

# Glass ceramic ZERODUR®: Even closer to zero thermal expansion: a review, part 1

Peter Hartmann<sup>1</sup>,\* Ralf Jedamzik, Antoine Carré, Janina Krieg, and  
Thomas Westerhoff  
SCHOTT AG, Mainz, Germany

**Abstract.** Observational astronomy has striven for better telescopes with higher resolutions from its start. This needs ever-larger mirrors with stable high precision surfaces. The extremely low expansion glass ceramic ZERODUR® enables such mirrors with more than 50 years of significant improvements in size and quality since its development. We provide a survey of the progress achieved in the last 15 years. The narrowest coefficient of thermal expansion (CTE) tolerance is now  $\pm 7$  ppb/K. It is possible to adapt the material for lowest expansion to temperature-time courses given for special environments. At cryo temperatures, expansion is low and adaptable. Improved measurement capabilities allow for absolute CTE uncertainty of 3 ppb/K and reproducibility of 1 ppb/K ( $2\sigma$ ). The influence of ionizing radiation on the surface figure integrity is subject to new investigations. Improved measurement capabilities increase the reliability of structure designs. Some outstanding examples are given for applications of ZERODUR in astronomy and in the very important high technology industry. The progress of thermal expansion homogeneity, the mechanical strength of ZERODUR, production capabilities in melting, precision machining, light-weighting, and dimensional metrology is presented in the second part of the paper. © The Authors. Published by SPIE under a Creative Commons Attribution 4.0 Unported License. Distribution or reproduction of this work in whole or in part requires full attribution of the original publication, including its DOI. [DOI: [10.1117/1.JATIS.7.2.020901](https://doi.org/10.1117/1.JATIS.7.2.020901)]

**Keywords:** ZERODUR; extremely low expansion; glass ceramic; coefficient of thermal expansion; homogeneity; large size; monolithic; light-weighting.

Paper 20171V received Nov. 24, 2020; accepted for publication May 3, 2021; published online Jun. 3, 2021.

## 1 Introduction

The lithium-aluminum-silicate (LAS) glass ceramic ZERODUR® (Trademark of SCHOTT AG, Germany) is a material full of innovations even more than 50 years after its introduction. This is possible because its properties are not fixed completely just by its composition as is the case with crystals, for example. A glass ceramic is a material between two extremes: glass and crystal, which are free from each opposite configuration. A glass ceramic consists of a glassy phase and a crystalline phase combined. A temper process allows for the creation of nanometer-sized crystals within a pure glass, which are interspersed homogeneously throughout the total volume. With LAS glass ceramics such as ZERODUR, the nano-crystals exhibit a negative thermal expansion. A fine balanced mixture of the crystal phase and the residual glass with positive expansion will lead to a net thermal expansion, which is very close to zero. The control parameters are crystal size and number per volume. For additional detailed information, refer to the book of H. Bach, (Ed.)<sup>1</sup> on low thermal expansion glass ceramics.

Melting, casting, and precision tempering, especially of large ZERODUR items, benefit strongly from SCHOTT's long-time experience gained from optical glass production. Thus, many outstanding properties of optical glass could be transferred to ZERODUR. This is, for example, the very high controllability of the absolute value and the outstanding homogeneity of its key property. For optical glass, this is the index of refraction, and for ZERODUR, it is its low thermal expansion. Further parallels are the very high internal material quality (low bubble and inclusion, striae, and stress content) and the capability of producing large meter-sized items.

\*Address all correspondence to Peter Hartmann, [peterhartmann17@gmail.com](mailto:peterhartmann17@gmail.com); [peter1.hartmann@schott.com](mailto:peter1.hartmann@schott.com)

Only the combination of all of these aspects and the capability of achieving such quality with high reproducibility enabled the material to find very successful applications in earth-bound and space-borne astronomical telescopes and in the high-tech industry.<sup>2,3</sup>

In the last decade, further progress has led to astonishing improvements in several aspects that are of high importance for leading-edge application.

First, the key property low expansion could be restricted even closer to zero, leading to narrower tolerances for the coefficient of thermal expansion (CTE). Moreover, a new method allows for adapting specific batches to the temperature profile prevailing in specific applications. This enables a further reduction of the expansion by a factor of up to ten with respect to the lowest value previously possible. The denomination of the corresponding quality grade is ZERODUR TAILORED.

Second, the improved expansion homogeneity now lies within the single digit parts per billion per Kelvin (ppb/K) range. This holds true for short distances of centimeters and up to 4 m in diameter in the radial and in axial directions. Dilatometers with improved statistical reproducibility allow for proving this.

Third, developments in mechanical grinding allow for producing structures with rib thicknesses of only 2 mm and yielding 200 mm in height, which enables mirrors with a weight reduction close to 90%. Generating such structures from a cast item removes any doubts about integrity, which are common with structures made by joining elements.

Fourth, extensive research on the mechanical strength of ZERODUR has shown that it is suitable for applications with much higher bending stress loads than previously thought. A new approach allows for calculating the minimum lifetime of loaded structures in a deterministic and reliable way. Finally, a meta-study has proven a much higher resistance of ZERODUR against a radioactive environment, which is subject to confirmation by ongoing further research.

Due to the plethora of available information, this paper comes in two parts. The first part concentrates on the low thermal expansion under ambient conditions, within the cryo temperature range, and on the expansion measurement methods used. Additionally, it treats the resistance of ZERODUR against ionizing radiation present in space applications. The second part highlights the homogeneity of the thermal expansion, the material homogeneity, and the mechanical strength. It presents the progress in manufacturing technology that enables extremely light-weighted mirror blanks and large 4-m mirror blanks to generate surface geometry and texture ready for polishing.

## 2 Low Thermal Expansion Material—A Continuous Challenge

### 2.1 *Low Thermal Expansion Material—Essential for Technical Progress*

Thermal expansion of technical materials has been an obstacle for progress for a long time. The consequences of high thermal expansion such as deformation and warping have been the insuperable obstacles for many precision applications in the past. Internal stress caused by high thermal gradients might even lead to destruction by breakage. Hence, finding a material with low thermal expansion can lead to a breakthrough and start a technical revolution. One example is Otto Schott's (1851–1935) invention of borosilicate glass, which made it possible to illuminate cities at night with hot gas lamps by the turn of the 20th century.<sup>4</sup>

The mirror of the Mount Palomar 5-m Hale telescope was the largest astronomical in the world for a long time. It is made from borosilicate glass.<sup>5</sup> Even 8-m mirrors of present and planned telescopes still use such material.<sup>6</sup> The CTE of borosilicate glass of the DURAN®/PYREX® type is 3.3 ppm/K. This is not much more than a factor of 2 smaller than that of ordinary glass. The borosilicate glass used for present day telescopes lies within 2.2 ppm/K or somewhat lower. Fused silica provides seven times smaller CTE than the borosilicate glass, and titanium doped fused silica can get even lower. For a small temperature interval, it can get very close to zero. Due to the extremely high processing temperatures of fused silica type materials, the achievable sizes of monolithic blanks remain limited. However, even with this restriction, a wide range of application for large mirrors was possible.

## 2.2 Discovery of Low Thermal Expansion Glass Ceramic

In the 1950s, S. Donald Stookey (1915–2014) of the company, Corning USA, made a serendipitous discovery while researching light sensitive glass.<sup>7</sup> He found a new type of material: glass ceramics. This is the combination of material types usually seen as opponents: glass and crystal. Glass ceramics consist of a glassy phase that is interspersed with tiny crystals. Due to their high number per volume, the crystals occupy a large part of the total volume.<sup>8</sup> This discovery might have remained a laboratory curiosity. However, one property raised special interest in this material: its low thermal expansion. This comes from the, at first sight, strange behavior of the nanocrystals. They contract while being heated up. A closer look at the crystals, which ideally have the shape of a hexagonal double pyramid with one long and three short axes, reveals that this holds only for the long crystal axis. The short axes behave normally. However, their length change is much smaller. Hence, the net effect of the crystals while heating is contraction. With the suitable sizes and number per volume in the macroscopic view, the crystals are able to compensate for the positive thermal expansion of the surrounding glass.<sup>1</sup>

The first application for low expansion glass ceramics was and still is household glassware. Soon their use for astronomical mirrors seemed attractive.<sup>1,9</sup> Due to the initiative of the astronomer Hans Elsässer (1929–2003) from the Max-Planck-Institute for Astronomy in Heidelberg, Germany, SCHOTT developed ZERODUR in 1968 for mirror substrates to meet the requirements of the lowest possible and homogenous CTE together with a high Young's modulus.<sup>1,2,10</sup> They should be easy to be inspected and polished and coated with aluminum or silver many times.

## 2.3 Large Astronomical Mirrors from Low Expansion Glass Ceramic

Casting and ceramizing, which means controlled the growth of crystals, of small and thin laboratory samples is not very difficult. Casting of 4-m diameter and of 600-mm thickness items and tempering them for low CTE is a challenge, especially when high homogeneity is required throughout the total volume. First, producing a large extremely homogeneous melt of many tons of weight and casting it into a mold without impairing it needs outstanding skills. Second, tempering low thermal conducting materials such as glass and glass ceramics requires special measures. They react with high internal temperature gradients on rapid heating or cooling. If such gradients occur in temperature ranges that determine material properties, this will lead to inhomogeneity in these properties. For glass ceramics, the temperature ranges of crystal nuclei formation and of crystal growths are the important ones. Fortunately, these two temperature ranges have only a small overlap, thus allowing for a controlled crystal formation and subsequent growth. The company SCHOTT was well prepared for this challenge because of their long experience with casting of large optical glass items requiring subsequent precision tempering. This so-called fine annealing serves for controlling the absolute value of the refractive index as well as its homogeneity.

