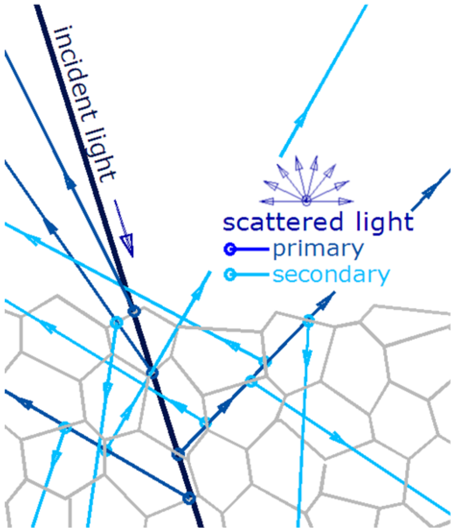
A ‘visual’ example: relation between roughness and optical properties

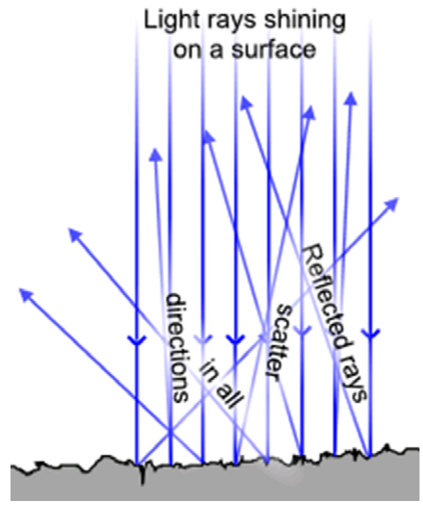
Let us now consider optical properties of surfaces. Here, we will limit ourselves to the case of diffusive and non-diffusive surfaces.

These two situations are illustrated above, where in the left a ‘frosted’ glass bottle is shown – representative of a diffusive surface, while in the right, a transparent glass bottle is shown that is characteristic of a non-diffusive surface.

In both cases, the material is strictly identical, but the surfaces have been prepared differently. Obviously, the two surfaces have different optical properties.

Phenomenological understanding of diffusion effects





(source Wiki)

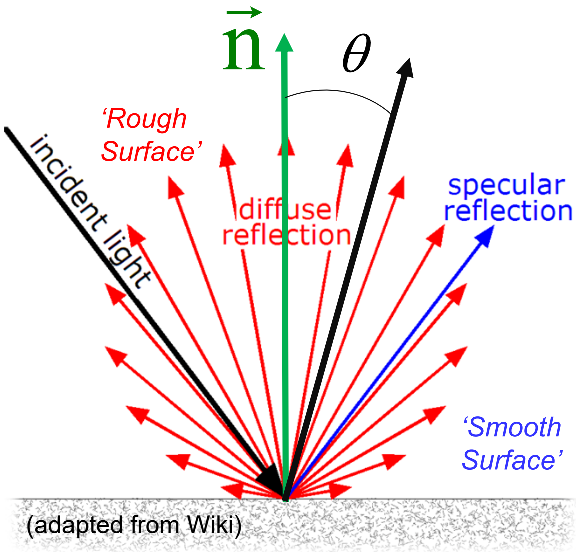
Whether it is in transmission or in reflection, the diffusivity of a surface, for simplicity, can be intuitively and phenomenologically understood by imagining rays falling on a rough surface but not transparent or going across a granular material partially transparent, but causing multiple reflections.

Note that this phenomenological interpretation based on ray optics is simplistic but remains sufficient at this stage to grasp these concepts.

As they hit an interface having multiple surfaces that are not perpendicular to the rays propagation direction, they reflect in arbitrary, and somewhat random directions, causing a hallow 'light' effect on the surface.

Hence, the term 'diffusive reflection' as opposed to 'specular' reflection that corresponds to the traditional reflection predicted by Snell-Descartes law.

Diffuse reflection: Lambert model



$$I_d(\theta) \propto I_0 \cos\theta$$

The diffuse reflection at an angle from the normal of the surface is proportional to the cosine of this angle.

Johann Heinrich Lambert (1728–1777)

The phenomenon of diffuse reflection was discussed by Johann Heinrich Lambert.

Lambert's law states that the intensity of light diffusing in a direction defined by an angle θ from the normal of the surface plane, is proportional to the incident intensity and the cosine of the angle θ.

In the illustration above, the specular reflection, according Snell-Descartes law, is shown in blue, while the diffusive reflections are shown in red.

A surface is said to be 'Lambertian', if the model proposed by Lambert applies (which is very often true, but not always). Note, there exists other models to describe the diffusivity of a given surface, but the Lambert-mode remains the most common.

How ‘smooth’ should a surface be?

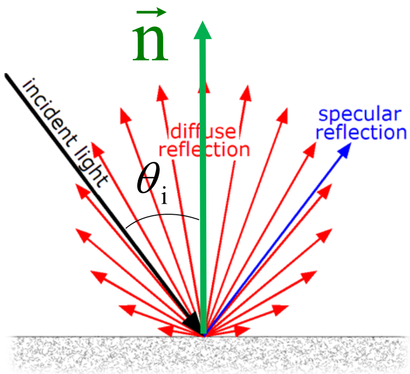
Bennett and Porteus model

$$R_s\left(R_q\right) = R_0\left(1 - e^{-\left[4\pi\left(\frac{R_q}{\lambda}\right)\cos\theta_i\right]^2}\right)$$

Total scattered reflection

Theoretical reflectance

Ratio of the roughness versus wavelength



Bennett & Porteus, “Relation Between Surface Roughness and Specular Reflection at Normal Incidence,” JOSA 51, 123 (1961)

An important underlying question related to diffusivity is to find a relation with the material roughness. In other words, can we consider a model where we can correlate some of the surface characteristic, capture with some simple metric, with intensity scattered from the surface.

The mathematical treatment is non-trivial and can be found in the paper above for the reader interested in getting into more details.

This important relation connects the total scattered reflection for a given angle of incident light with the roughness (Rq – see previous slides) and the wavelength of light λ considered, and the theoretical reflectance of the material considered.

Note that this research is relatively recent, a little more than 60 years ago...

Observations

- Optical scattering is proportional to reflectance ⇔ surfaces that reflects light will scatter more light than transmissive surfaces.
- Scattered intensity is related to Rq
- Shorter wavelengths scatters more than longer ones.
- Light scatters more at normal incidence than grazing incidence

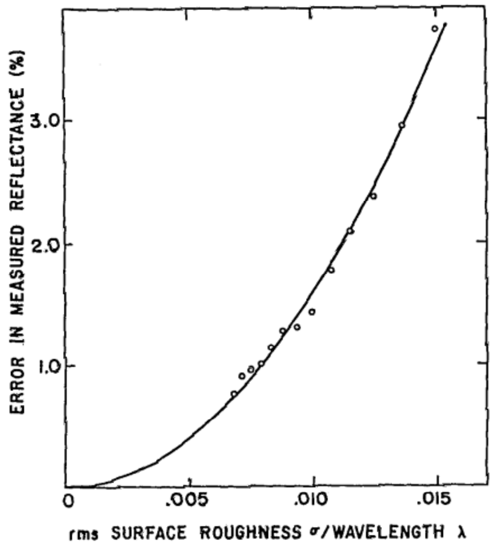
Bennett and Porteus model allows us to draw a few important conclusions.

Optical scattering is proportional to the material reflectance. In other words, a material that naturally reflects well is likely to also diffuse well. Consider for instance, polished aluminum versus sand-blasted aluminum.

The scattered intensity is related to the ratio between the roughness (Rq) over the wavelength. As one would expect, the higher the roughness, the more scattering will be observed.

Shorter wavelength will also scatter more. For instance, UV light will more easily scatter than infrared light.

Importance



Rq = 25 nm scatters about 10% @ 500 nm !

Single Point Diamond turned surfaces can achieve Rq = 5 nm

Bennett & Porteus, "Relation Between Surface Roughness and Specular Reflection at Normal Incidence," JOSA 51, 123 (1961)

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This graph above illustrates that even for small roughness, it rapidly impacts the intensity of light being diffused and not reflected, even though the roughness is an order of magnitude less than the wavelength. (In the example above, the green 'color'.)

For instance, a Rq of 25 nm (which is already very smooth and not too easy to achieve as a surface quality) still will result in 10% of scattered at the green wavelength.

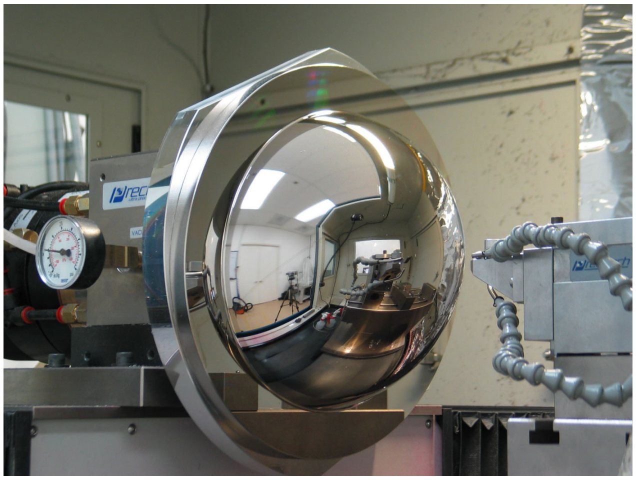
To achieve such low roughness, single point diamond turning is one of the process used.

The importance is that such models help properly defining optimal requirements for an optical roughness finish.

Indeed, one could think that after all, we should always aim at the smoothest surface as possible, with the lowest Ra as possible. However, the cost and manufacturing time to achieve the lowest possible roughness can be extremely high.

Hence, using such model can help satisfying both requirements and economical constraints.

About diamond turning...



Source : NiPro

YouTube illustration from Thorlabs:

https://youtu.be/6iRohI_jaYg

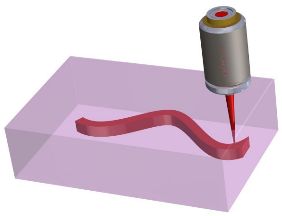
Video illustration of the single point diamond turning process to achieve high quality optical surfaces.

Single point diamond turning process is one of the only process capable of achieving optical surface quality on complex and curved shapes. The process can also be applied to a variety of substrates.

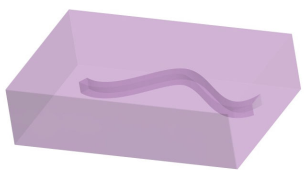
Illustration 3: effect of surface properties on fracture mechanics

3D machining illustration: two-step laser manufacturing

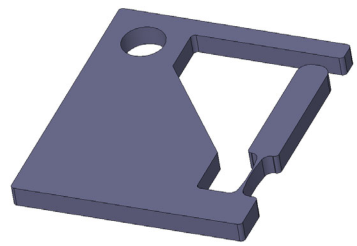
1/ Femtosecond laser exposure **(no ablation)**



2/ Chemical etching **('development step')**



Arbitrarily shaped parts



We will discuss it more in the laser-manufacturing lectures!

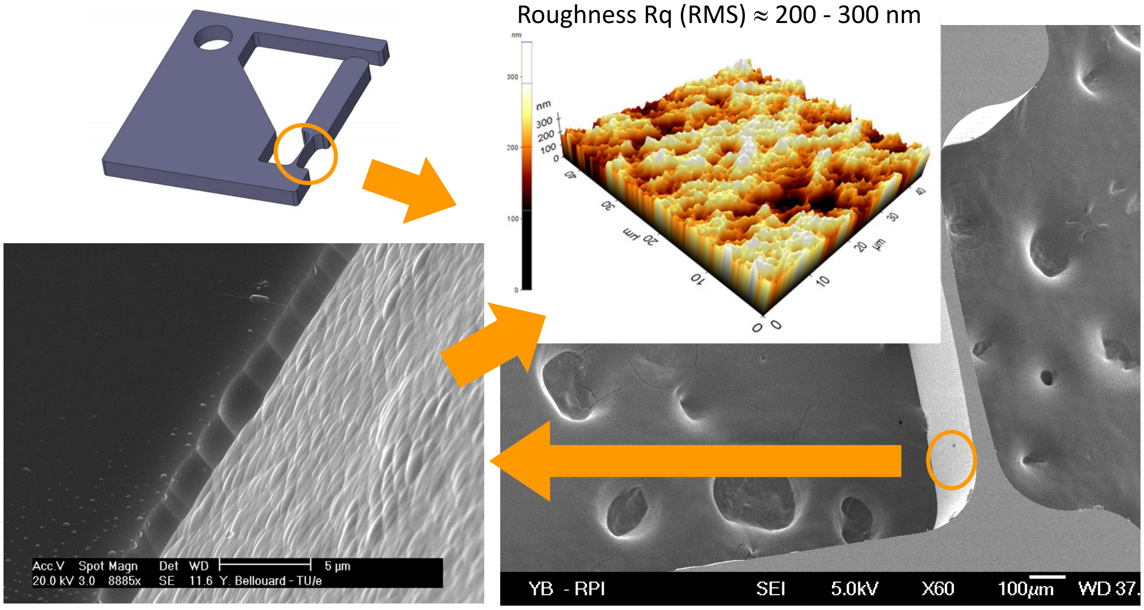
*Y. Bellouard, A. Said, M. Dugan, P. Bado, Optics Express, **12**, 2120-2129 (2004).*

Let us discuss a practical example, here for a process for manufacturing glass that combines femtosecond laser exposure and chemical etching. This process will be discussed in the next lectures.

