Technical Note

Roll-Off of Sliding Angle and Dynamic Contact Angles

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Inclination to know-how

Professional measurement of the roll-off or sliding angle and dynamic contact angle

Surfaces from which raindrops simply roll off and take the dirt with them – the popular trademark Lotus-Effekt® describes the properties of hydrophobic surfaces which have a nanostructure similar to that of the lotus leaf. Manufacturers make use of research into this plant for façade paints and other coatings such as nanoimpregnations or surface-treated glass for windscreens.

Fig. 1: The lotus leaf: a symbol of purity thanks to natural "nanotechnology"

A measuring method for hydrophobic and self-cleaning surfaces – nanostructured or not – is the study of the roll-off behavior of drops on inclined surfaces. However, to date there has been no standard procedure that adequately takes such limiting conditions as drop volume or tilting speed into consideration. In this article we present the results of a comprehensive series of measurements that indicate an approach to the comparability of measurements of the roll-off angle and the dynamic contact angle of drops on inclined surfaces.

Inclined measurements with KRÜSS contact angle instruments

KRÜSS contact angle measuring instruments investigate the wettability of solids by depositing drops on them whose video image is then analyzed. The measured contact angle is the angle between the drop contour and the surface line of the sample (baseline); it is a measure of the wettability and allows – for measurements with different test liquids – the calculation of the surface free energy of the solid.

In inclined measurements the focus is on the behavior of the drop when the surface forms an inclined plane. Attention is given to both the sliding angle (SA) and the dynamic contact angles.

Sliding angle

Initially the drop is normally first deposited on a level surface. The table is then slowly tilted – the angle of inclination increases. At first the drop does not move, but becomes deformed – to an extent that varies according to its density, volume and surface tension. At a particular angle of inclination the drop starts to move and slides or rolls across the surface.

The sliding angle SA is the tilt angle TA at which the movement of the drop across the surface begins.

Fig. 2: Continual inclination until SA is achieved

Dynamic contact angle

The dynamic contact angle is the contact angle during the wetting (advancing angle θ_{Adv}) and dewetting (receding angle θ_{Rec}) processes, i.e. during the movement of the drop across the surface (Fig. 5 right).

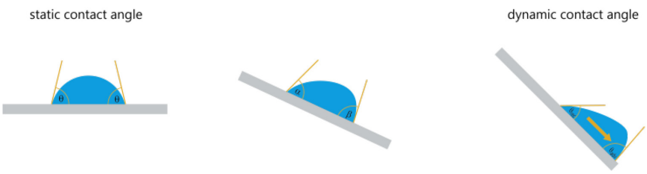

Fig. 3: Measuring the dynamic contact angle during roll-off

Available tilting tables

There are two different tilting tables with differing arrangements available for KRÜSS contact angle measuring systems. With the *external* tilting table PA3220 for the DSA100 the whole instrument is placed on the tilting table. The video camera also rotates so that the position of the sample in the video image does not alter. This makes it easier to determine the baseline and evaluate the image. The dosing system for depositing the drop also rotates, so that it only makes sense to deposit the drop on a level table.

Fig. 4: DSA100 with external tilting device PA3220

With the *internal* tilting table PA3240/4240 for the measuring systems DSA100 and DSA30 only the support surface of the table with the sample rotates; the camera and dosing system remain stationary. This means that the inclination is also visible in the image. The drop can be deposited on a level or already tilted surface, as required.

Fig. 5: DSA30 with internal tilting device PA3230

Manual and software-controlled dosing units are available for both instruments; both tilting tables are controlled by the drop shape analysis software DSA4.

The studied system

For the measurements presented here, KASI® coated PMMA was used as the solid with water as the drop liquid. The method described for use with this system is intended to find the measuring conditions under which the SA and dynamic contact angles can be reliably determined.

Determining the sliding angle

When evaluating a sequence of images the SA can be determined by drop shape analysis. The recommended criterion is the movement of the three-phase point (3PP), i.e. the left- and right-hand phase transition points between solid, liquid and the surrounding gas phase.