## 2.4 New Applications Led to Milestones in Development

The first milestone was the 4-m mirror blank in 1974 and scaling up the production size from a laboratory sample to a large mirror blank. About ten years later, another step in the development of ZERODUR manufacturing became necessary. Two astronomical projects needed not only one single high-quality blank. In fact, they needed a large series of blanks with equally high quality.

One was the x-ray space telescope CHANDRA, at that time still called AXAF. The original design asked for two times six hollow conical cylinders as components of an x-ray imaging Wolters telescope. A second set served as the spare. This required the production of 24 large high-quality blanks in total. The height was 1 m for all cylinders. The diameters varied from 0.6 to 1.3 m. All cylinders were drilled out of full material boules cast with 1.5-m diameter and 1.2-m height.<sup>11</sup> The special challenge was the precise control of the thermal processes with such extremely bulky pieces of a low thermal conducting material.

The other project was the Keck I telescope. To overcome the diameter limit for monolithic mirrors of 5 m as reached by the Hale telescope mirror, Jerry Nelson (1944–2017) of the University of Santa Cruz designed the primary mirror as a segmented mirror consisting of 36 hexagons each of ca. 2 m size.<sup>12</sup> Together with spare elements, this needed casting of 42 disks of 2-m size with high requirements on CTE, CTE homogeneity, and low residual internal stress. The key word for this progress step is reproducibility. Contrasting with science, where reproducibility confirms the validity of an experimental result by repeated outcome, in industry it means being capable of achieving the same high quality at any time for an arbitrary number of products. Usually, the development effort needed to achieve this capability is considerably higher than that for making just the prototype.

The development of extremely large high-quality ZERODUR blanks culminated with the production of the four largest directly cast monolithic mirror blanks for the ESO-VLT telescope. One 8.2 m diameter  $\times$  177 mm thick meniscus blank was made from a casting of 19 m<sup>3</sup> volume or 48 tons weight.<sup>1,13–15</sup> The overall quality of all four blanks was much better than originally specified. The 8.2-m diameter did not mark the maximum achievable size for monolithic cast blanks. The practical limit is set by the restrictions with transporting such huge items from their production site to the polishing site and finally to the telescope.

## 2.5 Future Applications—New Challenging Demands

The challenges continue. New questions came up in the early planning phase of the next-generation astronomical telescopes, the extremely large telescopes (ELTs) with 30-m primary mirror diameter and even larger. How close to zero is the ZERODUR CTE within the special temperature range in which the ELTs will operate? How homogeneous is the CTE of 1.5-m segments in the lateral and axial directions? Many hundreds of mirror segments have to be kept focused simultaneously while being in a temperature field varying in space and time. The CTE and its homogeneity have to be guaranteed in narrow limits for all segments. This means an extraordinarily high requirement on mastering material properties and on their reproduction.

Satellite missions and secondary mirrors of earth-bound telescopes ask for structures combining low weight with high stiffness, ionizing radiation resistance, and mechanical strength. Efforts building lightweight filigree structures by bonding turned out to work only for small test pieces. For larger sizes, costs and risks of imperfect bonding rise sharply. Therefore, the present way of producing such structures is grinding them from full material blanks using sophisticated machining processes. The filigree structures are subject to strong acceleration forces in many cases. They must survive rocket launches. Wobbling vibrations affect secondary mirrors of ground-based telescopes for many years of operation. Fixtures of ELT segments providing mechanical support and deformation capability for online surface figure optimization must maintain their bonding strength for the total lifetime of the telescope. This needs knowledge and reliable data about the mechanical strength of ZERODUR items. Extensive research on this topic has led to considerable improvements with respect to allowable loads and reliability in strength design. These continuing challenges have led to extraordinary progress in the last decade. The following sections will present this in detail.

## 3 Thermal Expansion

### 3.1 Coefficient of Thermal Expansion

The most important property of ZERODUR is its very low relative expansion with temperature change characterized by the extremely low value of the CTE  $\alpha$ . It is defined according to Eq. (1). The length  $l_0$  of an object at a given temperature  $T_0$  will change to the length  $l_1$  at the temperature  $T_1$ .

$$l_1(T_1) = l_0(T_0) \cdot (1 + \alpha \cdot \Delta T) \quad \text{with} \quad \Delta T = T_1 - T_0. \quad (1)$$

Equation (2) is a commonly used reformulation of Eq. (1).

$$\Delta l/l_0 = \alpha \cdot \Delta T \quad \text{with } \Delta l = l_1 - l_0. \quad (2)$$

In the following, the frequently used abbreviation CTE for the coefficient of thermal expansion and  $\alpha$  will be used as synonyms. Its physical unit is 1/Kelvin or 1/K. The values are small. Hence, for convenience, they are multiplied by the factor one million or in the case of the very low expansion materials by one billion. This is indicated by a factor in the unit such as  $10^{-6} \cdot 1/\text{K}$  or, using the abbreviation ppm for parts per million, ppm/K (parts per million per Kelvin). For the very low expansion materials, the indications are  $10^{-9} \cdot 1/\text{K}$  or ppb/K (parts per billion per Kelvin).

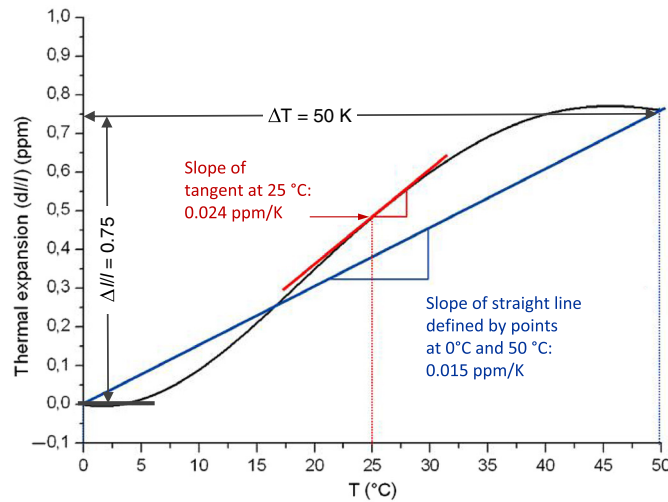
The values of  $\alpha$  for different materials used for astronomical telescope mirrors or instrument components cover the wide range of four orders of magnitude; see Table 1. The first telescope mirrors in history consisted of a bronze-like metal. Early glass mirrors were made from the optical glass type with the lowest CTE, which is N-ZK7 in its modern denomination. Table 1 shows the tremendous improvement with reducing thermal expansion achieved within the last 50 years since the discovery of the very low expansion materials. In the case of ZERODUR, it is more than the factor 600 with respect to the glass type N-ZK7.

The measurement method that SCHOTT introduced for the CTE uses a push-rod dilatometer with a highly sensitive sensor; see Sec. 4. The length change of 100-mm long samples will be recorded at 50°C related to 0°C while cycling temperature between these two values. This range leads to length changes that are large enough to provide high accuracy and reproducibility. Moreover, it covers the typical application temperature range for ZERODUR. The thermal expansion coefficient measured in this way is denominated specifically as CTE (0°C, 50°C). The expansion quality grades of SCHOTT are based on this definition. The three expansion classes 2, 1, and 0 stand for the tolerance limits of  $\pm 0.1$ ,  $\pm 0.05$ , and  $\pm 0.02$  ppm/K, respectively.

The CTE follows simply from dividing the difference of the measured relative sample lengths by the temperature interval according to Eq. (2). This is equivalent to assuming that the CTE changes linearly between the two temperatures. The method neglects the real expansion changes within the temperature interval. Figure 1 gives an example of the temperature behavior in detail. In this case, the expansion curve is steeper than the average value of 0.015 ppm/K in the temperatures range from 10°C to 35°C. For example, at 25°C it is 0.024 ppm/K.

**Table 1** CTE for selected materials frequently used for astronomical equipment applications.

Material	$\alpha$ (ppm/K)
Polycarbonate <sup>16</sup>	70
Aluminum <sup>16</sup>	22.7
Bronze <sup>16</sup>	≈ 17
Steel <sup>16</sup>	10–17
Optical Glass N-BK7 <sup>17</sup>	7.1
Optical Glass N-ZK7 <sup>17</sup>	4.5
Borosilicate Glass Duran®/Pyrex® <sup>18</sup>	3.3
Borosilicate glass E-6 <sup>19</sup>	2.9
Silicon carbide Boostec® SIC <sup>20</sup>	2.2
Fused silica <sup>21</sup>	0.51
Titanium-doped fused silica ULE® <sup>22</sup>	0 ± 0.030
ZERODUR	0 ± 0.007



**Fig. 1** ZERODUR CTE in the interval 0°C to 50°C detailed and mean value defined by the 0°C and 50°C values (blue line), example. Other shapes and slopes are possible depending on the ceramization process.

