APPLICATION NOTE

HAAKE RheoWin Software - Features for Quality Control and Routine Measurements

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Introduction
Rheology is used in more and more industries for product development and quality control. A trend is to develop standard operation procedures like it is common for many other analytical methods. Standardized measuring procedures are necessary to optimize product properties and to determine possible qualitative fluctuations of a product.

By determining the relevant rheological parameters, relationships between structure, process behavior and final product properties can be established. Quality standards can be fulfilled and new products launched on the market faster. This applies to many industrial fields such as plastics and adhesives, paints and coatings, personal care and detergents, foodstuffs or even building materials.

Rheological measurements provide information about storage stability, processability or flow properties of liquid and semi-solid formulations, which are essential for quality control and further product development.

However, due to the increasing complexity of the materials and the high performance requirements, it is not always easy to develop the optimal testing method for a particular application. In this context, a versatile measurement and evaluation software can provide useful support to both, beginners and experts in the field of rheology, in order to establish and execute suitable procedures. The Thermo Scientific™ HAAKE™ RheoWin™ is the instrument control and data evaluation software for all Thermo Scientific™ HAAKE™ rheometers and is used to set up and run measurement procedures with subsequent automatic data evaluation and report generation.

The following article is intended to provide an overview of the possibilities offered by the HAAKE RheoWin software in order to be able to perform a comprehensive and meaningful rheological characterization in quality control.

General structure of the HAAKE RheoWin software
The HAAKE RheoWin software can be changed to 12 different languages with the touch of a button and consists of three different modules:

- The HAAKE RheoWin JobManager for creating and executing measurement and evaluation routines - so-called Jobs. The JobManager also allows for controlling all functionalities of the rheometer individually and outside of complete measurement and evaluation routines.
- The HAAKE RheoWin DataManager for displaying and further evaluating measured data. Different data sets can be overlaid and compared. Graphic and table layout can be formatted and data be transferred into various formats.
- The HAAKE RheoWin UserManager for creating user accounts of different levels and assigning specific and individual user privileges.

In addition, the following optional software modules are available for specific demands and applications:

- A CFR Part 11 tool to meet the requirements of US FDA 21 CFR Part 11
- Additional modules for polymer analysis: Time Temperature Superposition (TTS), generating relaxation spectra and the calculation of molecular weight distribution (MWD)
- Software module for interfacial rheology
- Software module for combination with FT-IR spectrometer

For creating Jobs with the HAAKE RheoWin JobManager, predefined graphical elements are used (Fig. 1). These elements cover all aspects of a complete standard operation procedure, including:

- pre- and post experimental instrument settings
- experimental settings
- data evaluation
- data handling, export
- report creation

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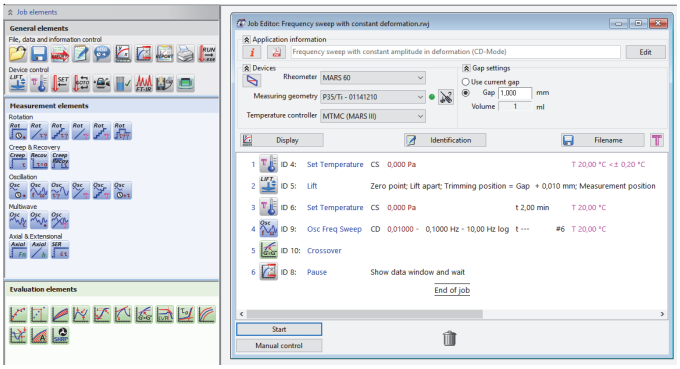

Fig. 1: Graphical elements for creating measurement and evaluation routines in the HAAKE RheoWin JobManager.

All elements can be selected quickly and comfortably via a drag & drop method. All measurements and evaluations can be carried out fully automatically in a single Job sequence. A manual operation mode can be used to perform rheological pre-tests or for gap setting and temperature control outside a measurement and evaluation routine.
The HAAKE RheoWin software allows for simultaneous sample testing and data evaluation or multiple measurements with different rheometers connected to the same PC. Measurement results can be exported and stored in various formats (ASCII, MS-Excel or PDF). The connection to a laboratory information management system (LIMS) is also possible.

Selected measuring and evaluation elements for rheological characterization in quality control and product development

Important product properties for consumers and manufacturers are for instance storage stability, viscosity, yield stress, thixotropy or curing behavior. The determination of these rheological parameters allows for improving product performance as well as for an effective and reproducible evaluation during quality control.

How does a product react under stress or strain? How do different additives, such as fillers or pigments affect the properties of a material? Which thixotropic agent and how much of it has to be added to a product in order to maintain good flow properties or to prevent sagging effects? How should a yield stress be adjusted in order to ensure good shelf life and transportability of a product? How quickly should a micro structure rebuild, after a material was exposed to high shear?

These are just some of the many questions that product manufacturers have to deal with over and over again. The HAAKE RheoWin software provides evaluation routines with the possibility of defining QC criteria for many standard testing methods (Fig.2).
The following section presents selected options offered by the HAAKE RheoWin software to perform effective quality control test routines. The predefined measuring and evaluation elements only have to be adapted to the respective product and the corresponding quality criteria have to be defined accordingly.

Reference curve with deviation tolerance

By using the reference curve element, new measuring results can be compared with previous measurement

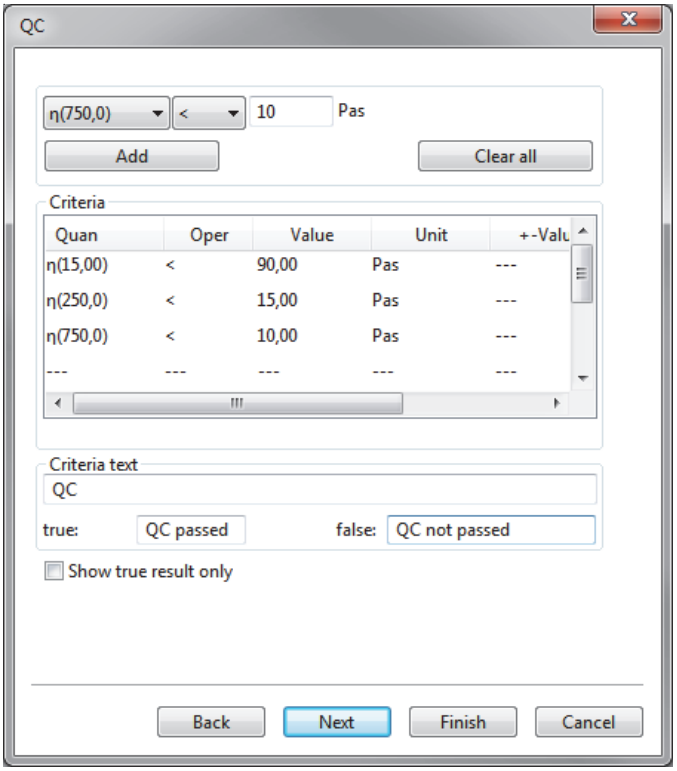

Fig. 2: Definition of Quality Control criteria in a measurement and evaluation routine.

data (Fig.3). A reference curve can be either a fixed data file or selected individually for every measurement by the operator.
The tolerance by which the measured values may deviate from the reference curve can be specified either as percentage or absolute values. Furthermore, the deviation tolerance be can set to either a linear or a logarithmic scale.