Such process are typically used for producing a variety of devices and parts, such as for instance micro-flexures as illustrated in the right CAD rendering.

It belongs to the category of 3D-printing and has enabled numerous innovations in micro-manufacturing.

Micro-manufacturing illustration



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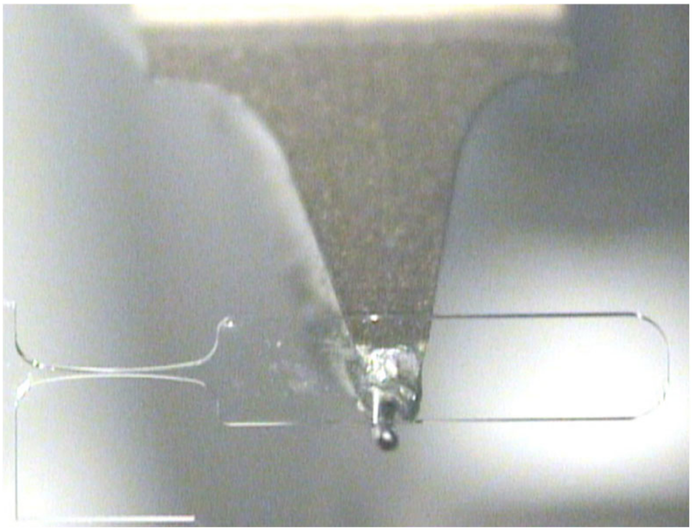
The roughness in this illustration will be analyzed in the exercise related roughness metrics and that uses the open-source software Gwyddion. (https://gwyddion.net/)

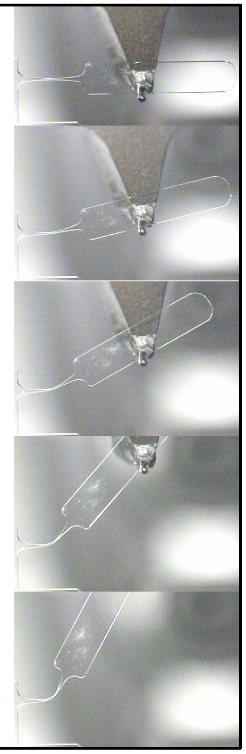
This illustration concerns a glass flexure and the influence of surface machining roughness on their performances in term of fracture limit.

Although it is a glass (fused silica), and although it may sound counter-intuitive, they exhibit remarkable elastic performances, comparable to the best steel, if not better.

Glass flexure strength depends on etching time...



Stress locally well above 2 GPa!

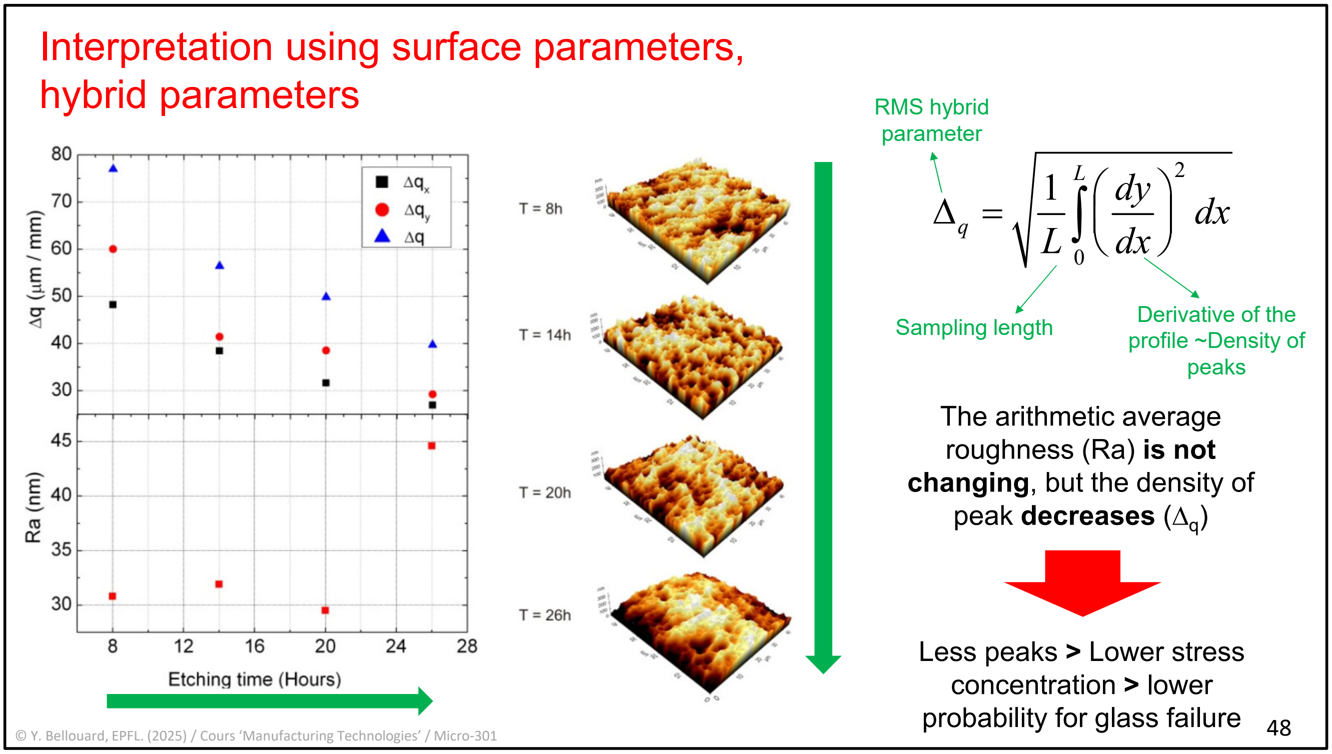


Observation: The more we etch, the higher the strength. Why is it such?

This illustration shows a glass flexure being severely bend elastically, without breaking. In the example here, the stress locally reaches 2 GPa.

What was observed in these series of experiments is that the longer the etching time, the higher the strength.

The question is understand why longer etching has an impact of the mechanical strength.



Interestingly, it was noticed that etching does impact the breaking limit of the glass significantly.

Intuitively, one may think that increasing the etching time of the glass may improve the roughness. In fact, it is not the case, and the roughness, Ra, does not change noticeably.

However, etching does have an effect on the surface as can be seen on the topography images.

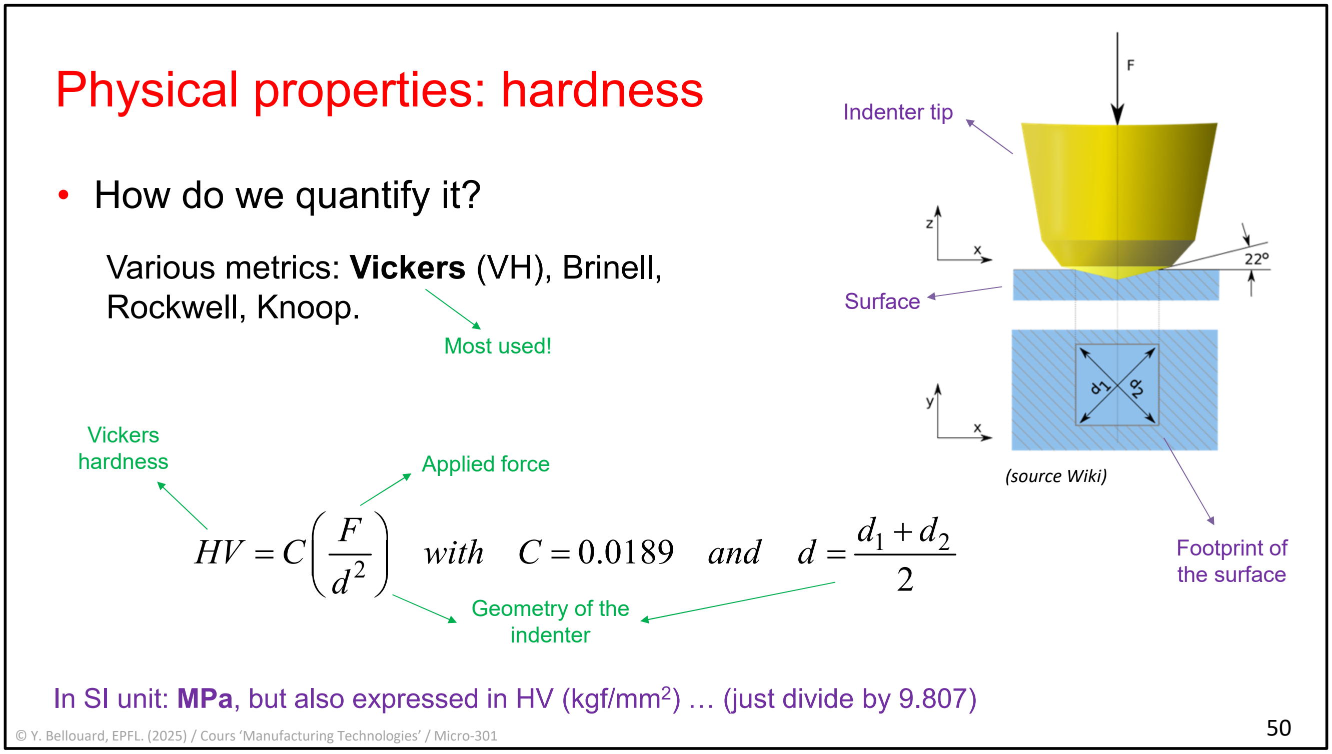
In this example, one needs another surface metrics to understand the behavior.

In fact, the etchant decreases the number of peaks, but not their highs, which explains why the Ra is not changing, since it is an average of the height.

Considering the Δq metrics (so called 'hybrid parameters'), it is possible to capture this decrease of peak densities that seems to explain why the glass becomes increasingly more resistant after a certain number of hours. The Δq gives a metric of the root mean square (RMS) distribution of the derivative of the profile, instead of the profile itself. Somehow, it provides a metric of the 'sharpness' of the peaks in the profile. Unlike the Ra, this metric does decrease with the increased etching time. It suggests that the smoothing of the peak (not their amplitude that do not change), reduces the likelihood for a part to break.

Finally, this example illustrates the importance of using various surface metrics in the context of surface analysis to be able to interpret various experimental observations.

Example of a surface functional properties characterization: the hardness of a surface



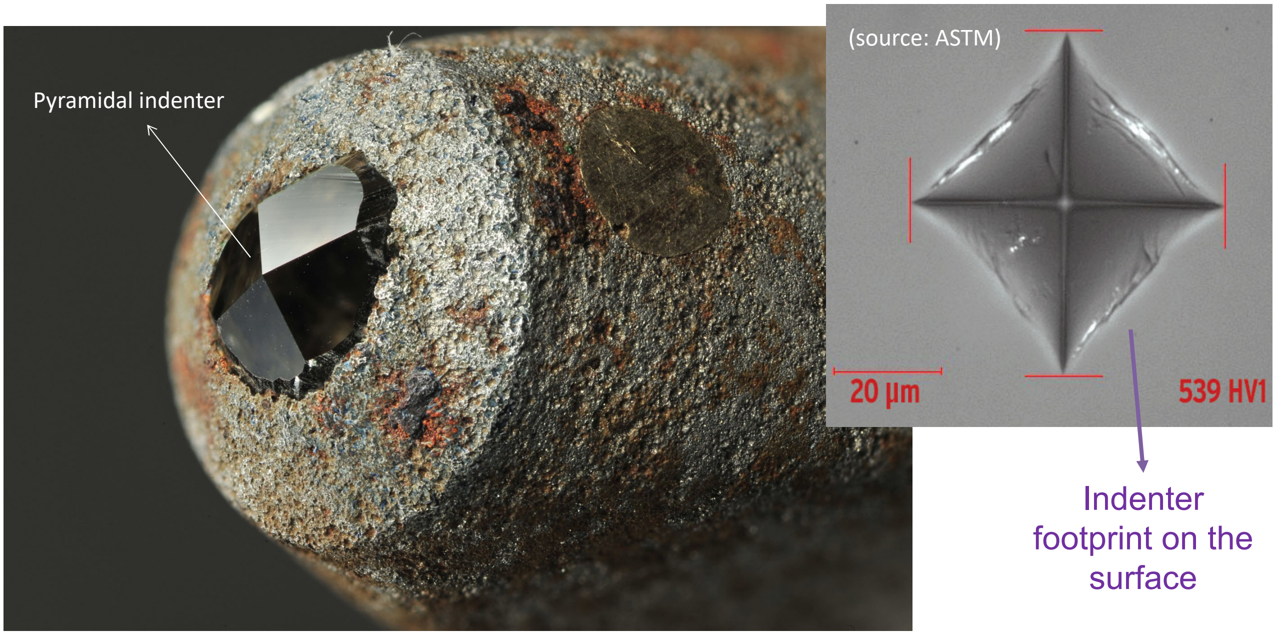
An important parameters defining the strength of a surface is its hardness. It describes 'how hard it is to plastically deform a surface'.

