

Homogeneity of the coefficient of linear thermal expansion of ZERODUR[®]

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ABSTRACT

The low thermal expansion glass ceramic ZERODUR[®] is the material of choice for many big astronomical telescope projects like VLT, Keck I + II, HET, LAMOST and GRANTECAN (GTC). For future giant telescope projects like OWL or TMT with at least several hundreds of mirror blanks the CTE homogeneity within a single blank and from blank to blank is an crucial issue.

The ZERODUR[®] production process is based on established and proven methods used in the production of high homogeneity optical glasses. Therefore ZERODUR[®] itself is a material of highest homogeneity even in large dimensions and huge quantities. This paper presents an evaluation of the homogeneity of the thermal expansion coefficient within more than 250 mirror blanks. The observed homogeneity range is only slightly larger than the repeatability of the standard dilatometer measurement of $\pm 0.005 \cdot 10^{-6} \text{ K}^{-1}$.

To improve the accuracy of measurement and to get a deeper understanding of the thermal expansion behaviour of ZERODUR[®] a new dilatometer was built exhibiting a repeatability of $\pm 0.001 \cdot 10^{-6} \text{ K}^{-1}$. Detailed evaluations of the thermal expansion coefficient homogeneity of a 100 mm x 100 mm ZERODUR[®] test block showed no variation within the repeatability of measurement of the improved dilatometer.

Keywords: Zerodur, astronomy, thermal expansion, homogeneity, repeatability

1. INTRODUCTION

Schott has a long history in the delivery of Zerodur mirror blanks starting with the delivery of a 3602 mm diameter and 593 mm thick mirror blank for the Calar Alto Observatory of the MPIA in 1973. With the production of 4 m monolithic mirrors in the 1980s the era of massive blanks had reached a limit. Significantly larger mirrors stiff by themselves by their 6:1 diameter to thickness ratio would come out extremely heavy needing outstanding mechanical supports and directional control. Two possible ways to overcome the size restriction have been pursued. Large telescopes have been built using ZERODUR[®] segments: The two Keck telescopes at the Mauna Kea peak of Hawaii, the Hobby-Eberly Telescope (HET) and the GranTeCan (GTC) telescope, which is presently under construction at La Palma, one of the Canary Islands.

The other approach is based on thin flexible monolithic mirrors. The European Southern Observatory ESO succeeded to demonstrate the feasibility of high quality telescopes with the ESO-NTT (New Technology Telescope, 3.6 m ZERODUR[®] primary mirror, La Silla, Chile). This was the first large telescope using active optics to control the primary mirror shape online. In 1988 ESO placed an order to SCHOTT to manufacture 4 thin ZERODUR[®] meniscus shaped blanks of 8.2 m diameter. They were meant for the ambitious project ESO –VLT Very Large Telescope at the Paranal peak in Chile [1,2]. This proceeding gives an overview on the CTE measurement results of the VLT mirror blanks and the segments from the Keck, HET and GTC telescopes and tries to give a general impression on the homogeneity of the coefficient of thermal expansion (CTE) of ZERODUR[®]. Figure 1 gives a summary on these mirror projects including the overall CTE specifications.

The ZERODUR[®] production process is based on established and proven methods used in the production of high homogeneity optical glasses. Therefore ZERODUR[®] itself is a material of highest homogeneity, and even in large pieces with dimensions of several meters exhibits thermal and mechanical characteristics with nearly unmeasurable relative deviations. The optimized processing sequence starts with the discontinuous melting of the raw glass material. During the melting process several measures are taken to achieve an excellent homogenization of the melt. After the

casting process the hot ZERODUR glass melt is cooled down inside an annealing oven in a controlled way. A first processing step has to be carried out after the coarse annealing process to get rid of any pre-ceramized outer material layers to prevent any uncontrolled ceramization in the following annealing process. During this processing step first CTE samples will be taken that can be added to the ceramization oven separately for a fast CTE measurement after finishing ceramization. The subsequent annealing process that is necessary for the ceramization of the glassy ZERODUR[®] influences the final CTE homogeneity of the blank and can last up to several months depending on the size of the ZERODUR[®] part. In the following processing steps CTE samples are taken directly from the final ZERODUR[®] material.

Project/Site	Dimensions of Primary Mirror Blank	Mean CTE [10 ⁻⁶ K ⁻¹] specification	CTE homogeneity [10 ⁻⁶ K ⁻¹] specification	Start of Production	Completed
VLT / Chile	4 blanks dia 8200 mm, thickness 177 mm and 1000 mm centre hole.	+/- 0.15	< 0.05	1988	1996
Keck I / Hawaii	43 segments dia. 1900 mm thickness 76,5 mm	+/- 0.10	≤ 0.02	09/1984	1990
Keck II / Hawaii	42 segments dia. 1900 mm thickness 75,8 mm			10/1991	1993
HET / Texas	96 hexagons, width 1019 mm, thickness 56 mm	+/- 0.15	≤ 0.01	02/1994	1995
GTC / La Palma	42 hexagons, width 1622 mm, thickness 83,5 mm	+/- 0.05	≤ 0.02	06/1999	2002

Figure 1: CTE specifications for the ZERODUR[®] mirror blanks of the VLT, Keck I+II, HET and GTC telescopes.

2. CTE METROLOGY

The CTE of ZERODUR[®] is measured by taking samples from the material and measure them in a precision dilatometer.

