

TIE-37: Thermal expansion of ZERODUR®

0. Introduction

This technical information gives an overview on the thermal expansion of ZERODUR®. This article begins with a description of the dilatometric measurement of the coefficient of linear thermal expansion (denoted as CTE in the following text). Then the specifications of the mean CTE and the CTE homogeneity are explained and an evaluation of the results for about 200 ZERODUR® mirror blanks is presented to give a statistical description of the achievable quality. At the end of this article is a short summary about the influence of thermal cycling and cryogenic temperatures on the CTE.

1. Production of ZERODUR®

ZERODUR® is an inorganic, non-porous glass ceramic, characterized by a phase of evenly distributed nano-crystals within a residual glass phase. ZERODUR® contains about 70-78 weight percent crystalline phase with a high quartz structure. This crystalline phase has a negative linear thermal expansion, while that of the glass phase is positive. The crystals have an average size of about 50 nm.

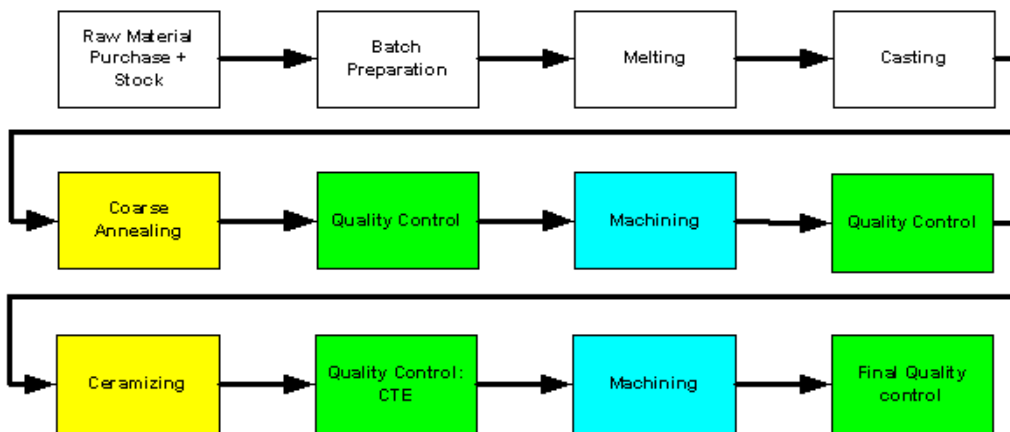


Figure 1: ZERODUR® production process schematic

The ZERODUR® manufacturing sequence is based on established and proven methods used in the production of high homogeneity optical glasses. The production starts with filling a large tank with raw material and heating it up to obtain a homogeneous melt (refer to figures 1-3).

The ZERODUR® melt is cast into molds and inserted into annealing ovens to cool the raw casting down to room temperature in a controlled way for several weeks. The glassy raw casting still has a coefficient of thermal expansion (CTE) of $\sim +3 \cdot 10^{-6} \text{ K}^{-1}$. Pre-crystallized outer material layers are removed in a subsequent processing step to prevent any uncontrolled ceramization during the following ceramization process.

During the ceramization process the material is heated up again to achieve controlled nucleation and growth of the crystal phase. The ceramization has a big influence on the final CTE homogeneity of the blank and can last up to several months depending on the size of the ZERODUR® part [1]. After ceramization the material can be processed to its final shape. During this processing step CTE samples are taken directly from the final ZERODUR® material to estimate the final CTE value and homogeneity.

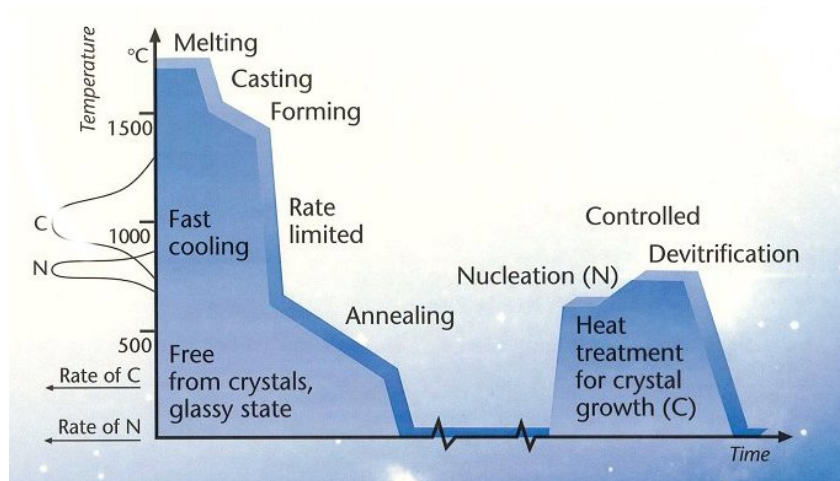


Figure 2: ZERODUR® annealing process schematic



Figure 3: ZERODUR® near net shape raw casting

2. CTE measurement

ZERODUR® is optimized for an application temperature range of 0°C to 50°C. The CTE is measured using a modified dilatometer setup. SCHOTT offers standard and improved measurement accuracy for the CTE (0°C, 50°C) measurement. The achievable accuracy is summarized in table 1:

	Accuracy [$\cdot 10^{-6} K^{-1}$]	Repeatability (95%) [$\cdot 10^{-6} K^{-1}$]
CTE (0°C, 50°C) Standard dilatometer	± 0.0100	± 0.0050
CTE (0°C, 50°C) Improved dilatometer	± 0.0062	± 0.0012

Table 1: Comparison of the standard and improved dilatometer setup

2.1 Standard dilatometer setup for ZERODUR® CTE measurement

The basic construction of the dilatometer used for the CTE measurement can be seen in figure 4. This setup is similar to the instrument presented by Plummer and Hagy [2]. The difference lies in the material used in the sample holder and the push rod in our setup – these are made of titanium silicate glass. The system is optimized for a sample length of 100 mm and 6 mm diameter. The temperature is measured with a platinum resistance thermometer (PT 100) mounted near the sample. The temperature of the dilatometer head is stabilized at better than 0.2 K. The whole equipment is set up in a laboratory where the temperature variation is less than 1 K.