Different methods exist to define it quantitatively (Vickers, Brinell, Rockwell, Knoop) and relies on a similar measurement concept.

The principle is to push an indenter (a hard material) into the material to measure by applying a measured force, the plasticity of the surface.

In practice, the size of the print that the indenter leaves in the material after applying a force, is measured to calculate how much the surface as plastically deformed (permanent deformation) under the application of the force.

In the case of the Vickers Hardness (VH) – which is the most commonly used metrics, the punch made of hard material has a pyramidal shape. The hardness HV is defined by a formula based on the force measured and by the characteristic dimensions d_1 and d_2 of the print that are measured at the end of the experiment.



Source R. Tanaka - http://www.flickr.com/photos/fluor_doublet/6864844960/

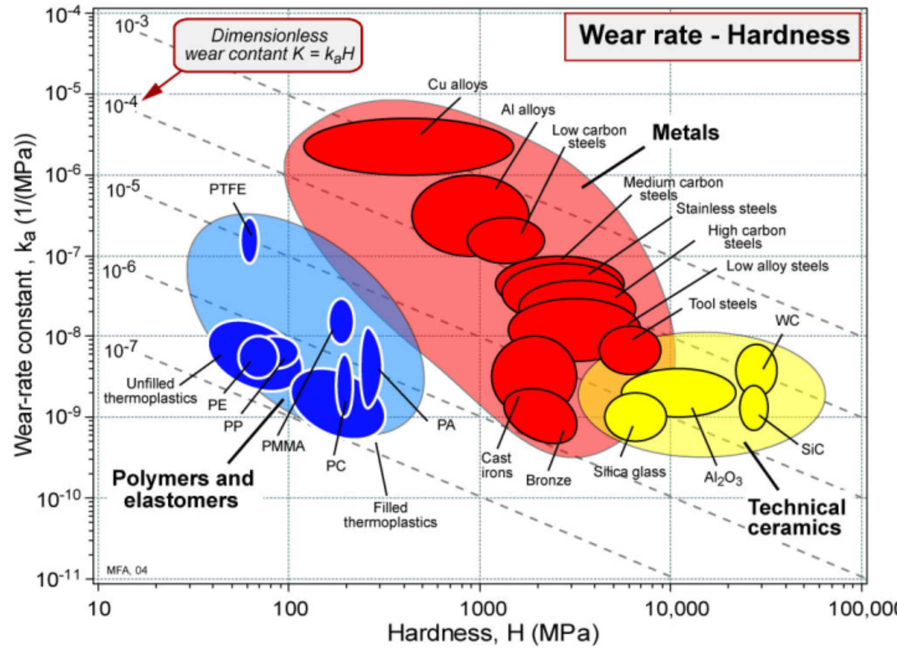
This image illustrates an example of pyramidal indenter and a typical example of a print it may leave on a material surface.

Naturally, the material used for the indenter must be itself significantly harder than the material to test. In a way, it is equivalent to consider that the indenter becomes infinitely stiffer compared to the surface to measure.

Typical materials used for the indenters are diamonds, tungsten-carbide, sapphire, etc.

Hardness versus wear rate plot

Perhaps counter-intuitive: the higher the hardness does not necessarily mean the most wear-resistant!



(source Ahsby)

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Using a Ashby plot (see previous lecture notes), one can visualize how various materials perform with respect to their hardness properties.

As hardness is often a property that is needed to dimension parts in contact one with another (like for instance in the case of roller-bearings), it is convenient to represent it on a common graph with the wear rate that measures how fast a given material wears for a given load.

For the metals, one can see that Cu-alloys are performing poorly as they tend to have the lowest hardness among metals and wear rapidly for a given load. At the other end of the spectrum for metals, 'tool steels' are the compromise in term of hardness and wear rate (not surprisingly as these alloys are precisely designed for this purpose, hence the name 'tool steels').

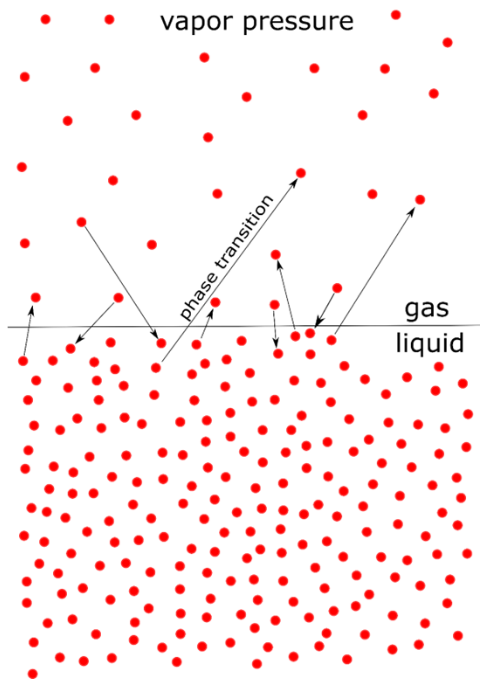
Among the hardest materials, one finds the technical ceramics and in particular Silicon-Carbides and Tungsten-Carbides.

As expected, polymers have low hardness in general (an order of magnitude less than metals), but on the other hand, can have very good wear resistance. Consequently, they can be an excellent material for bearing if the loads are not too high.

Example of a surface functional properties characterization: wetting properties of a surface

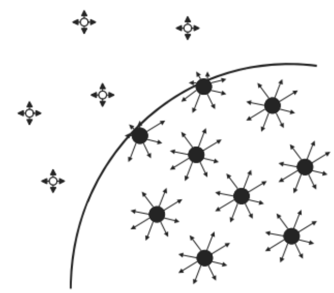
Among the other important physical properties of a surface are their wetting properties ('Propriétés de mouillage').

These properties describe how a surface will behave when a given liquid (that can be of any kind) is poured on it.



Surface Tension

- Molecules of a liquid attract each others
  - Hydrogen bonding forces for polar molecules
  - Van der Waals forces for other molecules



*Imbalance of this attractive force at an interface leads to **surface tension***

An important concept to understand when dealing with wettability is the concept of surface tension. In a liquid, molecules attract each others through different mechanism depending on their polar or non-polar nature.

Clearly, the balance force for an individual molecule is not the same, whether they are located in the volume of the liquid or at the surface defining the interface with a gas or solid phase surrounding the liquid.

The imbalance of attractive forces creates a surface tension that holds things together.

Important definition: Surface Tension

Surface tension has units of **N/m (J/m²)**. If *S* is the total surface are of an interface and *γ* is the surface tension, then the total **energy stored** in the interface is

Surface tension N/m (J/m²)

$$E = \gamma S$$

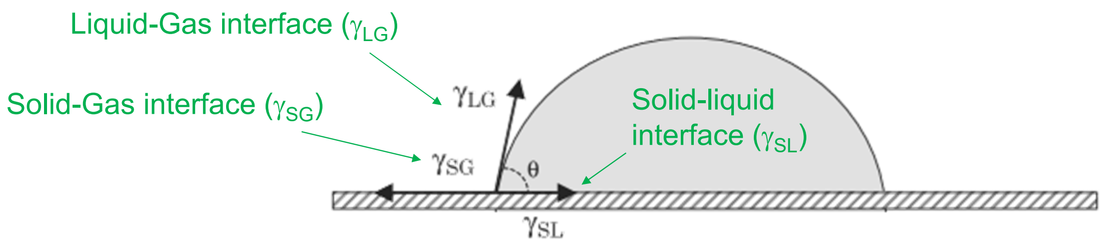
Energy (J)

Surface (m²)

Surface tension is defined as the force requires to break an interface, should we be able to pull on the interfacial layer. This force is defined in N/m.

The energy required (or work) to break the interface can be expressed as the product of surface tension time the surface considered.

Wettability: Young's law



The **contact angle** at the intersection of three interfaces is entirely determined by balancing the **surface tensions** of each interface. ('Equilibrium of forces')

$$\gamma_{\text{Liq}-\text{Gas}} \cos\theta = \gamma_{\text{Sol}-\text{Gas}} - \gamma_{\text{Sol}-\text{Liq}}$$

*If θ > 90, the surface is said to be '**hydrophobic**'*
*If θ < 90, the surface is said to be '**hydrophilic**'*

If we consider a liquid droplet on a surface, we can identify three interfaces between phases: the liquid-gas phase, the solid-gas phase and the solid-liquid one. The surface tensions between these phases can be expressed as forces at the point where the three phases meet. These forces are tangent to the interface considered.

At equilibrium, we obtain the Young's equation. This equation integrates the three characteristic surface tensions and an angle θ, also called 'contact angle'.

If the contact angle is greater than 90 degree, the surface is said to be hydrophobic (i.e. 'does not like water'), while if it is smaller than 90 degree, the surface is said to be hydrophilic (i.e. 'does like water').

Contact angle measurement is a common way for characterizing the wetting properties of a given surface.

Contact angle generally depends on the wetting history!

- Hysteresis in wetting
- Surface are not perfect and has defects
- The liquid has a certain mass / contacts may deform, etc.
- Concept of **advancing** and **receding** angles

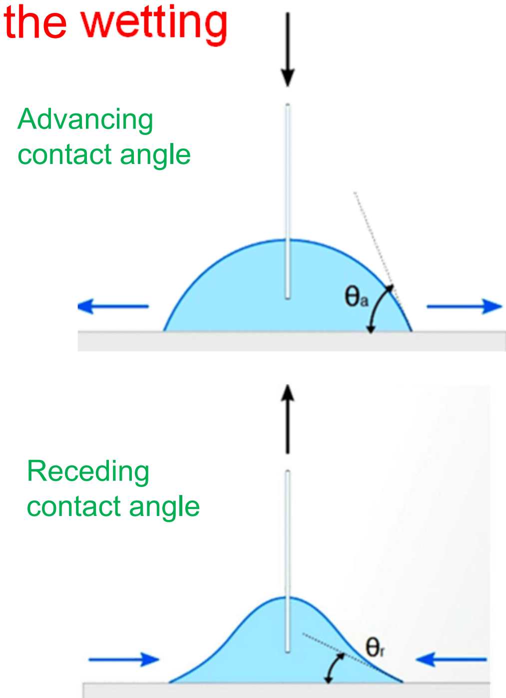


Figure: Fabio, Villa et al. (2014). doi: 10.1615/IHTC15.nmt.009823.

Note that contact angles depends on the wetting history as illustrated in the right figure. To capture this effect, one defines advancing and receding angles (in addition to the classical contact angle defined in the previous slide).

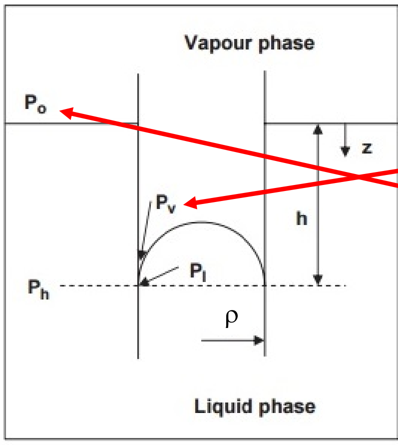
To understand this concept, consider the following experiment. A droplet (top-right) is first deposited on the surface and shows a given contact angle θ. Feeding the droplet with more water will make it inflating and the contact angle will first increase up to a certain value, θ_a until the droplet starts growing homogeneously, without further change of the contact angle.

