Meeting product quality demands by monitoring PU foam formation

By Bernd H. W. Hofmann, Format Messtechnik GmbH, Karlsruhe, Germany

about this feature

Key customer industries, such as automotive seat makers and furniture manufacturers, along with the insulation and construction industries, are setting increasingly stringent quality requirements for their suppliers, including the flexible foams used in their products. In some cases these go beyond the basic foam properties such as hardness and density, and require foamers to supply detailed information on the process parameters that applied during manufacture of the particular batch of foam.

At the same time, technologists want to gain a firmer control of the foam-making process, particularly in terms of ensuring that they consistently yield good products.

These various demands have led to the development and routine use of measurement devices for recording key characteristics such as foam rise height and changes in temperature, pressure, dielectric polarisation and mass loss during foaming. Additional information such as viscosity, gelling and curing behaviour can also be derived from the data determining the properties of the generated foam, making such measurements a valuable part of the process technologists' arsenal, as described in the article on pages 18-20.

And, of course, it is not just the foam sector that is putting increasing demands on its customers. Makers of products as diverse as cast elastomers, thermoset composites, adhesives and sealants also want to monitor their processes and characterise their products more comprehensively.

These various groups are capitalising on the characteristics of the scanning vibrating needle curemeter (SVNC) developed by Bryan Willoughby and colleagues at Rapra Technology Ltd, as described in the article on page 22.

David Reed, Editor

about this article

Format Messtechnik GmbH, based in Karlsruhe, Germany, has developed a measurement device, designated Foamat, for recording key foam-processing characteristics such as foam rise-height, reaction temperature, rise pressure, dielectric polarisation and mass loss during foaming.

Additional information such as viscosity, gelling and curing can also be derived from the data. This makes the instrument a valuable method for determining the properties of the generated foam, making such measurements a valuable part of the process technologist's arsenal, as described in the article below. **UT**

The quality of polyurethane foams depends on what happens during their formation. It therefore makes sense both to record the formation parameters by suitable means, and to check them regularly on representative samples.

A further benefit of such measurements is that the quality consistency of primary chemicals can be ensured, prior to their use in actual foam production.

Such monitoring is also very valuable when foam systems with special properties are being developed: measuring the formation parameters gives an insight into how the reaction is proceeding and how foam formation is affected by additives, blowing agents, and stabilisers, as well as changes in basic parameters such as the mixing ratio.

To achieve all these objectives, the Foamat measuring device can be used to show whether the foam fulfils stringent requirements in terms of measuring accuracy and reproducibility. It also offers versatility in accommodating different specimen shapes.

Rise profile: Foam fingerprint

The traditional method for characterising foams is to measure the rise height of an expanding foam sample in a cup, a cardboard box, or a cylindrical container over time. This yields what is generally referred to as a rise profile. The start time and the rise time are determined from the rise profile.

Although these terms have not been standardised, the start time is generally accepted to be the start of the reaction between the components A (polyol + additives) and B (isocyanate) after mixing. The foam rise continues until maximum expansion has been reached, and the time elapsed is called the rise time.

Ultrasonic sensors have proved to be most useful for measuring the height of the rising foam surface, replacing manual observation methods and their uncertainties. Bouncing an ultrasonic signal off the foam surface allows accurate monitoring of the rise process over time.

More recently, the accuracy of these devices has been significantly enhanced by the introduction of integrated temperature sensors and ventilation fans for temperature control to allow compensation for the fact that the velocity of sound is temperature dependent. Such units can be used for monitoring all types of foams, including rigid foams which tend to generate high reaction temperatures and may contain physical blowing agents (see Fig 1).

The rise profile is the fingerprint of the foam. The rise profile of a new formulation can be compared with that of a given master curve during quality assurance testing. The master curve has a tolerance band and the rise profile of a 'good' foam sample should lie within this band.

Using specially developed software, designated Foam, quality managers can generate master curves from a host of rise profiles for the range of representative formulations used in the company concerned. The various formulations used are documented, along with important measurement parameters such as mixing time and test time.

Change is coming

Although rise-height measurement remains the standard test method in foam qualification, this may be about to change. New techniques have become

Fig 1 The typical Foamat set-up ...

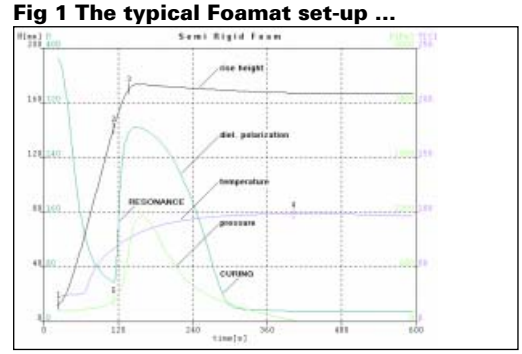

Fig 2 ...yields a wide range of data

Fig 3 System for pressure monitoring

available which, in addition to monitoring rise-height, simultaneously yield data for other physical values (see Fig. 2).

Parameters such as the rise pressure of the foam sample, the reaction temperature and the dielectric polarisation of the foam can all be monitored, and this extra data can be used to make inferences about the foaming reaction which cannot be gained solely from the rise profile.

