

The thermo mechanical and thermo dynamic analysis

Introduction

The dynamic mechanical analysis (DMA) is an excellent method to determine the physical properties of polymers. There are a great number of applications where it is necessary to determine the mechanical properties of a material for quality control purposes. In many cases it is necessary to use a standardized test procedure to determine if a material meets a quality standard. In addition, the TMA can perform long term aging studies.

Basic principle of operation

The DMA is the test method under all available thermo analytical methods that

The response to the applied signal is delayed on the time axis and the time delay is described through the phase shift. The relation of the dynamic deformation component to the elastic tension contains a complex value E^* , which is a vector that can be split into a real part E' (storage module) and an imaginary part E'' (loss module). The storage module describes the ability of the material to store energy and describes the stiffness of the material. The loss module describes the part of the applied mechanical energy which was transferred into heat within the sample.

All viscoelastic materials including polymers show a time and temperature dependency. This can be easily shown through DMA tests. The DMA measures the temperature of the

gives the highest resolution in order to find small changes in the material structures. In addition it is possible to retrieve good information about the stiffness and the damping behavior, or the energy absorbing properties of a material, as a function of temperature or as a function of frequency. When working with dynamic mechanical analysis a force with a sine wave shape (square wave) is applied to the sample. Then the response coming from the material is measured.

glasspoint were a material changes from a hard state into a soft and flexible condition. These measurements can be used to understand the changes in stiffness of a material as a function of temperature and time. The DMA measures the energy absorption or the damping characteristics of materials. It is also possible to study softening points below the glasspoint, that are in conjunction with user characteristics of material and even a ruff estimate of the life time of the product is possible.



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Technical data

- temperature range
 - 150° C to 500° C
 - 20° C to 1000° C
- applied force
 - static 0 to 500 mN
 - dynamic 0 to 500 mN
- wave form
 - off, square wave, sign wave,
 - triangle
- frequency
 - 0,001 to 1 Hz
- max. sample dimension
 - Ø 8,5 mm, length 30 mm
- measuring range
 - +50 µ to +2500 µm



Picture: measuring head

Concept and design of the LINSEIS TMA/DMA system L77

The complete instrument consist of 3 parts:

1. basic unit with measuring head and furnace
2. amplifier
3. PC [including controller card with screen and color ink jet printer)

The basic unit is always equipped to run static and dynamic forces. There is a possibility to run under vacuum and under different gases.

The automatic zero setting of the LVDT sensor is built in standard. Optionally the automatic sample length measurement is available. Also a controlled cooling up to -150 °C is possible (option). The amplifier which is housed in its own chassis includes the control circuits for the LVDT control of the linear motor applying the force and for the thermostat that controls the temperature of the measuring head. All controls and switches are placed for easy operation.

The computer system has a Pentium processor and includes the input card controller that needs two 8Bit ISA slots.

Accessories

A comprehensive package of accessories is available for the L77 system. Different fixtures are available as well as different cryo parts for low temperature.



The LINSEIS DMA L77 is especially suited to determine the viscoelastic properties of polymers. The measuring system has the ability to put a force with varying frequencies and different signal forms on to the sample. The heating rate is available in a wide range from 0,1 - 50 K/min. The measurement can be done through a dynamic as well as through a static test procedure, This enables the acquisition of floating long term expansions and the determination of transformations into a stress free condition. There are different mechanical measuring fixtures available: penetration measurements, expansion measurements, measurement under pressure and under tension. All these measurement modes enable a large variety of tests.



picture: sample holder with pushrod

The data is acquired in a computer system with a windows based software (windows95 or windows NT4.0). Here the E-modulus is computed and displayed either on a linear or logarithmic scale depending on the range of the expansion of the sample. Especially all results for on and after the glasspoint are being evaluated with highest resolution.

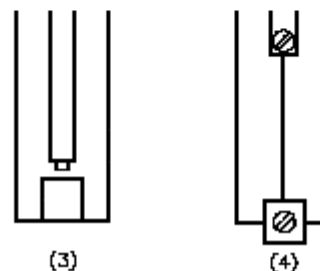
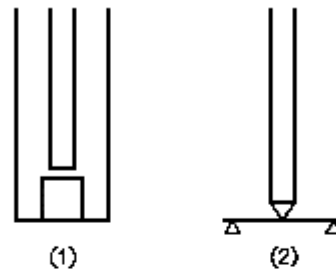
The DMA enables the measurement of deformations [change of depth or length] of a sample with a force applied, using different measuring fixtures. The temperature can either be fixed or variable.