If now, the water fed into the droplet is gradually sucked out, a new contact angle θ_r is observed in steady mode. This contact angle is called the receding contact angle.

The difference between the two angles, advancing and receding, defines the wetting hysteresis.

Change in vapor pressure due to contact angles

- Kelvin equation



(source Wiki)

$$\ln \frac{p_v}{p_0} = \frac{2\gamma V_m}{\rho R T}$$

Surface tension

Molar volume

Saturated vapor pressure

Meniscus radius

Universal gas constant

Temperature

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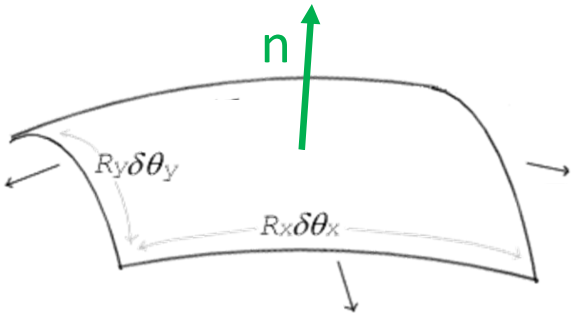
Related to surfaces and to how water behaves on surfaces is the change in vapor pressure due to contact angles. This phenomenon are responsible for capillary effects.

The Kelvin equation takes into account the equation of state that models the condensation of water as a function of pressure, temperature and volume. (R is a constant and γ the surface tension between the liquid and the surface considered).

In the figure above, the water meniscus in the capillary (tiny tube) denotes a material contact that has a hydrophobic behaviour, hence a convex profile. Due to this wetting behavior, if the capillary is small, the liquid surface will be moving downward compared to the liquid surface in a large liquid container. Conversely, if the material has a hydrophilic behavior, the meniscus will have a concave profile, and water will crawl up in the capillary. The smaller diameter of the capillary, the higher the water will move up.

This equation is used to access how water can move in capillaries, e.g., sub-mm tiny channels, in which water can crawl up againt gravity due to local effects of surface tension on the vapor pressure.



Laplace-Young equation

- States the difference of pressure at the interface between a gas and a liquid

$$\Delta p = \gamma\left(\vec{\nabla}\cdot\vec{n}\right) = \gamma\left(\frac{1}{R_x}+\frac{1}{R_y}\right) = 2\gamma H$$

Surface tension: γ

Normal to the surface: $\vec{n}$

Mean curvature: H

Difference of pressure across the interface: Δp

Principal radii: R_x, R_y

More generally, using these angles of contact and the radius of curvatures of the sphere and the droplet, we can estimate the difference of pressure (Laplace Young equation) accross a water interface.

Illustration on how processing can be used to engineering surface at the nano-scale

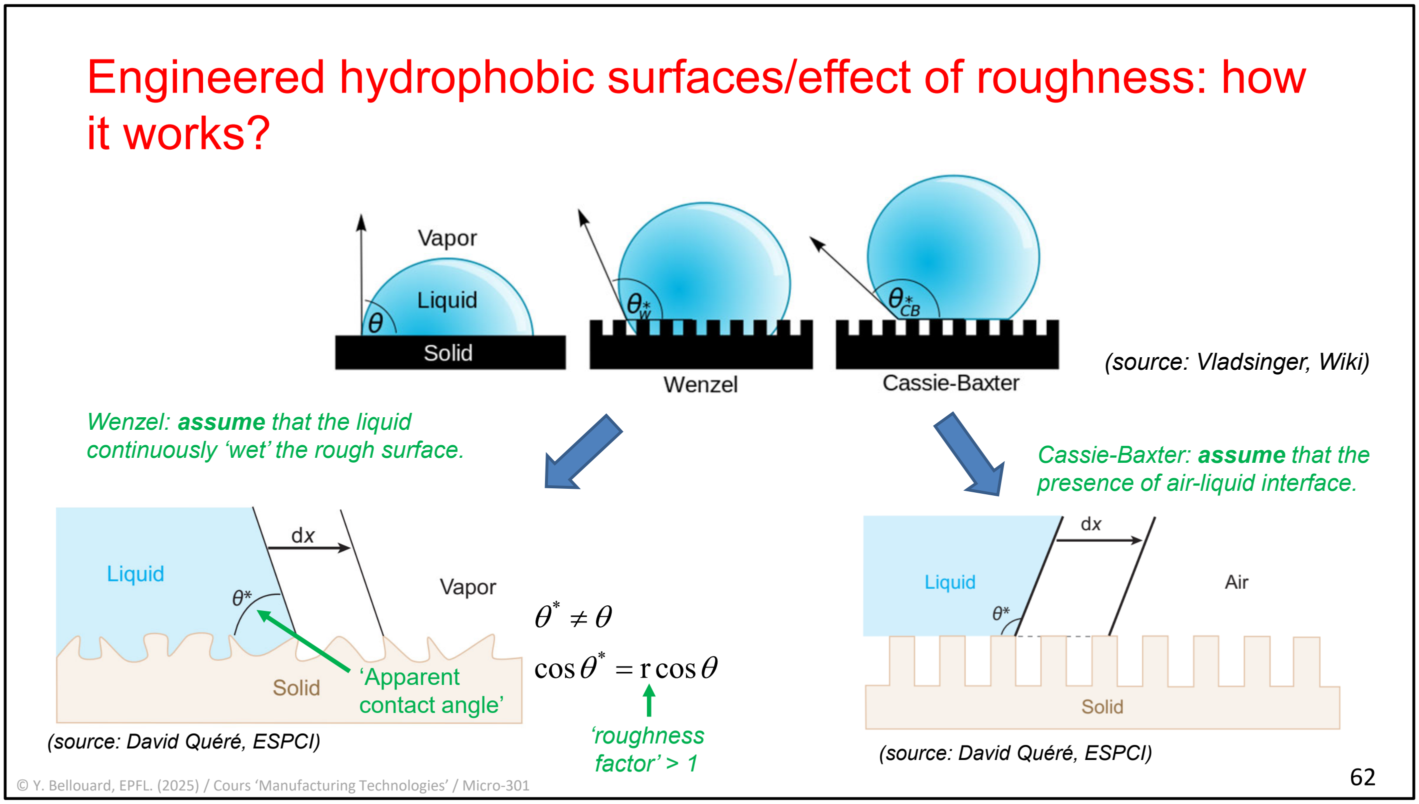
With the recent advances in micro- and nano-manufacturing, one can now 'engineer' surfaces to achieves novel properties.

Walking on water...

Bush JWM, Hu D, Prakash M. 2008. The integument of waterwalking arthropods: form and function. *Adv. Insect Physiol.* 34:117–92

A source of inspiration is nature. For instance, some arthropods are capable of walking on water, despite their weight.

The explanation of this phenomenon is related to the find cilia on the insect legs that cause a hydrophobic contact with water. The surface tension forces are then sufficient for preventing the water to rupture and allowing the insect to 'walk on water', just like walking on a carpet.



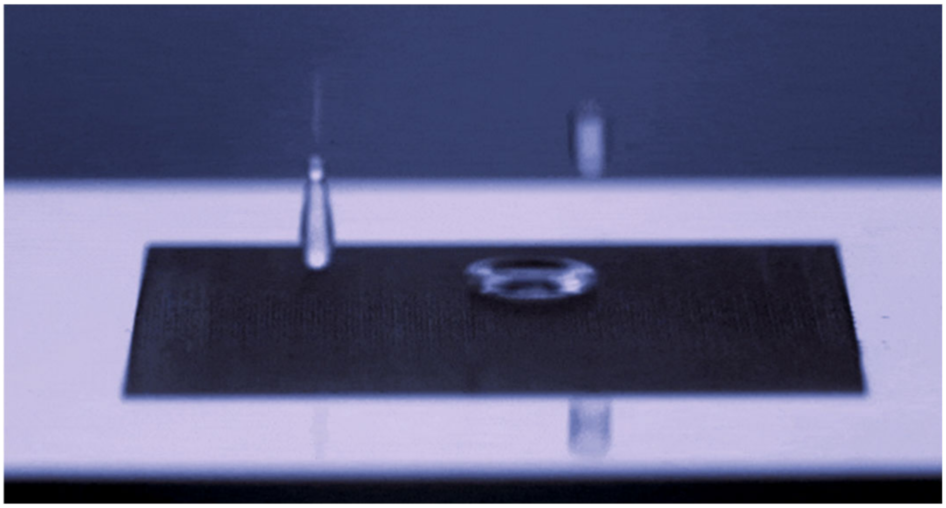
One way to engineer a surface of a solid to change its wetting behavior is to create '*nano-texture*' (smaller than the droplet sizes) as illustrated above. The fine analysis of the wetting properties is complex and a hot topic of research on its own.

Different wetting regimes have been observed.

In the case of the *Wenzel* regime, interstices on the surface are wet, while in the case of a *Cassie-Baxter* regime, the droplet 'sits' on the asperities.

Texturation of a surface with nanoscale resolution can be achieved by various means, ranging from laser surface texturing to lithographic means. The interest for such textures is for instance to turn an hydrophilic surface into an hydrophobic one, and doing so, to prevent water from wetting the surface which is useful for achieving self-cleaning surfaces. As a water droplet impact an hydrophobic surface, it can carry along dust on the surface as it rolls on the surface, achieving a cleaning action.

What about manufacturing? Engineered surfaces?

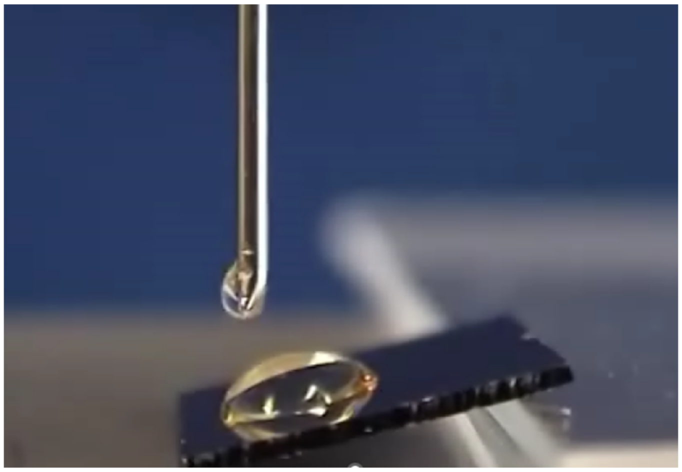


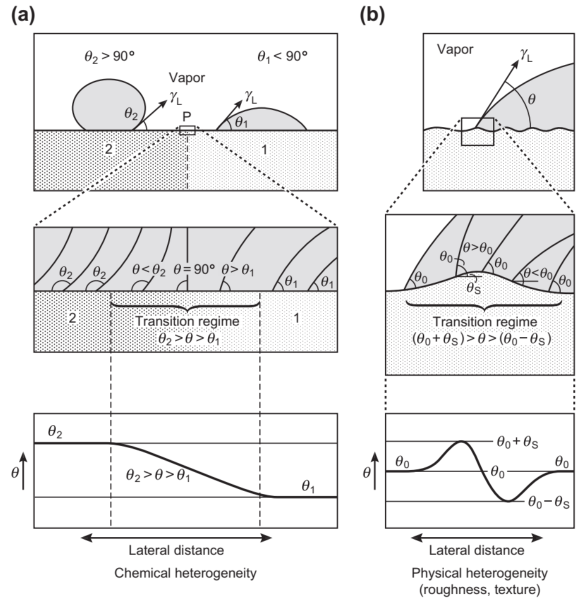
(source: Univ. of Rochester, Prof Guo) https://youtu.be/FLegmQ8_dHg

This example illustrate how the wetting behavior of a surface can be changed by engineering the surface that would otherwise be wetting.

In this example, nano-texturation of the surface using laser creates a periodic pattern that makes the surface highly hydrophobic (water droplets just bounce off the surface).