Interpolation with deviation tolerance

Interpolation is the calculation of a data point between two measured values. An interpolation can be performed automatically after a measurement (Fig. 4). Various methods are available for this purpose. For the interpolated values, a deviation tolerance can be defined in the HAAKE RheoWin software. Interpolated values need to be inside the deviation tolerance in order for the sample to pass the quality assessment.

Curve fitting

The HAAKE RheoWin software allows for performing curve fitting with various mathematical and rheological models (Fig. 5). Fitted curves can be extrapolated beyond the range of measured data. After a curve fitting was performed all calculated parameters are reported and stored along with the measured data.

Determination of the linear-viscoelastic range

Within the linear-viscoelastic range rheological parameters are independent of the applied stress or strain value. The linear-viscoelastic range of a sample can be determined by performing an oscillation amplitude sweep at constant frequency. The HAAKE RheoWin software allows for the automatic determination of the linear-viscoelastic range from amplitude sweep data (Fig. 6). The end of the linear-viscoelastic range is reached at stress or strain values, where the rheological parameters will start to change from a constant behavior.

Which parameter is used for the evaluation (G', G'' Iη*I, δ or tan δ) can be selected by the operator. The linear-viscoelastic range can be determined either as a stress or strain value. The HAAKE RheoWin software also allows for the determination of the linear-viscoelastic range according to the DIN 51810-2 standard procedure.

Area under a measuring curve

The area under a measurement curve or a selected curve section can be calculated by the HAAKE RheoWin software (Fig.7). Upper and lower deviation tolerances can be defined as a quality control criterion.

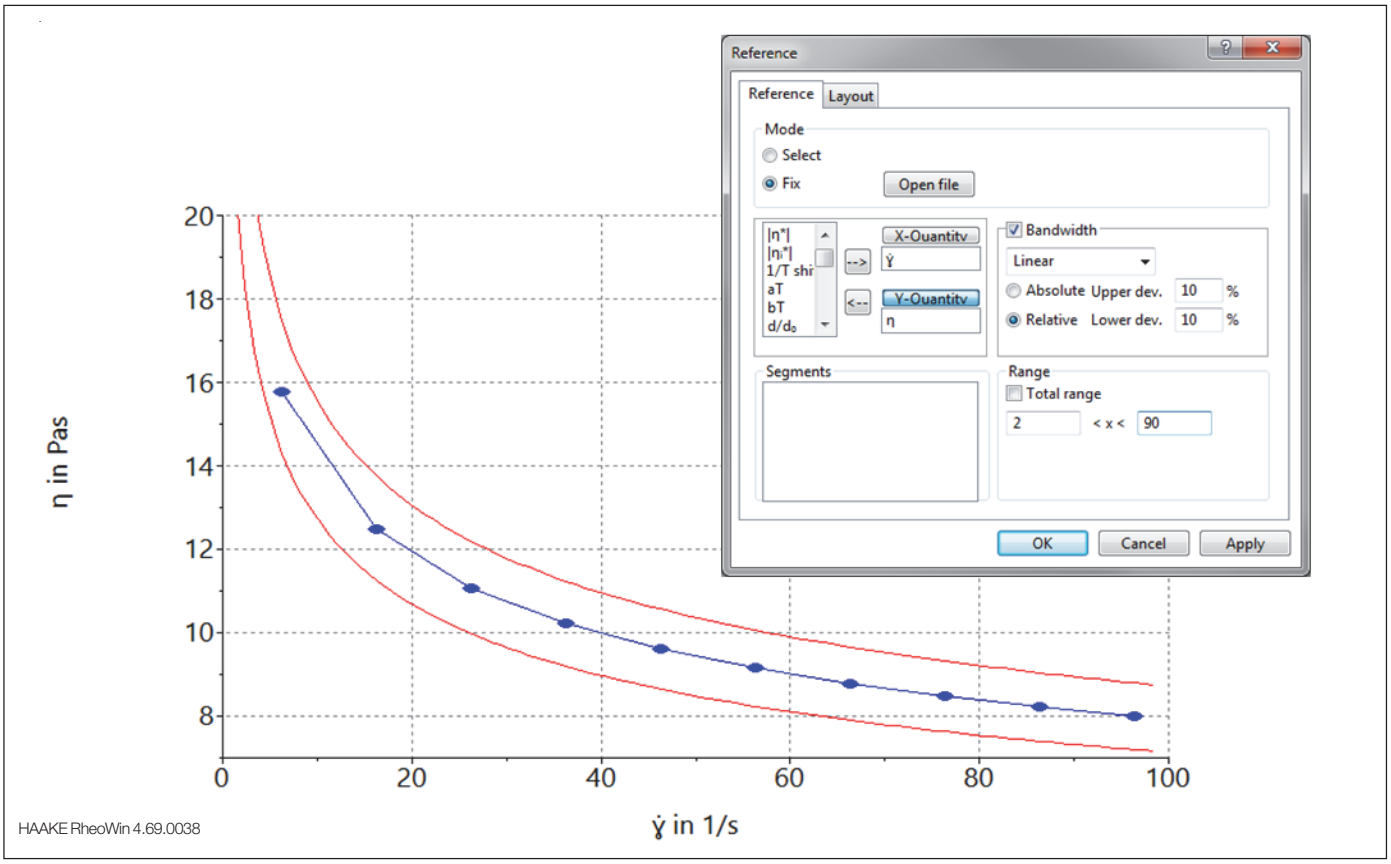

Fig. 3: Reference curve with deviation tolerance.

