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Inline process control of wettability by means of contact angle measurement on moving surfaces

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Contact angle measurement has completed the step from the test laboratory into the production hall with regard to quality assurance for the cleaning and pretreatment of surfaces. This has been made possible thanks to the development of fast and mobile measuring technologies. We go even further with this feasibility study and show a way for the potential use of contact angle measurements as a test method for uninterrupted inline process controls. Two recent innovations from KRÜSS open up this perspective: the extremely fast Liquid Needle technique that enables contact angle measurements on moving samples and the ADVANCE API software interface with which the integration of fully automatic analyses is possible in any complex information systems.

Background

Materials are subjected to surface treatment in many production processes, in order to prepare these for further processing steps. Typical examples here include the cleaning of surfaces before bonding or plasma treatment of a polymer in order to activate it for subsequent coating and thus achieve the best possible wettability and adhesion. As wetting is reflected directly by the contact angle, measuring the contact angle is an excellent way of testing the efficacy of such pretreatment methods [1].

Pretreatment is often carried out on samples that are continuously moving. Until now the inline control of such dynamic processes was not possible for the following two reasons:

1. Deposition of a drop with a classic needle dosing system is too slow for moving samples; the drop smears on the sample. Attempts to shoot the drops onto the sample fail due to excessive impact speeds that lead to falsified contact angles and larger error bars.

2. There was no technology for the integration of contact angle measurements in a comprehensive information system, nor the automatic synchronization of results with sample information or even for establishing feedbacks with process parameters such as cleaning and activation time.

KRÜSS has developed solutions for both impediments. The *Liquid Needle* technique [2] provides the necessary drop deposition on moving samples. It combines fast and contactless dosing with low dynamics that do not influence the shape of the drop.

The ADVANCE API interface of our software for contact angle measurement and many other measuring methods enables incorporation in integrated information systems. A typical procedure looks like this: an automation program created in ADVANCE specifically for the respective measuring task – one which for example carries out the dosing and analysis of a drop – is started from a different software via the API. The same external software can write sample information in a details field of the ADVANCE measurement, for example. When the measurement is completed ADVANCE exports the data automatically in a freely definable format and sends a completion message via the API. The external software can now gather and analyze these data and initiate any subsequent steps.

There are several possible ways of implementing such a process. For example, drops can be positioned onto a sample using the Liquid Needle while the sample moves through the frame of a Drop Shape Analyzer – DSA100. Figure 1 shows a scheme of such an installation. In this report we have simulated this setup as an example for measurements carried out on moving samples.

As an alternative solution, the software-controlled axes of a Large Surface Analyzer – LSA from KRÜSS, which move a measuring head equipped with the Liquid Needle, could be synchronized with the movement of the sample.

Experimental procedure

We used an off-the-shelf DIN-A4 PP foil that had been cleaned with a surfactant, warm water and isopropanol as the sample for the feasibility test. The foil was placed on a vacuum table mounted on a 170-mm automatic axis of the DSA100. The camera was set at a high image acquisition rate of 200 fps in order to prevent motion blur in the drop images. Once the axis started to move at a speed of 250 mm/min, 50 drops of water were dosed and measured at intervals of 0.8 seconds. We repeated the procedure on the same sample after subjecting three places on it to a plasma treatment (60 seconds) using a plasma pen.

Fig. 1: Schematic setup of a series contact angle measurement on a moving sample.

Results

Figure 2 shows a contact angle measurement being carried out on a moving sample by way of example. As can be seen the drop was positioned perfectly on the sample an analyzed in the center of the image. On the right edge of the image you can see the left side of the drop that was dosed and measured 0.8 seconds earlier.

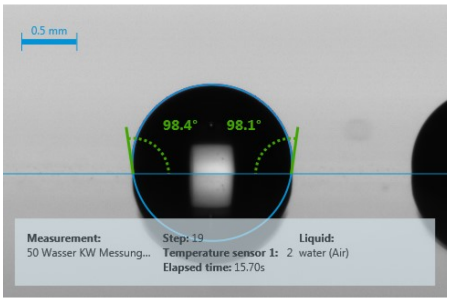

Fig. 2: Example image of the contact angle measurement of a sample moving at a speed of 250 mm/min. The drop was fitted to the asymmetrical ellipsis function (tangent-1).

Figure 3 shows the results of the contact angle measurements on the 50 drops on the freshly cleaned sample and the 50 drops on the partially plasma treated sample. Each row of drops was distributed evenly over a length of 167 mm and was measured within 40 seconds. The dark blue curve shows the contact angle on the untreated PP sample and the light blue curve the contact angle on the partially plasma treated one.

An atmospheric plasma treatment results in smaller water contact angles. A comparison of the two curves therefore clearly indicates the positions that did not undergo plasma treatment. It can now be assumed that wetting problems would lead to defects in quality at these places in a production process. However, the software can give feedback immediately after the measurement via the ADVANCE API, so that countermeasures can be taken in good time thus preventing greater loss of production.

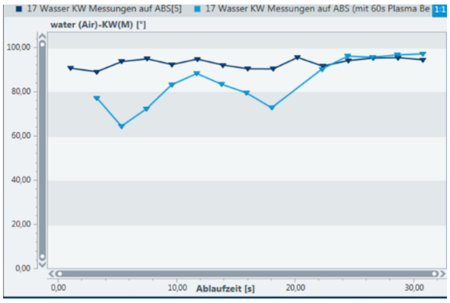

Fig. 3: The contact angle measured on each 50 drops on the moving sample. Measurement of the freshly cleaned PP sample is indicated in dark blue and that of the partially plasma treated sample in light blue.

In the tests carried out here the speed of the samples complied with the maximum DSA100 axis speed of 250 mm/min. Additional tests would be necessary with higher process speeds, in order to verify the reliability of this method. However, first tests on manually moved samples indicate that speeds of up to 1 m/min can be measured very well.

The size of the samples is the second limiting factor of the test setup, apart from the maximum speed of the samples. The maximum sample height is 27 cm and sample width 68 cm (with the DSA100 Large Frame). The dosing system must be refilled at the earliest after 2000 drops. However, two DSA100 arranged in tandem would enable uninterrupted quality testing.

Summary

It is possible to reliably measure the contact angle on samples moving at a speed of 250 mm/min with the aid of the ultra-fast Liquid Needle dosing technique. Process speeds of up to 1 m/min are conceivable, but require further testing. Samples with widths of up to 68 cm are possible with the DSA100 measuring instrument selected for our test setup.

The ADVANCE API software interface opens up flexible possibilities for embedding the start and progress of the measurement as well as exported results in an extensive information system for an inline process control. There are many conceivable scenarios, from a simple message after a warning limit is exceeded on through to include automatic adjustment of process parameters.

Literature

[1] KRÜSS Application Report AR272

[2] M. Jin, R. Sanedrin, D. Frese, C. Scheithauer and T. Willers, "Replacing the solid needle by a liquid one when measuring static and advancing contact angles", Colloid Polym. Sci. 294(4), 657-665 (2016).