LINSEIS RCS – Rate Controlled Sintering

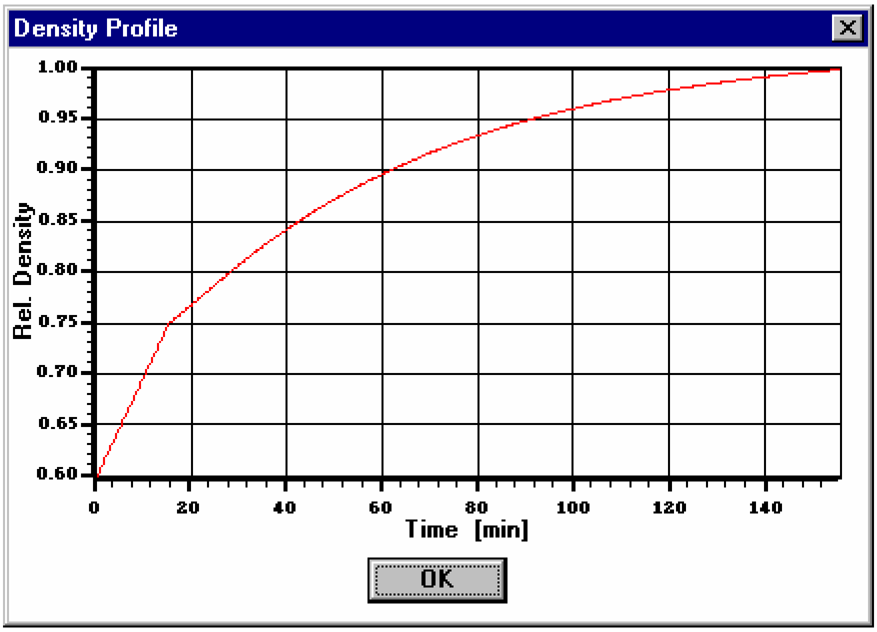
1. What is RCS

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/min, time: 15min
2. Stage: 75..80% density, densification rate: 4E-3/min, time: 12.5min
3. Stage: 80..100% density, final densification rate: 4e-4/min, time: app. 128min

2. 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):

$$d_t = \frac{da * l_0^3}{l_t^3}$$

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):

$$dt = da + \Delta d * t$$

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):

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

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], ι = time constant:

$$\tau = \frac{d_e - d_a}{\Delta d_a - \Delta d_e}$$

de = rel. final density, da = rel. initial density 3. sinter stage,
Δd_a = densification rate 2. sinter stage [1/min],
Δd_e = final densification rate [1/min],

The transition form 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:

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

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]

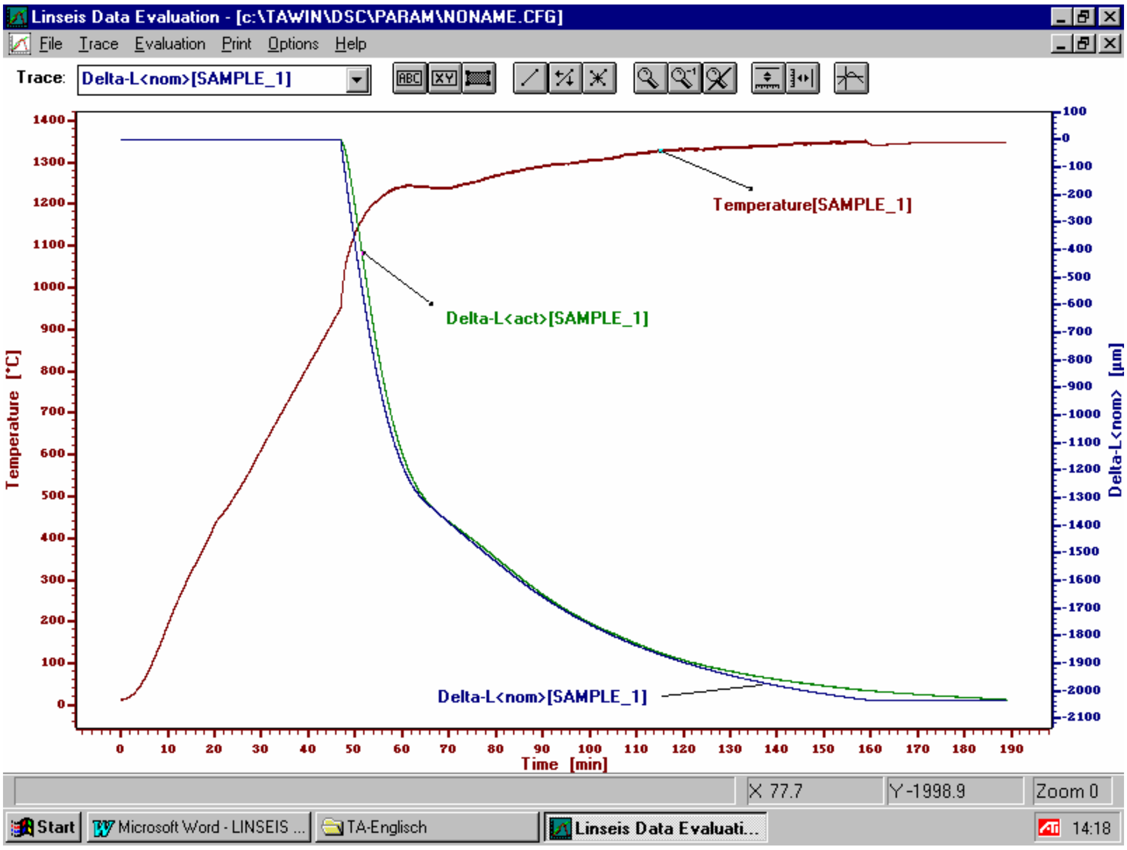
The resulting absolute error (due to the simplified correction method) is usually small, related to the change in length of the sample during sintering.

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.

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.

3. 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:

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

d_{arel} = relative initial density, d_{aabs} = absolute initial density [g/cm^3],
d_{eabs} = absolute final density [g/cm^3]

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.