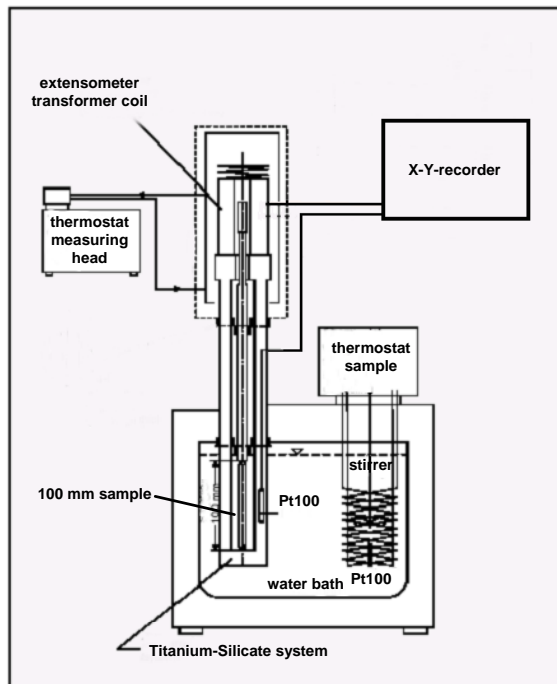


Figure 4: Basic construction of the push rod dilatometer [3]

During measurement, the sample and the sample holder are immersed into a water bath. A thermostat is programmed to heat and cool the water bath according to a defined procedure. When changing the temperature, the sample changes its length and the rod moves the coil of a Linear Variable Differential Transformer (LVDT). The signals of the LVDT and the temperature of the water bath are recorded and used for calculating the CTE. This system is optimized for the measurement of the CTE (0°C; 50°C)

The CTE or $\alpha(T_0;T_1)$ is calculated by

$$\alpha(T_0;T_1) = \frac{U(T_1) - U(T_0)}{E \cdot l_0 \cdot (T_1 - T_0)} + \alpha_{\text{syst}}(T_0;T_1)$$

where $U(T_i)$ is the voltage (mV) of the LVDT for the temperatures of T_0 and T_1 , l_0 is the sample length, E is the sensitivity of the LVDT (mV/ μm) and $\alpha_{\text{syst}}(T_0;T_1)$ is an expansion-correction of the system (sample holder).

The system is regularly calibrated using a certified titanium silicate reference sample that was measured at the Physikalisch-Technische Bundesanstalt (PTB) with a high-precision interferometer system. The reference sample was measured to an absolute accuracy of $\pm 0.005 \cdot 10^{-6} \text{K}^{-1}$. The overall accuracy of the dilatometer for the absolute CTE (0°C, 50°C) measurement is $\pm 0.010 \cdot 10^{-6} \text{K}^{-1}$ and the reproducibility is $\pm 0.005 \cdot 10^{-6} \text{K}^{-1}$ (95% confidence level).

2.2 Improved dilatometer setup for ZERODUR® CTE measurement

The improved dilatometer setup is based on the standard setup described in chapter 2.1. Pushing the limits for accuracy of the CTE measurement forward, the standard electro-mechanical LVDT was replaced by an interferometric measurement system. The interferometer measurement head can measure relative length changes to a high precision at an increased resolution. The resolution is a factor of 50 better as compared to the standard LVDT setup. This improved system was developed to cope with the demands of high precision CTE measurements of ZERODUR® used as mirror substrate in extreme UV microlithography applications [4].

The short term reproducibility of the new dilatometer setup is $\pm 0.0012 \cdot 10^{-6} \text{K}^{-1}$ based on a 95% confidence level. The overall accuracy of the improved dilatometer for the CTE (0°C, 50°C) measurement is $\pm 0.0062 \cdot 10^{-6} \text{K}^{-1}$. The repeatability has been improved by a factor of 4 as compared to the standard setup [14].

2.3 Measurement of the CTE (0°C, 50°C)

The temperature sequence for the measurement is as follows: First the system is heated up from room temperature to 50°C. The temperature is kept constant at 50°C for 20 minutes. Then the system is cooled down to 0°C at a rate of 0.6°C/min and kept constant for 20 minutes. At the end of the 20 minutes holding time, the length of the sample is virtually set to zero. Then the sample is heated up again to 50°C and the relative length change of the sample is measured after a holding time of 20 minutes. The CTE (0°C, 50°C) value can be calculated from the change of length afterwards.

2.4 Measurement of the total change of length curve (TCL)

To determine the total change in length, the relative change of length Δ/l_0 is generally measured within a temperature range between -50°C and $+100^{\circ}\text{C}$. The total change of length of ZERODUR[®] is the maximum change of length within this temperature range. In contrast to the static CTE (0°C , 50°C) measurement the TCL curve measurement is a dynamic measurement, therefore the measurement setup and sequence is different.

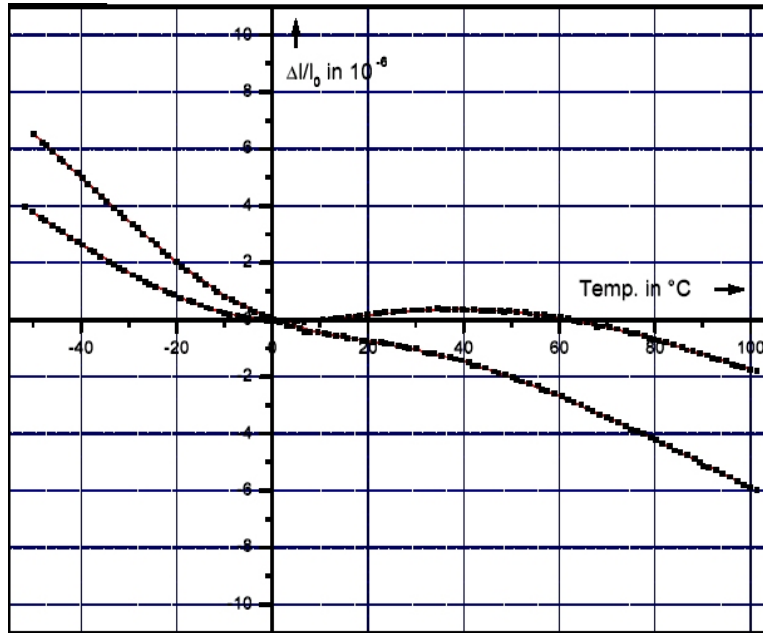


Figure 5: Two typical TCL curves of ZERODUR[®]

For measurements in the temperature range $[-50^{\circ}\text{C}$, $+100^{\circ}\text{C}]$ the standard dilatometer is equipped with a gas cooling system instead of a water cooling system. The sample is cooled down to -50°C first. Then the sample is heated up with a constant annealing rate of 1.3 K/min . The change of length is recorded continuously, approximately every hour, during the heating process. Two typical TCL curves with different CTE (0°C , 50°C) values are given in figure 5. The curves are typically slightly s-shaped and tilted around 0-expansion for different CTE (0°C , 50°C) values. The TCL value for the first curve is $\sim 13 \cdot 10^{-6}$ and the CTE (0°C , 50°C) derived from the curve is $-0.040 \cdot 10^{-6}\text{K}^{-1}$. The TCL value of the second curve is $\sim 6 \cdot 10^{-6}$ and the CTE (0°C , 50°C) value is $0.010 \cdot 10^{-6}\text{K}^{-1}$.

