

DILATOMETER

Special Software

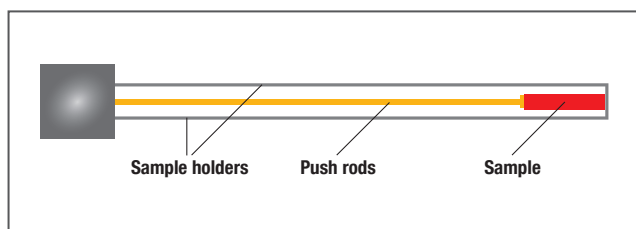


INSEIS

Software for Dilatometers

Softening Point Software for Dilatometers

All dilatometer delivered since 01. 11. 2002 has a new feature included in the WIN-DIL software. Softening point feature to determine the softening point of a sample is a standard feature now. It is now part of the standard functions of the WIN/DIL software program at no extra cost.



The feature not only determines the softening point of the sample, but it also protects the measuring system from a sample melting sample. With the softening point feature is activated, if a sample softens or shrinks more than the preset amount the controller will switch to a cooling cycle.

The softening of the sample is detected when the expansion of the sample reaches a maximum and starts shrinking again. The amount of shrinking after a maximum can be freely programmed. It is also possible to program different shrinkage factors for each heating stage.

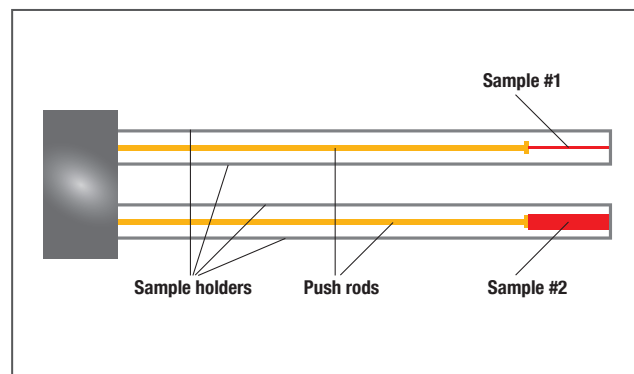
The normal temperature control program enables 16 different segments with different heating rates, cooling rates, and dwell times.

The same software feature can also control for expansion events. This is another way of controlling the dilatometer if an event occurs.

L75 / SDC Simultaneous Dilatometer Calorimeter

With the newly developed LINSEIS SDC Software it is possible to get results on a Dilatometer not only for expansion coefficients (CTE). Using the same hardware you can measure the caloric properties of the sample, heat capacities, enthalpies and phase transitions are available. The SDC Software utilizes the dual sample Dilatometer's capability. Two samples of the same material, same length, and different diameters are used.

Because both samples have different volume and mass, (different heat capacity) the temperature that can be measured at both samples during a heating or cooling phase are different. The larger sample is tempera-



ture wise "behind" the smaller sample. As the temperatures measured at both samples are thus different at specific times, the corresponding delta L / expansion signal is also different. The principle of the SDC Software is to maintain a constant length difference between sample one and sample two throughout the test. This is accomplished by varying the applied heating and/or cooling rate.

The heating and/or cooling profile is now proportional to the heat capacity of the sample material. The result are Endothermic and exothermic peaks displaying phase transitions of the sample based on the resulting heating and/or cooling rates. Absolute values can be obtained by running calibration two samples of known heat capacity and known mass.