The exothermic polymerisation reaction causes the temperature within the foam sample to rise.

However, the heat distribution within the sample is not uniform as it is affected by heat dissipation at the free upper surface of the foam, heat conduction through the container wall, the insulation properties of the foam, and the adiabatic expansion of the foam itself. The point at which the temperature change is measured within the rising foam can therefore be highly critical.

Experimentally, we have determined that the maximum core temperature is best measured by placing the thermocouple in the lower third of the total foam height. The exact location of the thermocouple above the bottom of the container can be established after the test: as the foam rises it meets the thermocouple and so causes a sudden temperature increase.

As the reaction continues, gelling of the foam components begins and pressure builds up within the foam. Since the foam is free to expand upwardly while the pressure is being measured, the ultrasonic fan sensor can measure the rise height simultaneously. In the gelling process, a matrix of stable cells forms which prevents the foam from expanding further and also stops any blowing agents from escaping. These two processes generate stress (pressure) within the foam which can be deleterious in practice.

For example, when making foam-backed wall elements with the foam providing insulation or rigidity, panels or sheet metal can become stressed at right angles to the direction of foam flow. In many cases, high forces can be generated, so that the production equipment has to be reinforced or supported until cure is complete. In extreme cases, these forces may even destroy a part, so monitoring this parameter has become an important requirement in product development of rigid foams.

The forces are measured as the rise pressure, so called because the local stresses arising after gelling are critically dependent on the foam rise height. Whereas the rise curve primarily records the dynamics of blowing agent formation, the rise pressure mirrors the properties of the foam cells, which are affected by the polymerisation reaction.

Rise pressure is measured using a special test mould or expansion container (FPM, Foam Pressure Measurement) into which the reactive foam components can be poured or injected (Fig. 3).

The expansion container replaces the standard test cups and comprises a cardboard cylinder that is put onto the flange of a lower metal cylinder bearing a force gauge, with a polyethylene film to protect the plate against contamination.

As the foam expands it stresses the bottom of the expansion container, and the force (pressure) is measured by the gauge.

When speciality systems are being developed, such as polyester foams with very coarse cell structure, measuring the rise pressure can yield important information about the effects of catalysts and stabilisers on gelling, tack-free time and post-expansion.

Pot life/curing monitor

By Bernd H. W. Hofmann, Format Messtechnik GmbH, Germany

Standard laboratory methods for examining the reaction profile of polyurethane-based formulations for CASE (coatings, adhesives, sealants and elastomers) and epoxy resins mainly aim to measure their mechanical and thermal parameters, such as viscosity build-up, pot life and cure time, and temperature changes during cure.

For example, the viscosity of the reacting liquid can be measured by using rotational or vibrational viscometers. The basic disadvantage of such devices is that the rotational or vibrational parts influence the setting of the material. On a practical level, the embedded parts are also difficult to recover after a test. And, finally, viscometers cannot be applied to very fast curing systems due to their slow response.

These difficulties can now be avoided, using a new technique we have developed: The pot life and curing monitor device, designated SubCASE (Fig. 1).

This provides both simple handling and reliable measurement data for PU-CASE formulations and epoxy resins. The unit combines a dielectric polarisation sensor with two temperature transducers. As discussed in the main article, dielectric polarisation gives insight into the changes as the mixture cures.

The test container is made of a disposable cardboard cylinder and a dielectric polarisation sensor, which forms the bottom of the container. The unit is mounted onto the heated base plate of the SubCASE unit. The dielectric polarisation sensor is covered with a thin plastic film to prevent any direct contact with the reactive mixture.

Polarisation data is obtained from the very beginning of the chemical reaction until the end of the curing process. A temperature probe is positioned in the centre of the dielectric polarisation sensor, with the core temperature being measured by an additional thermocouple positioned in the centre of the test sample. For process-near conditions, the base plate can be heated up to any controlled temperature, even as high as 110°C.

As an example of the use of the unit, measurements using SubCASE have been made with polyurethane cast skin formulations. Fig. 2 shows the test results of two formulations with different amounts of catalyst.

The black curves show a test with a standard formulation. The red curves show the reaction profile of a sample with a 200-percent excess of catalyst and show a significantly faster curing of the material. The temperature/time curves show that there is an exothermic reaction.

The software of the SubCASE unit analyses the data to determine the pot-life and curing behaviour of the system from the polarisation curves. The coloured areas around the 'good' curves are master curves used for quality control.

The new measurement technology thus gives detailed insight into the reaction profile of PU-based coating, adhesive, sealant and elastomer formulations as well as for epoxy resins. In addition, the SubCASE unit is easy to operate, making it suitable for use in quality control testing. **UT**

Fig 1 The SubCASE monitor

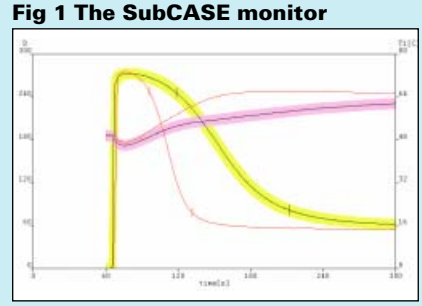

Fig 2 Display shows effect of catalyst level on cure

The pressure curve also yields valuable information for actual production purposes: it shows the pressure decay time, thus indicating when it is safe to open the mould. This helps processors avoid tearing of the foam and post-expansion problems; it can also allow processors to optimise processing time by preventing foams from spending too long in the mould.