It is important to note that the CTE depends on the temperature/time history and therefore also on the measurement sequence (the so-called CTE relaxation effect described in chapter 6). CTE (0°C ; 50°C) values derived from TCL-curves are therefore less accurate than those obtained with the 0°C – 50°C method. Due to the different temperature versus time program, CTE values may differ slightly even for the same sample.

The TCL curve of ZERODUR[®] can be adjusted to shift the CTE value to exactly zero at a given temperature under defined conditions [4]. Figure 6 shows an example of the two tailored CTE curves with “zero crossings” at two different temperatures. Such tailored “zero-crossings” may be important for different applications of ZERODUR[®]

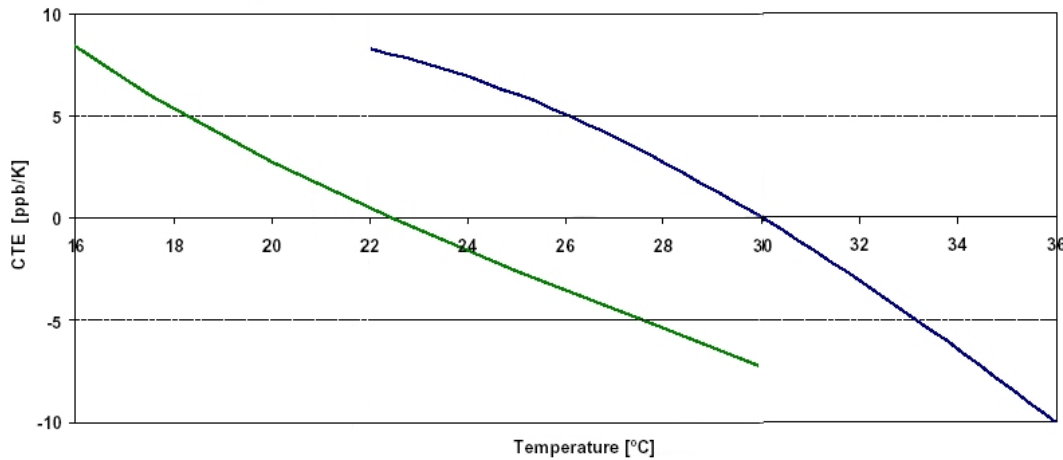


Figure 6: Two examples of tailored CTE “zero-crossings” of ZERODUR®

3. Sampling for CTE measurement of ZERODUR®

The following chapters present the results from CTE measurements of more than 200 astronomical mirror blanks with diameters between 1-2 m produced at SCHOTT [5]. Table 2 shows an overview of the mirror blank projects included in this presentation. A general overview on 100 mirror telescopes from SCHOTT can be found in [6].

Project/Site	Dimensions of Primary Mirror Blank	CTE [$10^{-6}K^{-1}$] specification	Δ CTE [$10^{-6}K^{-1}$] specification	samples per blank	Completed
Keck I / Hawaii	43 segments dia. 1900 mm thickness 76,5 mm	+/- 0.10	≤ 0.02	18 (9 from top and 9 from bottom, on two circles)	1990
Keck II / Hawaii	42 segments dia. 1900 mm thickness 75,8 mm				1993
HET / Texas	96 hexagons, width 1019 mm, thickness 56 mm	+/- 0.15	≤ 0.01	4 (2 from top and 2 from bottom per raw casting)	1995
GTC / La Palma	42 hexagons, width 1622 mm, thickness 83,5 mm	+/- 0.05	≤ 0.02	12 (6 from top, 6 from bottom)	2002

Table 2: Overview on the astronomical telescope mirror project results.

In general if there are no special requirements on the homogeneity of ZERODUR®, a single sample will be taken from each casting to estimate the CTE value. This sample will either be cut from the remaining material after ceramization or cut from the glassy ZERODUR® material before ceramization and placed in the same ceramization furnace.

Mirror blanks with dimensions up to 2.4 m are fabricated out of thick cylindrical or near net shaped ZERODUR® castings with a height sufficient to fabricate at least 3 or 4 mirror blank segments from it (figure 7). After ceramization the castings are cut into single blanks. Mirror blanks often have specific requirements on the homogeneity of CTE, therefore several samples representative for the total blank volume are needed. The samples have to be taken from the remaining material of the casting according to given sampling plans (figure 7). In general samples are evenly spread around the circumferential area of the mirror blanks. Samples between two blanks can be assigned either to the bottom of the upper blank or to the top of the lower blank. Table 2 gives detailed information on the numbers of samples per blank.

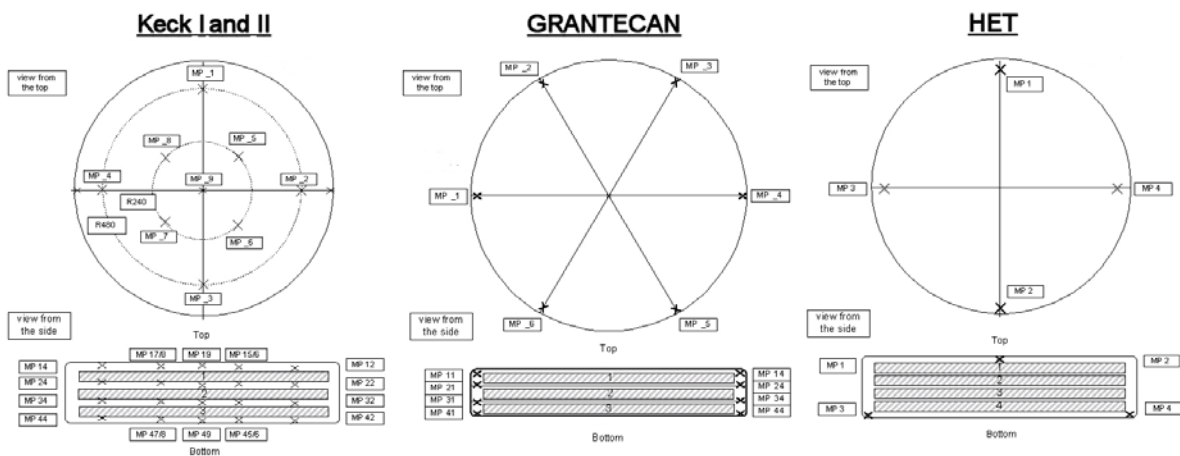


Figure 7: Sampling plans for the Keck, Grantecan and HET telescopes

4. Mean value of CTE

Individual pieces of ZERODUR® can be supplied with a mean CTE in the temperature range of 0° to 50°C in three expansion classes [13]:

Expansion class 0	$0 \pm 0.02 \cdot 10^{-6} \text{K}^{-1}$
Expansion class 1	$0 \pm 0.05 \cdot 10^{-6} \text{K}^{-1}$
Expansion class 2	$0 \pm 0.10 \cdot 10^{-6} \text{K}^{-1}$

Table 3: Expansion classes according ZERODUR® catalog [13]

If not otherwise expressly specified, expansion class 2 material will be supplied.