The basic construction of the dilatometer used for the CTE measurement can be seen in figure 2. This setup is similar to the instrument presented by Plummer and Hagy [3] but with the difference that the sample holder and the push rod in our setup is made of a titanium silicate glass. The system is optimized for a sample length of 100 mm and 6 mm diameter. The temperature is measured using a platinum resistance thermometer (PT 100) mounted near the sample. The temperature of the dilatometer head is stabilized better then 0.2 K. The whole equipment is in a lab where temperature varies less then 1K.

For the measurement the sample and the sample holder are immersed into a water bath. A thermostat is programmed to heat and cool the water bath following a defined procedure. When changing the temperature the sample changes its length and the rod moves the coil of a LVDT (Linear Variable Differential Transformer) which can measure relative length changes to a high precision.

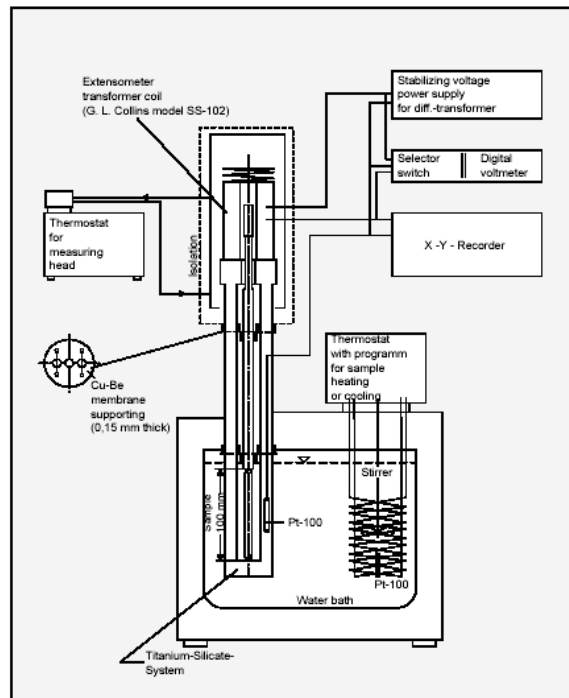


Figure 2: Basic construction of the push rod dilatometer [5]

The signals of the LVDT and the temperature of the water bath are recorded and used for calculating the CTE.

The system is optimized and calibrated for measurement of the CTE (0°C,50°C) value. The measurement is based relative length measurements. 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.

The system is regularly calibrated using certified reference samples. We use a titanium silicate reference sample that was measured at the PTB (Physikalisch Technische Bundesanstalt) with a high-precision interferometer system. The reference sample was measured to an accuracy of $\pm 0.004 \cdot 10^{-6} \text{K}^{-1}$. The overall accuracy of the dilatometer for the CTE (0°C,50°C) measurement is $\pm 0.01 \cdot 10^{-6} \text{K}^{-1}$ and the reproducibility is $\pm 0.005 \cdot 10^{-6} \text{K}^{-1}$ (95% confidence level).

3. CTE MEASUREMENT RESULTS OF SINGLE VLT MIRROR BLANKS

Four VLT blanks with a diameter of 8200 mm and a thickness of 177 mm have been produced and delivered to the ESO between 1988 and 1996. For the measurement of the CTE homogeneity of the VLT mirror blanks the ZERODUR® dilatometer samples have been taken from the outer diameter of the blank after the first coarse annealing of the glassy blank during raw glass machining. These samples were positioned on top and below the blank close to their original position during the ceramization process. The temperature homogeneity inside the ceramization oven has the biggest influence on the homogeneity of the ceramized ZERODUR® blank. The CTE pattern of the ZERODUR® samples therefore reflects the influence of the temperature distribution on the thermal expansion behaviour of the mirror at the measurement positions.

Radius [mm]	Positions on convex	Positions on concave	Sum
0	1	1	2
480	4	4	8
1800	8	8	16
3000	16	16	32
4125	32	32	64

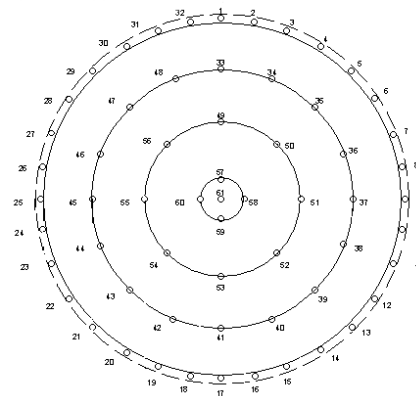


Figure 3: CTE sampling plan for the VLT 8 m mirror blanks

In total 122 samples have been positioned uniformly distributed on 5 circles with different radii on top and below the mirror blank. Figure 3 shows the sampling plan of the mirror and a table with the radial positions. Figure 4 shows a summary of the results for each of the 4 VLT mirror blank.

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

Figure 4: Summary of the CTE results for the VLT mirror blanks

The mean values are within expansion class 1 of the ZERODUR catalog. The CTE homogeneity, the peak to valley variation of the measured CTE values, is better than $0.03 \cdot 10^{-6} \text{K}^{-1}$. CTE and CTE homogeneity are well within specification.