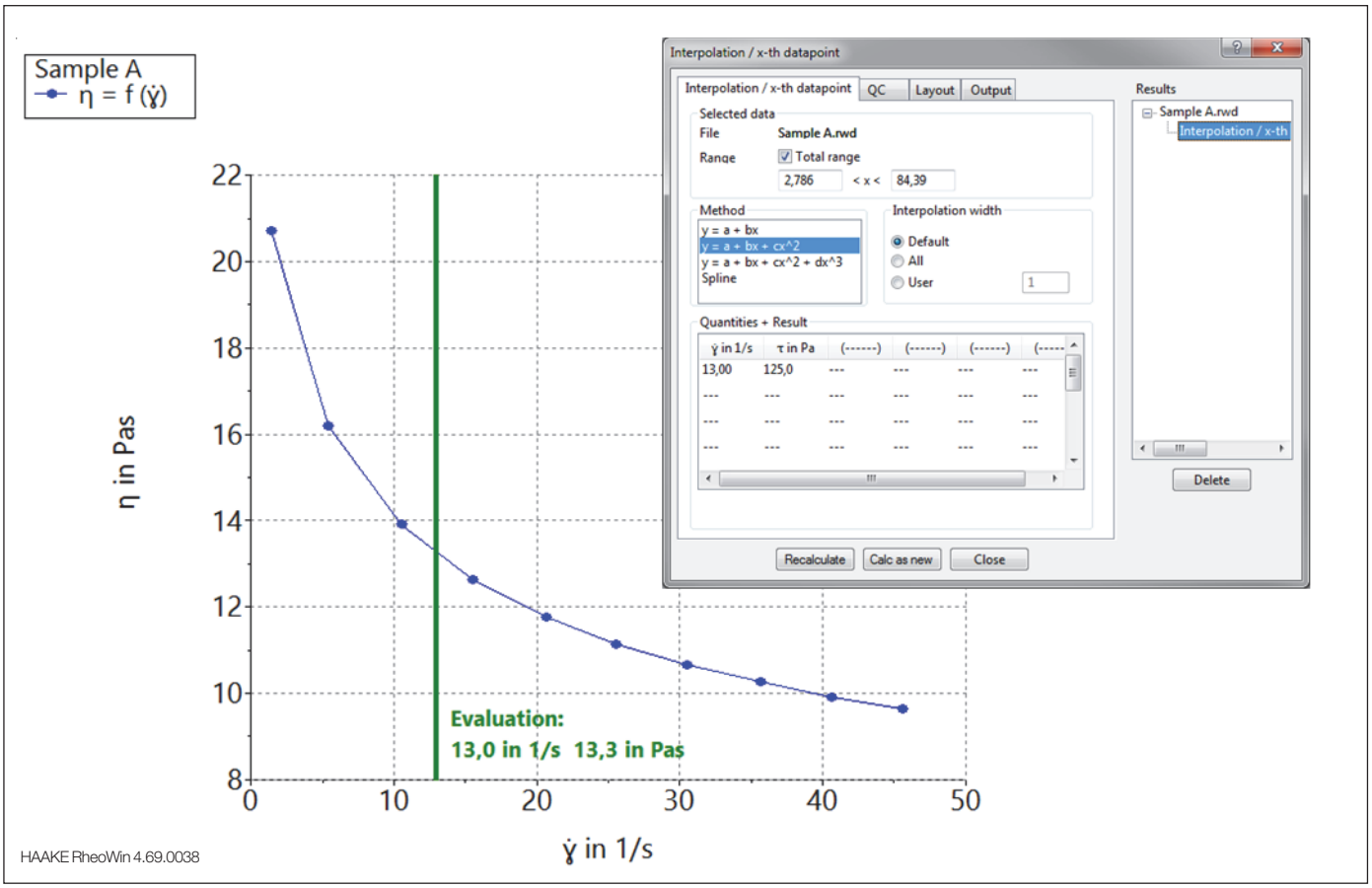

Fig. 4: Interpolation of a viscosity value at a defined shear rate.

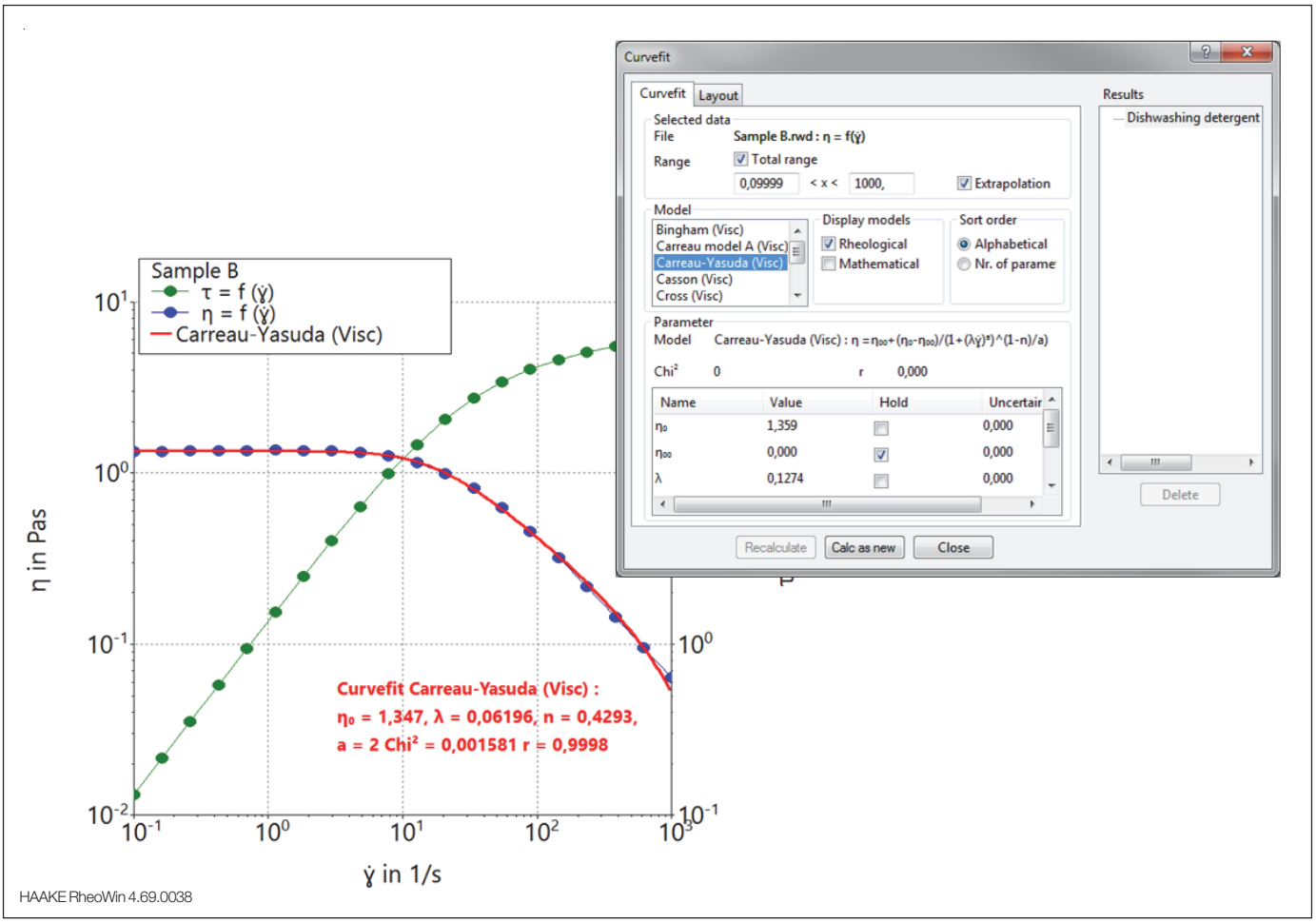

Fig. 5: Curve fitting of viscosity data with the Carreau-Yasuda model.

