Force controlled dilatometer

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Abstract
This article presents the advantages of a force-controlled measuring cell for dilatometers in terms of minimizing failure influences on the length signal. A function-integrated detection of the initial length with noticeable improved accuracy will be described.

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1. Introduction

In modern material research, e.g. zero-expansion materials, foams, polymers, ceramics, there is a need for higher precision in dilatometry and thus temperature driven dimensional changes. Following the state of the art, measurements of the thermal expansion $\alpha(T)$ and CTE $\alpha(\Delta T)$ can be determined and calculated as follows [1], [2] under a small load as defined in national standards:

$$\alpha(T) = \frac{1}{L_0} \frac{dL}{dT} \quad (1)$$

$$\alpha(\Delta T) = \frac{1}{L_0} \frac{dL}{d\Delta T} \quad (2)$$

$L$: measured length change of the sample
$L_0$: initial length of the sample
$T$: measured sample temperature

Figure 1 describes the principle of a dilatometer where a sample is placed in a sample holder inside of a moveable furnace which applies a controlled temperature [3].

Common dilatometers are designed with a guided pushrod which is in contact with the sample and transfers the length change of the sample to the measuring cell, where dimensional changes are detected by an LVDT. The contact force is applied by the preload of a spring. This design comprises several harmful disadvantages leading to deviations in the dimensional changes:

- Contact force is not measured and can only be adjusted manually within a small range ($0.15\,N - 0.45\,N$).
- Inadvertent changes of the contact force during the measurement are caused by the stiffness of the spring.
- Contact force changes caused by sliding and rolling friction as well as stick-slip-effects in the guidance of the pushrod.
- Determination of the sample length is executed manually by the use of a caliper or micrometer gauge with unknown contact forces.
- The used analog measuring principle of an LVDT (linear variable differential transducer) with high resolution but short measuring range strictly limits the application range.

2. Design of a force controlled measuring cell

A new design of a force controlled measuring cell for dilatometers with strongly reduced influence of the contact force on the length signal is shown in Figure 2.

The shown design has been developed in a systematically driven design process. Out of several functional schemes and technical principles the best has been chosen [5]. It includes an optical incremental linear encoder system placed in line to the sample to determine the initial sample length and the length change. This allows a widely extended measuring range and resolution. The contact force transmitted by the pushrod is measured by a moveable guided force sensor thus allowing a closed-loop-control. This predefined and adjustable contact force is applied by a piezo-drive, which also allows the adjustment to variable sample lengths.

3. Experimental results

3.1. Initial sample length detection
The influence of the contact force can be shown with the determination of the initial sample length $L_0$ of an insulating material sample (Styrodur®, BASF). Figure 4 shows the difference of manual measurements in comparison to the automatic mode with the force controlled measuring cell. The initial sample length can vary up to 5 % in dependency on the contact forces in a range of few mN up to 3 N. Measurements done manually with a calliper are marked in the shaded area with average and standard deviation. Contact forces of the manual measurement are in the range of approximately 0.85 mN to 1.05 mN.

![Figure 3. Determination of the initial sample length $L_0$ with different contact forces (Styrodur®, BASF)](image)

To demonstrate the performance of the new measuring cell to detect the initial sample length with a predefined contact force, the accuracy was analysed with an experimental setup (see Figure 5). A two-beam-interferometer (Figure 5, 2) was used to detect a fixed reflector at the sample holder. The second reflector is mounted on the movable bar, which is contacted on the backside by the pushrod of the force controlled measuring cell (Figure 5, 1). The position of the bar can be changed externally to simulate different sample lengths (Figure 5, 3). The comparison of the measuring cell signal with the length delta of beam I and II of the interferometer ensures that measuring errors caused by the basic body are eliminated.

![Figure 4. Experimental setup to analyse the accuracy of the initial sample length detection](image)

The experiment was executed with four different setups and two varying measuring cells. Measurement points with a distance of 5 mm-steps are detected up to 25 mm with a contact force of 0.5 N. Each setup was multiplied measured (ten times) and the corresponding average value including error bar is displayed in Figure 6.

The measurements show a systematic error smaller than 0.8 µm for an initial sample length up to 25 mm. A standard deviation for all measurement points in the range of ± 8 nm to ± 20 nm indicates a high repeatability. This experiment presents that the initial sample length can be detected with a much higher accuracy of about 0.003 % compared to common measurements by the use of callipers.

![Figure 5. Accuracy of the initial sample length determination of different setups](image)

### 3.2. The influence of the contact force

Measurements with packaging foam show the influence of the contact force on the CTE (coefficient of thermal expansion), see Figure 7. Therefore, different samples with an initial length of 25 mm were heated up from ambient to a temperature of 65 °C with different constant contact forces (0.01 N, 0.05 N, 0.15 N). Figure 7 shows that the expansion and shrinkage behaviour of foam strongly depends on the contact force. Not only the magnitude but also the start temperature of the sample shrinkage deviates extremely.

![Figure 6. Influence of different contact forces on the thermal expansion of foam](image)

### 4. Conclusion

A force controlled measuring cell for dilatometers has been developed for the automatic measurement of the initial sample length and its length changes caused by a controlled temperature change. As the new measuring cell is able to keep the contact force at a predefined constant level, failure impacts on the length signal can be strongly reduced. Furthermore, the new system allows the measurement of a wider range of sample materials with strongly improved precision.

### References