The next figures show more detailed results of the CTE measurements for the VLT mirror blanks. In figure 5a) the mean CTE values for each measurement radius on the concave and on the convex side of mirror blanks are displayed. On top of the figure the numbers of samples that are relevant for the mean value on a circle are given (half of the samples belong to the concave surface and half of the samples belong to the convex surface which has to be taken into account for statistical significance). The centre position of the mirror is represented by only one sample on the concave and one sample on the convex surface. The mean CTE value on the outer three diameters is nearly constant for every mirror. Deviations can be found between the two inner rings of measurement. Mirror number three exhibits a deviation of about $\sim 0.015 \cdot 10^{-6} \text{K}^{-1}$. Mirrors number one and two exhibit the smallest deviations, with values within the repeatability of measurement. Due to the low number of samples on the center (inner) circle it is difficult to draw any conclusion out of this trend. This is also valid for the deviation from the inner center circle to the center, because the center of the mirror is only represented by one sample on each surface. The maximum difference between the mean values of the concave surface and the mean values of the convex surface is $0.005 \cdot 10^{-6} \text{K}^{-1}$ (mirror 3).

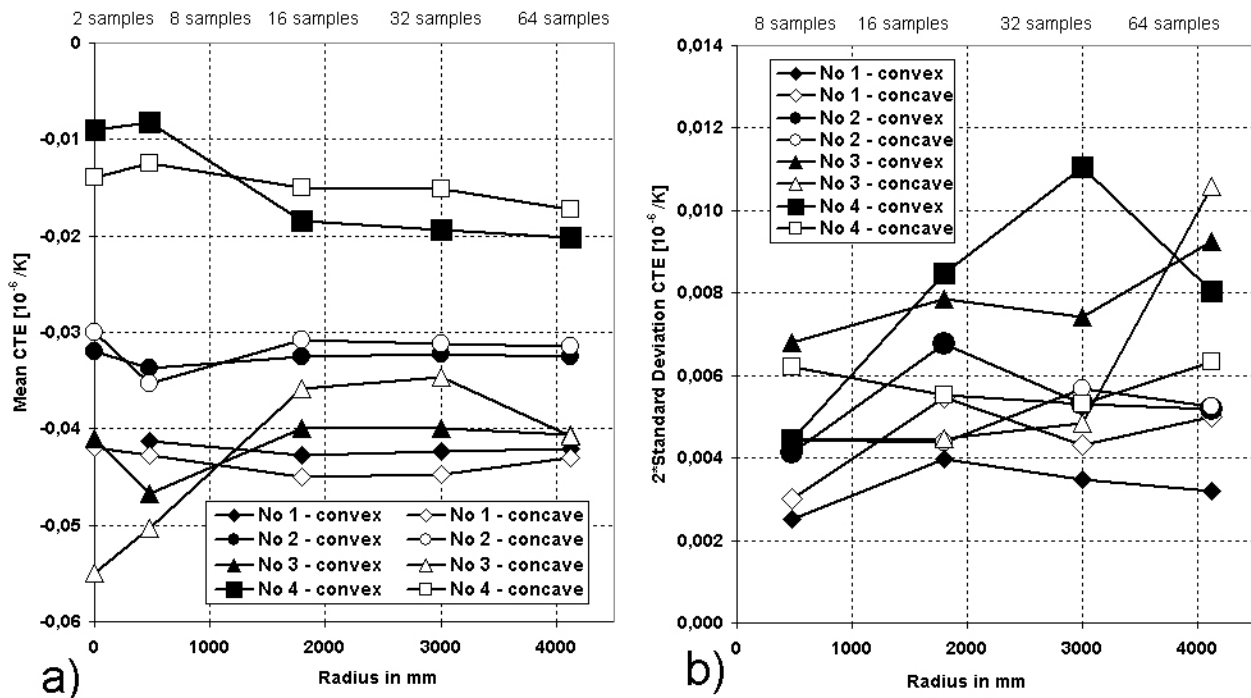


Figure 5: ESO VLT 8m mirror blanks mean CTE and 2*standard deviation over circles with different radii

Fig. 5 b) shows the double standard deviation over circles with different radii. Mirror 4 exhibits the maximum standard deviation value of $0.011 \cdot 10^{-6} \text{K}^{-1}$. Compared to the reproducibility of the single measurement ($0.005 \cdot 10^{-6} \text{K}^{-1}$) the observed standard deviations are very small. In general no obvious trend is visible. The standard deviation is different from mirror to mirror. No general trend of the standard deviations as a function of the radius is observable. The standard deviation at 480 mm radius is slightly lower but the reason for this might be the low number of measurements.

Figure 6 shows the distribution of all single measurements of mirror one as a function of the angle. There is no angular dependence of the CTE values observable. The values scatter nearly randomly between the minimum and maximum CTE value of the blank.