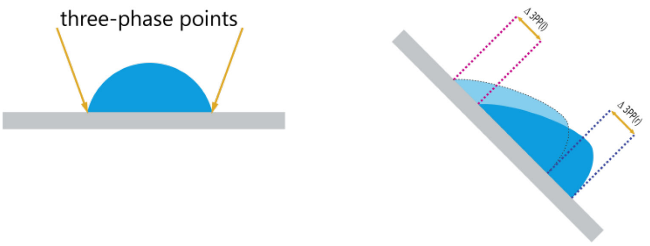

Fig. 6: Movement of the three-phase points as a criterion for SA

In the measurements described here under continuous tilting, the angle of inclination at which the position of the slowly moving three-phase point is displaced by 40 pixels is defined as being the SA.

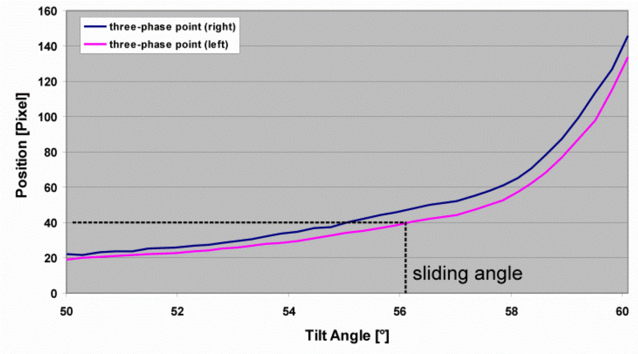

Fig. 7: Determination of the SA using the coordinates of the three-phase points in the video image

The SA is an empirical quantity that depends on the chosen measuring parameters, such as the tilting speed or drop volume. For comparative measurements between different samples it is therefore important to determine suitable measuring conditions according to the criteria mentioned below.

Influence of the tilting speed

One of the possible interfering factors in the measurement of the SA is a too high tilting speed; this influences the result because of the inertia of the drop. In order to compare different samples they should be measured in the slow tilting speed range in which the SA is still independent of the speed. As this speed range depends on various factors such as drop weight, contact angle and roughness, it should be investigated by measurements at different tilting speeds.

The influence of speed on the SA can be seen from the following curves showing the alteration of the three-phase point positions at different tilting speeds:

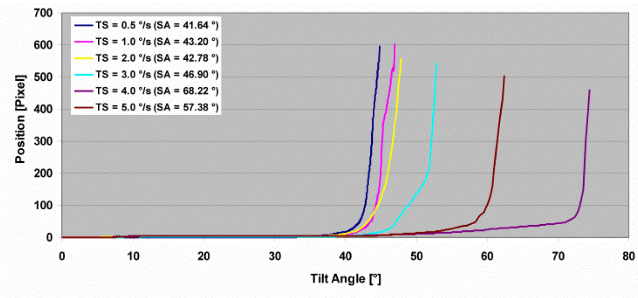

Fig. 8: Possible SA falsification resulting from too rapid tilting

Between 0.5 and 2°/s the differences in SA are within the measurement scatter range. From 3°/s the tilting speed shows a clear influence on the roll-off behavior.

Influence of the drop volume

The influence of the drop volume on the SA is obvious: the greater weight means that there will be a stronger force along the tilted surface, so that the drop will move at a slighter angle of inclination. This means that the SA sinks as the volume increases, as can be seen from the following plot:

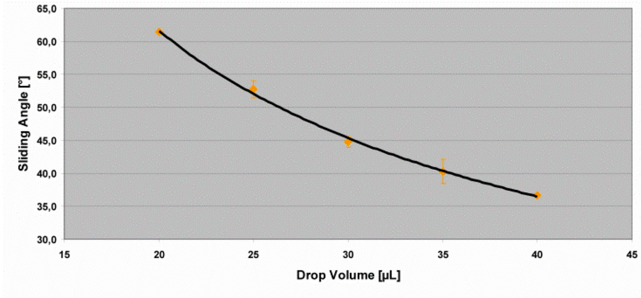

Fig. 9: SA dependency on drop volume

If different samples are to be compared then care must be taken to ensure a uniform dosing volume. Much higher dosing volumes are normally selected (between 20 and 40 µL) than for a standard contact angle measurement (1 to 3 µL).

