

Application Note 5

Surface tensions of molten polymers and hotmelts

The DataPhysics lamella method, a new method of determining the surface tension with increased temperatures

Problem

So far, the determination of the surface tension of molten polymers or hotmelt adhesives was only possible with considerable experimental efforts. The contact lamella method introduced by DataPhysics permits a rapid, accurate measurement of the surface tension with a considerably smaller experimental expense, compared to conventional methods, even at temperatures up to 400°C.

Traditional methods of determining the surface tension with gravimetric tensiometers are mostly limited to a temperature range up to max. 100°C. In suitable temperature control systems, the contour analysis of pendant drops (Pendant Drop Method) is also possible at higher temperatures. The problem here is the handling of highly viscous molten polymers. The dosing with heated syringes is troublesome, and especially the cleaning is difficult and takes a lot of time. Moreover problems are often caused by the formation of gas bubbles in the syringe, which make the dosing of drops of a defined size difficult or even impossible. The newly developed lamella method avoids these difficulties, because only easy to clean components come into contact with the molten polymer, and the sample container is open so that no gas bubbles can be trapped.

Method

Like the method of the pendant drop, the lamella method is based on the equilibrium between the weight and the surface tension. Fig. 1 shows schematically a liquid lamella, which will develop when a vertical test body is brought into contact with a liquid. With contact angles between 0° and 90° the lamella is curved upwards, whereas it is curved downwards for contact angles between 90° and 180°. If the contact angle is exactly 90°, there is no curvature of the liquid surface.

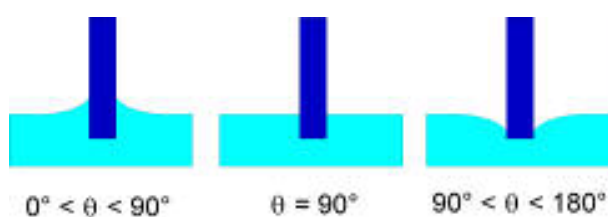


Fig. 1. Schematic illustration of the formation of a lamella

The lamella develops from the effort of the liquid to wet the test body. In this process the effect of the surface tension ensures that the newly created surface is kept as small as possible. At the same time, the weight tries to minimize the volume of a rising lamella. At equilibrium, the effects of surface tension and weight just counterbalance each other. When the weight is known, the surface tension of the liquid can be calculated from the exact knowledge of the lamella contour. The equation of the state of equilibrium forming the base of the calculation is the Young-Laplace differential equation for which there is no closed analytical solution under these boundary conditions. With recent, high-speed PC systems

and suitably optimized algorithms it is, however, possible to find the surface tension numerically exact within fractions of a second. Fig. 1 shows that with a contact angle of 90° and a straight cylindrical test body, no lamella can be created. If, however, instead of a cylindrical body a sphere is used, then a curved lamella will form even at a contact angle of 90° , because the spherical surface does not have a constant angle of 90° to the horizontal.

Procedure

In a specially shaped sample container the polymer or the hotmelt adhesive is molten. For this purpose the electrically heated temperature control system TEC 350 is used, which in this case replaces the sample stage of the optical contact angle measuring instrument OCA 20. The spherical test body is immersed into the molten sample so that its end is completely wetted. With the CCD camera a digital image of the lamella at the required temperature is recorded and saved. To calculate the surface tension, the program must work out the weight of the lamella. For this purpose the density of the molten material must be entered and the magnification ratio of the image taken must be determined. The density is entered by the experimenter, but the magnification factor is worked out from the test body geometry. After measurement, test body and sample container can be easily cleaned mechanically, for instance in an ultrasonic bath.

Results

By the example of a viscous hotmelt adhesive, below the lamella method is compared to that of the pendant drop. For this purpose, Fig. 2 shows the image of a pendant drop of the hotmelt at 120°C . Because of its rectangular format, the CCD camera was tilted by 90° from the horizontal to achieve the best possible image filling. From this image, a surface tension of $35.16 \pm 0.04 \text{ mN/m}$ was determined. A density of 1.0518 g/cm^3 was assumed.



Fig. 2: Pendant drop of a hotmelt 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 $34.81 \pm 0.35 \text{ mN/m}$ for the same density as in Fig. 2 was determined. Hence the two values agree within an error of 1% of the lamella method.



Fig. 3: Lamella of the hotmelt at 120°C

Summary

The new DataPhysics lamella method was introduced, which permits the rapid and easy determination of the surface tension of liquids at temperatures up to 400°C . With the example of a high viscous hotmelt adhesive, an accuracy of the method of 1% was achieved.