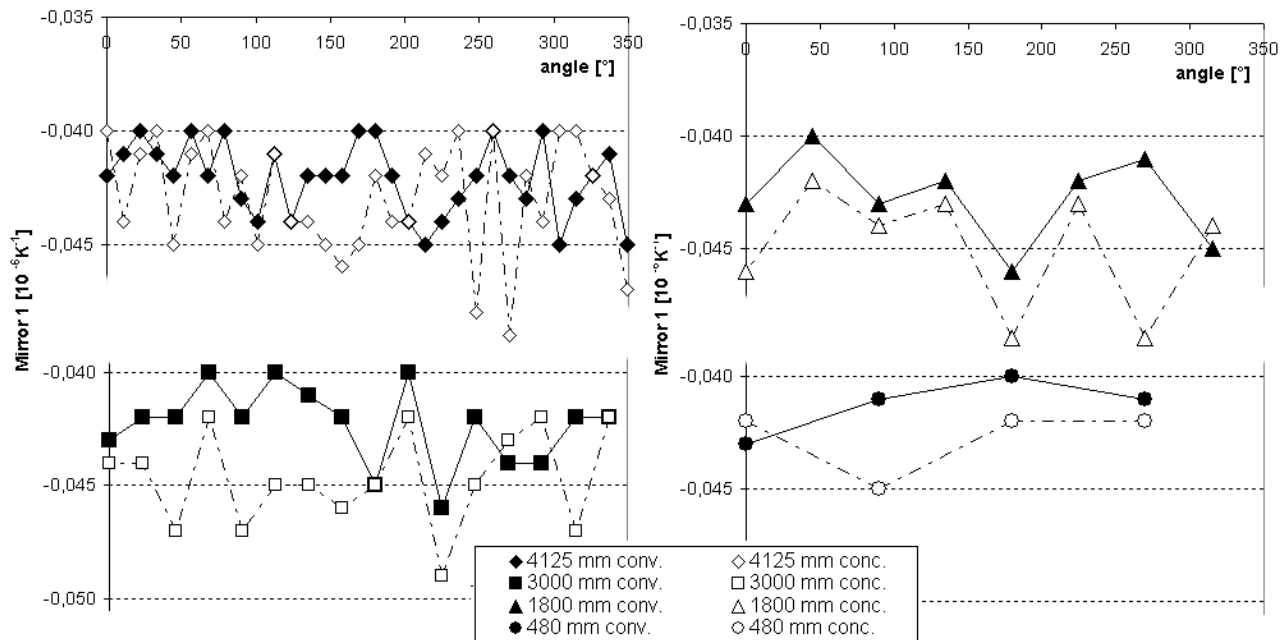


Figure 6: ESO VLT 8 m mirror 1: overview on all single measurement results

As a conclusion all the results show that the CTE variations within the 8 m mirror blanks are more or less randomly distributed within the mirrors. No general geometrical trend is observable because the deviations are either too small and therefore within the measurement repeatability or the amount of samples is too small (centre region).

3. COMBINED CTE RESULTS OF MIRROR BLANKS FOR SEGMENTED TELESCOPES

Up to now more than 250 ZERODUR[®] mirror blank segments with diameters between 1-2 m have been produced at SCHOTT. Mirror blanks with dimensions up to 2.4 m are fabricated out of thick cylindrical or near net shaped ZERODUR[®] castings with a thickness sufficient to fabricate at least 3 or 4 mirror blank segments out of it. After cermization the castings are cut into single blanks. The CTE samples are taken from the remaining material of the casting according the sampling plan. Figure 7 shows the sampling plans for the Keck, HET and GRANTECAN (GTC) mirror blanks. The sampling plans are not at scale. It should be noted that the HET mirror blanks are about half the diameter compared to the Keck mirror blanks. The sampling plan shows a top and a side view of the casting with the single raw blanks. SCHOTT delivered circular mirror blanks for the KECK project. The final Keck mirror blanks exhibit a hexagonal shape. Therefore the circular mirror blanks were stress polished later and cut to their final hexagonal dimensions by the customer. The CTE homogeneity of the 1900 mm diameter Keck mirror blanks was measured using 18 samples, 9 from the top and 9 from the bottom of each blank. The samples on the top and on the bottom side are evenly spread on two measurement circles. Additionally samples were taken from the center of the blanks. The samples have been cut from the surrounding material of the blank as indicated in the sampling plan. Therefore within a casting samples of a layer between two blanks are assigned to the bottom of the upper blank as well as to the top of the lower blank.

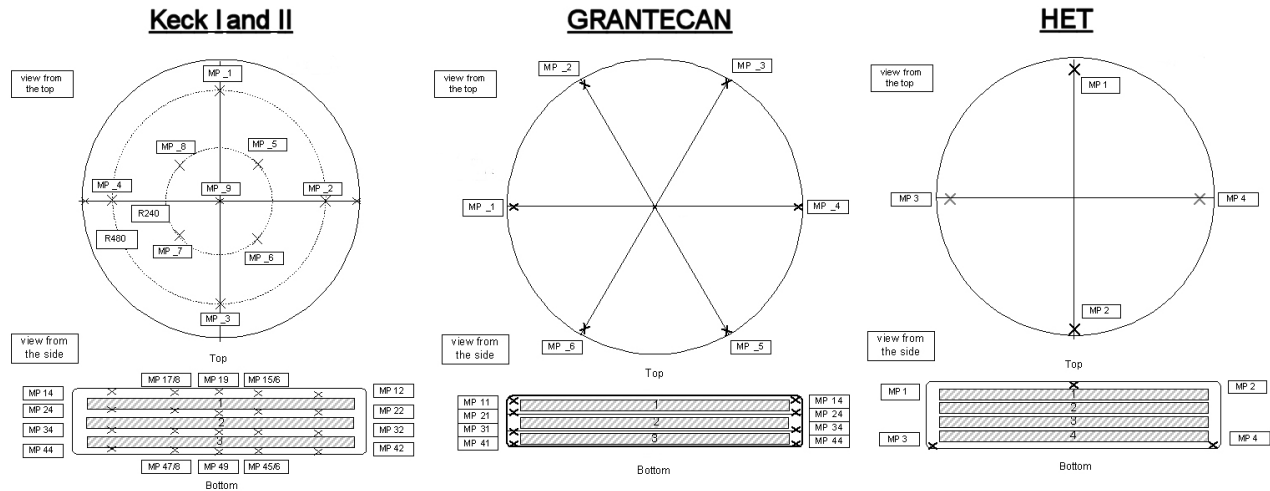


Figure 7: CTE sampling plans for the Keck, GTC and HET mirror blanks.

