Application Report

Contact Angle Measurements on Large Surfaces

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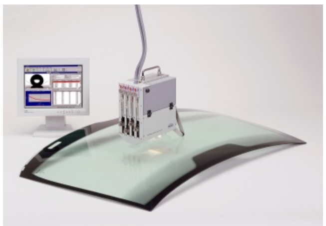


GH100

Method: SFE

Keywords: Mobile contact angle measurements, surface free energy, large surfaces, glass, windshield, paper roller, coating

Contact Angle Measurements on Large Surfaces

Abstract

The wetting properties of functional coatings on large surfaces can now be tested non-destructively under real conditions. Concrete results are presented using windshields and printing rollers as examples.

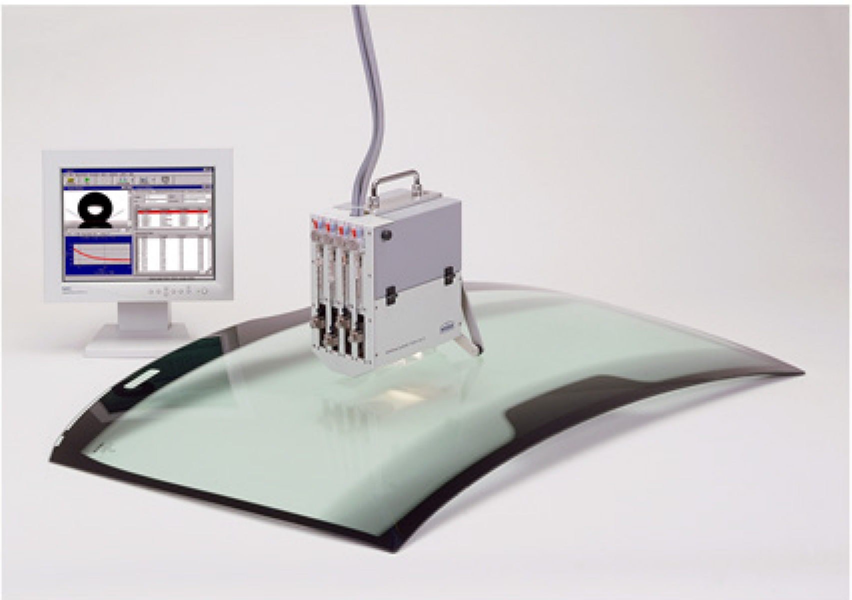
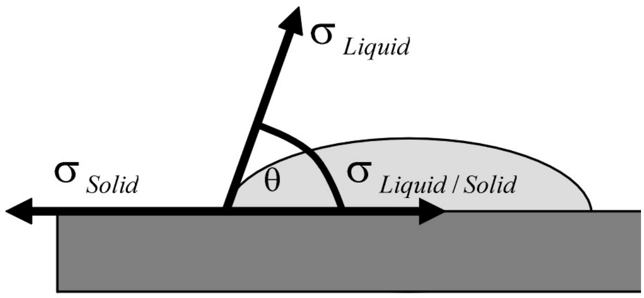


Fig. 1: GH1000 Universal Surface Tester as a mobile unit for testing a windshield

What do facades, windshields, shower side panels and TFT flat monitor screens have in common? The application properties of all these materials can be considerably improved by functional coatings. Silicone-based dirt-repellent and water-repellent facade colors maintain the long-term aesthetic value of a house by making it difficult for dirt, soot and moss to attach themselves to it. Hydrophobically coated windshields and side windows increase driving safety as rainwater pearls off them. Hydrophobically coated shower side panels, tiles and bathroom fittings avoid water stains.

On top of this defined and fixed wetting properties play a decisive role in the electronics industry, for example in the manufacture of TFT flat monitor screens, silicon chips and hard disks.

The surface energy is the decisive parameter in all these applications. It is technically easy to measure this quantity by making contact angle measurements with defined test liquids such as water, diiodomethane, ethylene glycol, formamide or glycerol. This is done by placing 2 to 10 µl droplets of liquids of differing polarity on the sample and determining the tangent of the wetting angle at the three-phase point by using a video system (Fig. 2).



$$\sigma_{Solid} = \sigma_{Liquid} \cos\theta + \sigma_{Liquid/Solid}$$

Fig. 2: Contact angle of a liquid on a solid together with the Young equation

Measuring problems with large samples

Because of the need to construct an optical bench, no instrument until now was capable of making measurements on a sample with a side length of more than 0.5 m without needing to cut up the sample in order to obtain a suitable size for the laboratory. This meant that quality assurance on complete windshields, printing rollers or baths was impossible.

Help was first provided by the GH100 Universal Surface Tester (Fig.1). Arranging the optical components in a new way meant that there was no longer any limitation on the sample size and that contact angle measurements could now be carried out on finished products, on large coated surfaces or even on-site with a mobile version, in factories or on test stands.

Below we give two examples to explain the practically relevant statements which can be obtained from making these measurements.