### 3.2 Coefficient of Thermal Expansion with Narrower Tolerances and for Arbitrary Temperature Intervals

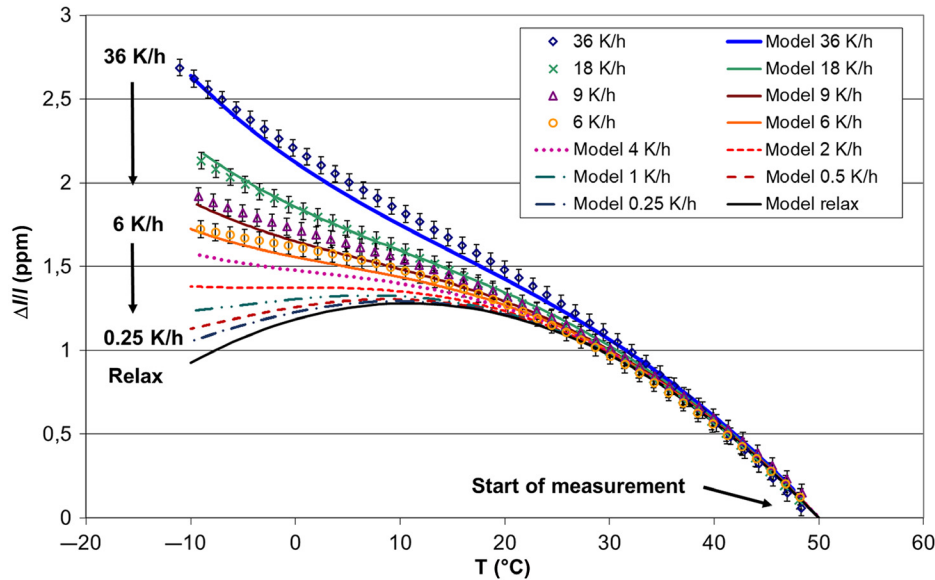
An increasing number of ambitious applications not only in astronomy but also in industry demanded a further reduction of the thermal expansion. For a long time, SCHOTT offered three different expansion classes that restricted the thermal expansion of ZERODUR within tolerance ranges of different size. The expansion class 0 being the narrowest one with tolerance limits of  $\pm 0.02$  ppm/K was not sufficient anymore.

Further decreasing the tolerance width needs controlling the absolute CTE value with higher precision. Related to manufacturing, this requires improving the mastering of the ceramization process. To monitor the progress and to enable routine quality assurance, the CTE measurement uncertainty had to be reduced considerably at the same time; see Sec. 4. The developments enabled SCHOTT in 2012 to introduce two new CTE (0°C, 50°C) tolerances for ZERODUR, the expansion class 0 SPECIAL with  $\pm 0.01$  ppm/K and EXTREME with  $\pm 0.007$  ppm/K.<sup>23</sup>

The linear approximation of the CTE for the interval between 0°C and 50°C (see Fig. 1) was sufficiently accurate for more than 30 years. That changed with the development of the extremely large telescopes of the 30-m class. The analysis of the thermal expansion behavior of many hundred mirror segments required more detailed data and focusing on a specific temperature interval of  $-5^{\circ}\text{C}$  to  $+15^{\circ}\text{C}$ . Quoting expansion in specific temperature intervals other than 0°C to 50°C needed the reconsideration of the material's behavior with temperature courses in more detail.

### 3.3 Detailed Thermal Expansion Behavior—The Structural Relaxation Effect of Low Expansion Glass Ceramics

As is generally the case with developments for achieving considerably higher accuracy, effects that were previously negligible step into the foreground. With the detailed thermal expansion behavior and the CTE measurement of low expansion glass ceramics, it is the structural relaxation effect that occurred as delayed expansion or contraction.<sup>23–27</sup> A sample cooled down from  $+50^{\circ}\text{C}$  to  $-10^{\circ}\text{C}$ , for example, does not acquire its equilibrium length for this temperature instantly but only after a certain period of contraction; see Fig. 2. The length of the delay period depends on the cooling rate and the final temperature. Similarly, while heating up, the material needs some time to expand to its final length. With higher temperatures, the delay becomes shorter. At  $40^{\circ}\text{C}$  and above, the delay vanishes.



**Fig. 2** Delayed contraction effect of ZERODUR due to structural relaxation.<sup>23,27</sup> Relative length change of a ZERODUR sample cooled down from 50°C to –10°C with different cooling rates. The data for 36 to 6 K/h are experimental results. The solid lines represent calculations based on the structural relaxation model. Kept at –10°C, the sample will contract towards the equilibrium length denoted with “relax.”

With thermal cycling of a sample, the effect is observable as a hysteresis. The hysteresis effect of ZERODUR was first observed in the 1980s. Lindig and Pannhorst<sup>28</sup> explained it as the structural relaxation of the amorphous glass phase. Similar effects in different material compositions have been well known for a long time. A prominent example is the mixed alkali effect. By the end of the 19th century, thermometer glasses showed systematic inaccuracies after calibration. Otto Schott explained this by the equal amounts of mobile sodium and potassium ions in the silica glass composition, and thus he solved the problem.<sup>29</sup>

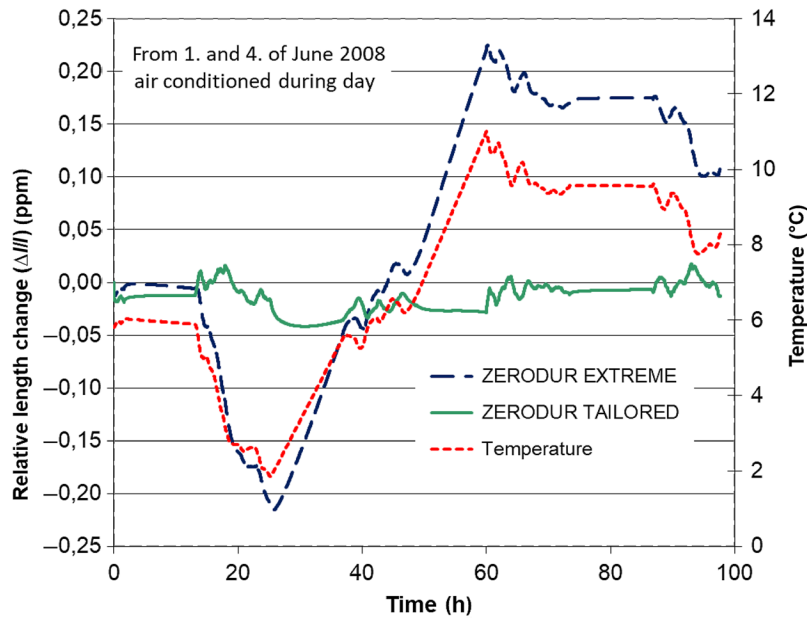
In the temperature range +40°C to +130°C, the relaxation processes in ZERODUR are fast enough so that the expansion delay is negligible. In the range –70°C to +40°C, they become slower and thus observable. The hysteresis in this temperature range is completely reversible. The effect does not impair the proper material function with most practical applications. In fact, the low thermal conductivity of glass-like materials is an advantage that prevents the material from following temperature changes instantly. Thus, the length changes of real larger-sized items will remain smaller than those derived from the behavior of the thin measurement samples as given in Fig. 2.

### 3.4 Customizing Thermal Expansion for Specific Temperature Profiles

To meet the requirement for quoting CTE data in temperature ranges other than 0°C and 50°C, SCHOTT came back to an existing physical model for the structural relaxation that also allows for calculating thermal expansion in the requested ranges. The model is based on a superposition of several processes with different parameters, relaxation times, and weight factors.<sup>27</sup> Refining this model together with determining more and better material parameters led to considerable progress reducing thermal expansion even further.<sup>23</sup> Moreover, the model allows for predicting the dimensional long-term behavior of ZERODUR with high accuracy.<sup>27</sup>

For a given temperature per time specification, the model renders the overall expansion curves for a given ZERODUR batch. By calculating such curves for different batches, the special one, which has the lowest overall peak-to-valley expansion, can be found. This optimum batch is not necessarily equal to the batch that meets the narrowest CTE (0°C, 50°C) tolerance.

Figure 3 shows the differences with an example. It shows the temperature course of four days at the summit of Cerro Armazones, the mountain in Chile selected as the site for the ESO 39-m



**Fig. 3** Relative thermal expansion of two ZERODUR batches with different grades as reaction on the temperature course on the Chilean mountain Cerro Armazones, the site of the European Extremely Large Telescope, as recorded from the first to fourth of June 2008 (dotted line with axis on the right side).<sup>30</sup> The grade ZERODUR TAILORED is adapted to the temperature course using the structural relaxation model. Its peak-to-valley expansion change is by almost a factor of ten smaller than that of the best CTE (0°C, 50°C) grade ZERODUR EXTREME.

ELT telescope. The scale for the dash-dotted temperature curve is on the right side. Two curves give the resulting expansions of different ZERODUR batches: one batch fulfilling the narrowest CTE (0°C, 50°C) tolerance and designated ZERODUR EXTREME and the other one denominated ZERODUR TAILORED and specially adapted to the temperature profile. The length change of the optimal adapted batch has a considerably lower relative expansion. The peak-to-valley expansion reduces by a factor close to ten. Unfortunately, the structural relaxation processes are too complex for an intuitive explanation of this observation.

Such tremendous improvement in expansion reduction for an even already extremely low expansion material was a fortunate finding. It is of outstanding value for all applications ultimately limited by thermal expansion. Consequently, within a short time the temperature course adapted grade ZERODUR TAILORED became a standard for important applications.