The sampling plan for the 1600 mm GRANTECAN (GTC) mirror blanks is displayed in the center of figure 7. 12 samples, taken from the outer diameter of the blank, are assigned to each mirror blank: 6 samples refer to the top and 6 samples refer to the bottom of the mirror blank. In contrast to the Keck mirror blanks the GTC mirror blanks were directly cast into a near net shape hexagonal mold. All samples were taken from the surrounding near the center of the hexagonal flats. Similar to the sampling of the KECK mirror blanks each layer of samples between two blanks inside a casting can be assigned either to the bottom of the upper blank and to the top of the lower blank.

The CTE of the HET mirror blanks is determined by only 4 samples of each casting. In figure 7 it can be seen that each casting consist of approximately 4 mirror blanks but with a final thickness that is much lower compared to the Keck and GTC mirror blanks. Two samples were taken from the top of the casting and two samples from the bottom of the sampling. The orientation between the top and the bottom samples is turned by 90°.

The CTE (0°C,50°C) values of all samples have been measured using the standard dilatometer setup mentioned above. The following frequency graphs show the distribution of the measurement results for the mean CTE value, the CTE homogeneity and the CTE axial gradient for all mirror blanks. 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 $0.06 \cdot 10^{-6} \text{K}^{-1}$.

For the GTC project each of the 42 delivered mirror blanks is represented by a mean value in the diagram. The mean value range, peak to valley from the lowest to the highest CTE mean value, covers about $0.06 \cdot 10^{-6} \text{K}^{-1}$. 33% of all mirrors are between $-0.01 \cdot 10^{-6} \text{K}^{-1}$ and $0.00 \cdot 10^{-6} \text{K}^{-1}$. The Keck values are combined values from two mirror projects: Keck I and Keck II with about 89 single mirrors (spare mirrors included). The overall CTE range is bigger with $0.16 \cdot 10^{-6} \text{K}^{-1}$. About 40% of all mirrors are within a range of $\pm 0.01 \cdot 10^{-6} \text{K}^{-1}$ around $0.00 \cdot 10^{-6} \text{K}^{-1}$. There are only 32 HET mean CTE values. As mentioned before the reason for this is that the CTE values were evaluated for the complete casting. Each casting consist of at least 3 mirror blanks. The values cover a range of $0.12 \cdot 10^{-6} \text{K}^{-1}$ but whereas the Keck and GTC values follow a slightly deformed Gaussian distribution the HET mean CTE value distribution seems to be bimodal. The maximum is located around $0.07 \cdot 10^{-6} \text{K}^{-1}$ with about 28% of the castings within a range of $0.1 \cdot 10^{-6} \text{K}^{-1}$.

The range of the mean value distributions reflects the CTE specification. If the CTE specification is relaxed (the HET specification represents such a broad mean CTE grade that it does not anymore exists in our current ZERODUR® catalog) 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 materials used may have different production histories, meaning that part of the mirror blanks may be from a new melt campaign and part of the mirror blanks are produced from material on stock with different mean value. By using defined casting formats, special ceramization setups the reproducibility of the ZERODUR® manufacturing can be further improved to keep a tighter mean CTE distribution. For

example most of the GTC mirror blanks have been exclusively produced out of special hexagonal shaped molds in a sequence of melting campaigns.

Figure 8 also shows that most mean CTE values can be found in the negative range. The reason for this is that a negative mean CTE permits a possible future adjustment of the mean CTE using an additional annealing process e.g. for special stress reduction. With the additional annealing process the mean CTE can be only increased. Therefore this re-annealing process is a tool to adjust the mean CTE and further minimize the distribution at the expense of an increasing of production time and cost.

