Application Note 21

Contact Angle Measurements on single fibres with the DataPhysics OCA50 Micro

Task

The optical contact angle systems of the DataPhysics OCA-series make it possible to dose drop volumes starting with a size of just a few nanolitres. Caused by the small diameter of single fibres (5-500 µm) the normal dosing of nano-/mikrolitres has no use due to the fact that the formed drops are just to big to be put onto the small fibres. The measurements of surface properties on small fibres are of interest as well as the analytics of large surfaces. So a possibility is needed to produce smallest drop volumes which will allow detecting the properties of the surface of single fibres.

Method

The DataPhysics OCA40 Micro with picodrop dosing system „DUP 2/xx" has been developed for contact angle measurements on single fibres. The system allows the dosing of drop volumes down to a volume about 10-20 pL. So it opens the possibility to measure contact angles on single fibres with a diameter of 5 µm or more.

After connecting of the dosing system to DataPhysics OCA40 Micro the measuring liquid will be filled into the storage vessel. The system has to be purged so no air bubbles will be left in the tubes. When the air has left the system a fine stream of liquid can be seen leaving the dosing capillary. The stream can be used to focus the camera exactly to the future plane of the generated drops.

The picodrop dosing system allows generating drops through a single or a triple pulse. For each dosing sequence the values for pulse width, pulse duration and delay can be varied. Depending on the used liquid it is needed to find the right pair of parameters that will generate a stable drop. To find the right settings of the pulse parameters the DataPhysics OCA40 Micro in combination with the picodrop dosing system is equipped with a stroboscope illumination. While using the function the parameter values can be changed until the camera shows the unchanging picture of a single drop. In this case the optimal parameters for the used liquid are found.

A stable drop under stroboscope illumination is seen in figure 1. The generated drops can vary its volume depending on the used set of pulse values. So it is possible that other sets of working parameters can generate an even smaller drop which will optimize the measurements. The image of the stable drop while using stroboscope illumination can also be used to adjust the focus of the camera exactly.

Is the system set up right and produces a stable drop the single fibre to be measured can be prepared. To tighten the fibre there is a special adjusted sample holder "FHO 40 Plus" for this application. A tight fixation of the fibre is important because even small vibrations or air streams will lead into a movement of the fibre which will move out of the focus plane.
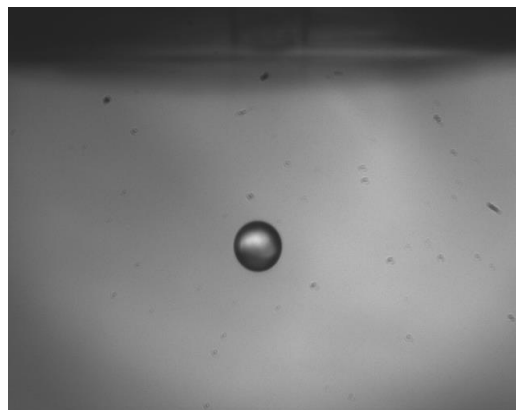

Figure 1: Stable drop under stroboscope illumination

If the volume of the drop is to be calculated the current magnification has to be calibrated. Using a know size like the diameter of the used dosing capillary (with the PDH50 it is 50 µm) or the thickness of the used fibre the calibration can be done.

Is the single fibre located within the focus the picodrop dosing system can generate a drop and put it onto the fibre. If the drop does not hit the fibre the position of it has to be adjusted until the drop hits the fibre in its centre.

The tinniest drops will have a volume of just a few picolitres so the vaporisation has a big influence on it. Because of that a movie is stared before generating a drop. Afterwards the recorded movie can be processed. The analysis works analogue to the analysis on larger samples. First the baseline will be detected automatically or if needed by hand. Also the shape of the drop will be detected automatically by the program. So the contact angle of the liquid on the single fibre will be calculated.

Results

Contact angle measurements of water on a carbon fibre with a diameter of 8 µm have been done. Therefore a DataPhysics OCA40 Micro in combination with the picodrop dosing system "DUP 2/E-MD" and the dispenser head "PHD 50" has been used. The drop was generated by using a triple pulse. Figure 2 shows an example of a water drop on the single fibre including the automatically contact angle analysis of the software.
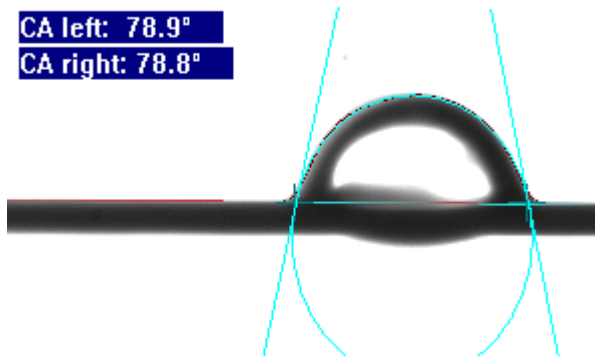

Figure 2: Resting drop on an 8 µm thick single fibre

The measurement has been repeated at different location on the same fibre to get trustworthy results. Even tinniest inhomogeneities of the surface will have an influence on the measured contact angle. So the standard deviation is an indicator for the homogeneity of the surface. As seen in table 1 the measured values vary very little even the used surface and drop volumes are very small. The calculated mean contact angle is about 79° with a standard deviation below 1°. So the value can be seen as a reliable indicator the wettability of the used fibre.

Table 1: Contact angle measurements

Measurement	Contact Angle [°]	Volume [pL]
1	80,55	32,98
2	78,68	36,98
3	78,09	38,87
4	78,87	37,77
5	78,66	37,51
Mean	78,97	36,82
SDDV	0,93	2,26

Looking at table 1 it is easily seen that the measurements of the contact angles are reproducible and vary just within the normal aberration. The high precision of the system can be seen by the sizes of the drop which are about the same size for every generated drop. The mean volume in this measurement was around 37 pL with a standard deviation of 2 pL.

Summary

Using the DataPhysics OCA40 Micro in combination with the picodrop dosing system „DUP 2/E-MD" water contact angles on a single fibre have been measured. The results show that the method works very precise and produces reproducible results even tinniest drop sizes are used. The system opens an easy way to measure on surfaces of small single fibres and analyze their surface properties.

Using different testing liquids it is possible to calculate the surface free energy of single fibres by using the software package SCA21. The measurements are done like on other surfaces too (compare application note 4).

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