Applications TMA:

- expansion and contraction behavior of fibers and foils
- determination of the softening behavior
- glass transition temperatures and secondary transitions
- determination of phase changes
- examination of mechanical behavior with materials under applied force
- determination of expansion coefficient of different materials
- sintering behavior

applications DMA:

- the DMA mode is used if the viscoelastic behavior has to be determined
- determination of the E-modulus (storage - and lossmodule) and loss factor tan
- elasticity
- slipping resistance and friction resistance



Different fixtures:

1. Expansion measurement
2. Elasticity measurement
3. Penetration measurement

4. Expansion of threads



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data acquisition, controlling, evaluation

All LINSEIS Thermal Analysis Instruments are controlled through a sophisticated Windows® software. The complete program consists of three sections; temperature control, data acquisition, and data evaluation. Essential sample information is entered in the data acquisition section.

Data acquisition section

Data Acquisition Setup - Sample Measurement

Operator:

Laboratory:

Atmosphere:

Flow Rate: [l/h]

Piston Material:

Comment:

Sample File:

Zero File:

Samp. Interval: [sec]

Duration: [min]

End Temp.: [°C]

Sample load

Force: [mN]

Frequency: [Hz]

Amplitude: [mN]

Off

Rectangular

Sine

Triangle

Sample

Name:

Length: [mm]

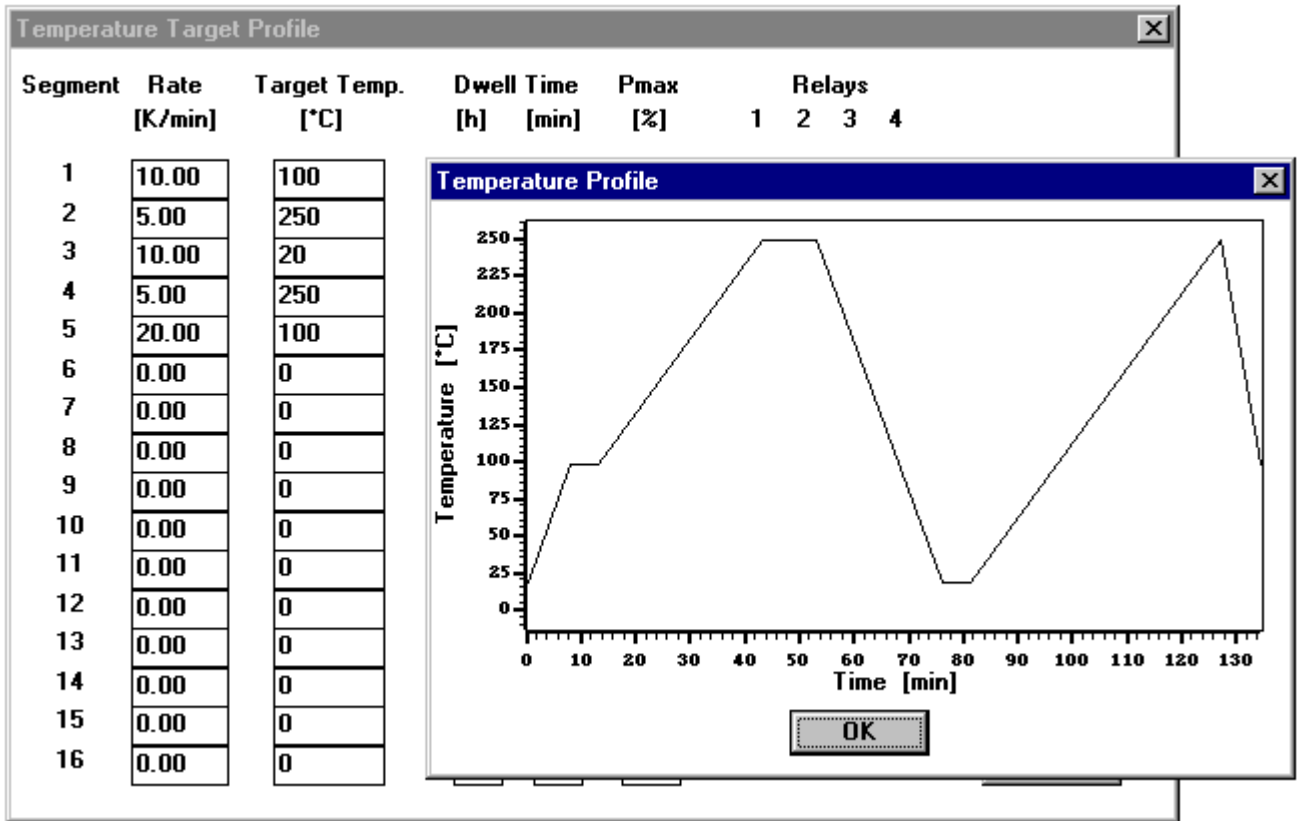
Range: [μm]

Picture 1: menu for the documentation set-up

Essential data for each sample test includes; operator, laboratory, atmosphere, gas flow, material, sample file name, zero file name, comments, sample length, measuring range, max. temperature, duration of run, sampling frequency, heating and cooling rates, number of cycles.