Wettability gradient





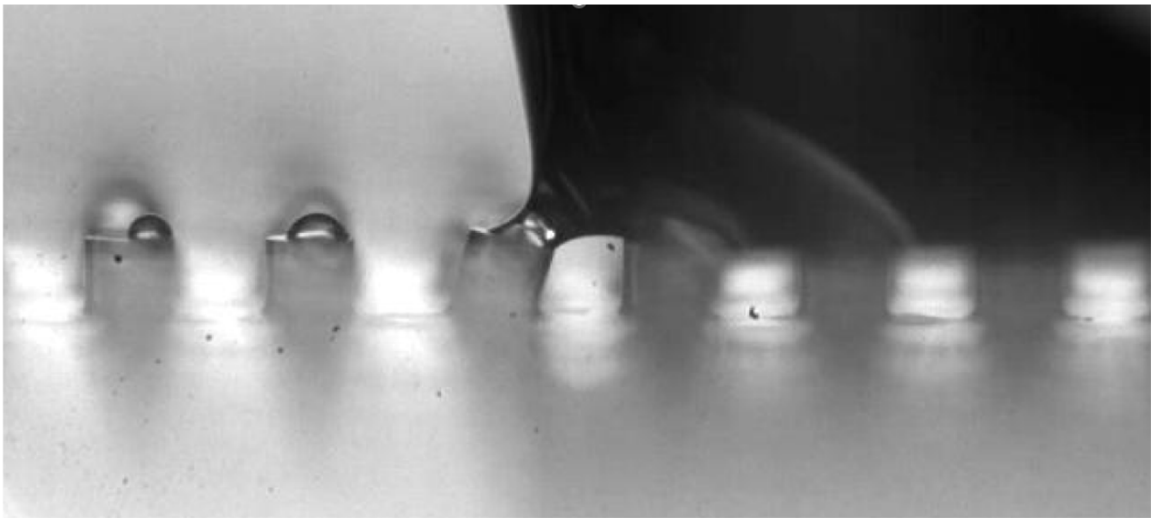
(Source D-MAVT / ETHZ)
https://youtu.be/puYo9w4cuOc

(source: J. Israelachvili, Adhesion and Wetting Phenomena)

The wettability can vary across the surface and so the contact angles. This can be achieved by either modulating the material properties on the surface (a) or by having a periodic or known texture on the surface (b).

These two modalities can be used to engineer wettability gradients. The video illustrates how such gradients can be used to make water droplet to move against gravity for instance.

Illustration of research at EPFL



(Courtesy Prof. Kolinsky, EMSI) / PNAS
https://doi.org/10.1073/pnas.2008683117

Illustration of a droplet 'walking on soft pillars' being deformed by surface tension.

Another illustration on research related to engineering surfaces. This example, from the laboratory EMSI at EPFL, shows how PDMS micropillar deforms as the droplet moves on the surface.

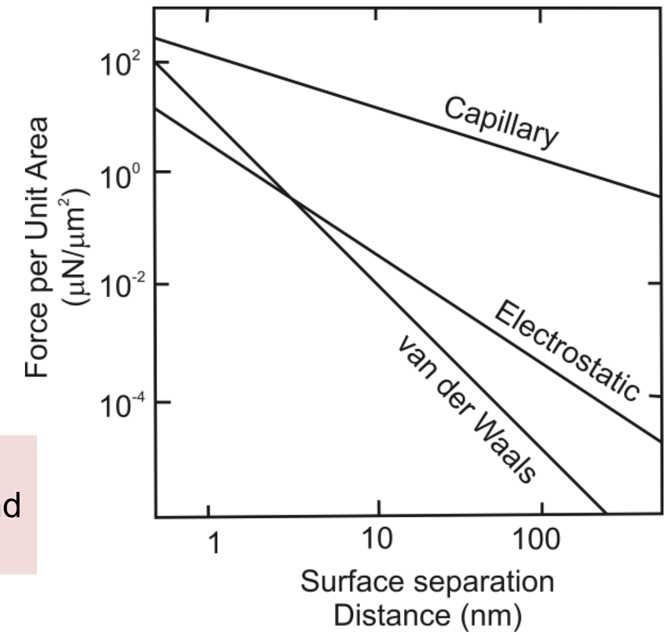
Adhesion properties of surfaces

So far, we have discussed about topology and some physical properties of surfaces, such as mechanical and optical properties.

Let us now examine the adhesion properties.

Adhesion force: ‘illustrative’ comparison

- Example for two SiO_2 surfaces (U=1V, AH = 1.7eV, γ = 73 dyne/cm.)



The nature of the adhesion forces changes with the type of surfaces and the surface separation distance!

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Adhesion forces scale with the distance between the two surfaces being brought close one to another. Here, a comparison is made for two SiO_2 surfaces that are brought close one to another.

In normal conditions (i.e., 50% humidity, etc.), there are typically three forces that come into play. The ratio of these forces changes with the interdistance between the surfaces.

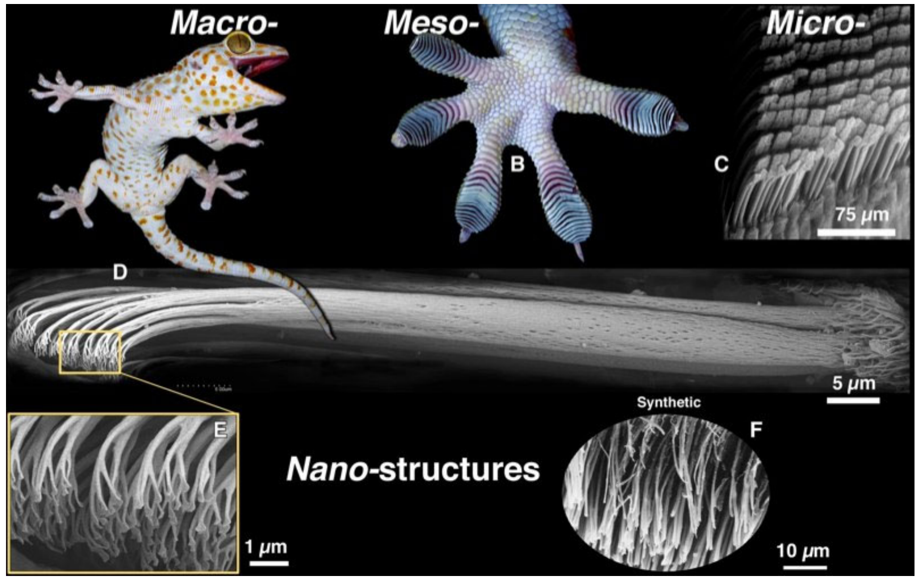
One can appreciate the ratio between forces as the surface separation distance is changed.

Down to the microscale separation, capillary forces (resulting from surface tension properties – see previous slides) domininate. In a normal environment (liveable for human being), we should keep in mind that there is always water present as a vapor. This water can locally condensate as two surfaces are brought together and turn into capillary attraction effects.

As the surfaces are brought closer together, electrostatic forces resulting from the presence of surface charges will dominate. As the distance is even further reduced, atomic interaction forces, so called van der Waals forces, will dominate.

Note that the simulation above is considering a very simple case. Perfectly flat surfaces of silicon dioxide. Investigating interaction forces on more complex cases, requires a refined analysis and advanced tools, such as AFMs, discussed before.

From the Gecko mystery… (a multiscale interaction)



(pictures source: K. Autumn, Lewis & Clark College, USA)

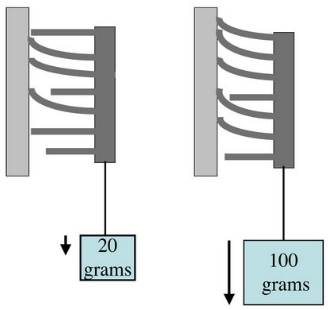
The 'gecko' (scientific name: gekkota) is capable of walking on vertical surfaces even if they are very clean, which is quite remarkable, considering the fact that the gecko is not particularly 'lightweight'. The animal does not fall because of the particular adhesive properties of his paws.

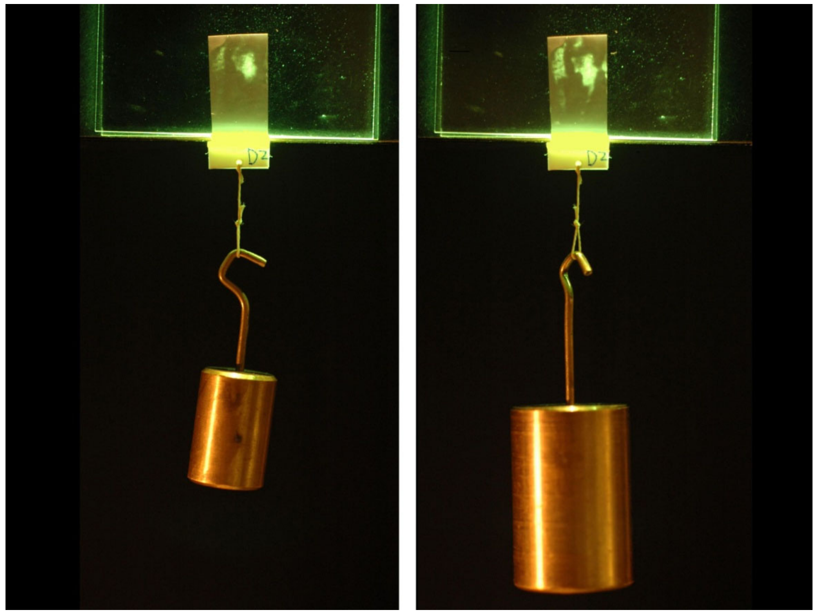
This remarkable ability was a mystery for some time and has been recently understood. It originates from the delicate and multiscale structure of his paws. Indeed, as illustrated above, the structure of the paws is complex, starting from microscale cilia, themselves made of nanostructures at his tips. These nanostructures get in intimate contact with surfaces, causing strong, short range, adhesion forces (van der Waals) to come into play. When the gecko walk, it gently changes the orientation of these structures (called spatulae), causing a change of the adhesion forces and allowing the animal to attach/detach his paw from the surface.

This observation has triggered numerous research worldwide to mimic this remarkable behavior, for instance to invent new adhesive principles.

Here is an educational video on the topic: (Public Broadcasting Service - PBS)
https://youtu.be/r5cqJTU5D5I?si=hC9CY1yNECt2M9Fn

To new adhesive principles….



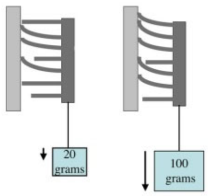


(source: Ron Fearing, UC, Berkeley)

For instance, in this new adhesive principle (from Prof. Ron Fearing at UC Berkeley) mimics the gecko feet behavior. It consists of a nanostructure layers with long pillars.

The adhesion force is very high in the vertical direction and very low in the perpendicular direction. It can hold strong shearing force, but not withstand gentle pulling force.

To new adhesive principles….

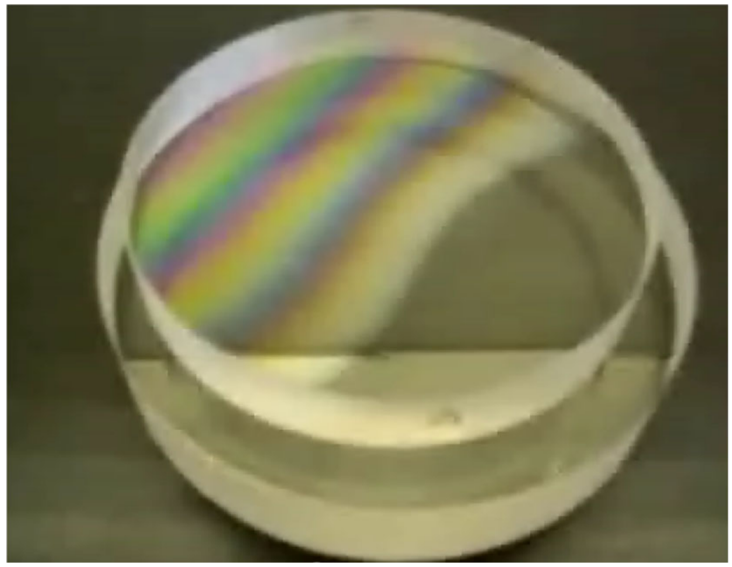


(source: Ron Fearing, UC, Berkeley)

This video illustrates the idea of using engineering nanoscale surface to create new adhesive based on van der Waals forces.

In industry: The optical contacting method

- Surface are so smooth that van der Waals surfaces prevails (sub-nm contact distances)



Source:
https://youtu.be/se3K_MWR488
https://youtu.be/hxGMA0nxdEU

Optical contacting is an important assembly method for many optical components. It gives the possibility to assembly optical interfaces *without* the use of glue or others adhesives.

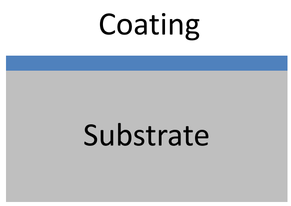
The working principle is to bring two surfaces (with excellent surface quality) as close as possible to reach a point where the interfacial forces (van der Waals) are significant enough to old the two objects together.

Even though the force are very localized, their intensity can be sufficient to old bulky objects together. This is an illustration on how nanoscale effects can have macroscale importance and can be used in practice.