A particular advantage of measuring the pressure at the bottom of a cylindrical expansion container is that this allows the viscosity of the generated foam to be calculated continuously from the experimental data.

Using traditional rotary or vibration viscometers to measure the viscosity of foams directly is difficult because the foam is increasing in volume and turning solid. When using such viscometers, the test probe either extends to different depths in the foam—giving variable results—or loses coupling with the sample. And, finally, the probe becomes stuck tight within the cured foam.

These technical and practical problems can be avoided by using Hagen-Poisseuille's viscosity model in conjunction with the FPM unit. This model assumes that the viscosity of the foam is determined by the force necessary to move a length element of foam at a specified speed through a tube—in this case the cardboard cylinder.

Naturally, the foam is not pushed through the cylinder by pressure from outside; rather, the foam itself exerts pressure by expanding. Only the reaction force is measured from the outside and it is obtained directly from the rise pressure.

As a result, monitoring the rise pressure and the rise profile in a cylindrical expansion container supplies all the data necessary for calculating the viscosity using the Hagen-Poisseuille model.

For optimising the foam propagation in a complex mould, the viscosity is an important piece of information that can be exploited, for example, in numerical foam expansion models.

The heart of the device

Another recent development in this field is the use of sensors that monitor the dielectric polarisation of the reacting system. The dielectric polarisation is measured as the change in capacity in the system under study relative to that of an empty container.

Because the parameter is strongly affected by the polar molecules of the base components, it decreases dramatically when polymerisation starts and the molecules become less mobile, so it can give information relating to the state of cure of the polyurethane molecules within the foam.

For any kind of foam the state of cure can be evaluated from the steady decrease of dielectric polarisation after maximum rise before the system finally gives a constant reading at a very low level. Dielectric polarisation sensors are thus the heart of any cure-monitoring device (CMD).

Independently of the dipole moments, the polarisation signal is also influenced by the foam density, which decreases during the expansion (rise) phase.

However, as shown in Fig. 2 on p18, although the signal falls dramatically in the first phase of the foaming process, there is often a sharp increase in dielectric polarisation during the rise phase. This is attributed to an increasing contribution from the dipoles of the intermediate polyurea molecules which are formed by the gas-generation reaction. When polymerisation is in progress, the polarisation signal falls again, as is also shown in Fig. 2 on p18.

For reproducible dielectric polarisation measurements, the foam must be in close contact with the sensor. This is achieved by positioning the sensor on the bottom of the expansion container (Fig. 4). Because of the rise pressure, the foam is pressed onto the sensing surface, thus ensuring the desired good contact.

While a key benefit of the systems so far described is that they reduce operator subjectivity, in order to obtain reproducible experimental data from the rise profiles and other parameters, the reaction components must be weighed out exactly. This is not so easy. Despite the utmost care by the operator, remnants of foam adhering to the mixer head and the mixing cup, as well as gas losses during foaming can lead to differences in the actual foam mass produced.

To minimise such factors, it is possible to integrate a laboratory balance into the measuring device, a process which can allow automated recording of the mass of each component during formulation. Additionally, mass loss due to the release of blowing agents and volatile components during foaming can be recorded on a continuous basis.

This approach is also useful when special additives are used to make undercrosslinked foams, such as viscoelastic/slow-recovery foams, or when polyester foams for reticulation are being made: the loss of mass during foaming provides data for assessing the evaporation rate of the whole system.

Another advantage of an integrated balance is the automated determination of the gross density from the mass of the finished foam sample and the measured final rise height. For simultaneous monitoring of rise pressure and dielectric polarisation using FPM and CMD, after pouring the mixture into the test container the mixing cup with the remaining foam mass is placed on the balance. All mass data is recorded and displayed together with the other measurement curves.

Fig 4 Cure-monitoring system

Software is critical

All of the above measurement and monitoring relies heavily on modern computer technology, as does the detailed analysis and presentation of the information in a usable form.

The Foamat unit uses PC software called Foam, which is Microsoft Windows-based and gives maximum objectivity of all measurement values. It controls the weighing and mixing stages as well as ensuring precise data acquisition and analysis, including the presentation of data in an understandable format and the display of curves.

If human intervention is required, each step can be performed in dialogue with the user. All data is saved automatically using an explicit format, which can be simply imported to any database program. Standard functions such as marker lines, zooming of curves and making comments on the results, are available at a mouse click, making the unit useful in both production and research environments.

The shrinkage of the foam sample can be measured at any time after the test. The generation of master curves is software-assisted using additional symbol-operated functions.

The reliable and rugged sensors of the Foamat unit, coupled with the user-friendly Foam software, make foam qualification an objective method according to the state of the art. **UT**

about the author

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