In the following CTE always denotes CTE (0°C; 50°C) values. Figure 8 shows the frequency distribution for the mean CTE values of the Keck, GTC and HET mirror blanks. The mean value data of all projects ranges from -0.109 up to $0.06 \cdot 10^{-6} \text{K}^{-1}$.

The range of the mean value distributions reflects the CTE specification. If the CTE specification is relaxed, the resulting mean values will have a boarder variation because no special material selection or other measures are necessary to fulfill the specification. In many cases the batches used may have different production histories. This means that part of the mirror blanks may be from a new melt campaign while the other part of the mirror blanks are produced from batches on stock with different mean value. By using defined casting formats and special ceramization setups, the reproducibility of the ZERODUR® manufacturing can be further improved to keep a tighter mean CTE distribution on request. For example most of the GTC mirror blanks have been exclusively produced out of special hexagonal shaped molds in a continuous sequence of melting campaigns.

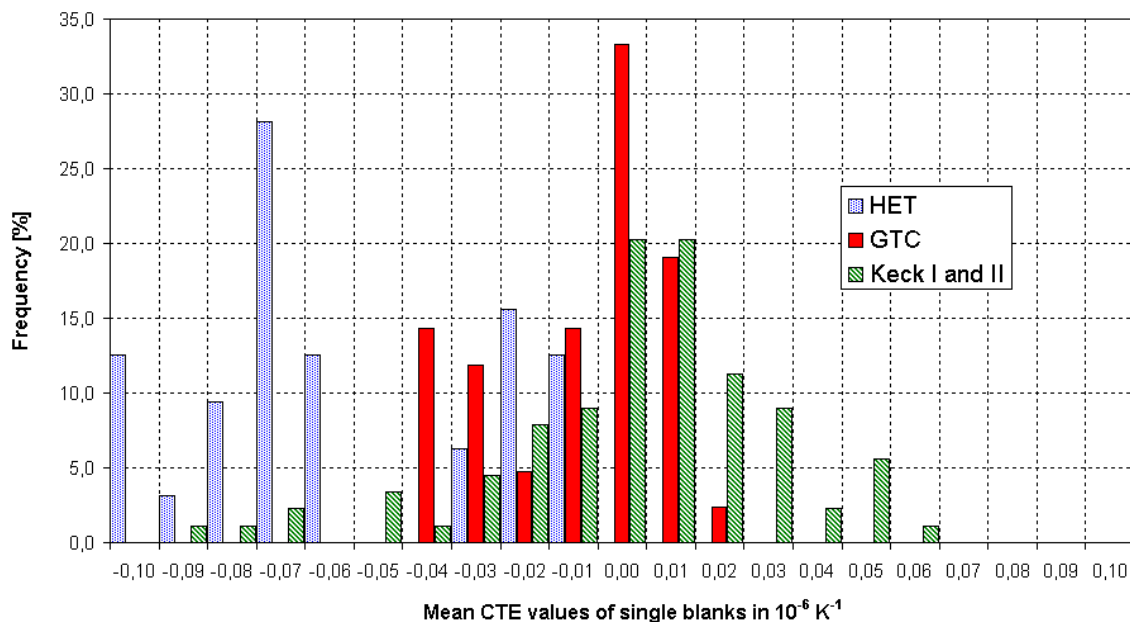


Figure 8: Frequency distribution of the mean CTE values of single blanks

Figure 8 shows that most mean CTE values are slightly negative. The reason is that a negative mean CTE permits a possible future adjustment of the mean CTE using an additional annealing process, which in any case leads to more positive CTE values. With the additional annealing process, the mean CTE can be increased to adjust the mean CTE and further minimize the distribution at the expense of an increase in production time.

5. Homogeneity of CTE

The homogeneity of CTE, the total variation of CTE values inside a specific batch, can be guaranteed in the following weight classes for each part, provided the diameter of the part is at least twice its thickness [13].

up to 18.0 tons	$\leq 0.03 \cdot 10^{-6} K^{-1}$
up to 6.0 tons	$\leq 0.02 \cdot 10^{-6} K^{-1}$
up to 0.3 tons	$\leq 0.01 \cdot 10^{-6} K^{-1}$

Table 4: Homogeneity of ZERODUR® depending on the weight according catalog [13]

The latest data evaluations and one of the most extensive homogeneity measurement ever made on a single ZERODUR® mirror blank with 1554 mm diameter clearly show the excellent homogeneity of ZERODUR®. The results fit the expectations that 95% of the standard ZERODUR® mirror blanks in the 1 to 2 m class exhibit a homogeneity even less than $\pm 0.010 \cdot 10^{-6} K^{-1}$. At 1300 mm diameter, a homogeneity quality of less than $0.004 \cdot 10^{-6} K^{-1}$ could be verified with the improved dilatometer measurement setup [14].

5.1 Statistical Homogeneity Evaluation

For future extremely large telescopes, with more than 3000 segmented mirror blanks, the reproducibility of the homogeneity from blank to blank is an important issue. Figures 9-11 show homogeneity evaluations of all segmented ZERODUR® mirror blank projects up to now (measured with the standard accuracy dilatometer).

Figure 9 shows the frequency distribution of the total variation of CTE values for HET (~115 kg/blank), Keck and GTC mirror blanks (~550 kg/blank). The HET data has the smallest total variation. Even if the maximum CTE variation of the GTC blanks is nearly the same as that for the Keck blanks, it seems that the GTC values are significantly better.

In general the distribution of the GTC values shows that ~60% of all total variations are better than $0.01 \cdot 10^{-6} K^{-1}$ (valid also for about 85% of the HET values – which are much smaller) whereas this is only valid for 22% of the Keck mirrors.