All menus are easily understood and intuitive. The software is quickly mastered with minimal training needed.

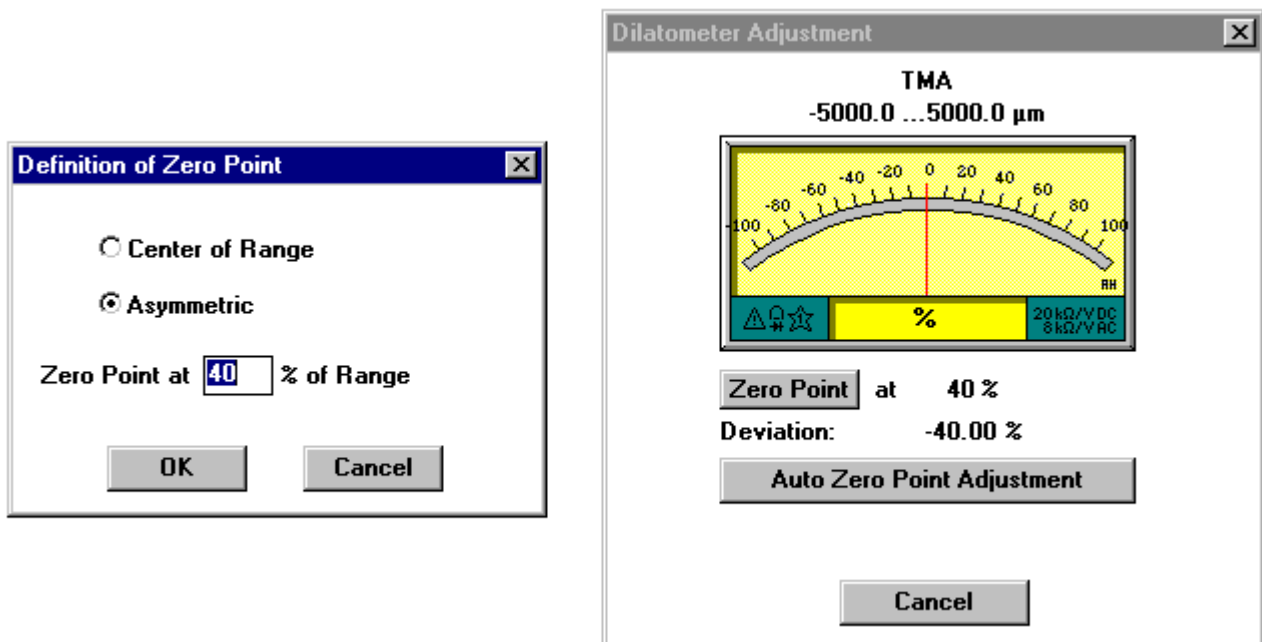
Evaluation section



Picture 2: results of a print-out

The evaluation is part of the complete windows software. It features a number of functions enabling a full evaluation of all types of data. All evaluation and data collection can be performed simultaneously. Data can be corrected using zero and calibration correction. Data evaluations include; absolute length change, relative length change, and coefficient of thermal expansion and the E-modulos. A mean curve with statistical analysis can be performed on multiple curves. Graphical displays can be printed on all windows compatible printers or plotters. Data can be displayed and printed in a table format. The software also includes an ASCII export feature.

Programming section



Picture 3: the evaluation menu

In this section the heat up speed, the target temperature and the dwell time are defined. Up to 16 segments can be programmed that can be run in up to 99 cycles. On top a max. of 4 relay outputs can be defined, that could be used for gascontrol etc.