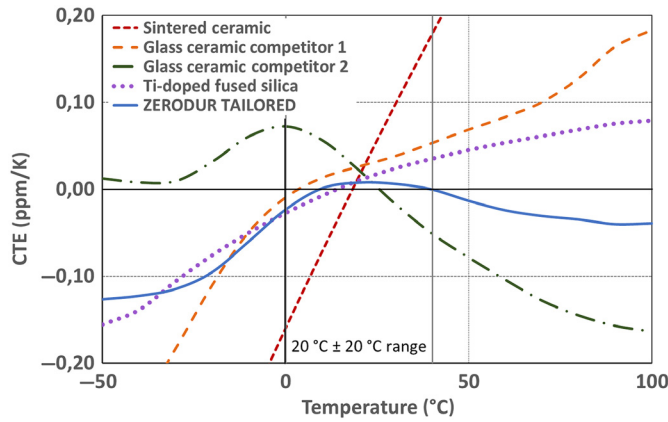
One example outside astronomy is the application of ZERODUR in microlithography. Figure 4 shows a comparison of different materials with the 22°C adapted ZERODUR TAILORED. The adapted batch has not only an extremely low expansion at 22°C but also the lowest expansion of all materials in a range as wide as 0°C to 40°C. Furthermore, the CTE change rate with temperature around 20°C is much lower than that of the other materials.

There is no general rule if either class ZERODUR class 0 EXTREME or ZERODUR TAILORED is always the best suitable grade for the most challenging applications. The choice of the adequate quality grade depends on the temperature changes in the environment.

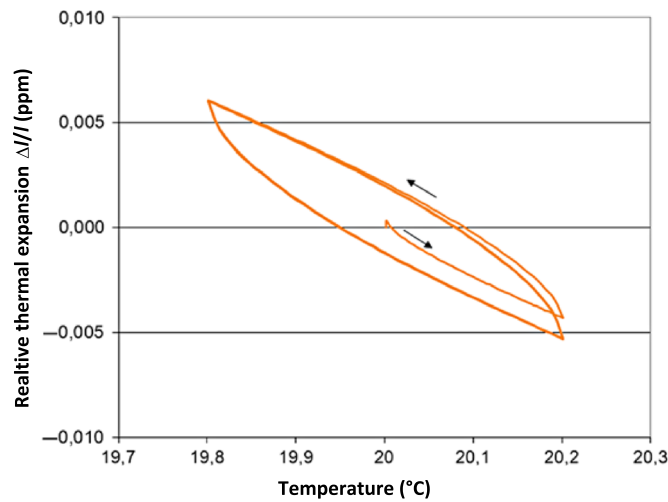
The quality grade ZERODUR TAILORED is optimum in an environment where temperature changes within a range of several degrees. However, there is one condition to be considered when choosing this grade. Obtaining the optimum batch for a given temperature course is not a selection from already produced batches. The thermal expansion adaptation is done with controlling the ceramization process. Therefore, the temperature course must be specified early enough to be taken into account during production.

For industrial applications in environments with highly stabilized temperatures, the temperature changes and change rates are very small. Figure 5 shows the relative expansion of a ZERODUR scale in a CNC measuring machine being cycled in the narrow interval of 19.8°C to 20.2°C. The temperature change rate is very slow at 0.2 K/h. A hysteresis shows up with





**Fig. 4** CTE for different low expansion materials compared with ZERODUR TAILORED optimized for operation in a 22°C temperature environment as used with wafer steppers in microlithography<sup>31</sup> and with lowest CTE in the 20°C ± 20°C range.

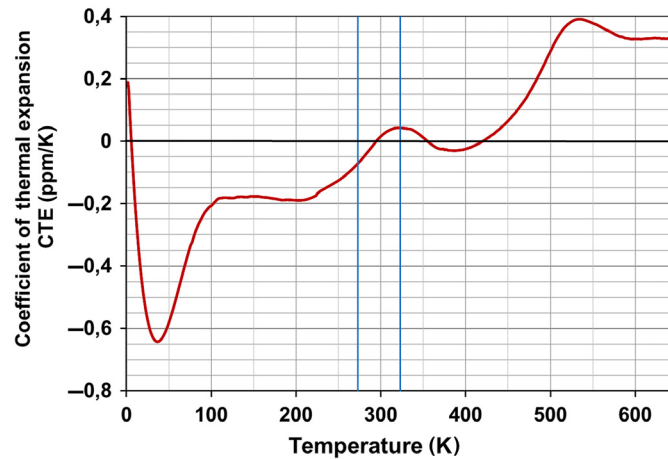


**Fig 5** Thermal expansion hysteresis of ZERODUR used as the length scale of a coordinate measuring machine operated in an air-conditioned lab room with 0.2 K/h change in the range 20°C ± 0.2°C.<sup>32</sup> The maximum difference is 0.003 ppm.

a range in the middle of the temperature cycle interval of about 0.003 ppm. During the first heating from 20°C to 20.2°C, the width is still smaller. In any case, the expansion lies fairly below any critical limits of this application. In this application, the mean CTE determines the total range of expansion change. Here, the grade ZERODUR EXTREME will be the adequate choice.

### 3.5 Application of ZERODUR at Temperatures Outside the Main Range of 0°C to 50°C

The overall CTE curve of ZERODUR covering the possible application temperature range from cryo temperatures up to 600 K shows that the CTE is generally very low (Fig. 6). Fine control of the ceramization process allows for adjusting CTE not only in the main application range around 0°C to 50°C but also in the cryo range (see below). Applications permanently above 50°C are not frequent. However, during mirror production, coating processes might need elevated temperatures. In such cases, another structural relaxation process happening in low expansion glass ceramics might come into consideration. It depends on the temperature range and the required precision in the application.



**Fig. 6** Thermal expansion coefficient of ZERODUR from cryo temperatures up to 650 K with the 0°C to 50°C range highlighted.

In the temperature range of +40°C to +130°C, the structural relaxation processes are fast enough that no delays in expansion occur. While cooling ZERODUR items down to room temperature from temperatures between +130°C and +320°C, two effects happen. The CTE and the length will be different from those before heating up, and the changes will be permanent.<sup>28,33</sup> Such effects will occur if the cooling rate in the temperature range mentioned above differs from that which SCHOTT used during the production of ZERODUR after the ceramization process. The change in CTE  $\Delta\alpha(0^\circ\text{C}, 50^\circ\text{C})$  can be calculated from the ratio of the application cooling rate  $v_{k1}$  and the original cooling rate  $v_{k0}$  according to Eq. (3).<sup>34</sup>

$$\Delta\alpha(0^\circ\text{C}, 50^\circ\text{C}) = 0.025 \cdot \log_{10}(v_{k1}/v_{k0}) \frac{\text{ppm}}{\text{K}}, \quad (3)$$

where  $v_{k0}$  is typically 3 K/h. If the same cooling rate is used, no changes will occur. Any changes that occur are fully reversible. Cooling with the adjusted rate will remove them.

These considerations hold only for items that underwent the different process temperatures throughout their total volume. If coating processes heat up only a very thin layer of the mirror, changes will remain minute. If the temperature-time course might lead to temperature gradients in the volume, especially of thick items, CTE and length changes will be distributed inhomogeneously. It is recommended to cool down such items slowly enough to avoid warping.

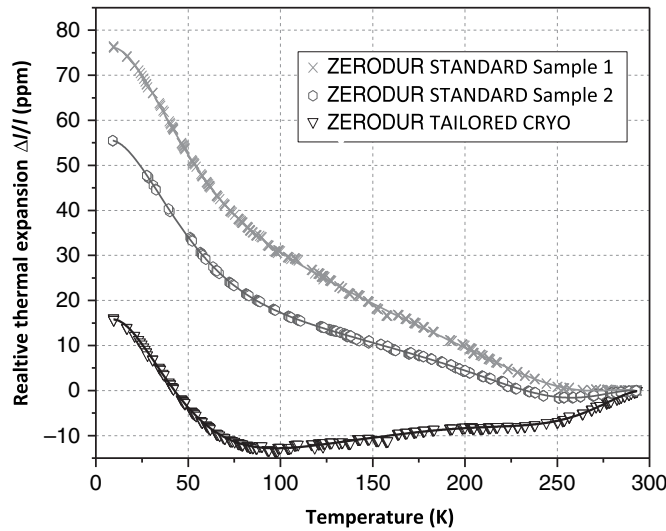
The highest temperature for applying ZERODUR is 600°C. Exceeding this temperature will cause further growth of the nano-crystals, which will change the material and its CTE irreversibly.

### 3.6 Thermal Expansion at Cryo Temperatures

In the cryo temperature range, it is also possible to reduce the thermal expansion of ZERODUR further.<sup>35</sup> Figure 7 shows the relative thermal expansion of three different ZERODUR samples from room temperature 293 K down to 10 K. Samples 1 and 2 represent batches of the standard CTE (0°C, 50°C) expansion class 2 ( $\pm 0.1$  ppm/K). The sample, ZERODUR TAILORED CRYO, has been adjusted for low-temperature applications.

The total thermal expansion of samples 1 and 2 lie at 75 and 55 ppm, when cooling them down from room temperature to 10 K. The ZERODUR TAILORED CRYO sample shows a considerably lower total thermal expansion within the range +15 to -13 ppm. In the temperature range down to 93 K, the total thermal expansion is <13 ppm.

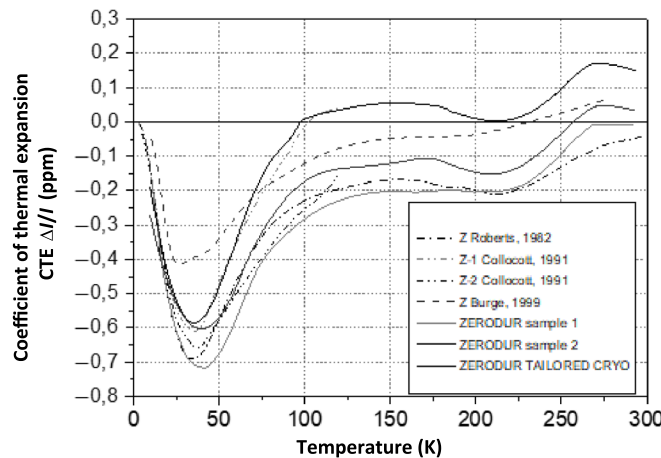
The expansion measurements were performed with the Ultra Precision Interferometer (UPI) of the PTB (Physikalisch-Technische Bundesanstalt, the national metrology institute of Germany). For more information, see Sec. 4.3.