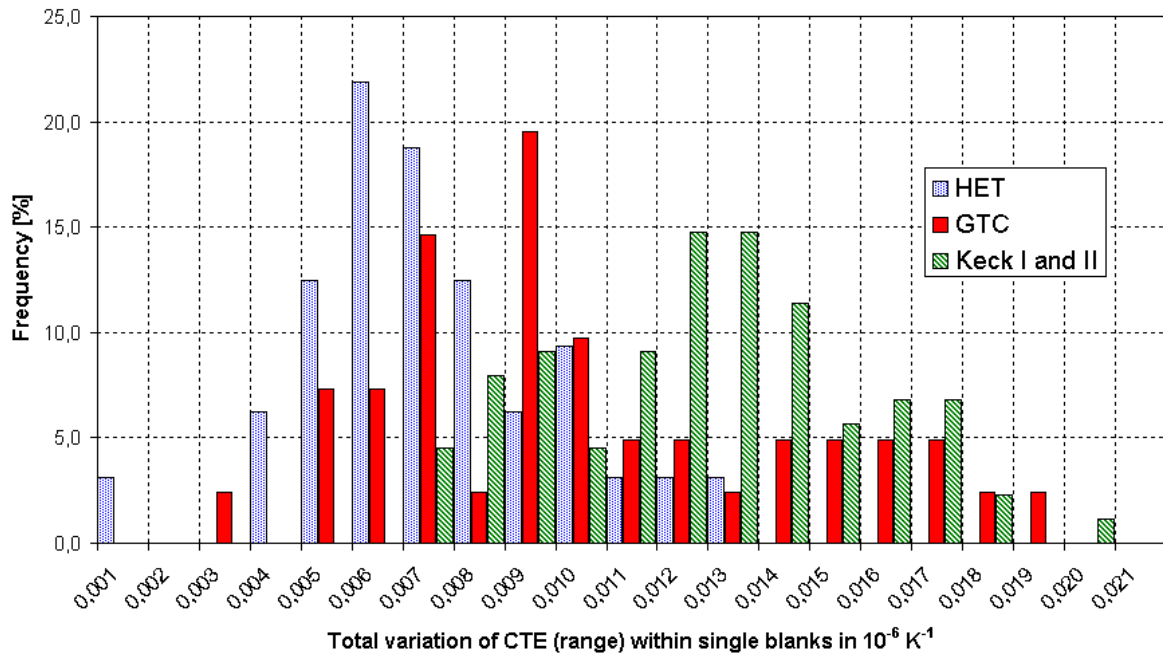


Figure 9: Frequency distribution of the total variation of CTE within single blanks

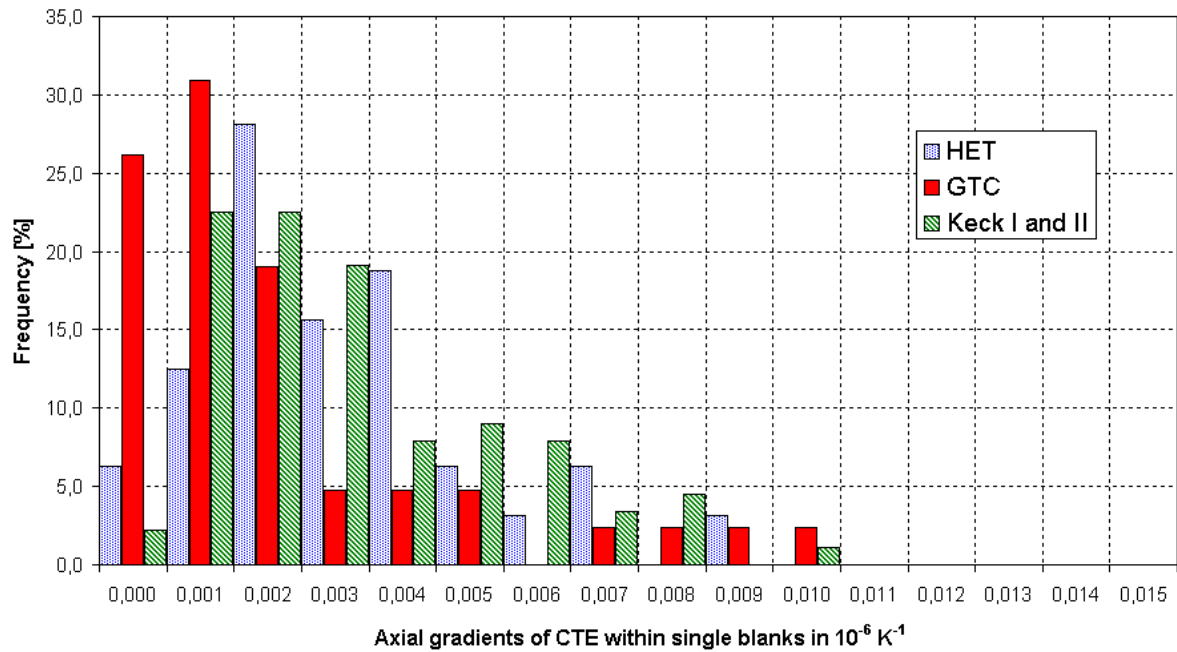


Figure 10: Frequency distribution of the axial gradients of CTE within single blanks

Figure 10 shows the axial gradient distribution of the mirror blanks. The maximum value for all mirror blanks is smaller than $0.01 \cdot 10^{-6} \text{ K}^{-1}$. Most values are even below the reproducibility of the standard dilatometer measurement ($\pm 0.005 \cdot 10^{-6} \text{ K}^{-1}$).

To get an overall impression of the general CTE homogeneity of ZERODUR® it is helpful to take the single measurements and normalize them to the mean values. Therefore the mean value of each mirror blank has been subtracted from the single measurements of this blank. These values can be displayed together for all mirror blanks of all projects in a single frequency diagram (figure 11).

After fitting a Gaussian distribution to the data set it is apparent that 95.5% (2*standard deviation) of all measurements are within $\pm 0.007 \cdot 10^{-6} \text{ K}^{-1}$ deviation from the mean value. In addition, the repeatability of the measurement (95.5%) is displayed in the diagram. The repeatability is only slightly lower than the double standard deviation. The complete curve is a convolution of the repeatability distribution of the standard dilatometer and the real distribution reflecting the measurement homogeneity. As a first approximation the 2*standard deviation of the de-convoluted real distribution without reproducibility effects is $\pm 0.005 \cdot 10^{-6} \text{ K}^{-1}$.

This shows that the homogeneity results so far are better than that reflected by the measurement due to the influence of the measurement repeatability of the dilatometer measurement system on the data.