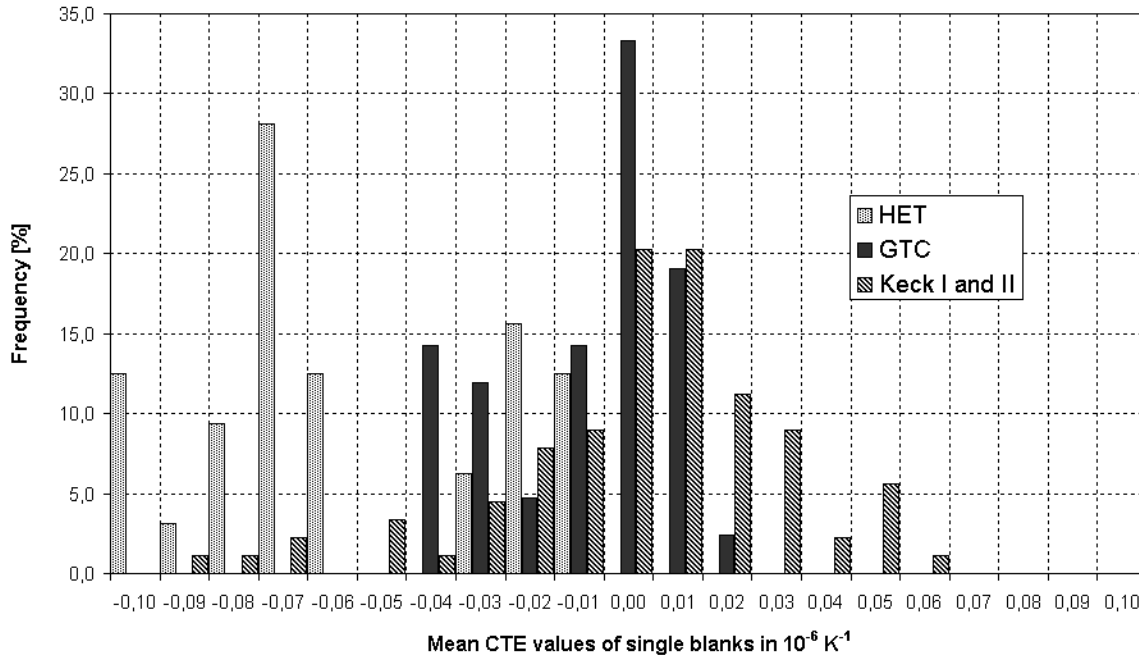


Figure 8: Frequency distribution of the mean CTE values of single blanks

Figure 9 shows the frequency distribution of the total variation of CTE values for all blanks. The total variation of CTE values within a single blank is determined by evaluating the largest difference between two single measurements.

As mentioned above each of the determined HET values represents a complete casting. The HET data also shows the lowest total variation. The maximum value is $\sim 0.013 \cdot 10^{-6} \text{K}^{-1}$. The distribution maximum is at $0.006 \cdot 10^{-6} \text{K}^{-1}$ representing 22% of all values. The largest value of the Keck mirrors is $\sim 0.021 \cdot 10^{-6} \text{K}^{-1}$ with a maximum between 0.013 and $0.014 \cdot 10^{-6} \text{K}^{-1}$ representing $\sim 30\%$ of all blanks. The largest value of the GTC mirrors is only slightly lower with $\sim 0.020 \cdot 10^{-6} \text{K}^{-1}$ but the distribution is broader with a maximum at $0.009 \cdot 10^{-6} \text{K}^{-1}$ representing 19% of the blanks. All distributions are slightly asymmetrical, because the total variation values cannot get smaller than 0.

Even if the maximum CTE variation of the GTC blanks is nearly the same as for the Keck blanks it seems that the GTC values are significantly better. In general the distribution of the GTC values shows that $\sim 60\%$ of all total variations are better than $0.01 \cdot 10^{-6} \text{K}^{-1}$ whereas this is only valid for 22% of the Keck mirrors but for 85% of the (much smaller) HET values. Nevertheless it should also be kept in mind that the Keck data is a combination of two mirror projects from two different melting campaigns of different time periods.

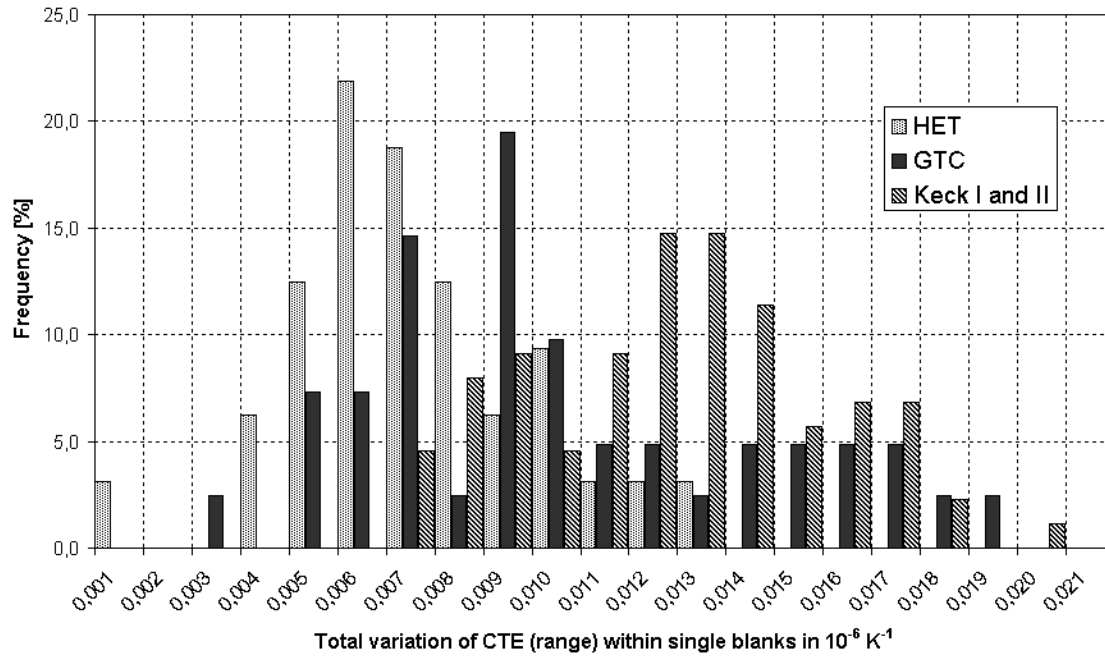


Figure 9: Frequency distribution of the total variation of CTE within single blanks