Measurements on windshields and printing rollers

With hydrophobically coated windshields, which provide excellent visibility at high speeds, the driver is chiefly interested in the stability and working life of such an expensive coating. As a result of UV-irradiation and the mechanical stress caused by the windshield wipers it must be expected that the coating will wear out in time; this can be quantified by making contact angle measurements on finished windshields.

It is obvious that this is not possible with laboratory instruments, as it is neither possible to cut a piece from the windshield nor do modern adhesive techniques permit non-destructive removal of the windshield from the automobile. A typical case for the GH100!

With printing rollers it is not so much the hydrophobicity of the coating which is of primary importance, but rather the uniformity of the interactions between the paper and the roller across its whole area. With the high paper web speeds of up to 60 m/s which are used today, different adhesive forces could lead to stresses or even to the paper web tearing. Owing to the immensely high operating costs it is apparent that knowledge about the distribution of the interactions, i.e. the condition of the surface and its degree of wear, is of fundamental importance to the manufacturers of such printing rollers.

Printing rollers are frequently up to 10 m long and can have a diameter of up to 2 m; this rules out laboratory measurements completely but is no problem at all for the GH100 (Fig. 3).

Experimental examples:

The sample was a windshield which had been subjected to defined windshield wiper load cycles of 2500, 5000, 10000, 15000, 20000, 22000, 25000 and 40000 cycles; after the corresponding number of cycles part of the windshield was covered up. This meant that the defined measurement of the coating properties at different load conditions was possible.

With the printing rollers a new hydrophobically-coated roller was investigated as well as the same roller after it had been in use for one week. The measurements were made on site; the wetting properties were determined every 100 cm across the roller width of 10 m.

In both cases the measurements were made using the GH100 as a hand-held instrument. Water (polar, high surface tension) and diiodomethane (nonpolar, medium surface tension) were used as the test liquids. Two measurements were made per cycle; these were repeated 10 times.

The analysis of the images recorded with the video system together with the calculation of the surface energy were carried out with the Krüss DSA II drop shape analysis software using the Young-Laplace equation.

$$\sigma_{Liquid/Solid} = \sigma_{Solid} + \sigma_{Liquid} - 2\sqrt{\sigma_{Liquid}^{Dispers} \cdot \sigma_{Solid}^{Dispers}} - 2\sqrt{\sigma_{Liquid}^{Polar} \cdot \sigma_{Solid}^{Polar}}$$

Using this equation in combination with the Young equation you obtain:

$$\frac{\sigma_{Liquid}(\cos\theta + 1)}{2\sqrt{\sigma_{Liquid}^{Dispers}}} = \sqrt{\sigma_{Solid}^{Polar}} \frac{\sqrt{\sigma_{Liquid}^{Polar}}}{\sqrt{\sigma_{Liquid}^{Dispers}}} + \sqrt{\sigma_{Solid}^{Dispers}}$$

$$y = m \cdot x + b$$

Surface energy

Just as for liquids, the interactions with the surrounding phase can also be characterized for solids. In the first case this is the surface tension (e.g. with a tensiometer); in the second case the surface energy. The contact angles measured with different liquids are directly related to the surface energy of the solid; this is shown in the equation in Figure 2. This equation can be solved by making independent measurements using two liquids with known but different surface tensions.

Small contact angles always mean good wettability and large surface energies; large contact angles therefore mean poor wettability and small surface energies.

In addition, different models (Owens/Wendt, Zismann, Wu or acid/base methods) can be used to obtain a more exact differentiation of the interactions in the polar and disperse fractions of the surface energy of the solid.
As an example the Owens/Wendt method is used together with the Young-Laplace equation (Fig. 2) to further break down the interfacial tension between the solid and liquid into its polar and disperse fractions:

[If this equation is included in the Young-Laplace equation we obtain:]
This can again be easily solved graphically by using test liquids with different polar and disperse fractions for the measurements. In the tests described here this was done by using water and diiodomethane as the test liquids.

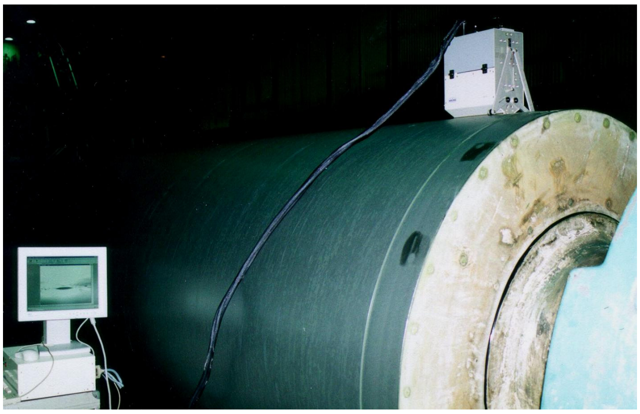


Fig. 3: GH100 being using for testing a printing roller at a production location.

Windshield results

The working life of the hydrophobic coating of a windshield can depend strongly on the mechanical stress to which it is subjected. This assumption has been confirmed experimentally, as is shown in Figure 4.