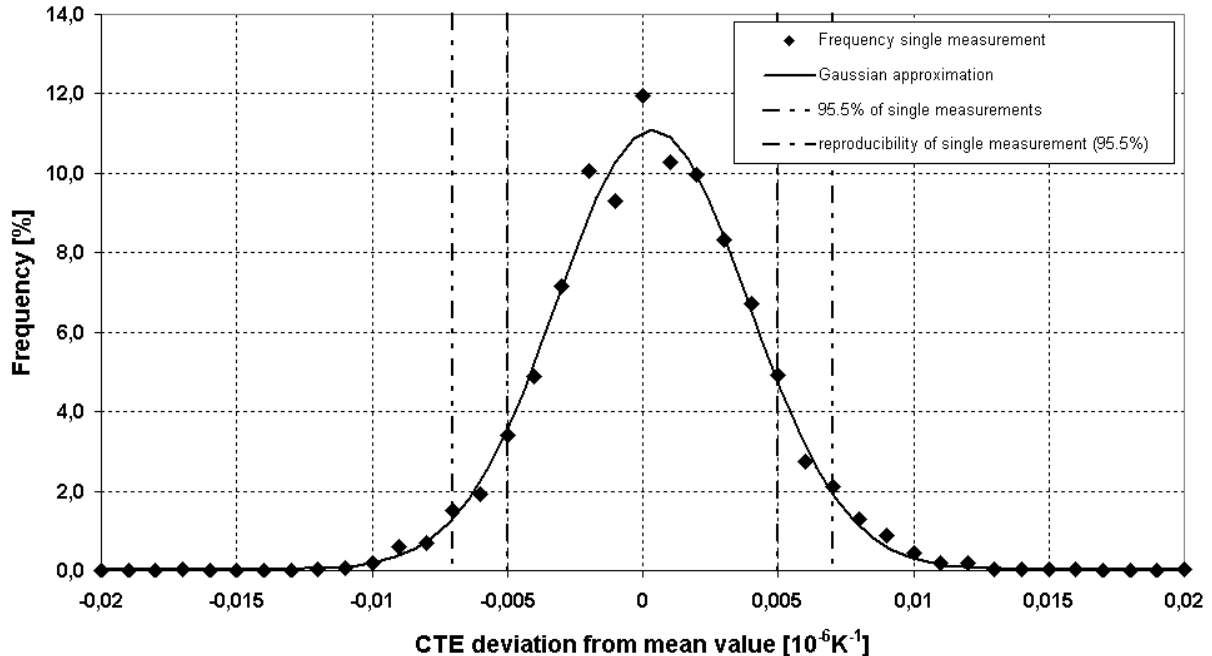


Figure 11: Homogeneity of the CTE: combined data from all mirror blanks of all projects.

5.2 Homogeneity of CTE in a single blank

Extensive measurements of the homogeneity inside a single mirror blank of ZERODUR® have been done for the four 8 m diameter VLT mirror blanks with the standard accuracy dilatometer setup. In total, 122 samples have been placed uniformly distributed on 5 circles of different radii on top and below the single mirror blank. Figure 12 shows the sampling plan of the mirror and a summary of the results for each of the 4 VLT mirror blank [6].

The single measurements are randomly distributed (no significant radial, angular or axial dependency observable) with a mean homogeneity in the range of $\sim 0.02 \cdot 10^{-6} K^{-1}$ peak to valley. Detailed results can be found in [6].

CTE (0°C,50°C) [10 ⁻⁶ K ⁻¹]	mean value	homogeneity (peak to valley)
Specification	0+/- 0.15	0.05
Mirror 1	-0.043	0.009
Mirror 2	-0.032	0.011
Mirror 3	-0.040	0.024
Mirror 4	-0.017	0.028

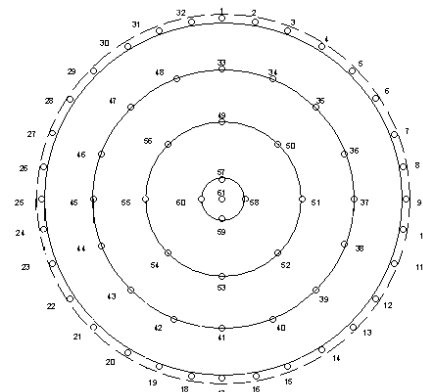


Figure 12: Sampling plan and results of the homogeneity for the 8 m VLT mirror blanks [6]

The homogeneity measurement results in the past were mainly limited by the reproducibility of the dilatometer measurement as shown in chapter 5.1. To overcome this problem, a dilatometer measurement setup with improved repeatability (chapter 2.2) has been constructed. This new setup has been applied to the CTE homogeneity measurement of 111 samples taken from a randomly selected striae free standard circular ZERODUR® blank with diameter 1554 mm and 345 mm thickness [14].

Figure 13 shows a 2D color plot of the CTE deviations from the mean value of all measurements. The CTE values are displayed in ppb/K units ($1 \text{ ppb/K} = 0.001 \cdot 10^{-6} K^{-1}$). Positive deviations in the diagram refer to larger CTE values as compared to the mean value. The total peak to valley homogeneity of the blank is $\pm 0.0064 \cdot 10^{-6} K^{-1}$ (The 2*standard deviation is $\pm 0.003 \cdot 10^{-6} K^{-1}$). The results show that for 70% of the ZERODUR® blanks, the peak to valley variation is less than $\pm 0.004 \cdot 10^{-6} K^{-1}$ representing a final mirror blank diameter of 1.3 m.