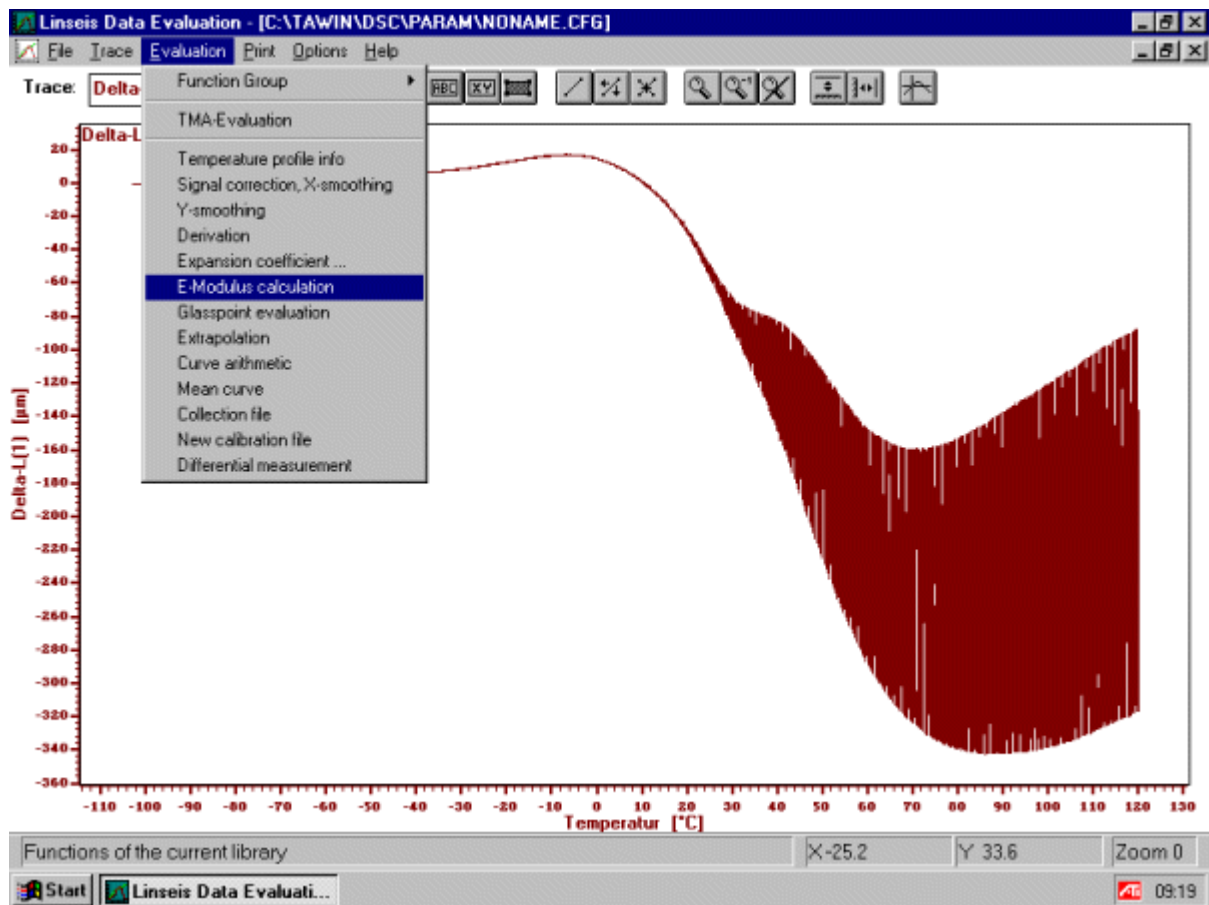


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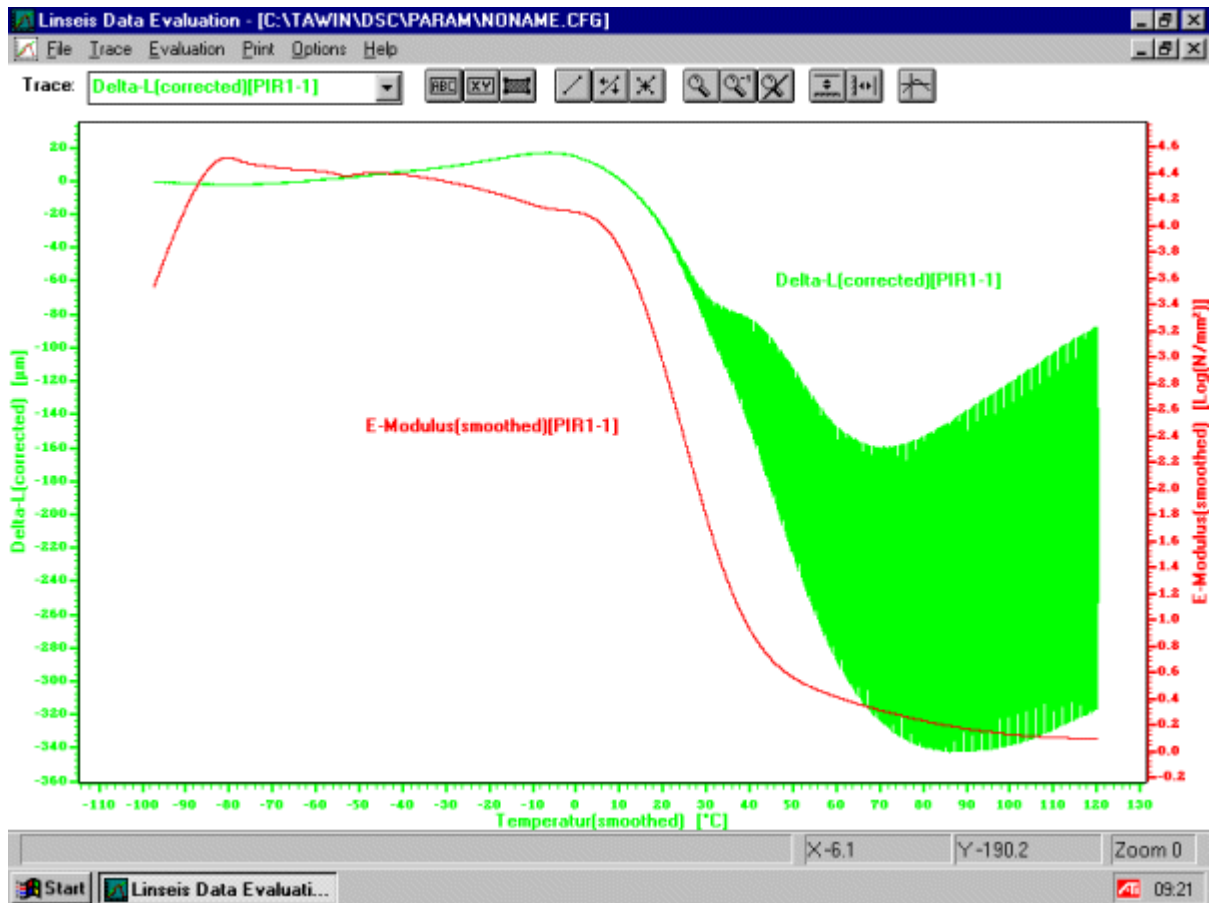
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Picture 4: evaluation menus

in picture 4 you can see the open pull down menu with the following points available: selection of function groups, TMA evaluation, adjusted heat up rates, signal correction, smoothing, evaluation of expansion coefficient, E-modulus, glass point, extrapolation, curve arithmetic, mean value of curve, collection file, new calibration file and differential measurement.