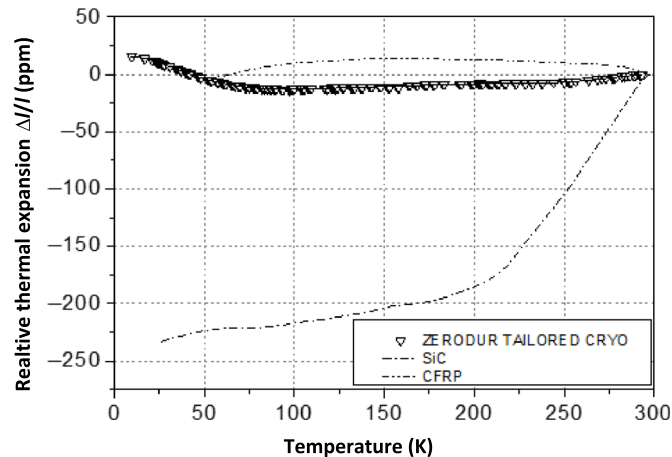
**Fig. 7** Thermal expansion measurements of three different ZERODUR samples cooled down from room temperature down to 10 K as provided by PTB.<sup>35</sup> ZERODUR TAILORED CRYO is optimized for low temperature applications. The thermal expansions  $\Delta l/l$  relate to the length at 20°C being set to zero.

Figure 8 shows the CTE curves corresponding to the expansion curves of Fig. 7 in comparison with other measurements found in the literature.<sup>36–38</sup> The general courses of the curves agree with each other. The data of the three samples of Fig. 7 demonstrate the large variation of the CTE values that are possible by adjusting the ceramization process. ZERODUR TAILORED CRYO shows a very low thermal expansion over a wide temperature range. The CTE is below 0.1 ppm/K in the temperature range from 250 K down to 70 K. In the temperature range from 90 K to 240 K, it is even below 0.05 ppm/K. Below 70 K, the thermal expansion increases to  $-0.6$  ppm/K. Below 25 K, the thermal expansion of all batches is the about same with respect to the measurement accuracy. The CTE of the displayed ZERODUR TAILORED CRYO was optimized for the temperature range 250 K to 70 K. It is possible to achieve even lower CTE for different temperature ranges.

Figure 9 shows the total thermal expansion of ZERODUR TAILORED CRYO in comparison with silicon carbide SiC and the carbon fiber reinforced polymer CFRP M55J/954-3, a unidirectional dimensionally stable material frequently used in optical telescope



**Fig. 8** CTE of the three samples of Fig. 7 (solid lines) compared with results from the literature (dashed lines).<sup>36–38</sup>



**Fig. 9** Relative thermal expansion ZERODUR TAILORED CRYO (symbols) compared with SiC and the carbon fiber reinforced polymer CFRP (M55J/954-3) (dashed lines).<sup>39</sup>

assemblies. The SiC and CFRP data are taken from Ref. 39. In contrast to SiC, the expansion of ZERODUR TAILORED CRYO fits well to that of CFRP.

The production of ZERODUR TAILORED CRYO is well controlled and highly reproducible. To obtain the best solution for a given application requirement, it is recommended to contact company SCHOTT at the earliest time possible.

## 4 Measurement of the Coefficient of Thermal Expansion

### 4.1 CTE Measurement with a Push-Rod Dilatometer

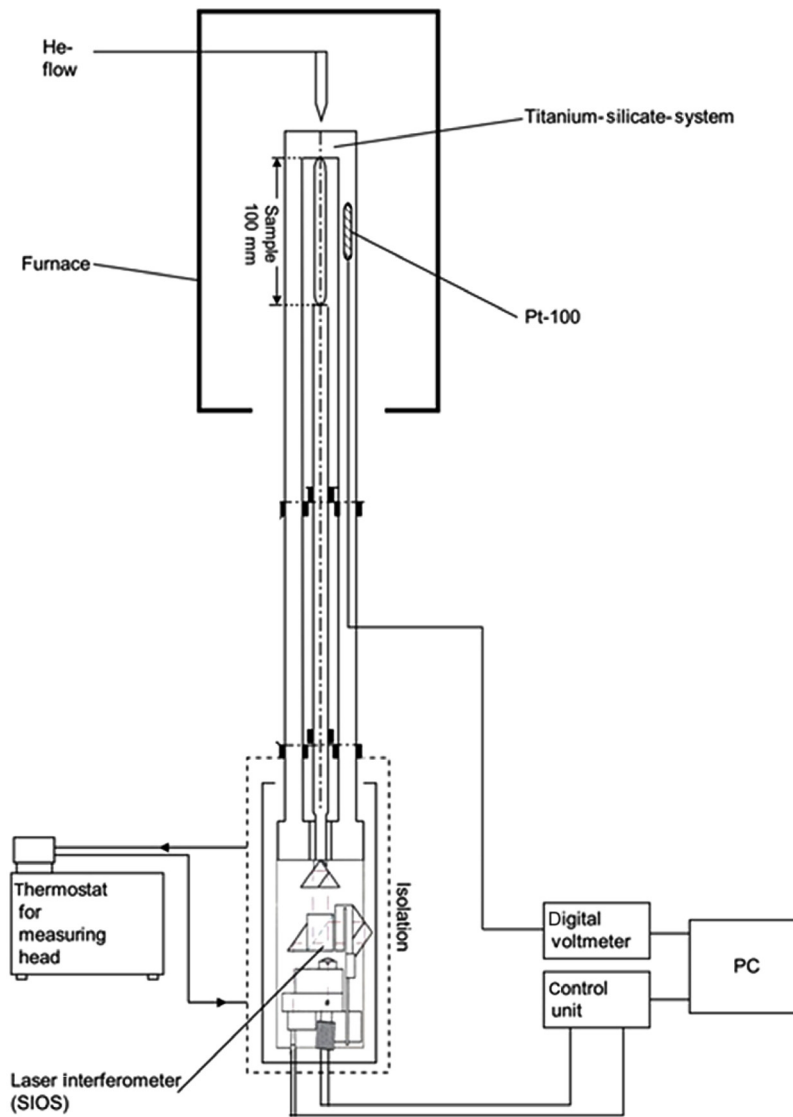
The key characteristic of ZERODUR, the CTE, is monitored closely to assure the required quality. This needs a measurement system that is fast, reliable, highly accurate, insensitive to industrial environment, and cost effective. Since commercially available systems do not meet all requirements together, SCHOTT developed its own equipment and method.<sup>40,41</sup>

Since the very beginning, the push-rod dilatometer described by Plummer and Hagy<sup>42</sup> was the method of choice. It allows for the use of simple samples without expensive and time-consuming surface preparations as, for example, the interferometric methods need. The length change of the 100-mm long sample during thermal cycling will be transferred from the thermal chamber to a highly sensitive sensor by a push-rod fixed in a fork-like titanium silicate glass frame; see Fig. 10. Reference samples calibrated by the PTB compensate for the thermal expansion of the dilatometer itself.

For a long time, the sensor was an inductive coil. Due to the increased requirements on absolute uncertainty and reproducibility, a new laser interferometric sensor was introduced in 2006.<sup>43</sup> This system is more sensitive by a factor of 50 compared with the inductive coil. However, unavoidable imperfections of the critical optical components prevented the full exploitation of the sensitivity gain for the overall measurement accuracy. Additionally, the long-term stability did not meet the expectations sufficiently. Therefore, SCHOTT continued developing the dilatometer system.

### 4.2 New Advanced Dilatometer of 2015

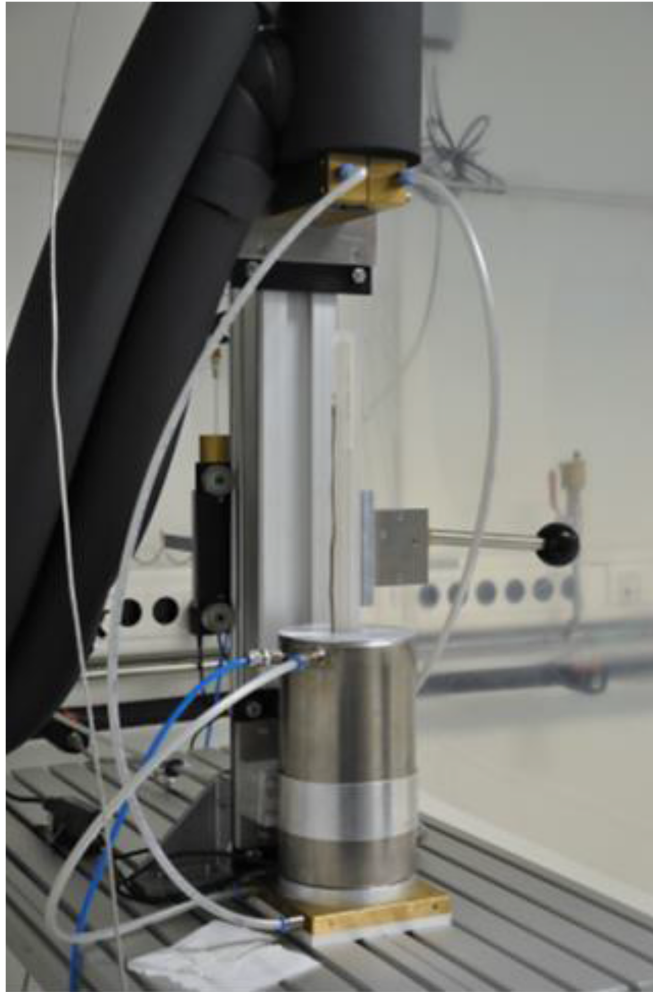
The targets of the new Advanced Dilatometer development were improving the reproducibility and reducing the absolute uncertainty.<sup>43</sup> A better long-time reproducibility allows the CTE homogeneity measurement to come closer to real variations in the material. There were strong indications that measurement statistics dominated CTE homogeneity results, thus presenting it worse than it really was; see part 2 of this article. Furthermore, the narrower CTE tolerances introduced in 2012 asked for a smaller absolute uncertainty.