At the start the windshield has good hydrophobic properties; these properties are clearly reduced after 20000 load cycles and it is apparent that the hydrophobic coating is being removed by mechanical means. Possible solutions to this problem could be, for example, improved surface bonding or cross-linking the hydrophobic groups.

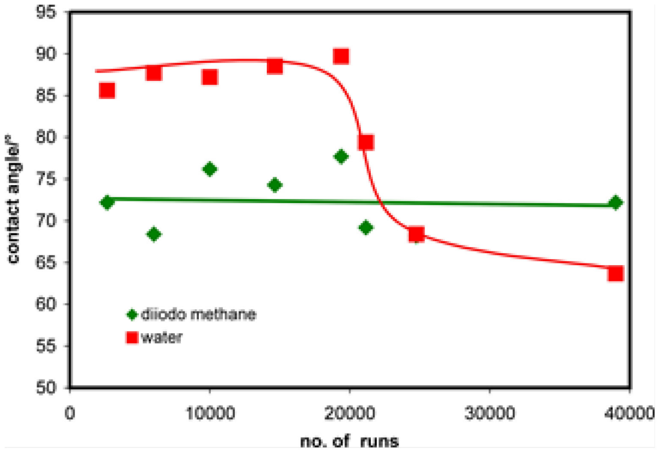


Fig. 4: Graph showing the contact angles of water and diiodomethane as a function of the number wiper load cycles on a windshield

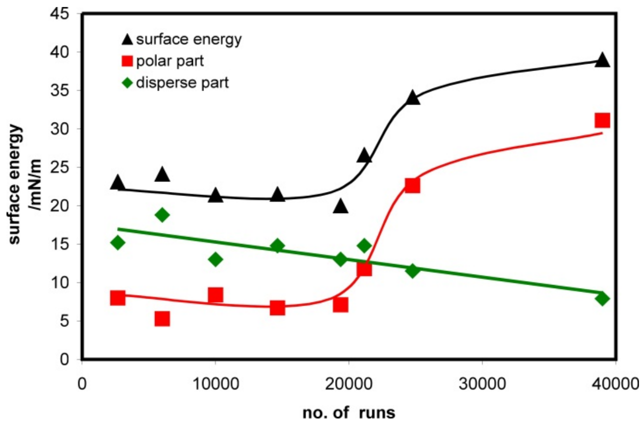


Fig. 5: Graph showing the total surface energy as well as its polar and disperse fractions as a function of the number wiper load cycles on a windshield

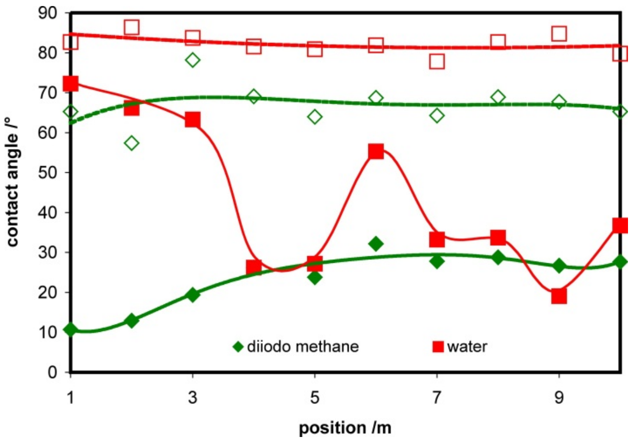


Fig. 6: Graph showing the contact angles of water and diiodomethane as a function of the position on a printing roller (Outline symbols = new roller; Solid symbols = roller after several weeks in use)

The contact angle of the nonpolar liquid diiodomethane shows no alteration as a result of mechanical stress. On the contrary, the variations which can be observed are the result of dirt on the surface and other inhomegeneities.

If the Owens/Wendt method is used to calculate the surface energy the values shown in Figure 5 are obtained. The increase in polar interactions above 20000 load cycles can be clearly seen, while the disperse fraction remains almost constant. As the load increases a better wettability and therefore poorer visibility can be predicted; this corresponds to an increase in surface energy or polarity.

Printing roller results

As can clearly be seen from Figure 6, in its new condition the roller has good hydrophobic properties, i.e. contact angles of about 90° are obtained for water. It is also obvious that this value is kept very constant across the whole roller width. The same can also be demonstrated with the nonpolar diiodomethane as the test liquid; its contact angles are also conspicuously high. However, this picture changes fundamentally after one week in use. The original properties are retained in some positions, but in many other positions considerably smaller contact angles are measured. The very inhomogeneous distribution of the surface properties is also conspicuous. It is just this sort of distribution which can cause stresses within the paper web as a result of the differing adhesion of the paper to the roller and lead to the paper tearing and machine downtime.

Summary

Contact angle measurements with the GH100 allow the exact characterization of wetting properties even on samples with large surfaces. Simple measurements on both the windshield and the printing roller show the mechanical loading problem spots. Concrete criteria for product improvement can be obtained under real conditions of use without any additional test runs being required.

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