A few words about surface treatment

Surface treatments: coatings

- ✓ **Two separate materials**, the substrate and the coating
- ✓ The coating **does not react** with the substrate
- ✓ Lot of available processes to deposit a coating



- **'Through a liquid phase'** : Electrolysis, Chemical, Painting, etc.
- **'Dry method'**: Physical Vapor Deposition, Chemical Vapor Deposition
- **'Thermal'**: flame, plasma, arc, explosive, etc.
- **'Beam assisted'**: laser, implantation, etc.
- ... and many others...

In a slide 35, related to mechanical properties, we have seen that a coating can have a very significant influence on the material properties. In the example before, we saw an illustration on how a stainless steel with a nitride-processed surface can outperform a plain stainless steel.

Coatings added to a material can have many different purposes, like improving mechanical properties, corrosion properties, optical properties, biological resistance, or simply for decorative purposes.

They represent an essential part of manufacturing and finishing processes. Surface treatments are manifold. A course alone would not suffice to consider all of them.

Here, we just provide a bird-eye view of the main generic methods used to produce these enhanced surface properties.

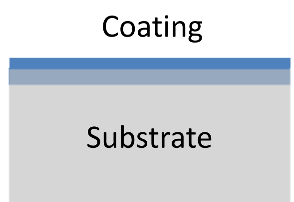
Coating can be divided into four main categories, for which it is useful to understand the main features and the general working principle.

A first group of coating processes are characterized by the use of two separate materials, the coating and the substrate, that do not react one with another. This first group includes paintings, PVD and CVD process, flame or laser-assisted deposition processes among others.

As there is no reaction between the two materials, these kinds of coatings may be subject to delamination in certain cases.

Surface treatments: conversion methods / reacting methods)

- ✓ **Two materials**, the feed-material reacts with the substrate to form a new compound.
- ✓ Localized chemical reaction



- **'Electrochemical conversion'** : anodizing of aluminum, titanium, zirconium... / Coloration of steel / Sulfurization in salt bath, etc.
- **'Chemical conversion'**

A second group of coating processes concerns also the case where two distinct materials are used, but this time, so that the two materials *react* one with another forming a new compound on the surface.

This is notably the case of anodization processes that are commonly found for coloring metals (see illustrations on the next page).

Examples

Anodized aluminum

(source Wiki)

Dye + anodization

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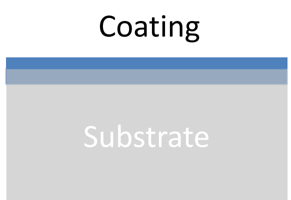
Two examples about the use of anodization processes to color metals like aluminum or magnesium. Here, the functional objective is aesthetic.

Surface treatments: thermo-chemical

- ✓ **Two materials**, the feed-material diffuses into the substrates (and may or may not form a new compound).
- ✓ Diffusion process



- **'Metal/ metalloid diffusion'** : chrome ('chromisation'), tantalum, vanadium, ...; ion mixing, laser-induced mixing,...; cementations, carbonitruration, etc.

A third category includes processes that will also involve two materials, the feed-material and the substrate, but this time involving a *diffusion* process.

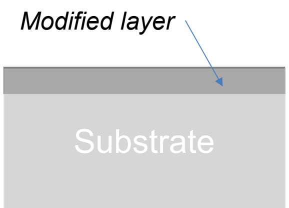
While the feed-material diffuses into the substrate, it does not necessarily react with it, and ions of the fed-material are simply *penetrating* inside the substrate.

Example of processes involving diffusion is the one used to obtain the Gorilla(tm) glass (from Corning), famous for its higher mechanical resistance and widely used for smart phone displays and displays in general.

In this example, the ions that diffuse inside the glass (an alumino-silicate) pre-stress the upper layer and induce a compressive stress that dramatically improve the mechanical resistance of the glass.

Surface treatments: thermal and mechanical, textured localized transformation

- ✓ **One material only** - the substrate
- ✓ **Local** process of mechanical hardening (‘écrouissage’), thermal annealing, texturing.



- **Mechanical hardening** : hardening, shock peening (‘grenaillage’), laser-shock peening, etc.
- **Localized thermal treatment** : local quenching, flame, laser-annealing, ...
- **Nanoscale texturing:** laser-induced ripples, ...

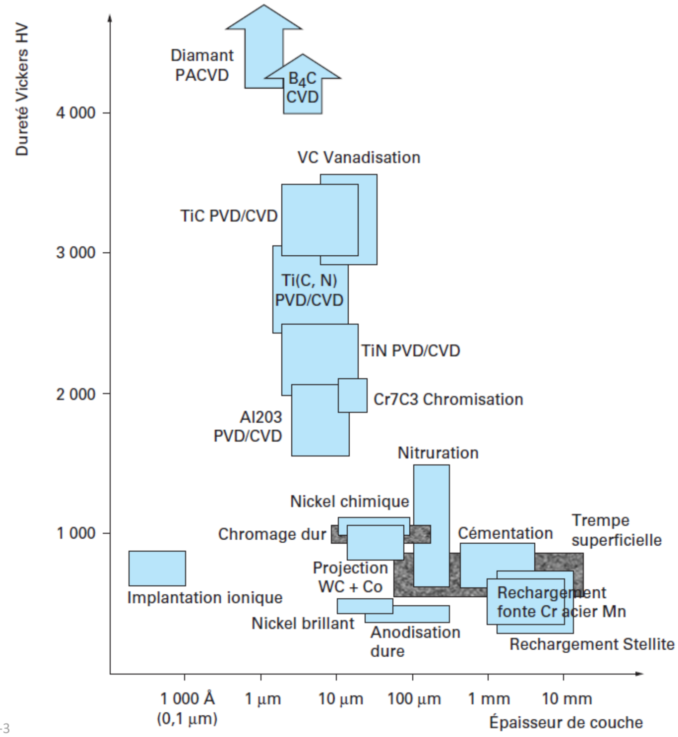
Finally, a fourth category, very important one, is one no additional material is used, but simply relies on localized treatments of the material surface. Mechanical hardening, thermal annealing or texturing are among the typical process that fall in this category.

For instance, in the case of shock peening, hard spheres bombard the surface causing localized plasticity and hardening in metals by inducing defects such as dislocations.

Nowadays, a variant of this process involves the us of laser to locally create shock-wave by vaporizing rapidly a sacrificial layer.

Other processes involve localized heat treatment, such as laser annealing that can for instance modify the grain size locally or induce localized precipitates.

Surface treatments to improve the hardness



(Source R. Gras, Techniques de l'ingénieur)

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This graph illustrates how various surface processes can dramatically increase the hardness (Vickers) of a material.

Particularly spectacular is the fact that even if the coating is very thin (a few microns only!), it can increase the hardness by an order of magnitude. In gray are represented surface treatment where no material is added.

Wrap-up

Key points to remember

- Importance of surface topological information and physical properties.
- Relation between surface and functionality
- How topography affects functional properties
- How do we measure the topography surfaces
  - Surface metrology principles
  - Roughness, waviness
  - Abbott-Firestone curves (bearing ratio)
- Role of manufacturing in engineering surfaces

Above are listed the main topics covered in the lecture and the key points to remember.

We will consider that these points as understood by the students at the end of the class.

'Lexique manufacturing'

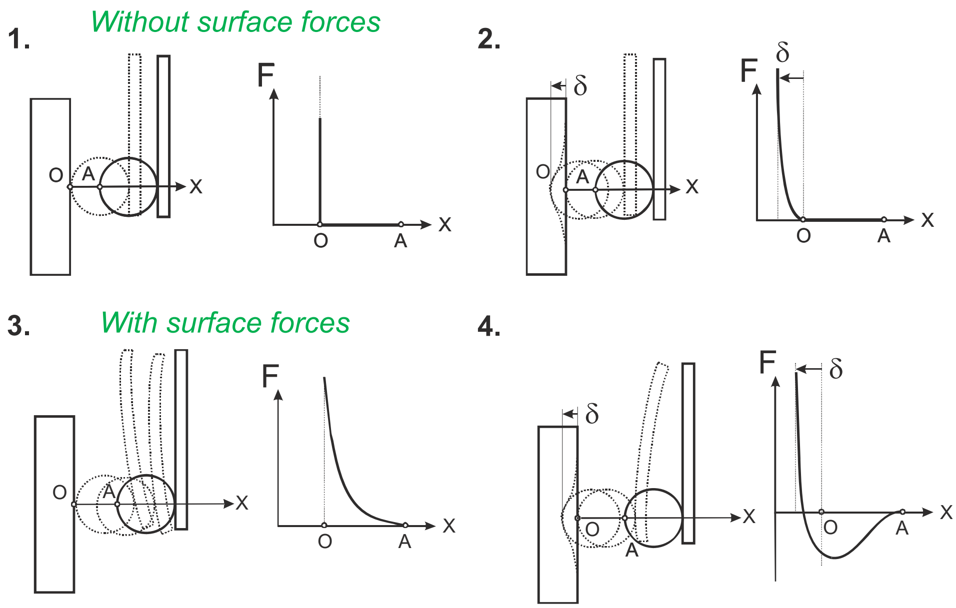
English (UK) > French

- Waviness: *Ondulation de surface*
- Roughness: *Rugosité*
- Toughness: *Tenacité*
- Wear: *Usure*
- Anodized: *~ Eloxé* (néologisme vient de 'Electricly oxidized') / Anodisé*
- Dye: *Colorant*
- Diffusivity: *Diffusivité*
- Scattering: *Lumière diffuse*
- Specular reflection: *Réflexion spéculaire*
- Grazing incidence: *Incidence rasante*
- Coatings: *Couches minces déposées*
- Diamond turning: *Tournage pointe diamant*

Annex on adhesion properties (to explore the topic further)

Let us know explore the surface adhesion properties.

Physics of adhesion (introduction)



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When objects are put in contact with a surface various situations can be considered. Here, we consider a sphere attached to a cantilever and look at the force that is applied on it resulting from its interaction with the surface.

Case 1. No adhesion forces are present and infinitely rigid surface is considered. This is an idealized case far from reality.

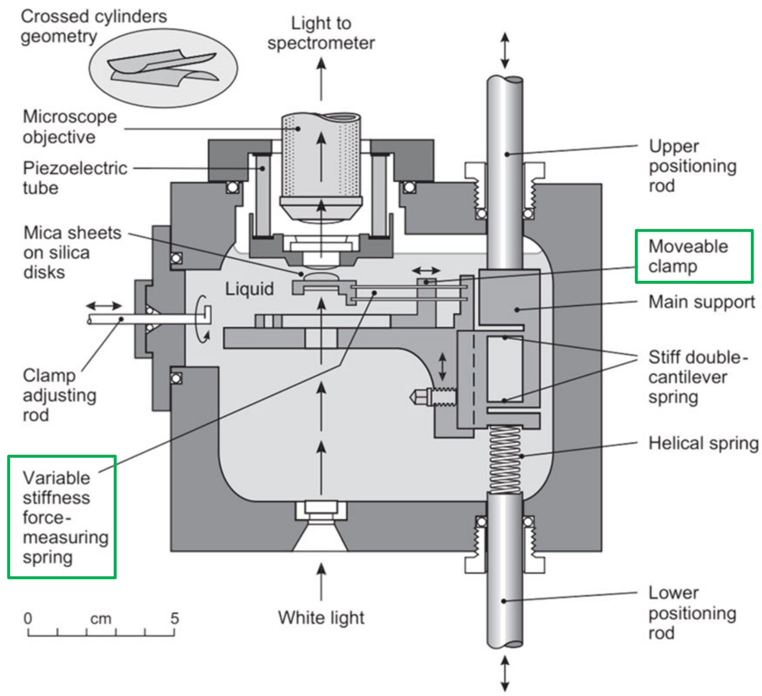
Case 2. A more realistic case: the surface does deform under the action of the force applied. There are no adhesion forces present.

Case 3. A repulsive force is present. As the cantivler moves toward the surface, the force that tends to push it away from it keeps on increasing.

Case 4. The most common case: adhesion forces are present. As the object is brought closer and closer to the surface, the sphere fills a force that attracts it to the surface. As it gets into contact, the surface deforms. This situations is a classical situation observed at the micro-scale.