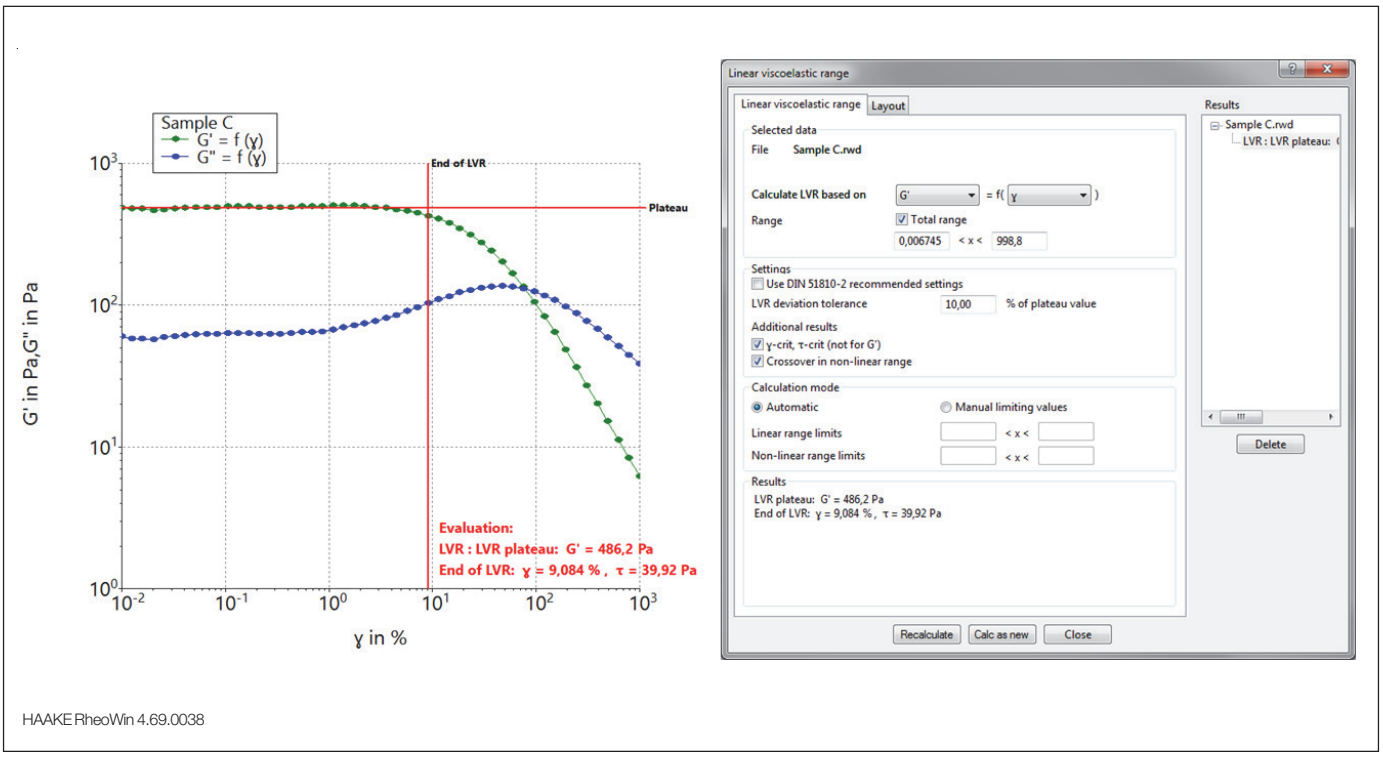

Fig. 6: Automatic determination of the linear-viscoelastic range from an amplitude sweep test.

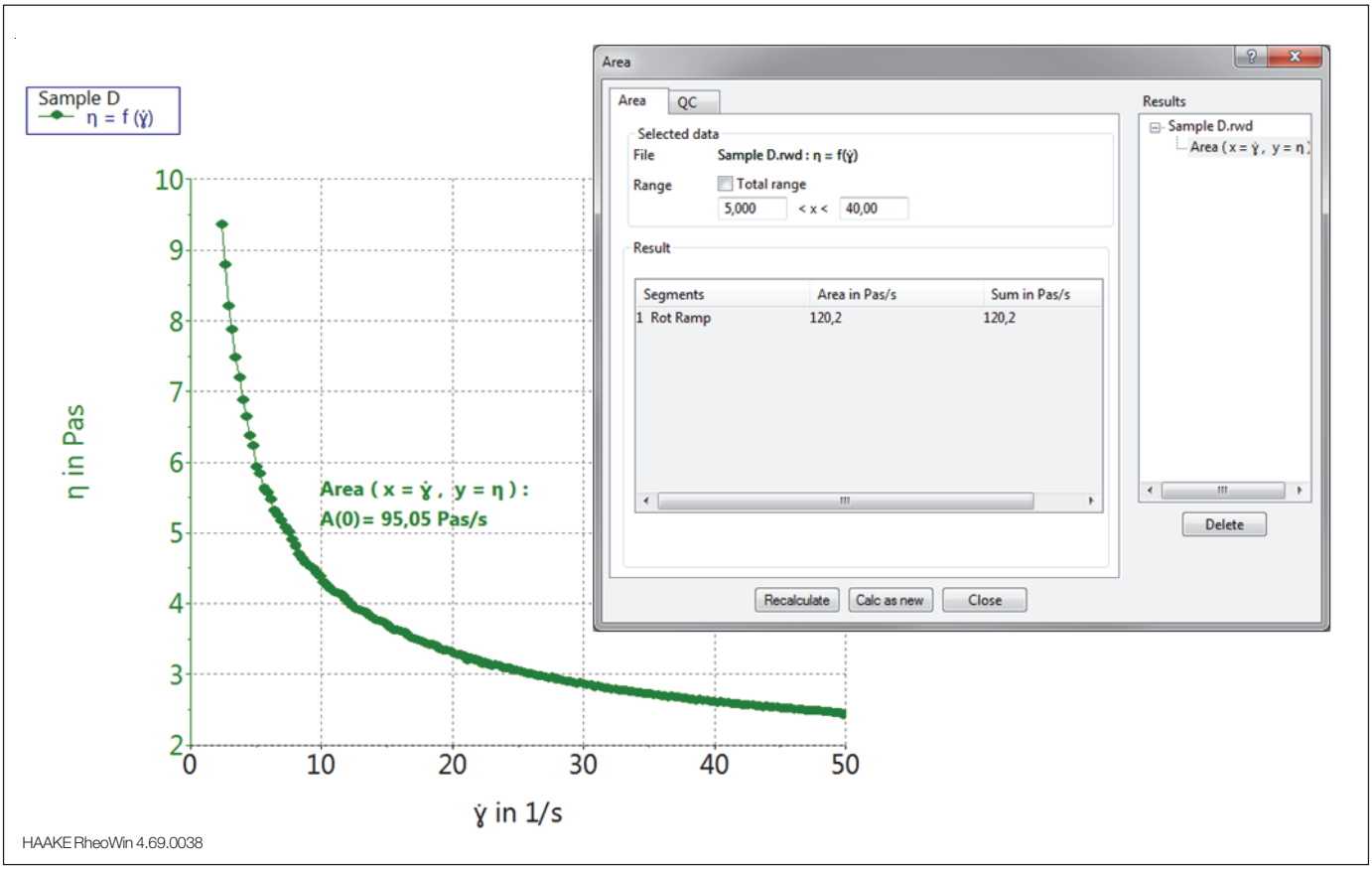

Fig. 7: Calculating the area under a viscosity curve.

Thixotropic loop test

Thixotropy refers to a shear rate and shear time depending decrease in viscosity of structured fluids. In a thixotropic loop test, a sample is exposed to an increasing followed by a decreasing shear rate ramp. The apparent viscosity and the shear stress are recorded as a function of shear rate. The hysteresis area that forms between the up and the down curve is a measure for the degree of thixotropy of the sample. When performing a thixotropic loop test, the HAAKE RheoWin software can determine the hysteresis area automatically for the viscosity or the shear stress data (Fig.8). Quality control criteria with deviation tolerance can be defined.