**Fig. 10** Basic construction of the Improved Dilatometer setup with a laser interferometric sensor.<sup>43</sup>

The push rod dilatometer design was maintained (see Fig. 11) because it enables fast measurement cycles and short downtimes. Improvements realized in the new advanced dilatometer are

1. Use of an extremely precise incremental linear encoder providing a shift resolution of 0.031 nm, a very low noise level, and a very low drift of <math>< 1.44 \text{ nm/day}</math>.
2. Monolithic design of the push-rod containing fork without bonded parts.
3. Thermally low sensitive fixation of the push rod within the fork.
4. Redesign of the thermal chamber and the temperature monitoring locations.
5. Installation of a new cryostat for measurements up to the full temperature range from  $-50^\circ\text{C}$  to  $+100^\circ\text{C}$ . The reproducibility of the temperature profiles is better than 0.2 deg.
6. Strictly controlled environment for the complete dilatometer: temperature variation  $< \pm 0.2^\circ\text{C}$  and humidity better than  $\pm 2\%$ .
7. New titanium-silicate reference samples measured at the PTB.



**Fig. 11** The new Advanced Dilatometer from SCHOTT.<sup>43</sup>

### 4.3 Dilatometer Measurement Uncertainty

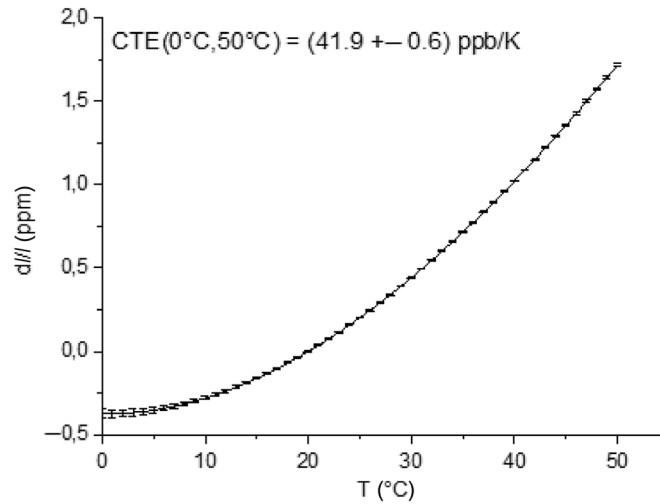
The absolute measurement uncertainty is determined by the measurement uncertainty of the titanium silicate calibration samples. Measuring them requires the utmost care. For this reason, the measurements were performed at the best available length change measurement facility, the PTB's UPI.<sup>44</sup> The samples having parallel polished faces at the ends are wrung to a polished reference plate. Two iodine-stabilized lasers at 532 and 633 nm provide the scale for their length changes. A rubidium-stabilized laser at 780 nm serves as a cross-check.

Figure 12 shows the results of a reference sample measured at the PTB in the interval between 0°C and 50°C. The length measurement uncertainties vary between  $\pm 0.003$  and  $\pm 0.06$  ppm with the highest accuracy achieved in the room temperature region. The uncertainty for the CTE (0°C, 50°C) measurement is 0.6 ppb/K (2 sigma, 95% confidence level).

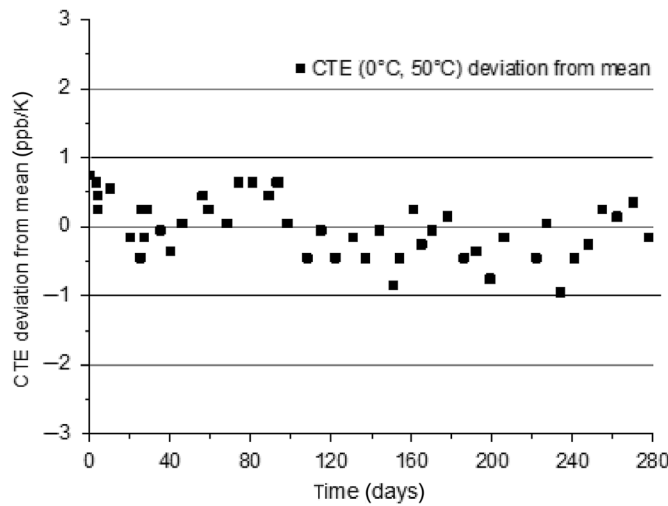
With all improvements realized in the advanced dilatometer setup, procedure, and calibration, the absolute measurement uncertainty for the CTE is now  $\pm 3$  ppb/K.

Figure 13 shows the results of the weekly reference sample CTE (0°C, 50°C) measurements of the Advanced Dilatometer for a time period of 280 days. The standard deviation of 0.5 ppb/K demonstrates the excellent long-term reproducibility achieved. The reproducibility within two to three days, which is relevant for CTE homogeneity measurements, is expected to be considerably better.

All three dilatometer types are in use depending on the required tolerances. Table 2 lists their absolute uncertainties and reproducibilities.



**Fig. 12** Calibration-sample measurement results PTB.<sup>43</sup>



**Fig. 13** Reproducibility measurement of the CTE (0°C, 50°C) of the Advanced Dilatometer shows a repeatability of less than 1 ppb/K for a 95% confidence level within a time of 280 days.

**Table 2** Progress of the CTE Metrology at SCHOTT. Uncertainty and reproducibility based on CTE (0°C, 50°C) measurements.<sup>43</sup>

Dilatometer	Measurement sensor	Uncertainty absolute (ppb/K)	Reproducibility ( $2\sigma$ ) (ppb/K)
Standard	Inductive coil	$\pm 10$	$\pm 5$
Improved (2006)	Laser interferometer	$\pm 6.2$	$\pm 1.2$
Advanced (2015)	Linear incremental encoder	$\pm 3$	$\pm 1$

## 5 Radioactive Radiation Stability

### 5.1 Ionizing Radiation Environment

Satellites orbiting around the earth are subject to ionizing irradiation. Charged particles of the solar wind and flares are trapped in the van-Allen belt. They are mainly electrons with the

maximum flux at 4.5 earth radii and energy below 2 MeV and protons with the maximum flux at 1.7 earth radii and energy below 1 MeV. When entering bulk material, these particles will lose their kinetic energy by forming mostly electron-hole pairs, which is called ionization. A marginal part of these particles will also induce atomic displacements and/or atomic reactions. Depending on the type of particle and its energy, it will penetrate into different depths and eventually be absorbed completely.

Application of ZERODUR in such an environment might raise the question of if it maintains its material properties on the microscopic scale. To be more specific, can ionizing radiations change the material's internal structure to an extent, that the precision surface figure might be warped and thus degraded in its function? Is there an irradiation triggered density increase, a compaction effect?

## 5.2 Existing Investigations Overestimate Ionizing Radiation Effects on ZERODUR

Even though ZERODUR has been used very successfully since 1978 in space missions, the question about its radioactive radiation stability comes up regularly if it is under consideration for another space application. The reason is that investigations about radiation effects with ZERODUR in the 1980s showed results that raised concerns. However, there is strong evidence that the effects found in the experiments are much too high. This comes from the limitations of the experimental methods and, in part, the very poor measurement accuracy. Furthermore, the investigation results are not satisfactorily consistent with each other.

The first experimental difficulty is irradiating samples with well-defined doses and sufficient homogeneity. The second one is measuring small density changes with very low uncertainty. This requires large samples (about  $1 \times 10^3$  g), which on the other hand are hard to be irradiated homogeneously.

Two error sources with irradiation had been overlooked in the early literature: the build-up effect caused by secondary gamma photons and the Bragg peaks with enhanced ionization density. The dose quoted in the early investigations is the dose detected in the ionization chamber located in front of or behind the irradiated samples. This dose is considerably lower than the dose absorbed within the ZERODUR bulk. Additionally, both effects introduce irradiation inhomogeneity, which prevents unique assignment of density changes to ionization densities.

It is not possible to conduct laboratory irradiation experiments over periods that are as long as the planned mission lifetimes. Furthermore, the density changes occurring with dose rates as low as in reality were too small to be measured with sufficient accuracy. Therefore, most of the experiments have been performed at radiation doses and dose rates that are higher than those in real applications by orders of magnitude. This presupposes that short-time, high dose rate measurements are equivalent to long-time, low dose rate measurements. Such equivalency has not been proven. In fact, there is reason to doubt it.

The conclusion is that extrapolation of compaction down to real radiation dose rates predicts effects that are much too high and with huge error ranges.<sup>45</sup>

One way to bypass the difficulties consists in measuring the change of camber induced by compaction on a precisely polished optical surface. This approach supposes some strong simplifying assumptions regarding the dose distribution within the material. The method and its limitation have been thoroughly discussed by Davis and Fainberg,<sup>46</sup> who showed that some misinterpretations over-evaluated the compaction by a factor of about 10.