Measuring surface forces

- ‘Large surface’
- Atomically smooth < nm roughness
- Typ. force resolution. 10^{-8} N



(source: J. Israelachvili, Adhesion and Wetting Phenomena) 84

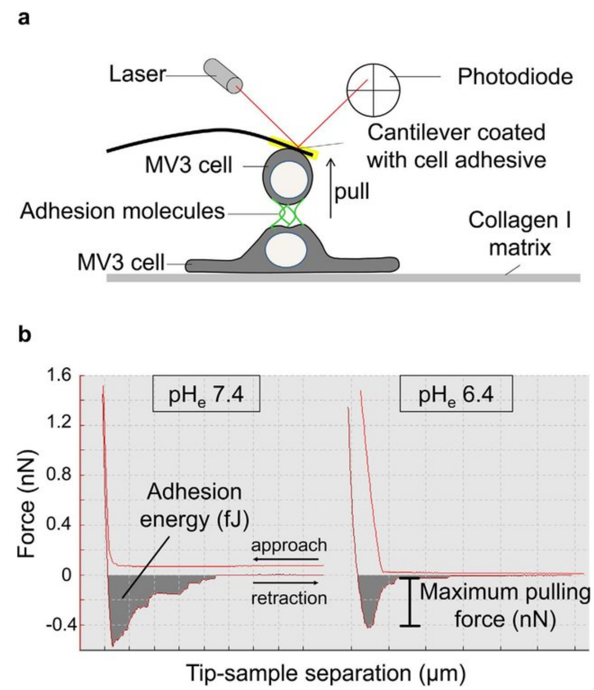
There are various methods for measuring surface adhesion properties. However, as the amplitude of these forces can be quite small, it requires highly sensitive metrology principles.

Furthermore, not only the force needs to be measured, but to be meaningful (and transposable to a force/surface unit, it also requires to observe or to know the actual contact surface.

The example above studies contact forces in a crossed-cylinders geometry.

Illustration of cell-cell adhesion

Schematic illustration of cell-cell adhesion analysis using AFM. (a) Single cell force spectroscopy. A single melanoma cell (MV3) attached to a flexible cantilever is brought into contact with another adherent melanoma cell of the same kind seeded on collagen



Hofschröer, V., et al. Extracellular protonation modulates cell-cell interaction mechanics and tissue invasion in human melanoma cells. Sci Rep ***7,*** *42369 (2017). https://doi.org/10.1038/srep42369*

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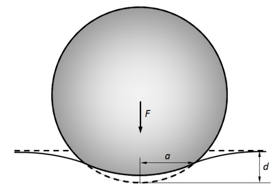
The method of using functionalized AFM is generic and is used in very diverse situations, from industry to research.

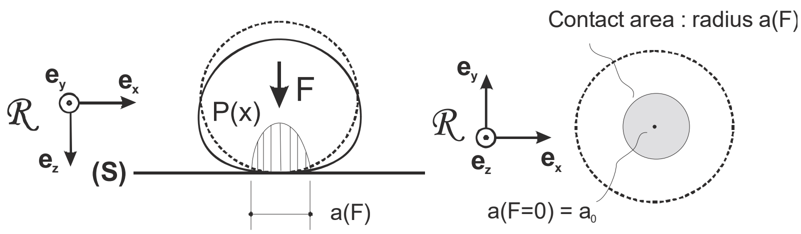
Here is an example in the context of bioengineering. The zone in gray is representing the 'adhesion energy' and tells, how a surface is adhesive for a given set of experimental conditions.

Contact surfaces

- Importance in manufacturing => Choice of material in contact areas
- *Without adhesion forces*
  - Hertz model (sphere on a plane / sphere deforms)
  - [variation: Sneddon model (sphere on a plane / plane deforms)]



(Sneddon model / sphere on a plane, assuming the plane deforms)



(Hertz model / sphere on a plane, assuming the sphere deforms)

AFM measures how much force is applied on the tip surface. Therefore, it measures a distribution of forces (i.e. a pressure) rather than point-forces. These distributions of forces depend on the surface of contact.

To be applicable, Hertz analysis supposes that:

- The strains are small and within the elastic limit.
- The surfaces are continuous and non-conforming (implying that the area of contact is much smaller than the characteristic dimensions of the contacting bodies).
- Each body can be considered an elastic half-space.
- The surfaces are frictionless.

It is important to estimate what is the contact area between a tip and the surface.
To simplify here, we consider a sphere on a plane.

Various models exist.

The first one considers the case where there is no adhesion force. As the sphere is pushed on the surface, the contact are evolves as the sphere is deformed. Hertz model considers a sphere that deforms and a plane infinitely rigid while Sneddon model considers the case of a plane that deforms and a sphere that does not.

Sphere on a plane (Hertz/Sneddon)

- *Assumes elastic deformation*
- *Small deformation*

Pressure distribution

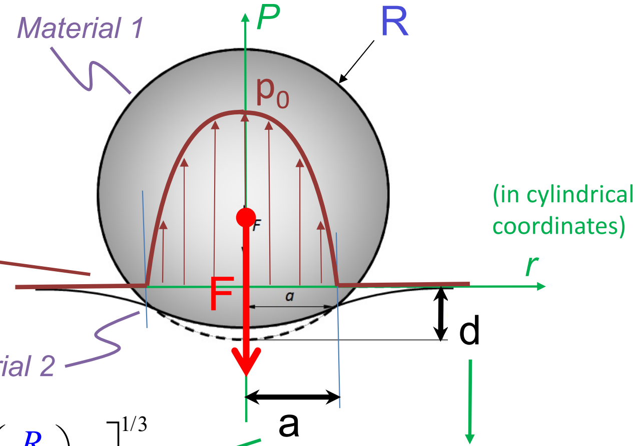
$$p(r) = p_0\left[1-\left(\frac{r}{a}\right)^2\right]^{\frac{1}{2}}$$

Effective Young modulus of the elastic interface:

$$\frac{1}{E^*} = \left(\frac{1-\nu_1^2}{E_1}\right) + \left(\frac{1-\nu_2^2}{E_2}\right)$$

Material 1 *(E_1, ν_1)* — *Material 2* *(E_2, ν_2)*

(ν Coefficient de Poisson)



$$a \simeq \left[\frac{3}{2}\left(\frac{R}{E^*}\right)F\right]^{1/3}$$

$$d \simeq \frac{a^2}{R} = \left[\left(\frac{2}{E^{*2}R}\right)F^2\right]^{1/3}$$

$$F = \frac{4}{3}E^* R^{\frac{1}{2}} d^{\frac{3}{2}}$$

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Let us look into the Hertz contact problem, for a sphere on a plane. This is a classical example that helps understanding the basic concept behind Hertz contact mechanics.

The important assumption is to assume elastic deformation. Hertz considers a parabolic distribution of pressure around the contact point. The pressure is maximum in the middle and zero at the boundary of the contact point.

Based on this assumption, the contact area of the deformed surface can be calculated as well as the penetration depth. One has to be careful to keep in mind that here the penetration depth considers an elastic deformation.

The Hertz model is useful for estimating the maximum stress applied locally and to verify if the materials consider for the design stay within their appropriate load limits.

Sphere on a plane (Hertz/Sneddon)

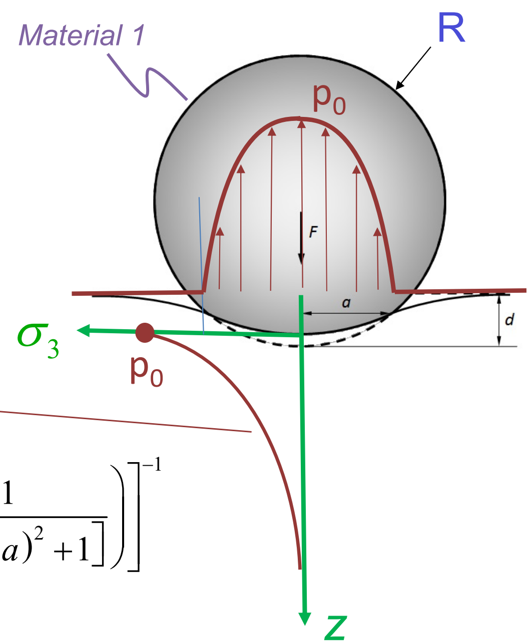
Principal stress (symmetric = cylindrical geometry)

$$\boldsymbol{\sigma} = \begin{pmatrix} \sigma_1 & 0 & 0 \\ 0 & \sigma_2 & 0 \\ 0 & 0 & \sigma_3 \end{pmatrix} = \begin{pmatrix} \sigma_\perp & 0 & 0 \\ 0 & \sigma_\perp & 0 \\ 0 & 0 & \sigma_\parallel \end{pmatrix}$$

$$\sigma_3 = \sigma_\parallel(z) = -p_0\left[1+\left(\frac{z}{a}\right)^2\right]^{-1}$$

$$\sigma_1 = \sigma_2 = \sigma_\perp(z) = -p_0\left[(1+\nu_2)\left(1-\left|\frac{z}{a}\right|^2\tan^{-1}\left(\left|\frac{z}{a}\right|\right)-\frac{1}{2\left[(z/a)^2+1\right]}\right)\right]^{-1}$$

Principal shear stresses: $|\tau_1| = |\tau_2| = \tau_{max} = \left|\frac{\sigma_\perp - \sigma_\parallel}{2}\right|$

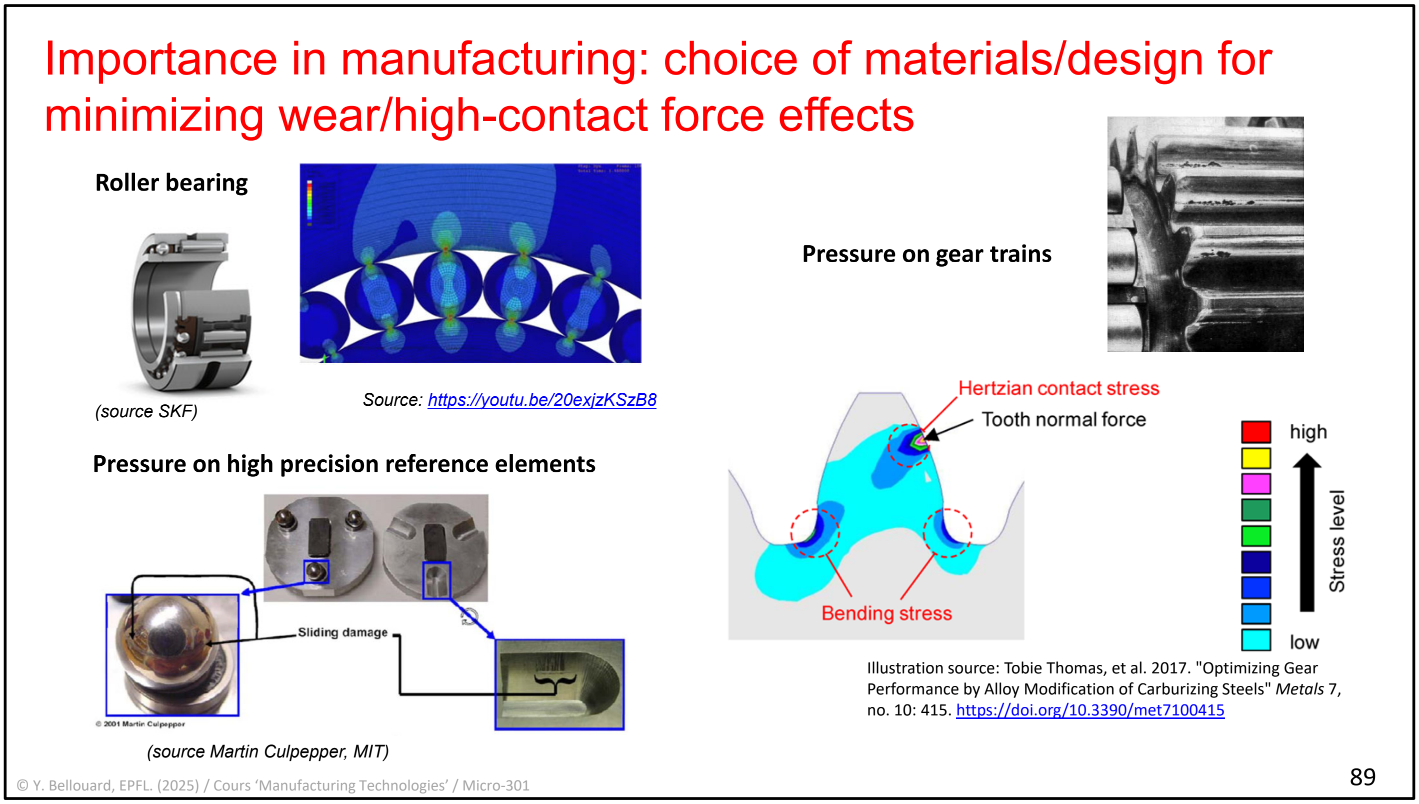


Similar models exist for other contact configuration (cylinder on a plane, sphere on a sphere, etc.)