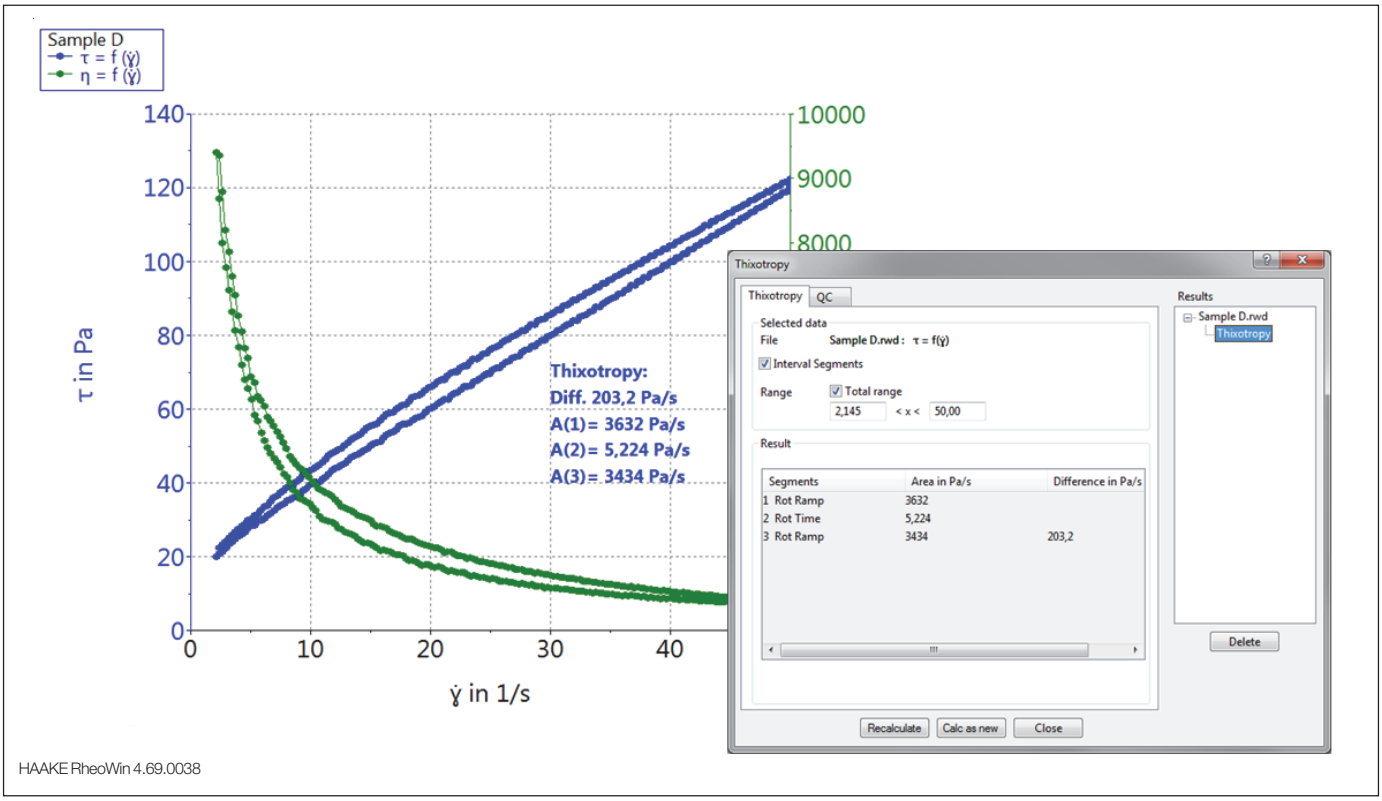

Fig. 8: Determination of the hysteresis area for shear stress data from a thixotropic loop test.

Yield stress

The yield stress of a sample can be determined by performing a shear stress ramp experiment where the deformation is recorded as a function of the increasing stress in a double logarithmic plot. At shear stresses below the yield stress, the deformation will increase linearly (slope of around 1 in a double logarithmic plot) with increasing shear stress. When the shear stress is approaching the yield stress the slope will increase and the sample will start to flow. The yield stress evaluation element in the HAAKE RheoWin software determines the yield stress by means of two tangents that are applied to the measuring curve. The intersection of these tangents is interpreted as the yield stress of the sample (Fig. 9). QC criteria with a deviation tolerance can be defined.

Curve discussion

Two different elements for curve discussion are available in the HAAKE RheoWin software. With the regular curve discussion element minima, maxima, smallest, highest and mean values of a measurement curve can be determined (Fig.10). With the advanced curve discussion element absolute and relative slopes, percentage of reference values as well as the intersections of tangents applied to the measured data can be calculated (Fig. 11). For both curve discussion elements quality control criteria can be defined.