The conclusion that radiation effects in ZERODUR are highly overestimated in the early literature is supported by the fact that there are a large number of highly successful space-borne applications of ZERODUR. Some of them have lasted for decades with no problems detected related to cosmic radiation.<sup>3</sup> The secondary mirror of the Hubble Space Telescope and the large mirror cylinders of the Chandra x-ray telescope are prominent examples operating in space since 1990 and 1999, respectively.



### 5.3 Radiation Loads—Compaction Effect

Strong ionization within glassy materials will lead to a density increase called compaction. This material shrinkage exists with almost all mirror and optical materials and is not specific to ZERODUR.<sup>47,48</sup>

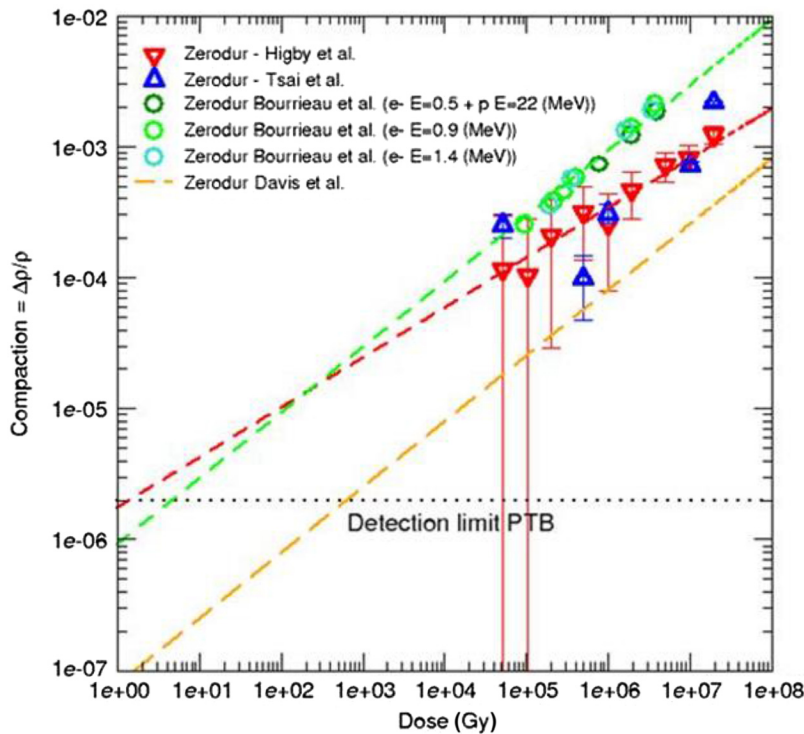
In earlier investigations, see the review by A. Carré (Ref. <sup>45</sup>), authors relate compaction  $\Delta\rho/\rho$  ( $\rho$  – density) with the absorbed dose D using a power law with the constants A and B [Eq. (4)] mostly used it in its logarithmic formulation [Eq. (5)]

$$\frac{\Delta\rho}{\rho} = A \cdot D^B, \tag{4}$$

$$\ln\left(\frac{\Delta\rho}{\rho}\right) = \ln(A) + B \cdot \ln(D). \tag{5}$$

Dose gradients gives rise to gradients of compaction. This leads to mechanical stress and distortion of highly precise surfaces, which is a concern.

Figure 14 shows the results of different compaction measurements. The absorbed dose ranges from  $10^5$  Gy to about  $10^7$  Gy. 1 Gy = 1 Gray is equivalent to 1 J energy deposited in 1 kg material. Compaction values below  $10^{-4}$  have not been measurable. Even up to the dose of  $10^6$  Gy, compaction values are uncertain by large factors. The data show large error bars—please note the logarithmic scale of the y axis—allowing for fits with very different lines. The power law description of the compaction is a very simplified approach that does not take into account the type of particles and the energy of the incident particles. The present state does not allow for deriving meaningful conclusions and thus asks for additional research.



**Fig. 14** Compaction of ZERODUR depending on the total absorbed dose of ionizing irradiation from the literature.<sup>45</sup> The practically important range for space missions  $5 \cdot 10^4$  to  $10^7$  Gy containing no data is presently subject to ongoing measurements.<sup>49</sup> Preliminary data obtained with a further improved detection limit lie close to the line extrapolated from the data of Davis and Fainberg.<sup>46</sup>

#### 5.4 Dose Rate Dependence—Recombination Effect

There are reasons to presume that the amount of compaction depends not only on the total absorbed dose but also on the dose rate. Until now, there has been no investigation on this possible influence with sufficient significance. In the cited experiments, the total doses have been absorbed in the material within a short time, which is hours or days. There is only one observation of dose rate dependency.<sup>50</sup> It used two different times for acquiring the same dose in 1 h and in 281 h. There were no detectable differences. However, this is by far shorter than the practical case of a ten-year or even longer operation.

Even though glass and glass ceramics are low electron conducting materials, thermally enhanced conductivity exists, which will lead to the recombination of electron–hole pairs thus reducing ionization. Such an effect is observable with the recovering of light transmission of irradiated optical glass. The recombination might prevent the total dose effect observed with short-time irradiation from ever being reached with long time low dose rate expositions.

#### 5.5 Radiation Load Design—Another Error Source for Material Validation

The analysis of radioactive irradiation effects on the mirror or structure material in a space mission includes calculating the expected dose. This is a complicated issue, especially with modeling the geometry and the shielding environment. Usually, the optical material will not be exposed directly to the environmental radiation except for a small solid angle. It will be shielded, for example, by the telescope's casing and metering structure. Typically, this corresponds to a 4-mm aluminium shielding ( $1 \text{ g} \cdot \text{cm}^{-2}$  equivalent mass thickness) leading to a significant decrease of the deposited dose in the optical structure by several orders of magnitude. The residual radiation will not penetrate deep into the material but will be absorbed within a few millimetres. The absorbed dose decreases exponentially with depth. Any radiation effect, if existent at all, will be restricted to a thin layer.

To keep the design calculations effort within reasonable limits, simplifications are introduced at certain stages. As a rule, each of the decisions will be conservative, which means that each time the worst case will be chosen. The accumulation of a series of conservative assumptions might lead to a tremendous overestimation of the radiation dose rate even by orders of magnitude. Combined with the uncertainties in the material reaction on irradiation, this might result in an overall inaccurate risk for applying the material that is several orders of magnitude too high. It is highly recommendable to perform a risk assessment only in the final stage to value all contributions together. This helps to avoid erroneously discarding a promising design solution.

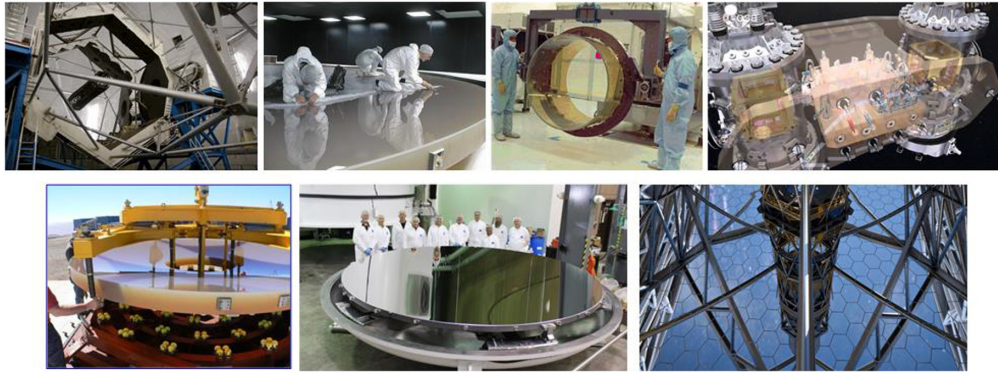
#### 5.6 Outlook

To achieve a reliable fact base, SCHOTT started a new compaction measurement campaign.<sup>49</sup> Considerable improvements are expected due to the availability of a long-time radiation source and the highly improved density measurement capability at the PTB, which allows for measuring a compaction of  $5 \cdot 10^{-7}$ . This enables data acquisition for compaction at low doses and low dose rates. It will remove the present huge uncertainties that exist in the range of practical applications coming from wide extrapolations of varying data sets. The initial results support the expectation that the compaction risks had been strongly overestimated in the radiation design analysis done in the past and explain the very successful use of ZERODUR within the last 40 years in space missions. The full report will be published soon.

### 6 ZERODUR—Proven in and Selected for Long-Term and Extremely Challenging Applications Not Only in Astronomy

#### 6.1 Long-Term Astronomical Applications of ZERODUR—in Operation and in Preparation

ZERODUR is widely used in astronomy. Prominent telescopes have operated ZERODUR mirror substrates for several decades. Examples are the earth bound segmented telescopes

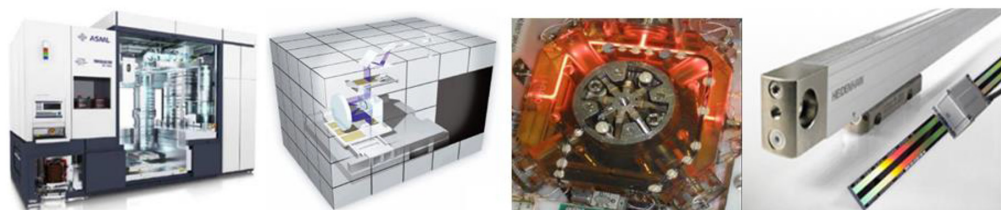


**Fig. 15** ZERODUR astronomical applications from left, upper row: KECK telescope 10-m segmented primary mirror, ESO-VLT 8.2-m monolithic primary mirror, NASA CHANDRA Wolter x-ray telescope mirror element 1.3-m diameter, ESA LISA Pathfinder optical bench and precision frame, lower row: ESO VISTA 4-m primary mirror, NSO DKIST solar telescope 4.25-m primary mirror, ESO ELT large segmented 39-m primary.