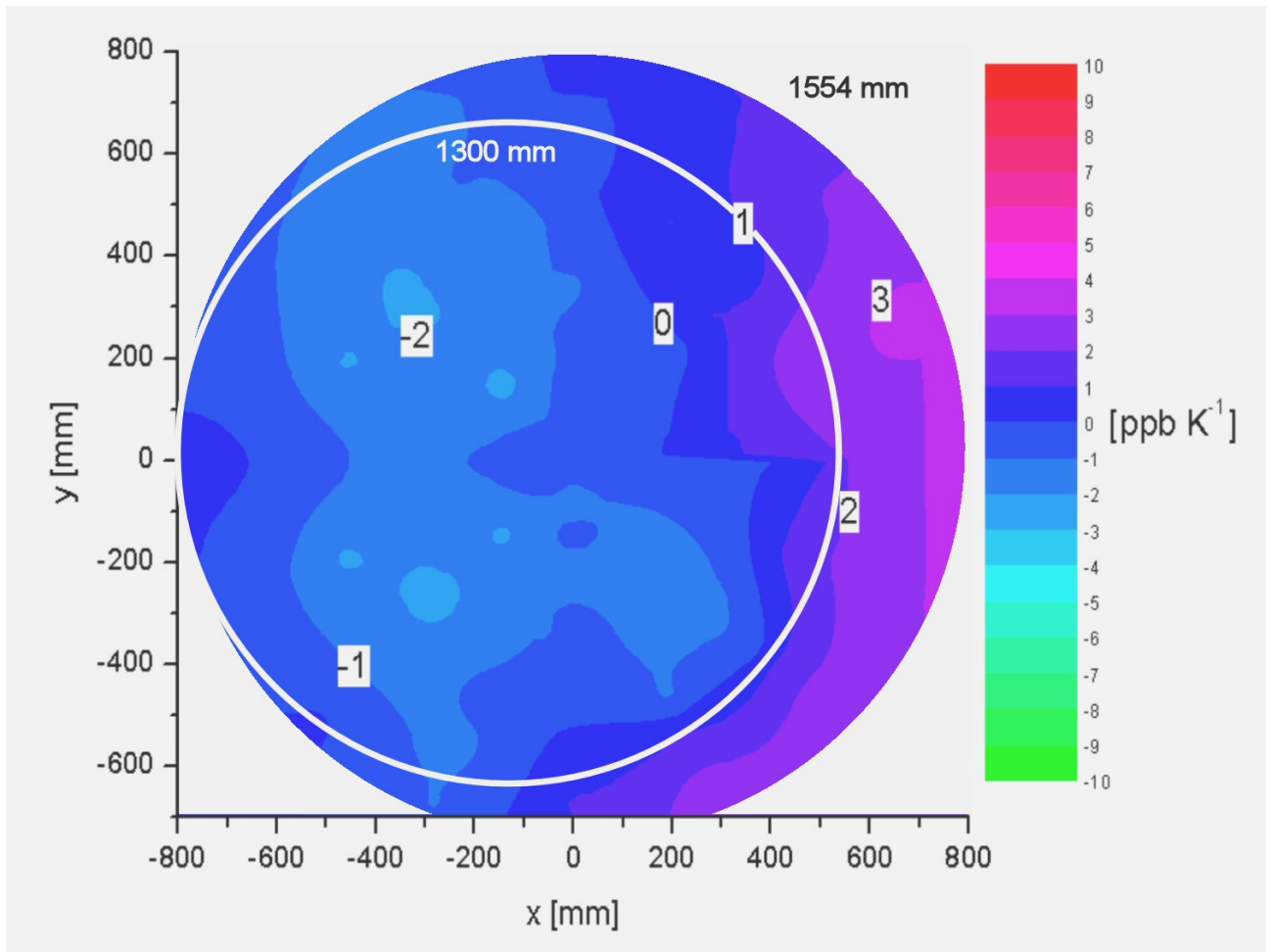


Figure 13: Two-dimensional variation of the CTE deviations from the mean value over the horizontal cut section [14]

Table 5 shows a summary of the results including the mean value over all measurements and axial gradient based on a final blank thickness of 70 mm. The axial gradient measurement results are in good agreement with the results shown in figure 10.

	[*10⁻⁶K⁻¹]
Mean CTE (0°C, 50°C)	0.042
Homogeneity (peak to valley) total	0.0064
2*standard deviation (95%)	± 0.003
Homogeneity (peak to valley) on 1.3 m	0.004
Axial CTE based on 70 mm thickness	0.0022 (max)

Table 5: Overview on the homogeneity measurement results on a single ZERODUR® blank [14]

6. Thermal cycling

ZERODUR® may be used as a mechanical component as well as a window at temperatures up to 600°C. Slight changes may arise in the CTE that can affect components of the highest optical precision, due to temperature treatments by the customer and user.

There are three temperature ranges where length stability may be diminished by changes of expansion or CTE [7]:

1. $T > 700^{\circ}\text{C}$ (ceramization range)
2. $130^{\circ}\text{C} \leq T \leq 320^{\circ}\text{C}$ (upper relaxation range)
3. $-70^{\circ}\text{C} \leq T \leq +40^{\circ}\text{C}$ (lower relaxation range)

6.1 Ceramization range

At temperatures above 700°C ceramization of the material continues and leads to irreversible changes of the dimensional and the material properties; the CTE for all temperature ranges is changed irreversibly.

6.2 Upper relaxation range

Figure 14 shows the limits and the influence of this temperature range on the dimensional stability. Initially well-annealed samples were quenched from different temperatures in an ambient room atmosphere and the CTE (0°C, 50°C) was measured. Up till temperatures of ~130°C, there was no change observed in the CTE (0°C, 50°C). With increased temperatures, the CTE (0°C, 50°C) after quenching also increased until a saturation value is achieved at 320°C.

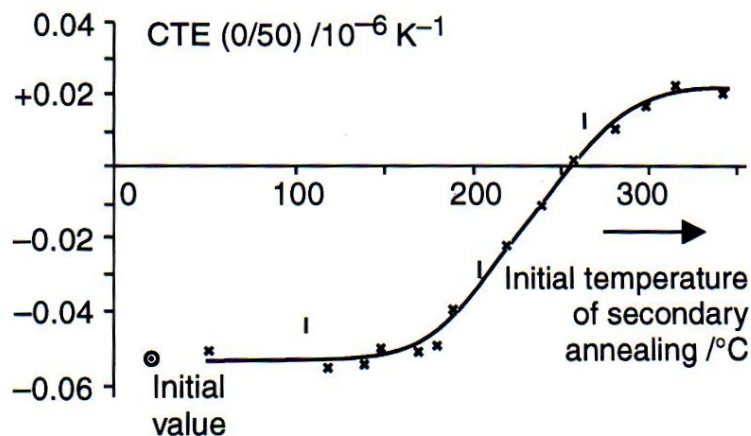


Figure 14: Variation of the coefficient of thermal expansion CTE (0°C; 50°C) – first cooling at 0.1 K/min- as a function of the initial temperature of a secondary cooling in open air to room temperature [7].

Different cooling rates influence the final value of CTE (0°C; 50°C) when cooling starts from the same temperature. If the final cooling rate R_E differs from the initial cooling rate R_P (ZERODUR® is typically cooled during production at an initial cooling rate between 1 K/h and 6 K/h), the change of the CTE (0°C; 50°C) starting from 320°C can be estimated by using the following equation:

$$CTE(0^\circ C, 50^\circ C) = 0,025 * \log_{10} \left(\frac{R_E}{R_P} \right) 10^{-6} K^{-1}$$

The CTE value changes with temperature cycles in the range between 130° and 320°C, so does the sample length at room temperature. Fast cooling in general leads to a greater length. In the given example 30 K/h fast cooling from a temperature of 300°C to room temperature leads to a relative elongation Δ/l of $14 \cdot 10^{-6}$ for a sample which has originally been annealed with a rate of 1 K/h [7].

Thermal cycling in the upper relaxation range leads to permanent changes of CTE and relative length at room temperature. These effects are reversible as soon as the sample is heated up to above 320°C and cooled down with the initial cooling rate of the ceramization process.

For most mirror applications ZERODUR® is exposed to elevated temperatures during coating. These processes are short in time and only a layer with small thickness really sees these high temperatures. Such processes have only negligible effect on the surface figures as experience demonstrates.

6.3 Lower relaxation range

Thermal cycles in the lower relaxation range lead to hysteresis shapes of the $\Delta/l(T)$ curve as shown in figure 15. The vertical opening of this hysteresis is of the order of $\Delta/l = 10^{-6}$. Like the effects of the upper relaxation range these effects are also strictly reversible. However, whereas the effects of the upper relaxation range are stable at room temperature, those in the lower range relax in a relatively short time even at rather low temperatures.