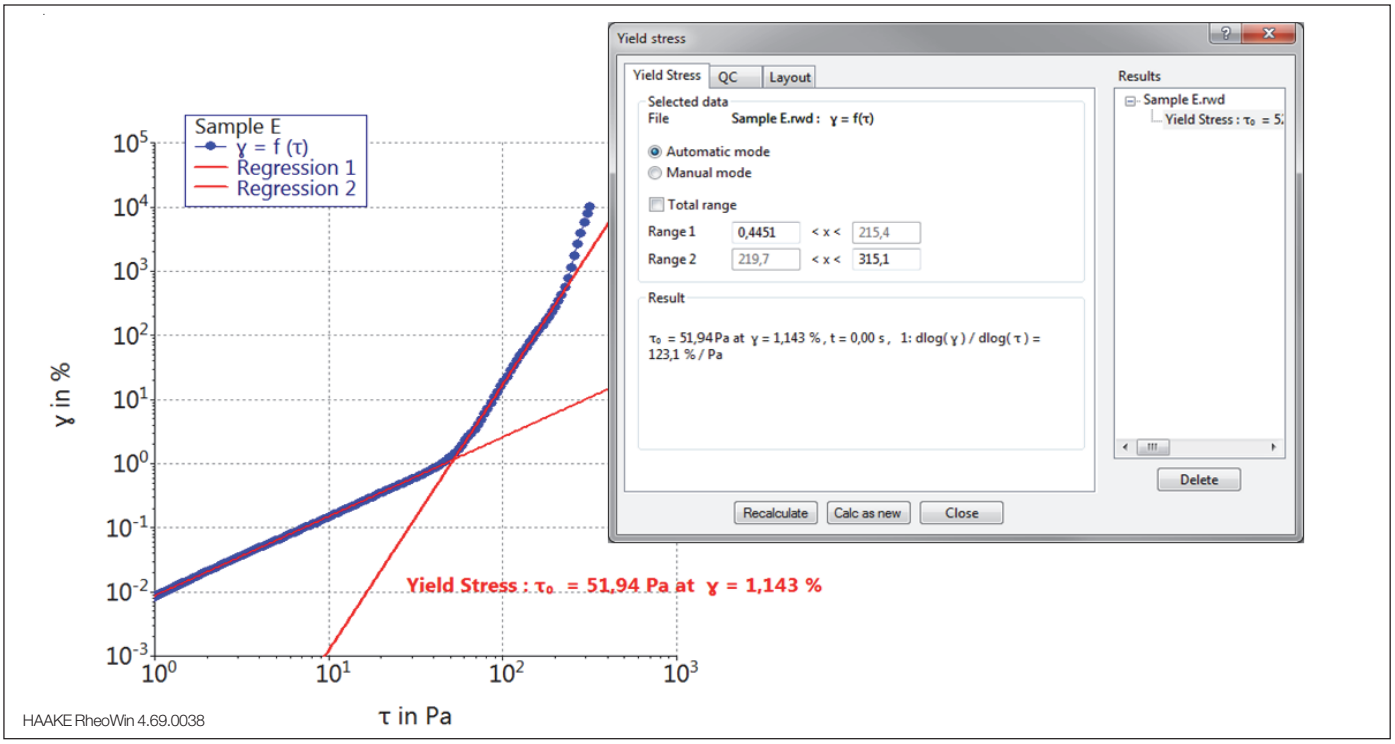

Fig. 9: Automatic determination of yield stress from shear stress ramp data with tangent intersection method.

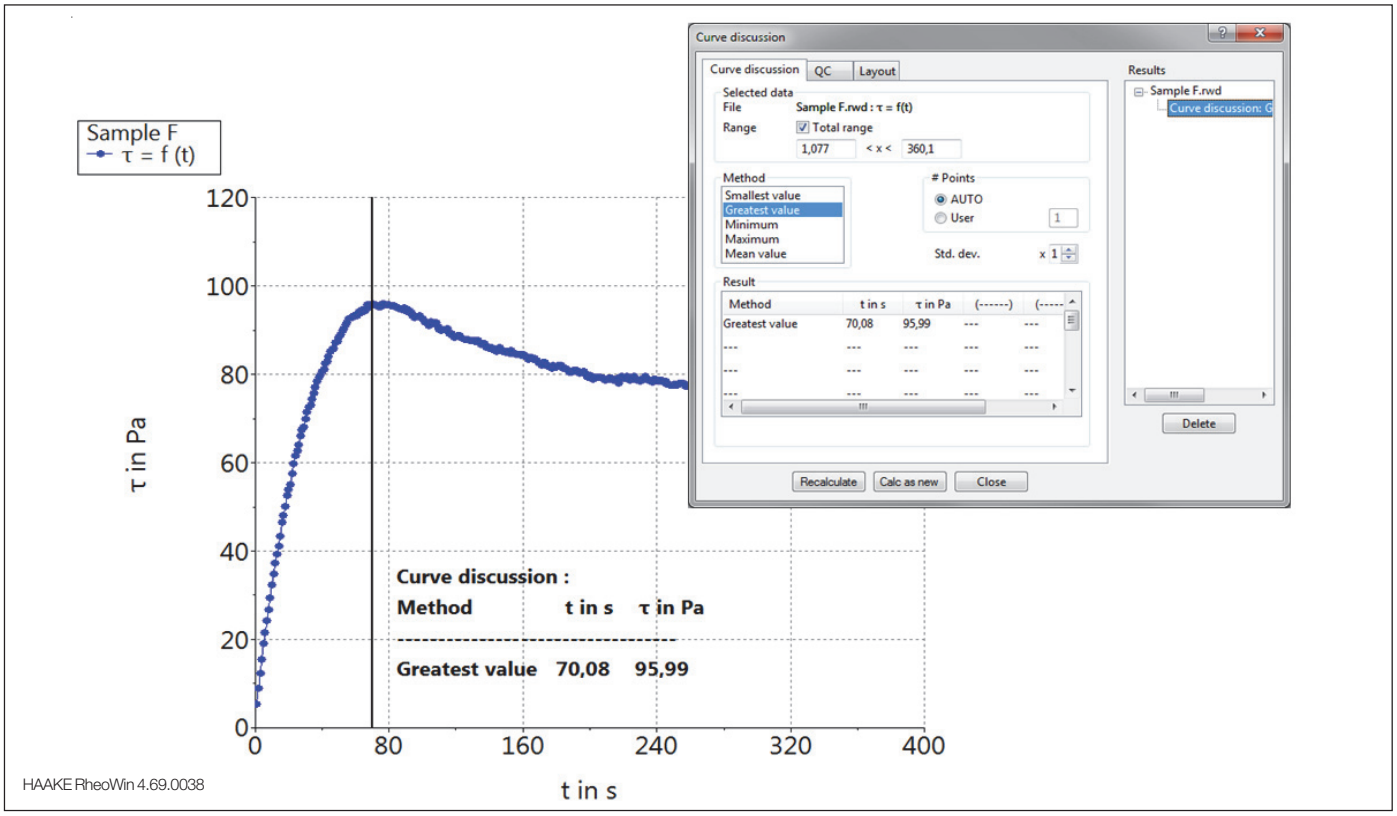

Fig. 10: Determination of the greatest value for the shear stress with the curve discussion evaluation element.