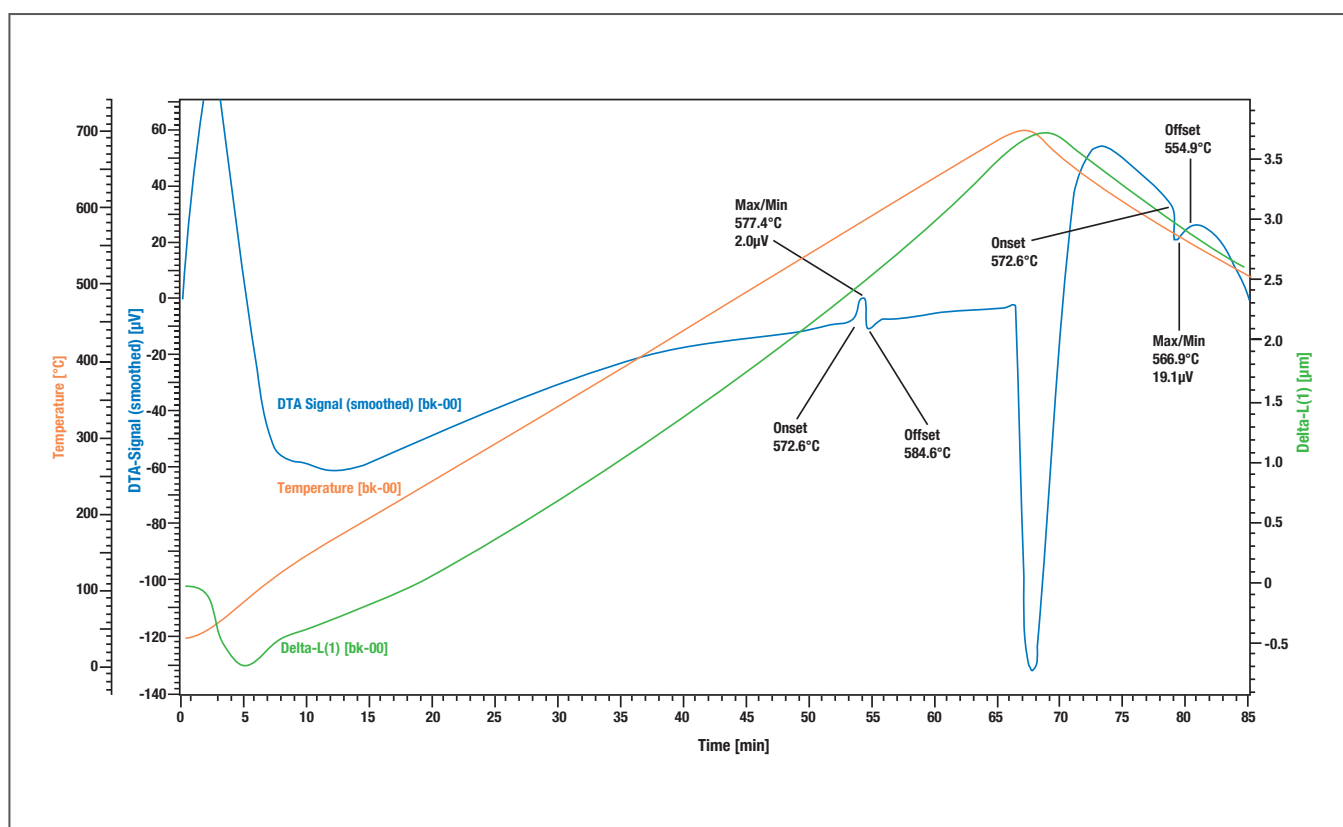
L75 / SDD Simultaneous Dilatometer DTA

Standard dilatometer measurement with additional DTA evaluation.

By means of this software any normal installed Linseis Dilatometer is now able to achieve additional DTA information / enthalpy values out of Dilatometer runs. Typical Dilatometer measurements usually determine the expansion delta L and expansion coefficient CTE of a sample. Very often the samples display an endothermal or exothermal reaction. These reactions cause a very small effect to the normally constant heating or cooling rate. For example if the sample requires energy during a phase transition, it will draw this energy from of the Dilatometer measuring system. In turn the furnace controller will compensate for this heat loss in order to obtain a constant heat up rate. These small deviations in the constant heating rate and the real temperature signal which is obtained from a heat loss or gain result in the DTA signal. The actual measured values are compared to a calculated heating or cooling curve. Using this procedure it is possible to get caloric enthalpy measurements from a Dilatometer. On next page we show an example of this software where the Alpha / Beta transition of quartz is shown. The sample had a mass

of 450mg and a volume of 3x4x5mm. The transition is endothermic during the heating phase and exothermic during the cooling phase. The measured value of the onset temperature is 573°C and it is identical to the literature value. To get these results, a normal Dilatometer measure-

ment file is loaded. Next the Dilatometer file is changed into a DTA curve format. DTA evaluation can now be displayed using normal procedure for evaluating onset and offset temperatures.