The principal stress can be deduced from the pressure distribution. The stress state has a cylindrical geometry.

If z defines the vertical axes, with its 0 at the interface between the two materials, the principal stress along z is maximum at z=0 and decays as z increases.

The shear stresses are, and logically, maximum at the boundary of the contact area.



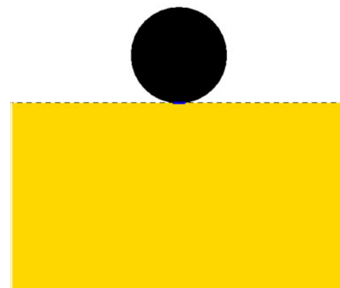
Hertz contact pressure is important for dimensioning important components, such as gear trains, high-precision positioning elements (kinematics coupler), roller bearing, etc.

It helps choosing appropriate materials and defining maximum operating loading conditions.

Models of adhesion forces

Hertz contact theory assumes no contact forces. The presence of adhesion forces modifies the contact area and hence, how the stress is distributed and evolves.

- Johnson-Kendall-Roberts (JKR)
  - Contact area (with adhesion forces **in** the contact area only)
- Derjaguin-Müller-Toporov (DMT)
  - Contact area (with uniform adhesion forces)
- Maugis / Dugdale
  - Contact area (with annular adhesion forces)



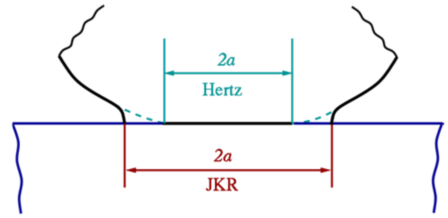
(illustration Wikipedia)

The case where adhesion forces are present is more complex and has been studied by various research teams.

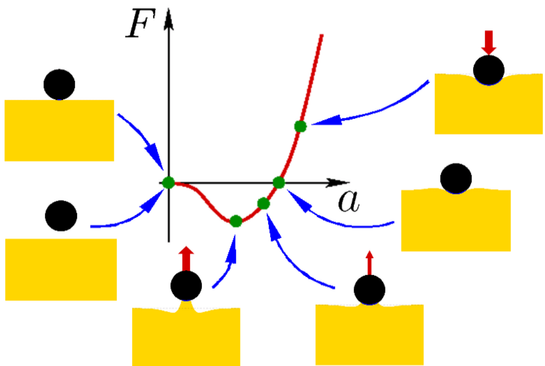
There are three main models with different level of sophistication. We will not review in details these models since it goes beyond the scope of this course.

We just examine the general concept of one of this model in the next slide.

Johnson-Kendall-Roberts (JKR) model



- Adhesion in the contact area only



(source Wikipedia)

Surface energy

$$a^3 = \frac{3R}{4E^*}\left(F + 6\pi\gamma R + \sqrt{12\pi\gamma RF + (6\pi\gamma R)^2}\right)$$

Hertz mode (sphere-sphere)

Effect of adhesion forces

$$\frac{1}{R} = \frac{1}{R_1} + \frac{1}{R_2}$$

(for two spheres R_1 and R_2)

When no force is applied (F=0):

$$a^3 = \frac{9R^2\pi\gamma}{4E^*}$$

Pull-off force: $F_c = -3\gamma\pi R$

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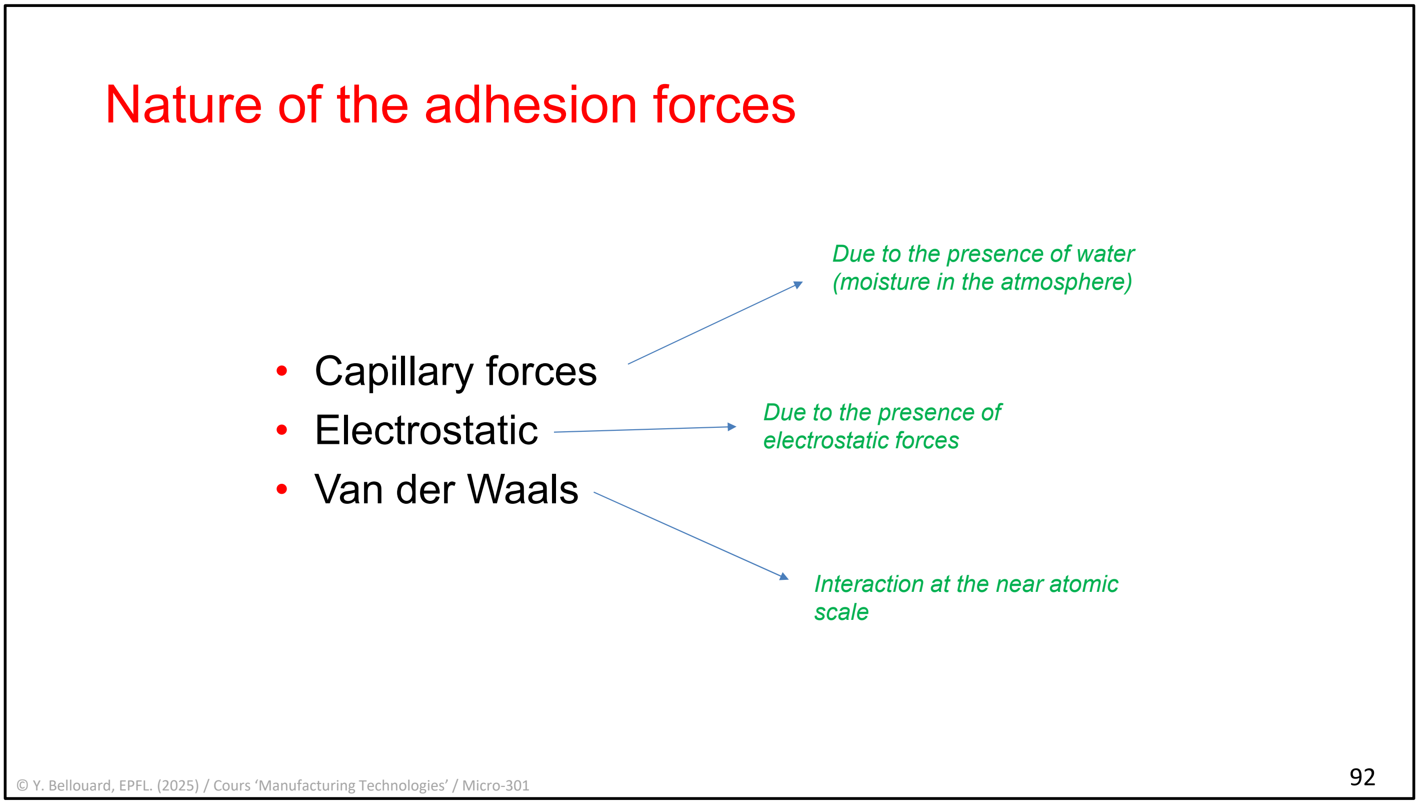
Johnson-Kendall-Rogers (or JKR) considers adhesion forces.

It estimates the contact area as the surface deforms due to the adhesion forces represented by the red curve. As the object moves inside the material (for which the deformation is exagerated for illustrative purpose, the contact area dynamically evolves.

What differs from the Hertz model is the area of contact that is now dependant also on the surface energy.



There are three main adhesion forces present at the micro-scale.

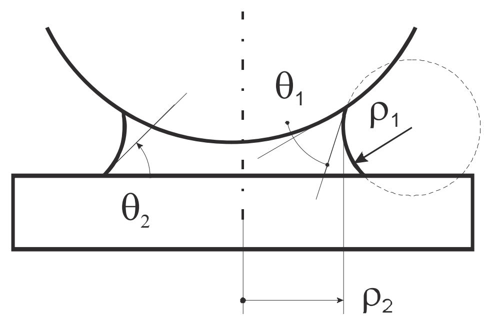
Capillary forces – due to the presence of a liquid interface, the electrostatic force – resulting from the presence of surface charges and van der Waals forces – that are a direct consequences of the atomic structure of matter.

Capillary forces

- Vapor pressure is smaller on curve surface (Kelvin equation)

➢ Formation of liquid meniscus at interface

➢ Pressure difference vapor/liquid => Attractive force



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In this slide, we examine how to estimate capillary forces in simple cases. Here we consider the case of a sphere in contact with a surface and we look at how much force will be generated and pull the sphere towards the surface.

Surface tension forces can be estimated based on the angle of contact between the liquid and the objects. Here we can define two angles of contacts (theta 1 and theta 2).

Illustration of capillary forces

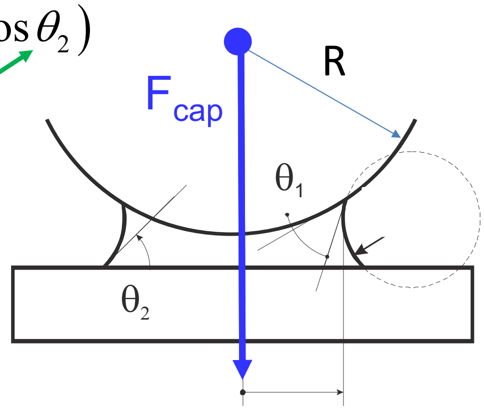
- Example of a water meniscus that pulls a sphere towards a plane

Sphere radius

$$F_{cap} = \gamma\left(2\pi R\right)\left(\cos\theta_1 + \cos\theta_2\right)$$

Surface tension of the liquid meniscus

Contact angles



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Using these angles of contact and the radius of curvatures of the sphere and the droplet, we can estimate the difference of pressure (Laplace Young equation).

The Kelvin equation takes into account the equation of state that models the condensation of water as a function of pressure, temperature and volume.
(R is a constant and γ the surface tension between the liquid and the surface considered).

The third equation gives the capillary force that keeps the sphere in contact with the surface.

Electrostatic forces

- Due to presence of surface charges
- Example: force due to a uniformed density of charges on a flat surface (from Gauss theorem)

Density of charge (C/m²)

Distance to the plane

$$F_{elec,z} = qE_z = \frac{(\sigma_s S)^2}{4\pi\varepsilon}\left(\frac{1}{r^2}\right)$$

Dielectric constant

- Pressure induced

$$p = \left(\frac{\sigma_S^2}{2\varepsilon}\right)$$

Electrostatic forces result from the presence of surface charges.

The force and the pressure induced due to a uniformely distributed charges on a flat surface can be estimated using Gauss theorem and Coulomb force.

Electrostatic forces

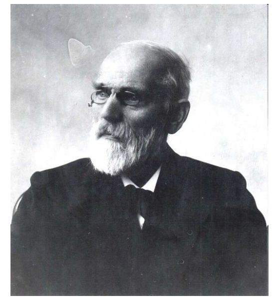
Orders of magnitude

Air	$\sigma_s = 3\ 10^{-5}\ C/m^2$	$p \approx 50$ Pa (gap typ. $1\mu m$)
Good insulator	$\sigma_s = 10\ mC/m^2$	$p \approx 1$ MPa (gap typ. $1\mu m$)

This slide illustrates two example of order of magnitude for the pressure due to charges surfaces.

(Note: 1 MPa = $1N/mm^2$.)

Van der Waals forces



Johannes Diderik van der Waals (1837 – 1923)

- Present in any solids
- Interaction between dipole moments of atoms
- Three components:
  - Keesom: dipole-dipole (C_K)
  - Debye: dipole-dipole induced (C_D)
  - Dispersion: instantaneous dipole-dipole induced (C_L)

van der Waals Potential:

$$U_{vdW} = -\frac{C_K + C_D + C_L}{\boxed{r^6}}$$

Short distance interaction ! (rapidy decay)

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Van der Waals forces are always present and resutl from interactions between atoms.

It is a very short range interaction that rapidly decreases ($1/r^6$). There are three different types of interactions.

Van der Waals forces (integrated forms for various geometries)

Geometry	Force
Two flat surfaces (per unit area dS)	$F = f \cdot dS \quad \text{with} \quad f = -\frac{A_H}{6\pi D^3}$
Two spheres (diameters R_1 and R_2)	$F = -\frac{A_H}{6D^2}\frac{R_1 R_2}{R_1 + R_2}$
Sphere (diameter R) – flat surface	$F = -\frac{A_H R}{6D^2}$
Cylinder (diameter R) – flat surface	$F = -\frac{A_H R^2}{6D^3}$

A_H is the Hamaker constants (material dependant)

These forces are difficult to estimate globally and are material dependant.

Approximated formula have been established for a variety of situations as illustrated in this table.