Static and dynamic wetting of porous Teflon® surfaces
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Chapter 3

Measurement Techniques

This chapter describes the techniques that were used to measure the contact angles and the surface texture of the prepared surfaces, as well as the high-speed imaging techniques.

3.1 Contact Angle Measurement

Two common methods to measure the contact angle of a liquid on a solid surface are tensiometry and goniometry. For the work described in this thesis goniometry was used to measure contact angles. With goniometry, the drop shape is analysed by observing the drop from the side. The contact angle can be determined by measuring the angle between the tangent of the drop surface at the contact line and the surface. This can be done manually, using a horizontal microscope with a goniometer. The drop shape can also be analysed by a computer. In this case, the horizontal microscope is replaced by a camera connected to a computer. The contact angle is measured by fitting a circle to the drop cross-section and determining the intersection with the projection of the surface.
For this work a KSV\textsuperscript{i} CAM 200 system was used to perform contact angle measurements. The system is equipped with a CCD camera connected to a computer and with an automatic liquid dispenser. The automatic liquid dispenser is convenient for the determination of the advancing and receding contact angle.

The advancing contact angle was determined by placing a 5 µl drop of water on the surface using a syringe. Subsequently the drop volume is increased by adding more water to the drop. For the determination of the receding contact angle some water was drawn out of the drop causing the drop to reduce in size.

### 3.2 Optical Microscopy

Apart from using a standard upright optical microscope, an inverted reflective optical microscope was used to characterise samples. An inverted microscope has its objectives positioned below the sample stage pointing upwards. This setup makes it possible to observe a sample from underneath rather than from above. This makes the inverted microscope very useful to investigate the behaviour of a drop of water on top of a self-cleaning surface.

For this work, an Olympus\textsuperscript{ii} BX60 upright microscope and an Olympus GX51 inverted reflective light microscope were used.

### 3.3 Scanning Electron Microscopy

The resolution of an optical microscope is limited by the diffraction of the used light. For an optical microscope, the maximum resolution is about 0.2 µm. In order to obtain a higher resolution, electromagnetic

\textsuperscript{i}KSV Instruments Ltd, Helsinki, Finland

\textsuperscript{ii}Olympus Corporation, Tokyo, Japan
waves with a shorter wavelength should be used. This is the idea behind electron microscopy. An electron microscope uses electrons with a De Broglie wavelength which is much shorter than the wavelength of light. The first electron microscopes developed at the beginning of the 20th century were the so-called conventional electron microscopes. Just like optical microscopes, these conventional electron microscopes give a direct enlarged image of the sample. Later the scanning electron microscope (SEM) was developed. In a SEM the electron beam is rastered across the sample and the released secondary electrons are detected, this way building up an image of the sample. Even though conventional electron microscopes reach higher resolutions, a SEM has some major advantages:

- A SEM can take images of thick samples. While a conventional electron microscope detects the electrons transmitted through the sample, a SEM detects secondary electrons that are released from the atoms on the surface of the sample, comparable to measuring in reflection mode with an optical microscope.

- A SEM has a greater focal depth compared to both optical microscopy and conventional electron microscopy. This is first of all because the incident beam is focused instead of a less homogeneous transmitted beam, and secondly because not the entire sample has to be in focus at once. A SEM only focuses on the spot that is being scanned at that moment. That spot is only several nanometers in size. Compared to an optical microscope, the SEM has a focal depth of about 1 mm at a magnification of 100x, while an optical microscope has a focal depth of only 1 μm at the same magnification.26

The sample is placed in a high vacuum chamber to prevent the scattering of electrons by air molecules. An electron beam is generated by applying a voltage between the cathode and the sample. The sample has to be conductive. In order to image a non-conductive sample, a several
A nanometer thick layer of a conductive material can be sputtered on top of the sample. Electric or magnetic fields are used as lenses to focus the beam onto the sample. A typical lens system in a SEM consists of 2 condensor lenses and 1 objective lens. The electrons impinge onto the sample causing the emission of secondary electrons. These secondary electrons are detected by a sensor such as a charge-coupled device.

For the images in this work two different SEM’s were used. The first SEM used was a Jeol\textsuperscript{iii} 6320F field emission SEM with electromagnetic lenses. The applied acceleration voltage was 2.0 kV, and the working distance was set to 5 mm. The other SEM was a FEI Philips\textsuperscript{iv} XL30 FEG Environmental SEM. The applied acceleration voltage was 7.5 kV, the chamber pressure was set to 1.9 Torr, and the working distance was set to 2 mm.

\section*{3.4 Atomic Force Microscopy}

As opposed to optical and electron microscopy, atomic force microscopy does not use electromagnetic waves but a scanning probe to image a sample. This can be compared to a record-player scanning a gramophone.