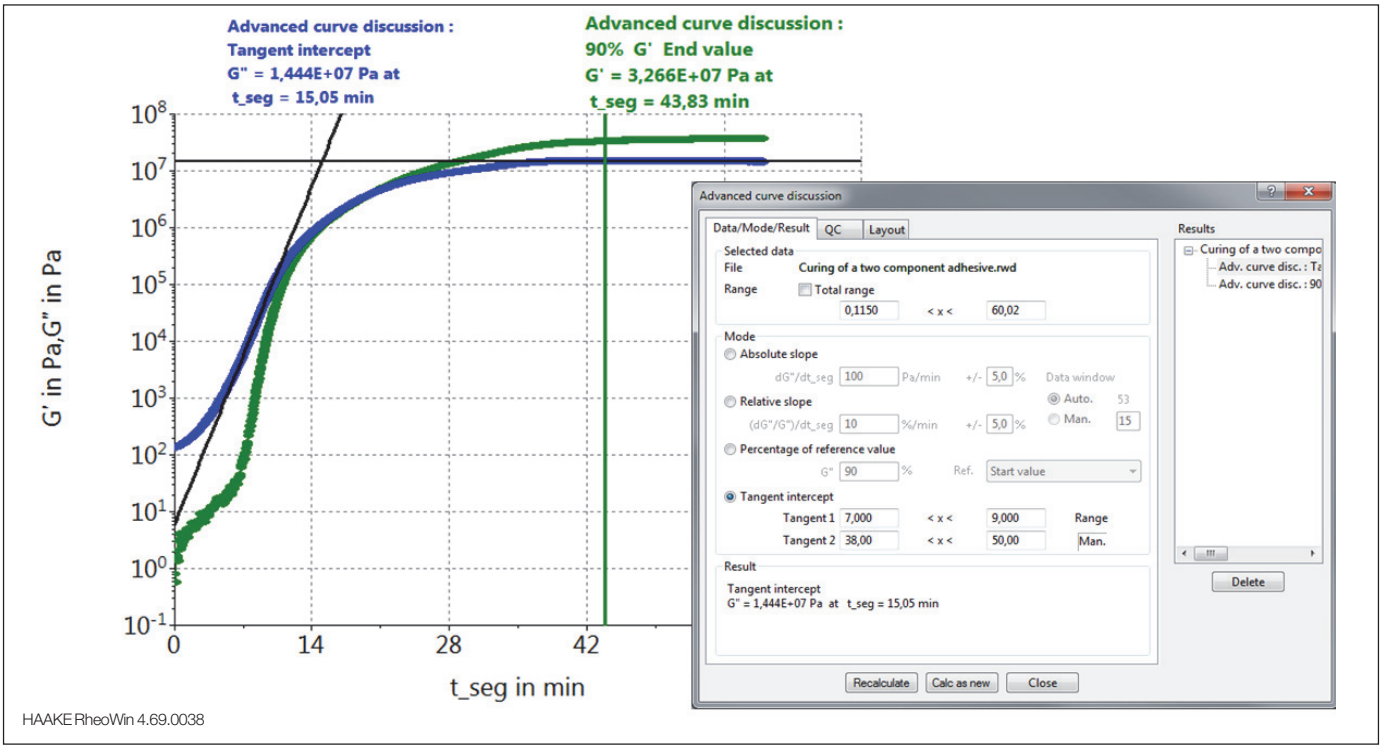

Fig. 11: Advanced curve discussion with G' and G'' data from a curing experiment.

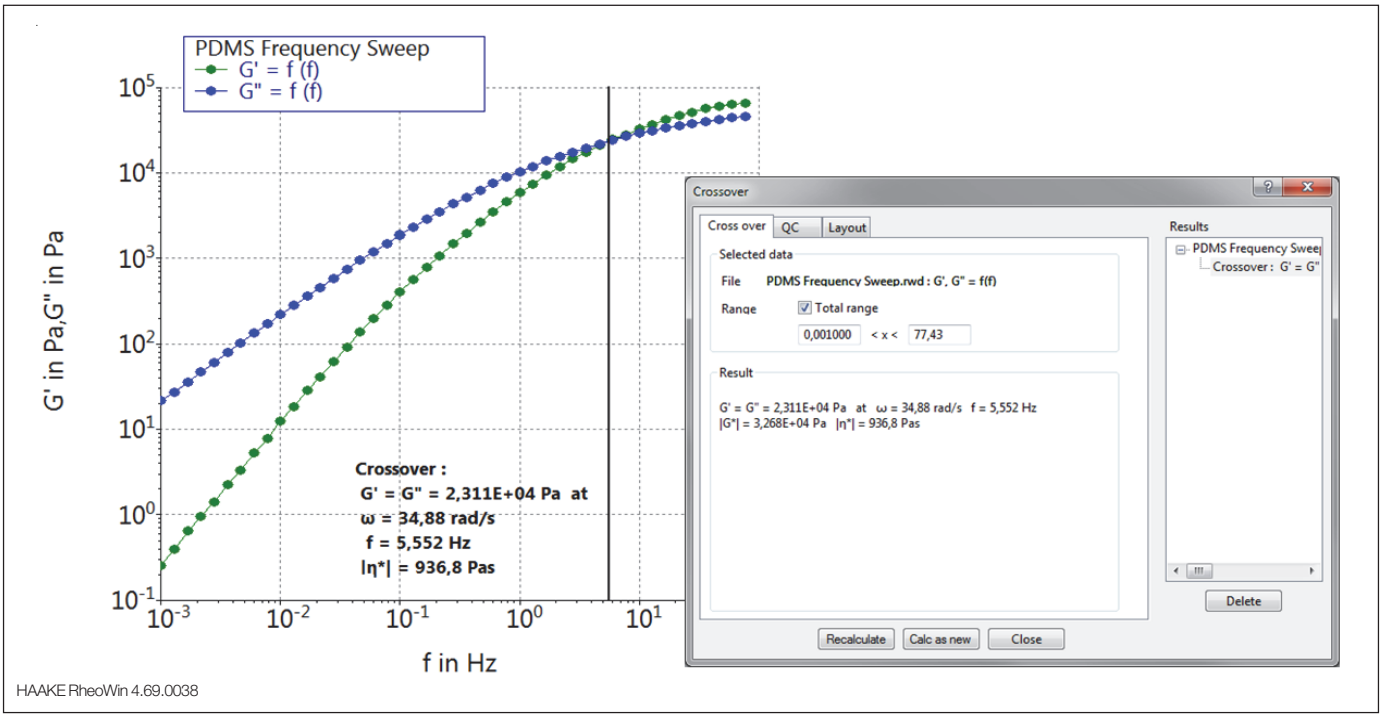

Fig. 12: Determination of the crossover of G' and G'' in a frequency sweep test.

Crossover

The crossover evaluation element determines the intersection point of the storage modulus G' and the loss modulus G" curve from an oscillatory shear measurement. These include amplitude, frequency, time and temperature sweep experiments (Fig. 12). Quality control criteria with deviation tolerance can be defined.

Structure recovery

The structure recovery element provides information on how quickly and how well the structure of a sample recovers after it was exposed to a high shear rate. A structure recovery test consists of three steps. In an initial step the viscosity or complex viscosity of a material with an intact structure is measured as a reference. The second step is a high shear rate period to break down the microstructure of the sample. In the third step the applied stress or strain signal is reduced to the initial value again and the recovery of the sample after a high shear impact is monitored. The structure recovery evaluation element of the HAAKE Rheowin software compares the data from the first and the third element to assess the recovery (Fig. 13). The evaluation options include the absolute change from the first to the end of the third step, relative recovery after a defined period of time and a relative recovery back to a defined percentage. Additionally, the time until the crossover of G' and G'' occurs (sample becomes predominantly elastic again) in the recovery step can be detected automatically.