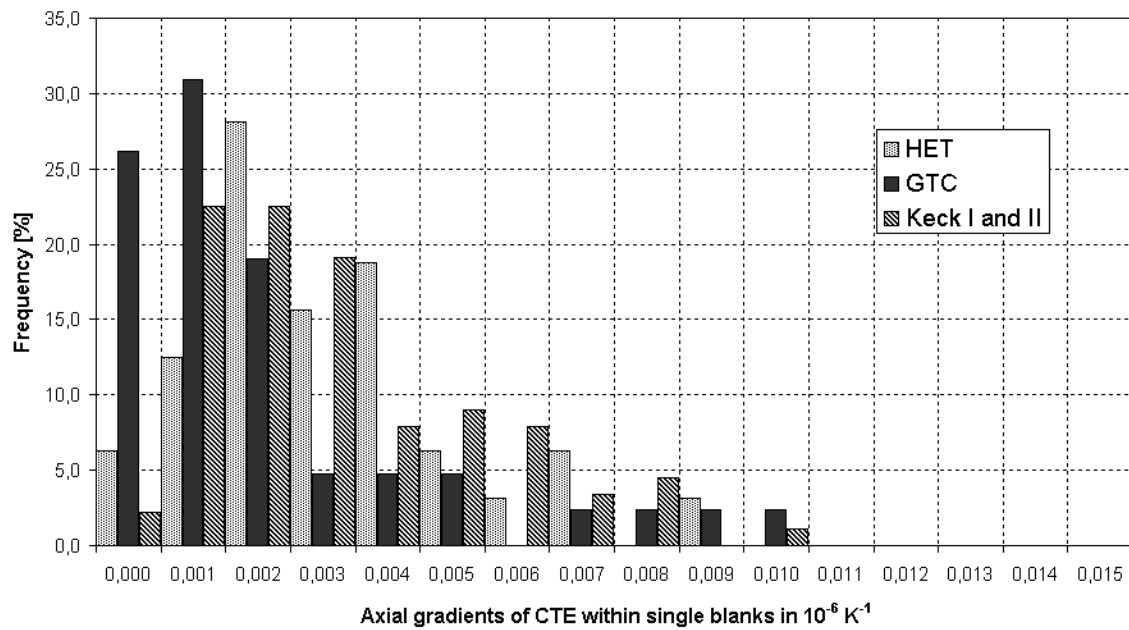


Figure 10: Frequency distribution of the axial gradients of CTE within single blanks

The low maximum value of total variation within the HET blanks can either be explained by smaller dimensions of the blank or be the low amount samples per raw casting. The VLT measurement results showed that there seems to be no clear trend between the sample position and the measured value. The values seem to be almost independent to radial or angular variations. But nevertheless it should also be pointed out that the radial distance between two samples on the VLT mirror is almost the size of the HET blanks and referring to such small length scales the overall differences between the CTE values seem to be smaller than for large mirror dimensions.

Figure 10 shows the axial gradient distribution of the mirror blanks. To estimate the absolute axial gradient for each mirror the mean CTE from the top side of the mirror was subtracted from the mean CTE of the bottom side of the mirror blanks. The thickness of the mirror blanks varies depending on the project from 56 mm (HET) to 86 mm (GTC). The figure shows one sided asymmetrical distributions. The maximum value for all mirror blanks is smaller than $0.01 \cdot 10^{-6} K^{-1}$. Most values are even below the reproducibility of measurement ($0.005 \cdot 10^{-6} K^{-1}$).

Figure 11 shows a summary of the results from the above diagrams.

	no. of values	CTE mean value range [$10^{-6} K^{-1}$]	CTE mean frequency maximum [$10^{-6} K^{-1}$]	CTE variation maximum [$10^{-6} K^{-1}$]	CTE variation frequency maximum [$10^{-6} K^{-1}$]
GTC	42	-0.049 .. +0.01 (~ 0.06)	0.000 (33 %)	0.020 (60% < 0.01)	0.009 (19%)
KECK	89	-0.099 .. +0.06 (~ 0.16)	0.010 (20 %)	0.021 (22% < 0.01)	0.013-0.014 (30%)
HET	32	-0.109 .. -0.01 (~ 0.12)	-0.070 (28 %)	0.013 (85% < 0.01)	0.006 (22%)

Figure 11: Frequency distribution of the axial gradients of CTE within single blanks

4. HOMOGENEITY OF ZERODUR

To get an overall impression of the general CTE homogeneity of ZERODUR® it is helpful to look at 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 KECK, GTC, HET and VLT mirror blanks in a single frequency diagram (figure 12).