An atomic force microscope (AFM) uses a sharp tip usually made from silicon or silicon nitride, attached to a cantilever to scan the surface of a sample. The tip is normally pyramid shaped with a radius of curvature of the tip of 10 nm. When the tip is brought near a sample, the sample and the tip interact causing the cantilever to bend. The interaction between the tip and the sample result from several forces, such as van der Waals forces (attractive) and the electrostatic Coulomb force (attractive or repulsive). The bending of the cantilever can be detected by measuring the deflection of a laser beam. The laser beam is reflected off the back of the cantilever onto a segmented photodiode. The working

\textsuperscript{iii}JEOL, Tokyo, Japan
\textsuperscript{iv}FEI, Hillsboro, Oregon, USA
of an AFM is illustrated in Figure 3.1.

![Figure 3.1: Typical set-up of an AFM.](image)

An AFM can be operated in different modes, namely static and dynamic modes. The static mode usually is referred to as the contact mode. To build up an image, the constant force mode can be used. In this mode the force on the cantilever is kept constant by feedback electronics moving the cantilever up and down using a piezo. By monitoring the motion of the scanner while rastering the cantilever across the sample, an image of the sample is constructed.
As the name suggests, the tip is in constant contact with the sample. This has some disadvantages: the tip can deform or even destroy the sample surface, especially if the sample is soft. The AFM scans in this work were made by using a dynamic mode, the so-called tapping mode. The cantilever is connected to a piezo crystal which oscillates in the vertical direction at a frequency close to the resonant frequency of the cantilever. The resonant frequency typically is in the 100 - 400 kHz range. When the tip comes close to a sample, the oscillation of the cantilever is damped. The oscillation of the cantilever is monitored by a segmented photodiode. When scanning the tip across the sample surface, the amplitude of oscillation of the cantilever is kept constant by moving the scanner up and down. For this work a Veeco® Dimension 3100 Scanning Probe Microscope was used.

3.5 High-Speed Imaging

The first cameras had very long shutter times and small aperture lenses, and were therefore only suitable for taking photographs of still objects. Nevertheless, a pioneer of photography, William Henry Fox Talbot, used such a camera in 1852 to make the first sharp picture of a moving object. He did this by illuminating the object, which was placed in a dark room, with a very short intense flash. The shutter of the camera was left open. He demonstrated the principle by taking a picture of a page from “The Times” newspaper, that was mounted on a rotating wheel.

The principle used by William Henry Fox Talbot is still used today. Usually stroboscopes are used to illuminate the object. A stroboscope sends out flashes of duration of only tenths of microseconds, at a flash rate up to a 1000 per second. Images can be captured by either using a standard camera, or, to collect a sequence of images, a video camera. It is possible to make multi-time exposure pictures to show motion. In this

\(^{v}\text{Veeco Metrology Inc., Santa Barbara, California, USA}\)
work several pictures have been taken using a stroboscope and a video camera attached to a microscope.

A famous pioneer in motion analysis was Eadweard Muybridge. In 1878, he proved that there is a phase where none of a horse’s hoofs touch the ground, by placing a battery of cameras connected to tripwires parallel to the racetrack. Later in his career he made a large number of motion pictures of other animals and humans.

Later, high-speed videography, with frame rates of 160 frames per second (fps) and above, was developed. Special high-speed cameras were used. In a high-speed camera the shutter is usually replaced by a rotating prism or mirror. Frame rates of several thousands or even tens of thousands fps can be reached. Some ultra high-speed cameras can reach stunning frame rates of several millions of frames per second. A breakthrough in high-speed videography was the invention of the charge-coupled device (CCD). In a CCD high-speed camera the frames are stored in the internal memory of the camera. In order to read out the CCD image fast enough, several readout channels are used. The frames can be read out from the camera memory to either an analog or a digital device at a lower speed, once the recording has stopped. Major benefits of CCD cameras are the ease of use, and the direct availability of the captured frames, not to mention the vast amount of tape that is saved.

Because of the short exposure time at these high frame rates, very intense illumination has to be used. Care has to be taken that the filmed object is not destroyed by the heat generated by the illuminating lights.

For this work, both a Kodak\textsuperscript{vi} EktaPro EM Motion Analyzer and a Kodak EktaPro HS 4540 Motion Analyzer were used. The Kodak EktaPro EM Motion Analyzer was used at a frame rate of 1000 fps, a shutter speed of 1000 s\(^{-1}\), and a recording time of 1.6 seconds. The Kodak EktaPro HS 4540 was used at a frame rate of 1125 fps, a shutter speed of 1125 s\(^{-1}\), and a recording time of 0.9 seconds.

\textsuperscript{vi}Eastman Kodak Company, Rochester, New York, USA