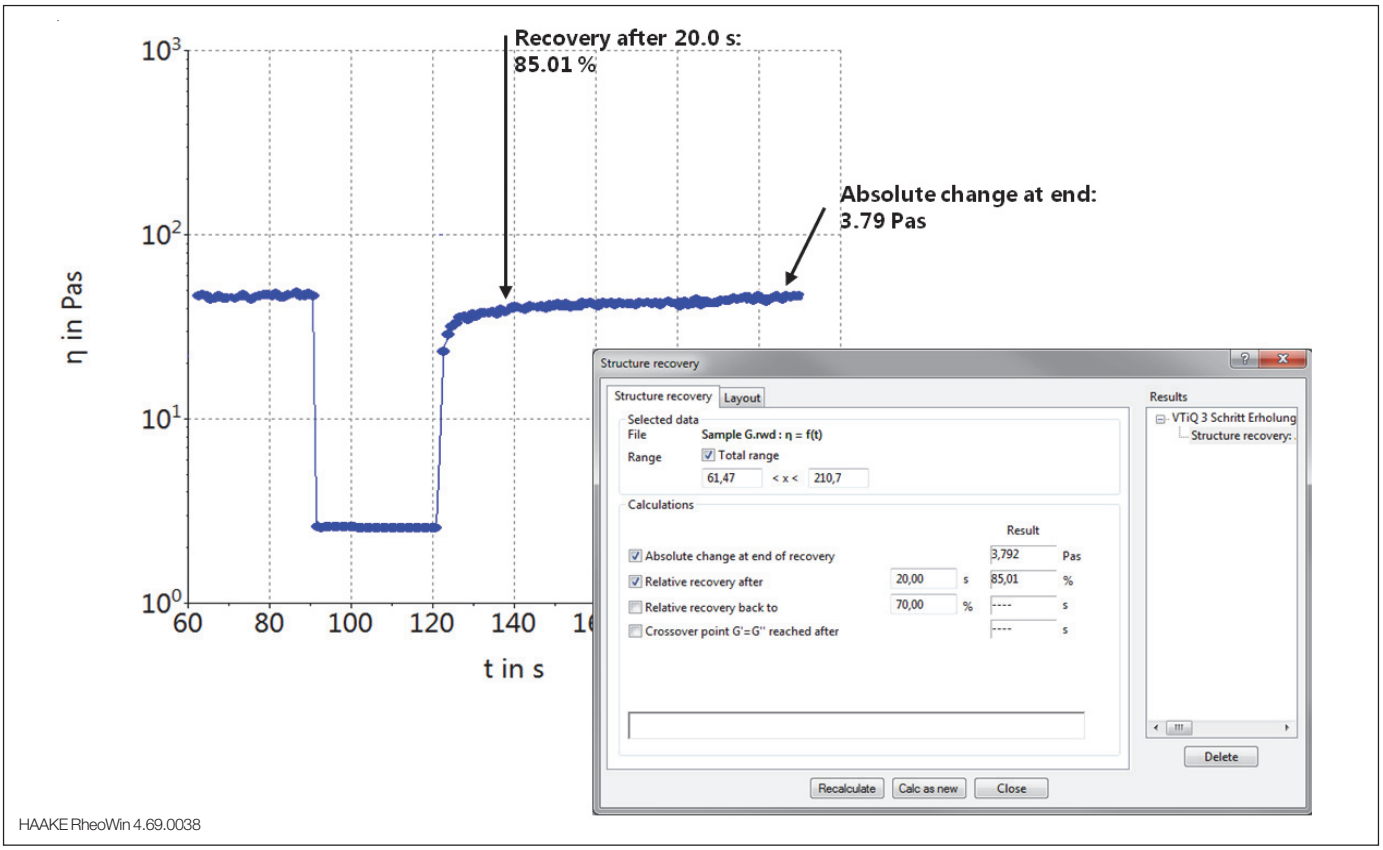

Fig. 13: Evaluation of the structure recovery after a sample was exposed to a high shear rate.

Creep recovery

Creep and recovery tests are the most direct way in rheology to qualify and quantify the elasticity of a material. The experiment is divided into two segments. During the first part, the creep, an instantaneous stress signal is applied to the sample for a defined period of time. In the second part, the stress is removed again and the recovery of the sample is monitored. The response of the sample is a deformation curve with a shape depending on both, the amount of stress applied by the rheometer and the microstructure of the sample.

The creep and recovery evaluation element allows for an automatic and comprehensive creep analysis (see Fig. 14). Parameters like the zero shear viscosity, recoverable deformation or equilibrium compliance can be extracted from the creep and recovery curves. Quality control criteria with deviation tolerance can be defined for the different evaluation parameters.

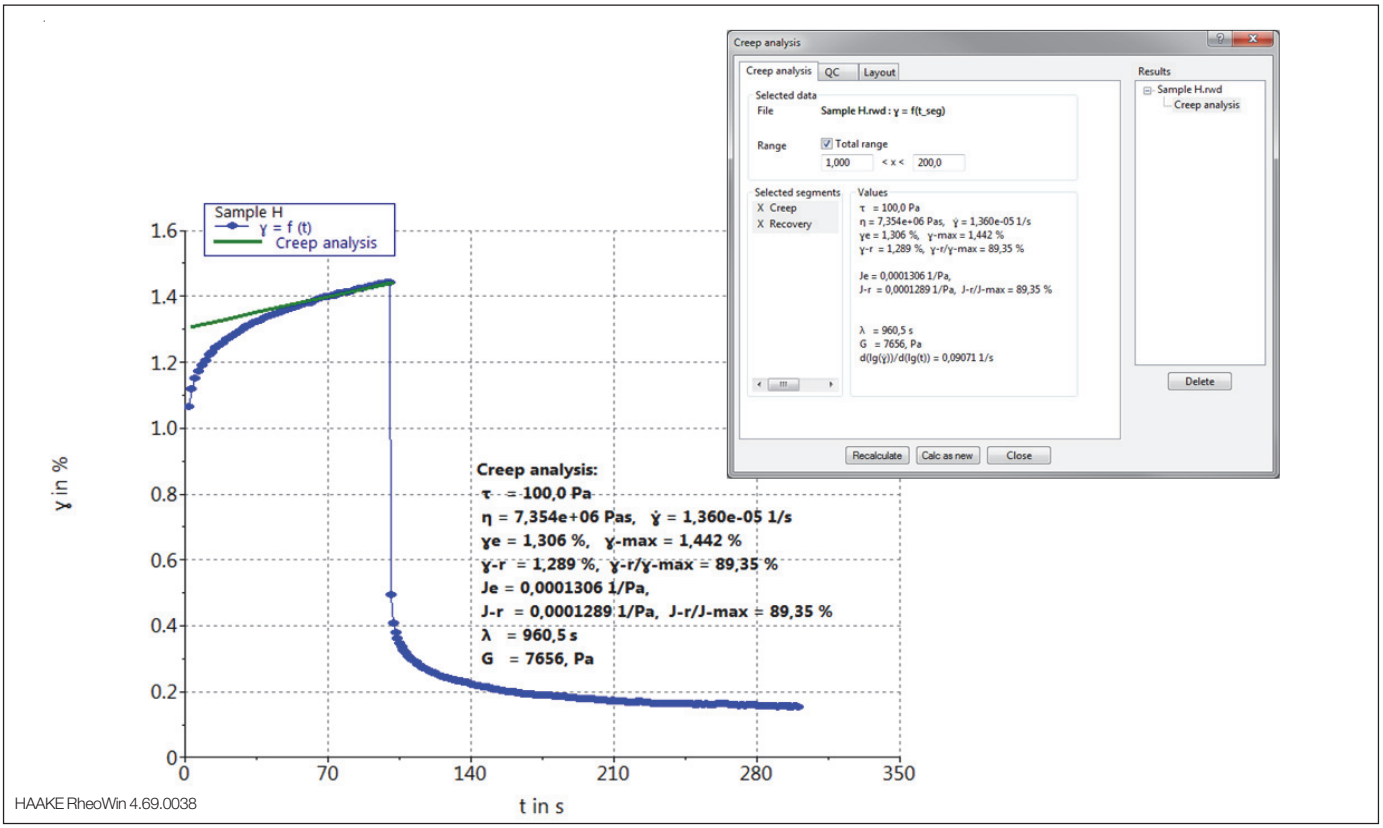

Fig. 14: Analysis of a creep and recovery test.

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Summary

The HAAKE RheoWin software enables a comprehensive rheological characterization of liquid, semi-solid and solid materials. Users can quickly and easily create suitable measurement procedures for various products and applications. A broad range of data evaluation routines allow for an automatic data analysis and the integrated QC criteria for a convenient operation in Quality Control.

Find out more at thermofisher.com/rheometers

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