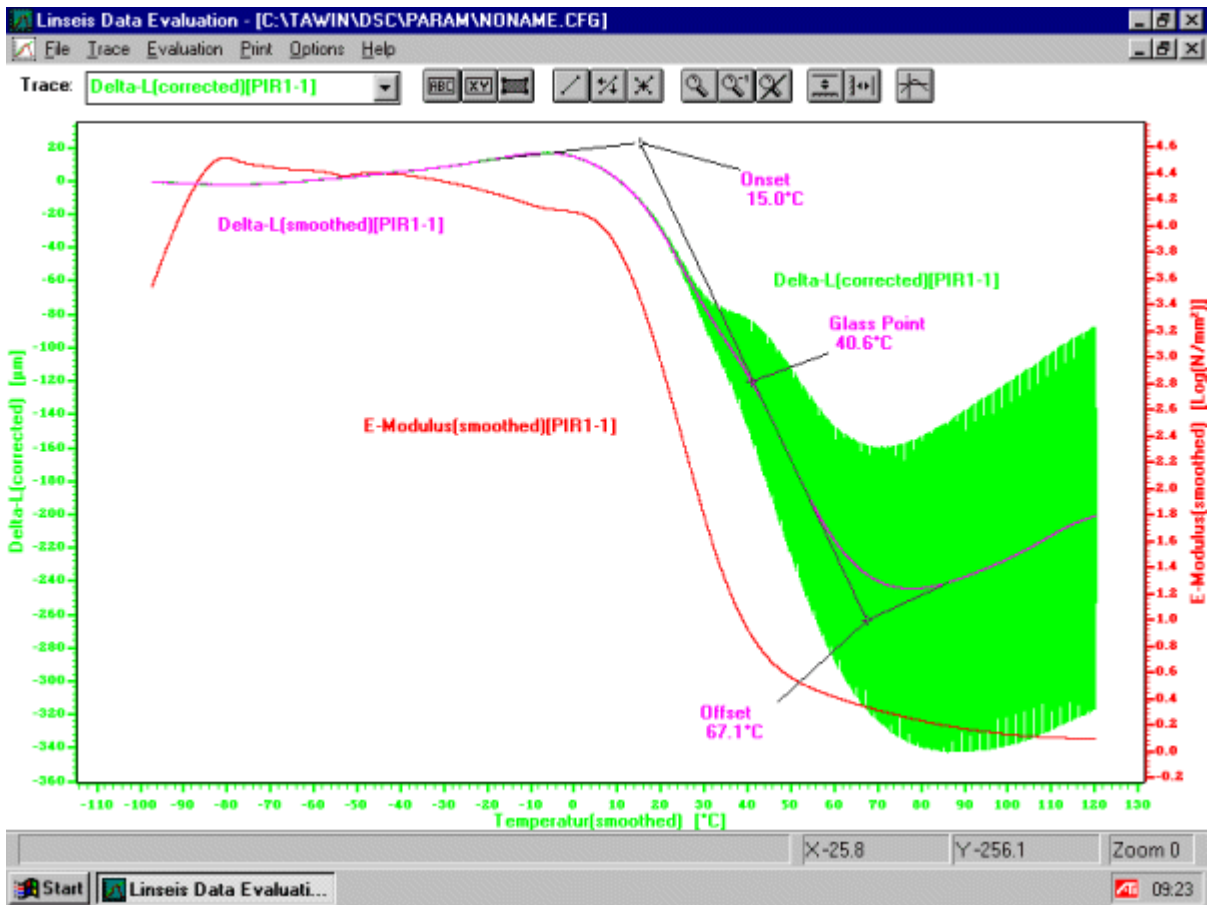


Picture 5: calculation of the E-modulus

In picture 5 the following steps are being made in order to calculate the E-modulus

1. zero correction
2. x-scale smoothing
3. calculation of E-modulus
4. smoothing of the E-modulus

You can see the measurement of an elastomer in the temperature range from -100°C to 120°C . In picture 5 the penetration characteristic of the push rod is shown (175μ diameter of the pin). The applied force is a sign wave (100mN, 0,05 Hz). The second curve shows the calculated E-modulus over the full temperature range.



Picture 6: evaluation of the glass point

in picture 6 you can see the same measurement as in picture 5. In addition to the E-modulus you can also see the evaluation of the glass point. Very good visible is the determination of the onset. You can see also the glass point which is defined in this application as the value where the step reaches 50 %. Another way of calculation for the glass point can also be selected where it is defined as the point of inflection. Also the determination of the offset is being shown.

