dataphysics

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Application Note 5

Surface tension of molten polymers and hot-melts.

Here we describe the lamella method; a new procedure, from DataPhysics, for the determination of surface tension.

Problem

Traditionally, the determination of the surface tension, of molten polymers or hot-melt adhesives, was only possible with considerable experimental effort. The new contact lamella method, introduced by DataPhysics, facilitates a rapid, accurate, measurement of surface tension with a considerably smaller experimental error, compared to those traditional methods (even at temperatures up to 400°C).

Traditional methods for the determination of surface or interfacial tensions, with gravimetric tensiometers, are conventionally limited to an experimental temperature range up to max. 100°C. In conjunction with suitable control systems the alternative, contour analysis of pendant drops (optical) method, allows for the determination of surface and interfacial tensions to higher temperatures. A crucial limitation of the pendant drop method is in the handling of highly viscous molten polymers. The required dosing, with heated syringes and needles, can be troublesome and will add expense to the instrument configuration. The requirement for optimised cleaning may also present considerable challenges. In extreme cases, problems can occur with the formation of gas bubbles in the syringe, making the dosing of drops, of a defined size, difficult or even impossible. The newly developed lamella method avoids these difficulties. Only parts of the device that can be cleaned with ease come into contact with the sample under test and the sample container is open, to no gas bubbles can be trapped.

Method

Like measurements carried out during the pendant drop procedure, the lamella method is based on the equilibrium between the forces exerted by the weight of the material, under test, and its surface tension. Fig. 1 shows, schematically, the lamella which will develop when a vertical test piece is brought into contact with a liquid.

With liquid/test piece contact angles between 0° and 90° the resulting lamella is curved upwards. The lamella is curved downwards for contact angles between 90° and 180° and if the contact angle is exactly 90°, there is no curvature of the liquid surface.

The lamella develops from the tendency of the liquid to wet the test body. In this process the effect of the liquids surface tension ensures that the newly created surface is kept as small as possible. At the same time, the weight of the liquid tries to minimize the volume of a rising lamella. At equilibrium, the effects of surface tension and weight counterbalance each other. When the weight is known (from the density and 'size' of the lamella contour), the surface tension of the liquid can be calculated. (The Young-Laplace differential equation forms the basis of the calculation).

With recent developments in image capture hardware, high-speed PC systems andoptimised software algorithms it is possible to calculate the surface tension, numerically, within fractions of a second.

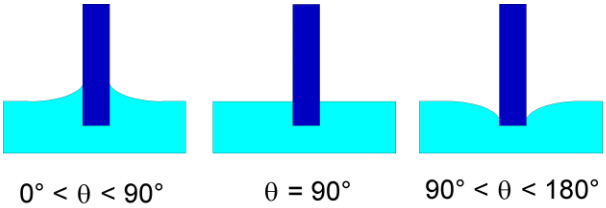

Fig. 1. Schematic illustration of the formation of a lamella

Fig. 1 shows that with a contact angle of 90° and a straight cylindrical test body, no lamella can be created. If, however, a spherical body is used, instead of a straight cylindrical body, then a curved lamella will form even when the contact angle is 90°, because the spherical surface does not have a constant angle of 90° to the horizontal.

Procedure

The molten polymer or hot-melt adhesive is placed in the 'well' of a specially shaped sample container. In order to maintain the measurement environment at 120°C the electrically heated temperature control system, TEC 350, is employed. This replaces the sample stage of the optical contact angle measuring instrument OCA xx. The spherical test body is immersed into the molten sample so that its end is completely wetted. The test piece is then withdrawn, vertically, to produce the lamella illustrated in Fig. 3 A digital image of the lamella is recorded and saved, with the chosen camera system, at the required temperature. To calculate the surface tension, the program must work out the weight of the lamella. For this purpose the density of the molten material and the magnification ratio of the image taken must known. The sample's density is entered by the operator, the magnification factor is calculated from the number of on-screen pixels that the upper part of test body takes up compared to it's known size (this being pre-determined with a micrometer or similar device).

After measurement the test body and sample container are easily cleaned in an ultrasonic bath.

Results

With a viscous hot-melt adhesive as our example, the lamella method is compared to that of the pendant drop procedure. Fig. 2 shows the image of a pendant drop of our hot-melt at 120°C. (Because of its rectangular format, the chosen camera system was tilted by 90°, from the horizontal, to provide an image that will ultimately facilitate the most accurate software driven analysis).

From this image, a surface tension of 35.16 ± 0.04 mN/m was determined. (A density of 1.0518 g/cm^3 was assumed).

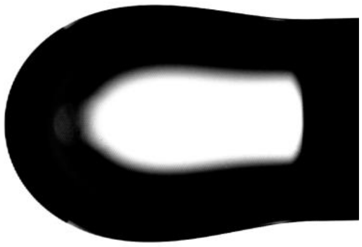

Fig. 2: Pendant drop of a hot-melt at 120°C

The lamella method was applied to the same material. The corresponding image is shown in Fig. 3. The lamella and the spherical test body are clearly visible.

From this image, a surface tension of 34.81 ± 0.35 mN/m was determined. (The same density being assumed).

Hence the two values agree within an error of 1%.

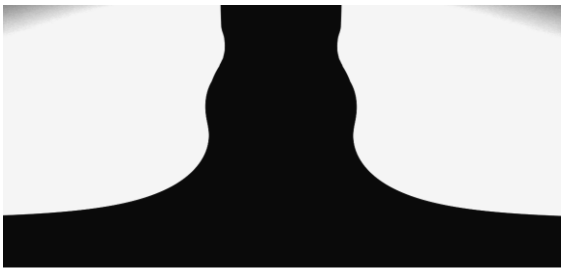

Fig. 3: Lamella of the hot-melt at 120°C

Summary

The new DataPhysics lamella method has been introduced here. It permits the rapid, robust and easy determination of the surface tension of viscous liquids, at temperatures up to 400 °C.

With the example of a high viscous hot-melt adhesive, an accuracy of the method of 1% has been achieved.