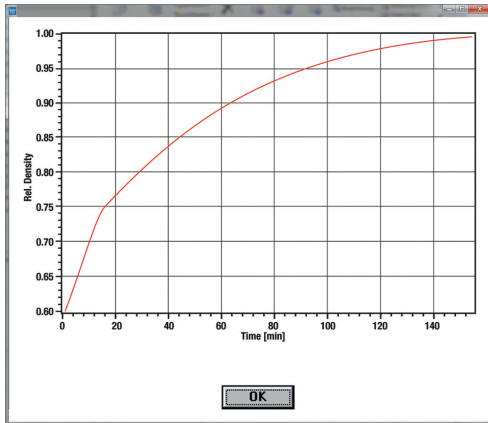
LINSEIS RCS – Rate Controlled Sintering

What is RCS

RCS is an add on for the standard dilatometer software. While during a dilatometer measurement the change in length of the sample during a given temperature profile is measured, RCS uses a quite different approach: For a given change in length profile (densification profile) the required temperature profile is determined.

The purpose of RCS is to determine the optimal sinter process, especially the optimal temperature/time profile. The aim of this optimization is to achieve the most possible final density in the shortest time at least energy consumption. The procedure used is based on the sinter theory of PALMOUR III (CERAM.MICROSTRUCT., PROC.INT. MATER.SYMP.6th 1976, WESTVIEW PRESS). According to this method, the sinter process is performed in a given densification profile. This profile is (with some restrictions) user definable, according to PALMOUR III:

- Two densification stages with a high and constant densification rate
- A third stage with a linear decreasing densification rate, until the desired final density is reached



Sample of a sinter profile

- 1. Stage:** 60..75% density, densification rate: $1E^2/\text{min}$, time: 15min
- 2. Stage:** 75..80% density, densification rate: $4E^{-3}/\text{min}$, time: 12.5min
- 3. Stage:** 80..100% density, final densification rate: $4e^{-4}/\text{min}$, time: app. 128min

How RCS works

By varying the sample temperature it is attempted to adjust the real densification of the sample to the given densification profile: If the real density is lower than the set density, the temperature is raised to achieve a higher densification rate. If the real density higher than the set density, the temperature is lowered to decrease the densification rate. The result is a temperature/time profile for an optimal sinter process.

The determination of the actual density is done by a length measurement of the sample, similar to a dilatometer measurement. An isometric sinter behavior is assumed (same densification in all three axis):

$$d_t = \frac{d_a \cdot l_0^3}{l_t^3}$$

d_t = rel. density at time t ,

d_a = rel. initial density,

l_0 = initial length [mm],

l_t = length at time t [mm]

The calculation of the set density as a function of time is done by the following equations:

- 1. and 2. Sinter stage (linear densification):

$$d_t = d_a + \Delta d \cdot t$$

d_t = rel. density at time t , d_a = rel. initial density of actual sinter stage,

Δd = densification rate [1/min], t = time [min]

- 3. Sinter stage (exponential decreasing densification rate):

$$d_t = d_a + \tau \cdot \Delta d_e \cdot (1 - e^{-\frac{t}{\tau}})$$

d_t = rel. density at time t

d_a = rel. initial density 3. sinter stage

Δd_e = final densification rate [1/min]

t = time [min]