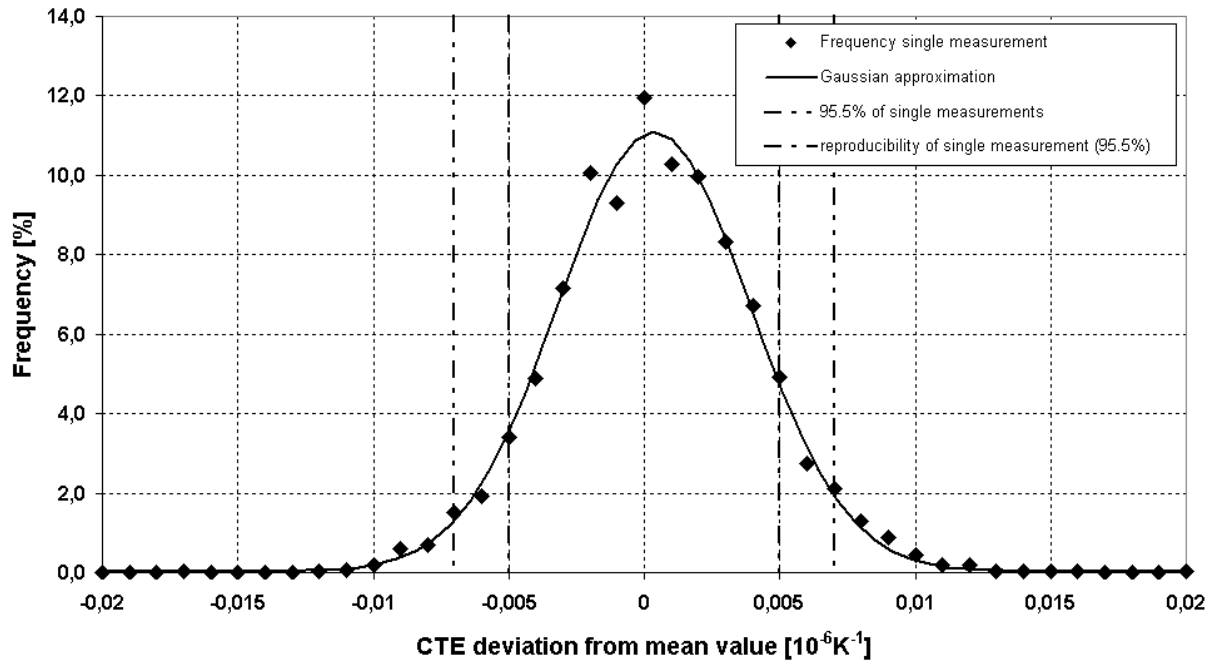


Figure 12: Homogeneity of the CTE: combined data from all HET, KECK, GTC und VLT measurements.

After fitting a Gaussian distribution to the data set it is apparent that 95,5% (2*standard deviation) of all measurements are within $\pm 0.007*10^{-6}K^{-1}$ deviation from the mean value. Additionally 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 and the real distribution reflecting the measurement homogeneity. As a first approximation the standard deviation of the real distribution without reproducibility effects can be calculated by using the following formulae: $((\text{std. dev. of the distribution})^2 - (\text{std. dev. of the measurement system})^2)^{1/2} = (0,0035^2 - 0,0025^2)^{1/2} \approx 0,0024 * 10^{-6}K^{-1}$. The 2*standard deviation is $0,005 * 10^{-6}K^{-1}$ accordingly.

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

To further improve the measurement accuracy a measurement setup was developed where the electro mechanical transformer was replaced by an interferometer system increasing the resolution by a factor of 50. A reproducibility of $\pm 0.001 * 10^{-6}K^{-1}$ was realized [6]. Currently this system is not used for standard measurements.

In order to demonstrate feasibility of the new measurement setup and to further demonstrate the homogeneity of CTE in ZERODUR[®], one block of material with $100 \times 100 \times 100 \text{ mm}^3$ dimensions has been totally cut to 100 rod like samples with a gauge length of 100 mm. With the standard dilatometer no CTE variation was observed within the repeatability of measurement. Parts of these samples were reanalyzed using the improved dilatometer. Again, no CTE variation was observed within the error of measurement of $\pm 0.001 * 10^{-6}K^{-1}$ [6]. This is in agreement with the above-mentioned observation on the HET values, the CTE variations are smaller on shorter length scales.

5. SUMMARY AND CONCLUSION

As mentioned before ZERODUR[®] manufacturing is based on established and proven methods used in the production of optical glasses. Therefore the homogeneity is similar to the homogeneity of optical glass. Even within a large ZERODUR[®] VLT mirror blank with 8 m diameter the CTE values are randomly distributed (no significant radial, angular or axial dependency observable) with a mean homogeneity in the range of $\sim 0.02 * 10^{-6}K^{-1}$ peak to valley. More than 60% of all 2 m blanks produced for the GTC project even showed homogeneity $< 0.01 * 10^{-6}K^{-1}$. The GTC project also proved that the mean CTE can be kept constant is a small range ($0.06 * 10^{-6}K^{-1}$).

95.5% of all single deviations of all projects fall within an interval of $\pm 0.007 * 10^{-6}K^{-1}$ representing a homogeneity of $< 0.015 * 10^{-6}K^{-1}$. These homogeneity values are largely influenced by the restrictions of the measurement repeatability. By improving the repeatability of the standard measurement dilatometer to about $\pm 0.001 * 10^{-6}K^{-1}$ it might be possible to achieve 2 m class ZERODUR[®] mirror blanks with a homogeneity of $0.01 * 10^{-6}K^{-1}$ for 95.5%. Therefore ZERODUR[®] seems to be a promising candidate for the up-coming extremely large telescope projects.

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