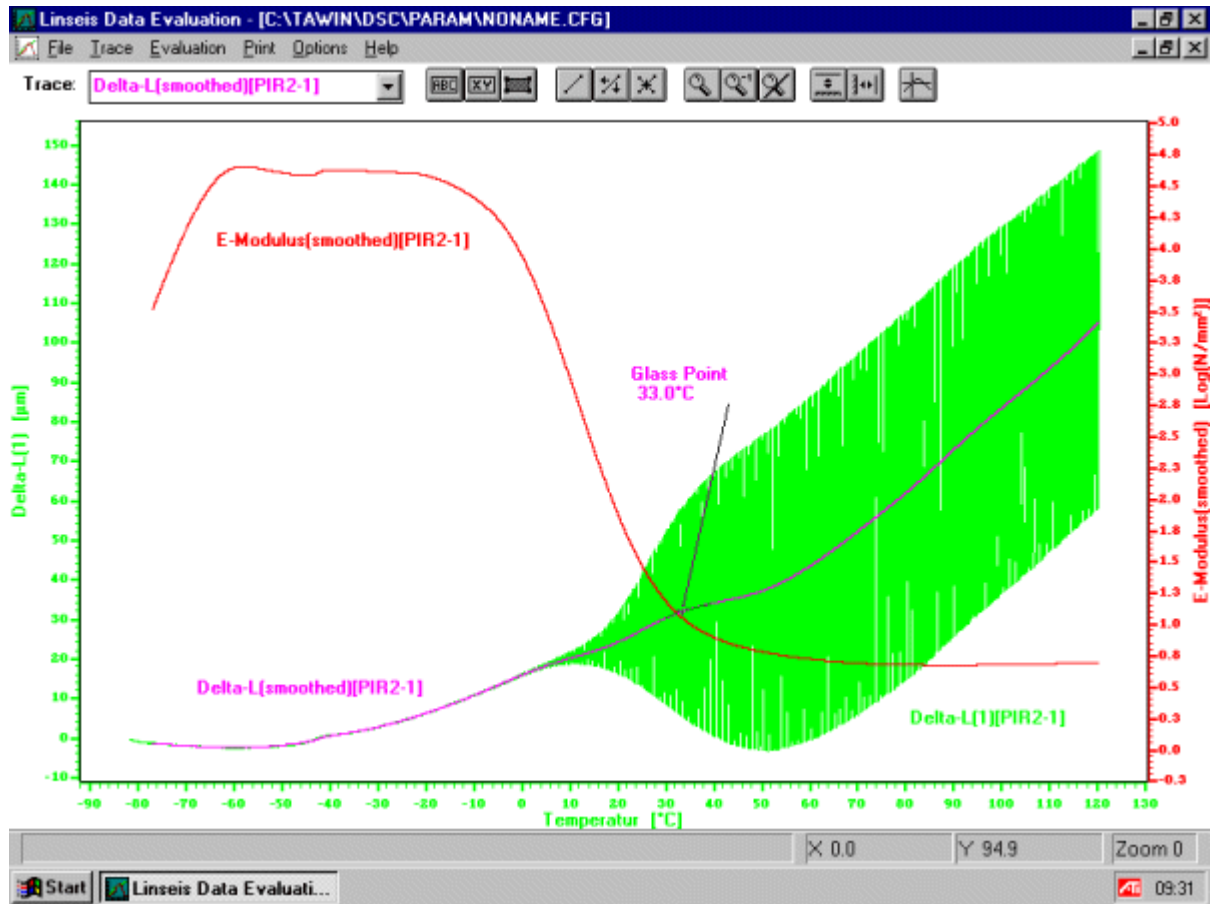


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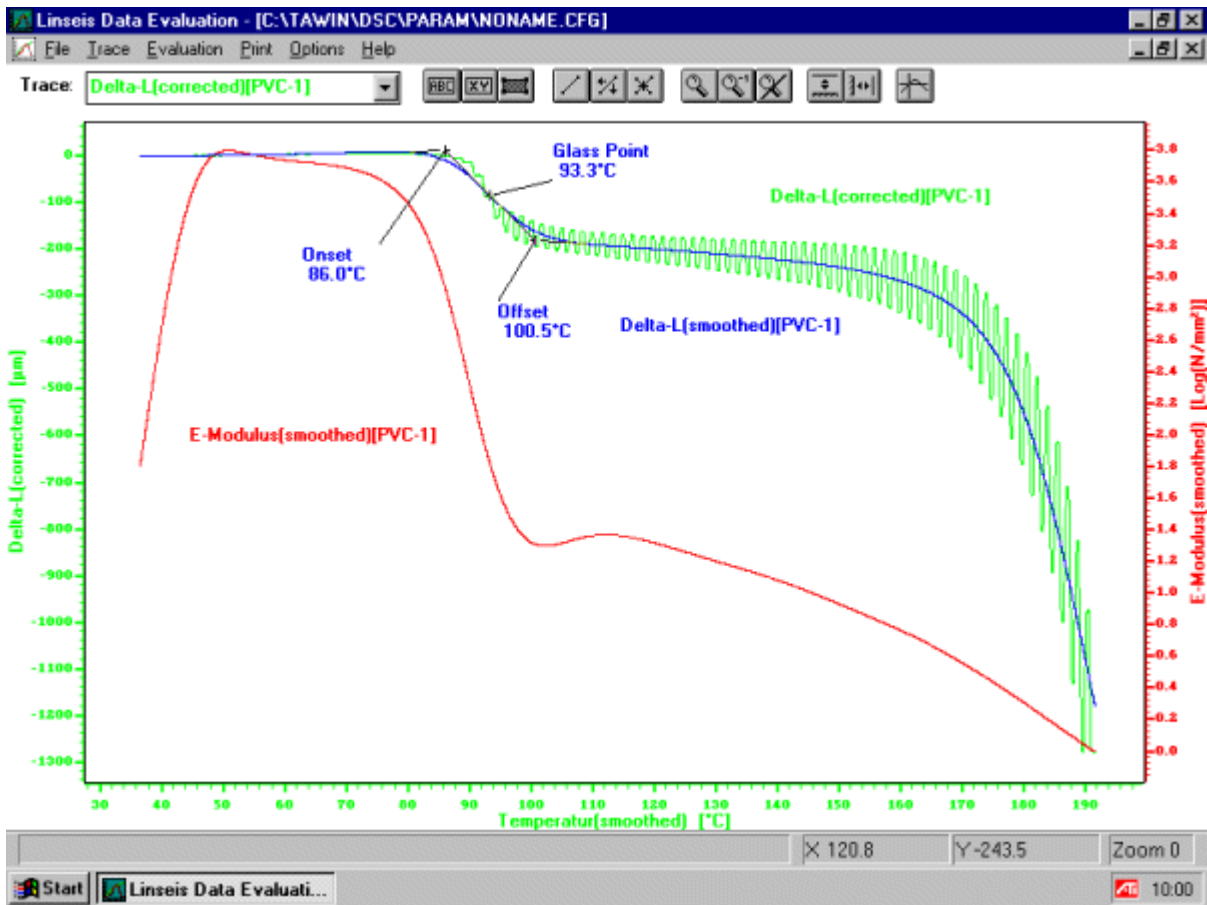
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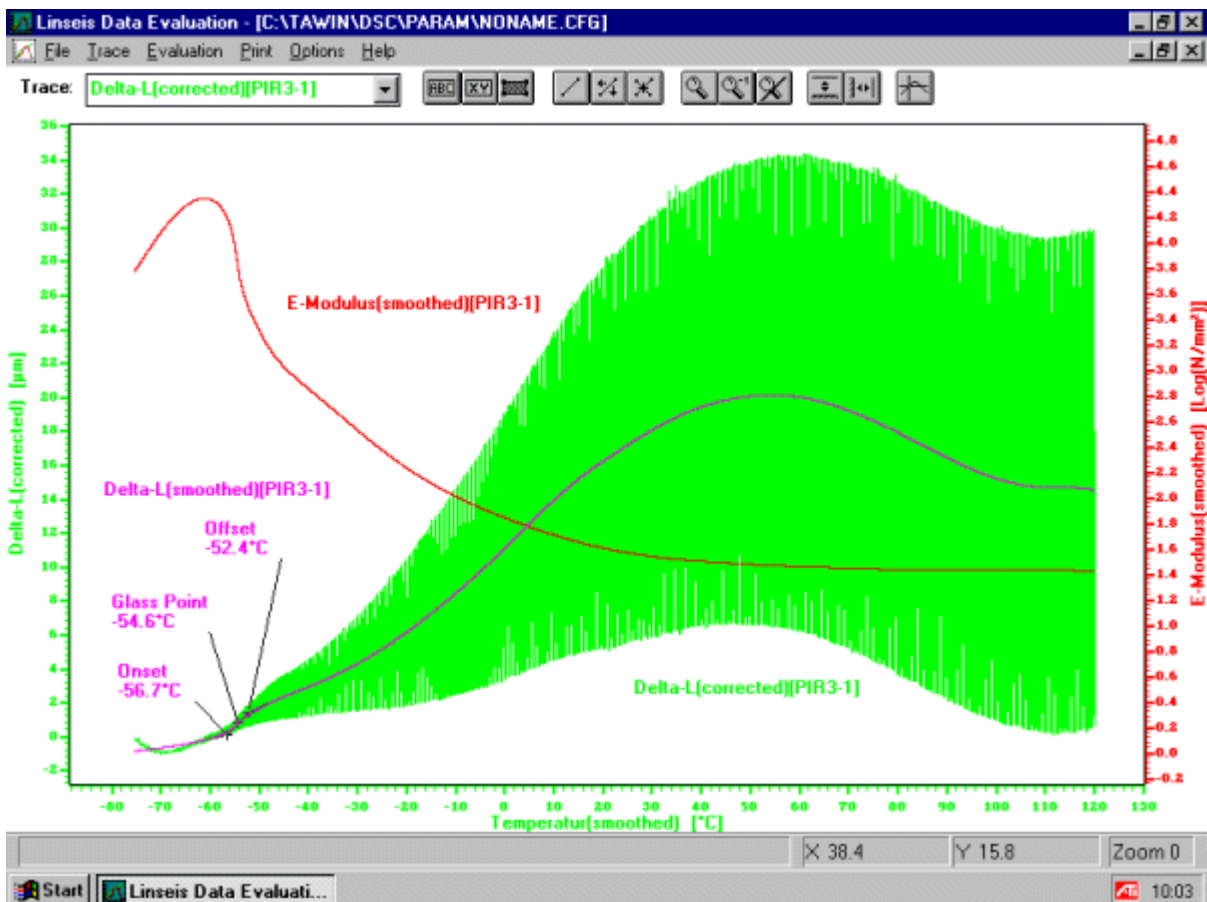
picture 7: elastomer

In picture 7 you can see an elastomer that was specifically developed for use at temperatures above 0°C. The glass point is at 29,9 °C. If the temperature is further increased an additional dilatation of the material in the elastic range is visible. The plastic range of the material is hereby not yet reached.



picture 8: PVC sample

In picture 8 you can see the test run of a PVC sample starting from room temperature. The glass point is at 93,3° C. The elastic range starts from approx. 150°C. At higher temperatures the change into the plastic range is very good visible



Picture: 9 silicon kautschuk

In picture 9 the evaluation of a silicon kautschuk is shown which was especially developed for use at low temperatures. The glass point is at $-54,9^{\circ}$ C. Furthermore the E-modules is shown over the complete temperature range and the mean value of the expansion. The change of length is up to about 50° C an expansion (elastic range), and thereafter a change into the plastic range is visible



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