τ = time constant: $\frac{d_e - d_a}{\Delta d_e - \Delta d_a}$

d_e = rel. final density

d_a = rel. initial density 3. sinter stage,

Δd_a = densification rate 2. sinter stage [1/min]

Δd_e = final densification rate [1/min]

The transition from the first to the second sinter stage can be smoothed if desired – a moving average is calculated over app. 5% of the time before and after the transition, to achieve a steady course of the sinter profile. For determining the sample length, a simplified correction method (related to the standard dilatometer method) is used, since the correction must be performed online during the measurement: No zero correction is performed, the correction of the expansion of the sample holder and the sample itself is done by a single, constant factor:

$$l_{\text{corr}} = (l_0 + \Delta l) \cdot K \cdot (T - 20)$$

l_{corr} = corrected length [mm]

l_0 = initial length [mm]

Δl = measured change in length [mm]

K = expansion coefficient entered [1/K]

T = actual temperature [°C]

The resulting absolute error (due to the simplified correction method) is usually small, related to the change in length of the sample during sintering. The complete sinter procedure consists out of three phases:

- Preheating of the sample, up to the temperature the sinter process starts
- The sinter phase itself
- A dwell time, holding the final temperature reached during sintering
- A cooling phase

The acquisition and storage of the measured values may be selected for the different phases of the sinter procedure. The time, the temperature, the measured change in length and the theoretical length for the given sinter profile are stored.

Determination of the required parameters for sintering

For sintering there are, except the desired sinter profile, some additional parameters required also:

- The sample length in [mm]
- The relative initial density (green density) of the sample

- The temperature value, sintering starts
- The expansion coefficient required for correction of the sample length

The sample length is usually measured with a slide gauge or a micrometer screw, like done for a dilatometer measurement also. The initial density is calculated from the ratio of the absolute initial density to the absolute, theoretical final density of the sample:

$$d_{\text{arel}} = \frac{d_{\text{aabs}}}{d_{\text{eabs}}}$$

d_{arel} = relative initial density

d_{aabs} = absolute initial density [g/cm³]

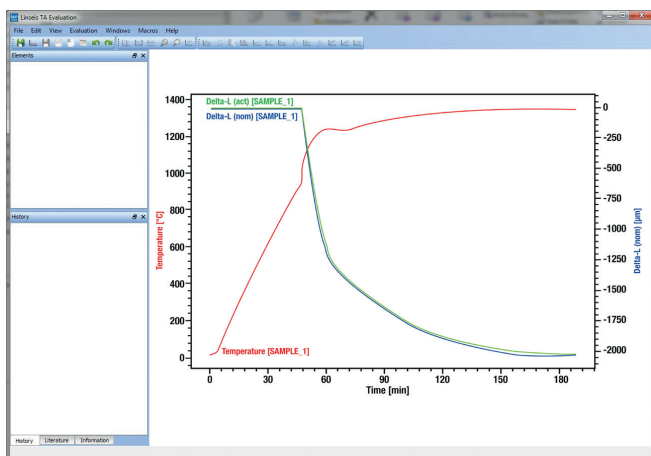
d_{eabs} = absolute final density [g/cm³]

For determination of the start temperature and the expansion coefficient two standard

dilatometer measurements are required:

- Measurement and evaluation of a none-sintered (green).sample, determination of the temperature where expansion of the sample changes to shrinkage
- Measurement and evaluation of a sintered sample. Determination of the expansion coefficient (Ak_{tech}) at the maximum required sintering temperature. If the evaluation of the measurement is performed without piston correction, the resulting Ak_{tech} is the coefficient to be entered directly for correction purpose (difference between expansion sample – expansion sample holder).

All determined parameters are entered together with the desired sinter profile in the sinter parameter dialog.



Sample sinter measurement:

The trace Delta-L<act> shows the measured and corrected length of the sample, Delta-L<nom> is the theoretical length profile for the desired sinter behavior. The trace Temperature is the resulting temperature profile.



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