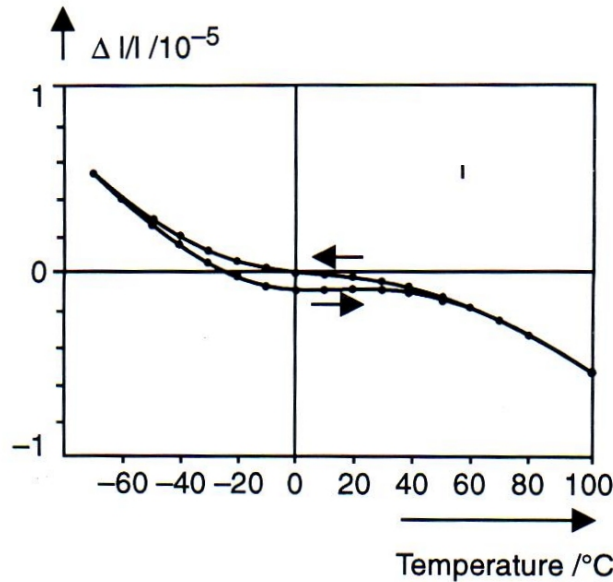


Figure 15: Hysteresis in the course of relative length change $\Delta l/l$ during a thermal cycling process of ZERODUR[®], temperature rate = 1.25 K/min [7]

6.4 Isothermal long-term stability

The length of a 400 mm long ZERODUR[®] bar was examined at a constant temperature of 20°C at the PTB Braunschweig from September 1973 for about 10 years [8]. Over a long period of time, ZERODUR[®] shows a continuous, monotone decrease in length (often referred to as aging). The slope of the decrease in length depends on the age of the sample after the last thermal treatment and the thermal treatment itself. A slow cooling after thermal treatment decreases the slope (the sample appears older).

A second thermal treatment will start the aging process anew. In general it can be said that the isothermal long-term stability of ZERODUR[®] is affected by the thermal cycling of the material but on a much longer time scale.

New investigations on the relaxation and isothermal long-term stability effects show that both effects can be described using the same mathematical model. This mathematical model is able to predict the relaxation behavior of a given ZERODUR[®] material under given thermal cycling conditions. This complete knowledge in the thermal expansion behavior of ZERODUR[®] even for small temperature changes is necessary especially for future EUVL applications.

7. Cryogenic behavior of ZERODUR®

At SCHOTT the cryogenic measurement of the thermal expansion is limited to temperatures of ~180°C (93 K). Therefore the description of the cryogenic behavior of ZERODUR® is based on known publications by other institutes [10, 11, 12].

Figure 16 shows the CTE of ZERODUR®, glassy ZERODUR® and 3 other materials in the temperature range of 10 to 300 K [11]. At low temperatures ZERODUR® normally expands upon cooling.

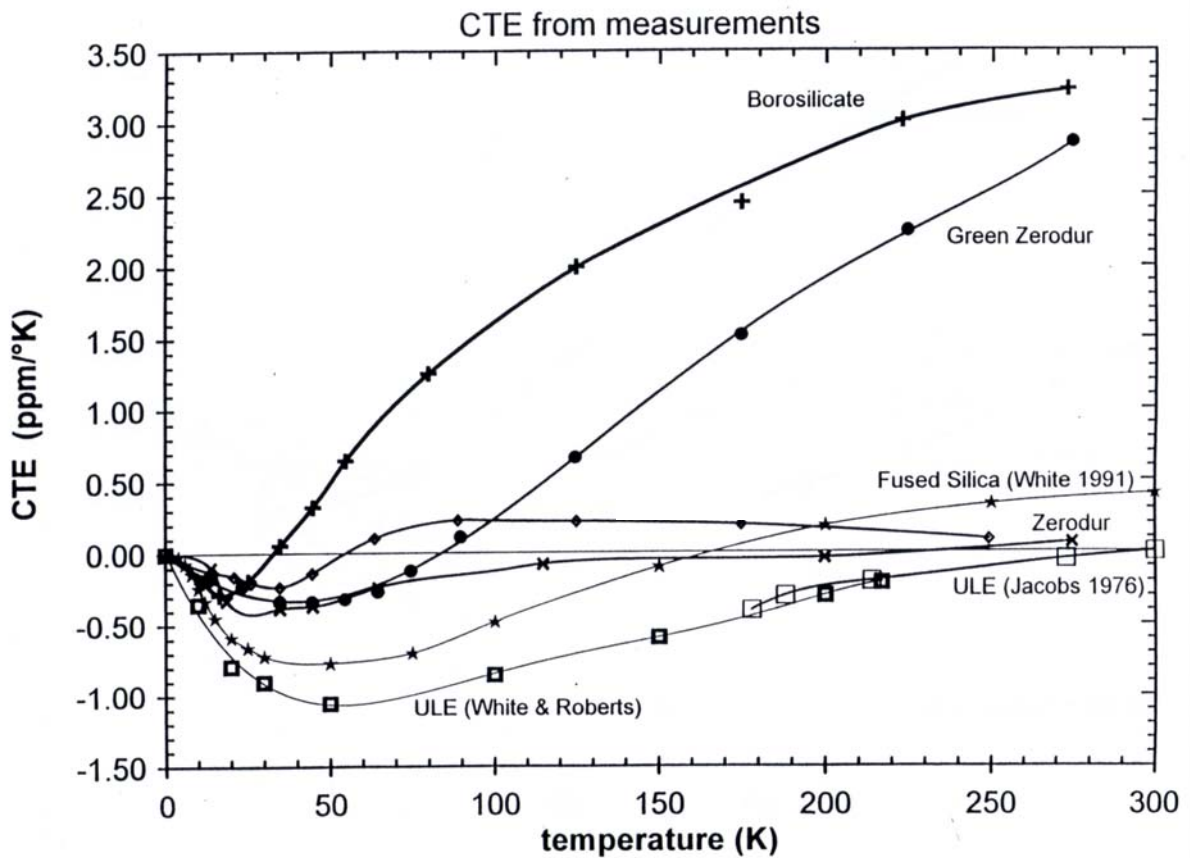


Figure 16: CTE of different materials with at temperatures between 10 and 300 K [11]

Burge, Peper and Jacobs observed a maximum CTE of $-0.4 \cdot 10^{-6} \text{K}^{-1}$ at ~20 K. They also observed some slight relaxation effects between 20 and 30 K amounting to $0.6 \cdot 10^{-6} \text{K}^{-1}$ relative length change.

One important question customers often ask is how stable ZERODUR® remains under cryogenic temperatures and temperature changes especially for the use as high precision astronomical mirror substrates under cryogenic conditions in space. J. W. Baer and W.P. Lotz observed mirror shape deviations of 300 mm diameter under temperature changes from room temperature to 130 K [12]. They found no significant distortion on two parabolic mirrors. These mirrors remained at or below 0.035 waves RMS figure irregularity over the 170 K cool-down from room to operational temperature.

8. Literature

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