Determining the dynamic contact angle

Dynamic contact angles are measured during the wetting and dewetting of a surface – in contrast to the static contact angle, in which the three-phase points do not move. A frequently used measuring method consists of the enlargement (advancing angle) or reduction (receding angle) of the dosing volume during the measurement on a level surface. Dynamic contact angles can also be measured as a drop moves across a tilted surface. This is why the measurement of the SA and the advancing and receding angles are closely related.

Determining the constant range

When tilting starts the drop does not move before the SA is reached, but it becomes deformed. The angle measured at the lower side becomes larger, that at the upper side smaller (see Fig. 3). However, these angles do not say a lot about the wetting properties, because they are primarily formed by the deformation resulting from the weight of the drop. Even when the drop starts moving above the SA, it takes some time before constant values are obtained at both sides of the drop. It is these plateau values that give required quantities: the advancing angle and receding angle.

The following illustration shows the gradual achievement of a constant dynamic contact angle during the tilting experiment. The upper plateau corresponds to the advancing angle θ_{Adv}, the lower to the receding angle θ_{Rec}.

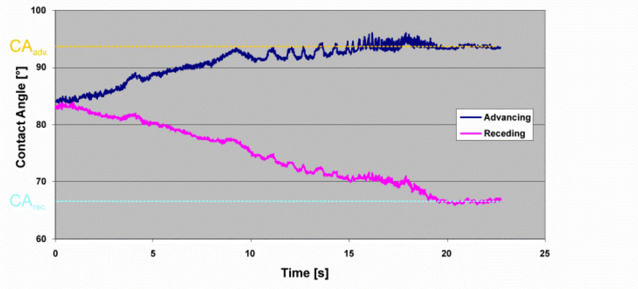

Fig. 10: Gradual achievement of plateau values in a tilting experiment (blue curve = advancing angle; red curve = receding angle)

Influence of the tilting angle

The greater the angle of inclination above the roll-off angle, the quicker the drop movement and the more difficult is the measurement of the constant dynamic contact angle. Ideally the tilting angle chosen for the measurement should be just above the SA.

Influence of the drop volume

It has been established that measurement of the advancing angle is largely independent of alterations to the drop volume. In the volume range from 20 to 40 µL the sought-after plateau was at the same level. In the medium volume range the receding angle was also independent of the drop volume, although there was a greater scatter than for the advancing angle. For small volumes the measured value tended to be higher. However, all in all the two dynamic contact angles were found to be independent of the volume throughout a wide range.

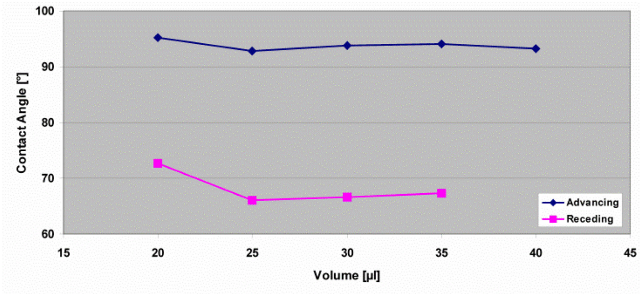

Fig. 11: Advancing and receding angles for different drop volumes

Summary

Measurements of the sliding angle (SA) and the dynamic contact angle were carried out on a KASI® coated PMMA surface using deposited water drops. The movement of the three-phase points in the video image by 40 pixels was first defined as being the criterion for reaching the SA.

As has been shown, the tilting speed must not exceed the particular critical value above which it influences the SA significantly.

As the SA depends on the drop volume, this must be kept constant for comparability reasons.

At tilt angles greater than the SA the advancing and receding angles can be measured as plateau values at the right and left of the drop image. The selected angle of inclination should be slightly larger than the SA, as otherwise the drop will move too quickly and the time window for the formation of the plateau will be too small.

The advancing angle is largely insensitive to alterations in volume; measurement of the receding angle is most reliable in a medium volume range.

The described procedure for determining favorable ranges for the experiment that are independent of critical measuring parameters can be transferred to other systems.

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