Keck I (28 years) and II (25 years), Grantecan (14 years), HET (25 years) and Lamost (13 years); the telescopes with the largest monolithic cast mirrors ESO VLT (23 years); and the space borne x-ray telescope Chandra (22 years) (see Fig. 15).<sup>2</sup> In addition, large solar telescopes chose ZERODUR as the mirror substrate, including Gregor (12 years) and Big Bear (12 years).<sup>51-53</sup> In 2020, the largest solar telescope (by far), DKIST with a 4-m diameter, saw its first light.<sup>54,55</sup> A whole set of 4-m mirrors from ZERODUR are in operation now. The oldest at the CALAR ALTO observatory has served the astronomical sciences for 37 years. A milestone within the 4 m series was the ESO-NTT telescope, established 32 years ago with considerably reduced thickness allowing for adjusting the mirror shape to the varying deformation with different azimuth angles. The next highlight is already on its way. The ESO-ELT ESO's 39-m telescope will consist of a primary mirror with 798 1.4-m segments, a 4.25-m convex secondary mirror, and a 4-m tertiary mirror.<sup>56</sup> The planned operation period is 50 years. The satellite mission LISA Pathfinder, the precursor experiment for the space-borne gravitational wave detection LISA (planned operation time 10 years<sup>57</sup>), used ZERODUR as ultra-stable optical bench<sup>58</sup> and as a mechanically strong<sup>59</sup> precision frame material.

## 6.2 Applications Outside Astronomy

For more than 25 years, ZERODUR has been a key material in many industrial high technology applications requiring the highest precision. The extremely low CTE of ZERODUR, its very low change with temperature, and its extremely high homogeneity even throughout large volumes (see Sec. 2 of this paper) enable metrology and alignment equipment to achieve perfect control of wafer positioning in microlithography; see Fig. 16. This is one of the key preconditions for the production of most advanced integrated circuits (IC) with feature sizes in the nanometer range.<sup>60</sup> In flat panel display production, large ZERODUR mirror substrates serve as key components of mask aligners of the machines projecting the patterns for the electronic wiring onto the flat



**Fig. 16** ZERODUR industrial applications from left: Microlithography precision support elements, flat panel lithography large optical elements, ring laser gyroscope helium-tight body and mirrors, linear encoder substrate material.

panels. In airplanes, ring laser gyroscopes of ZERODUR are part of the inertial navigation systems that provide information on airplane orientation and rotation in reference to the artificial horizon. An outstanding example of a ring laser gyroscope is the 4-m edge length ring-laser gyroscope in Wettzell, Germany, used for observing the earth's rotation speed.<sup>61,62</sup> Precision metrology systems, for example, three-dimensional coordinate measurement machines, use scales made of ZERODUR. Accuracy exceeding  $1:10^{-9}$  up to 1.6-m length has been achieved. A scale on a ZERODUR substrate enabled building a linear encoder with sub-nanometer positioning error in a visible tunable filter application.<sup>63</sup>

The industrial application-driven demand for ZERODUR exceeds its usage in astronomy by multiples. Even with the contracts signed in 2017 for the ESO ELT M1, M2, and M3, the industrial demand is by far larger than that of the telescope projects. This caused SCHOTT in 2019 to invest in extending the melting capacity as well as the machining and metrological capabilities, enabling the fulfillment of any future industrial or project demand.

## 7 Outlook to Part 2

The second part of this article comprises the following topics:

- i. **Homogeneity of the thermal expansion:** For precision dimensional and shape control of large items, CTE homogeneity is as important as the extremely low thermal expansion itself. The challenges for obtaining high homogeneity grow exponentially with the volume of the cast material. Process and metrology improvements allow for producing and verifying homogeneity deviations as low as 5 ppb/K throughout the total volume, even of a 4-m ZERODUR mirror blank. This value holds for long-range variations along meter distances and for short-range variations in the centimeter scale in the radial and axial directions.
- ii. **Material homogeneity:** This refers to the absence of material variations such as voids (pores, bubbles), alien material inclusions, hard micro-crystals, anisotropy, seams, flaws, layers, rings, short-range chemical variations (striae), and mechanical stress. ZERODUR is a highly isotropic, homogeneous material. Its content of bubbles, inclusions, striae, and stress is very low. Due to its transparency in the visible light, it can be thoroughly inspected, thus allowing for excluding any surprises in downstream processes.
- iii. **Mechanical strength:** ZERODUR is a brittle material with breakage behavior similar to glass. Just as with glass, its mechanical strength is considerably underestimated. Increasing demand to use the material at higher stress loads and to obtain statistically more reliable data initiated measurements with much larger numbers of specimens than in the past. The results prove the three-parameter Weibull distribution to be adequate for representing failure probability. Its threshold parameter divides tensile stress into two ranges, one with statistical behavior and one with deterministic behavior. With incorporating stress corrosion as the prevailing fatigue mechanism, the threshold model allows for calculating reliable minimum lifetimes for given constant stress loads.
- iv. **Manufacturing technology:** Advances in precision CNC grinding allow for filigree structures from ZERODUR with almost 90% weight reduction. Together with its favorable material properties and high strength, this makes ZERODUR a highly promising material for extremely light-weighted mirrors. At present, developments of the manufacturing and metrology capabilities are under way for increasing the size of such mirrors from about 1 m up to 4 m. For solid 4-m blanks, the shape manufacturing precision lies in the single digit micron range. This holds also for off-axis aspherical mirror faces. Such precision allows for polishing companies to omit all preparatory grinding processes, thus saving considerably cost and time.

## Acknowledgments

The authors thank all users of ZERODUR for their open and constructive feedback and their continuous challenging of the material's quality and properties and the company SCHOTT's production capabilities and capacities. This is the main driver behind the astonishing development the material has taken in the last two decades. We also thank the many colleagues at SCHOTT, who were the ones making it possible by courageous decisions, ingenious modeling,

metrology and engineering, outstanding craftsmanship, and maintaining close communication with the users. We thank especially Dietrich Lemke of the Max-Planck-Institute for Astronomy in Heidelberg, Germany, for initiating this paper.

## References

1. H. Bach, Ed., *Low Thermal Expansion Glass Ceramics*, Springer Science & Business Media (2013).
2. T. Döhring et al., “Forty years of ZERODUR® mirror substrates for astronomy—review and outlook,” *Proc. SPIE* **7018**, 70183B (2008).
3. T. Döhring et al., “Heritage of ZERODUR® glass ceramic for space applications,” *Proc. SPIE* **7425**, 74250L (2009).
4. J. Steiner, “Otto Schott (1851 to 1935): founder of modern glass science and glass technology,” *Glass Sci. Technol.* **74**(10), 292–302 (2001).
5. R. Florence, *The perfect machine*, Harper Collins, New York (1994).
6. J. R. P. Angel, “8 m Borosilicate honeycomb mirrors,” in *Proc. ESO*, p. 281 (1988).
7. C. R. Kurkjian and W. R. Prindle, “Perspectives on the history of glass composition,” *J. Am. Ceram. Soc.* **81**(4), 795–813 (1998).
8. S. D. Stookey, “Low expansion glass-ceramic and method of making it,” U.S. Patent Nr. 3,157,522 (1964).
9. R. C. Monnier and F. Cooke, “Fabrication of a 104-cm mirror from Cer-Vit® low expansion material,” *Appl. Opt.* **6**(8), 1437–1440 (1967).
10. P. Hartmann and H. F. Morian, “100 years of mirror blanks from SCHOTT,” *Proc. SPIE* **5382**, 331–337 (2004).
11. H. F. Morian, K. Knapp, and P. Hartmann, “Quality assurance demands and realization for thin-walled mirror blanks made of ZERODUR® for the AXAF project,” *Proc. SPIE* **1993**, 6–18 (1993).
12. T. S. Mast and J. E. Nelson, “Keck telescope primary mirror segments: fabrication and support,” in *Proc. ESO*, p. 411 (1988).
13. R. W. Mueller et al., “Manufacture of the first primary mirror blank for the Very Large Telescope (VLT),” *Proc. SPIE* **2199**, 164–176 (1994).
14. H. F. Morian et al., “Performance of the four 8.2-m ZERODUR® mirror blanks for the ESO/VLT,” *Proc. SPIE* **2871**, 405–416 (1997).
15. P. Dierickx et al., “VLT primary mirrors: mirror production and measured performance,” *Proc. SPIE* **2871**, 385–393 (1997).
16. F. Kohlrausch, V. Kose, and S. Wagner, *Praktische Physik*, B.G. Teubner, Stuttgart (1996).
17. SCHOTT, “Optical Glass Pocket catalog,” 2018, [https://www.schott.com/d/advanced\\_optics/1de0c3b6-522e-4ecd-b297-d1c10099a0c2/1.19/schott-optical-glass-pocket-catalog-february-2018-de.pdf](https://www.schott.com/d/advanced_optics/1de0c3b6-522e-4ecd-b297-d1c10099a0c2/1.19/schott-optical-glass-pocket-catalog-february-2018-de.pdf).
18. ISO 3585:1998 (en), *Borosilicate Glass 3.3 — Properties*, International Organization for Standardization, Geneva (1998).
19. S. F. Jacobs, D. Shough, and C. Connors, “Thermal expansion uniformity of materials for large telescope mirrors,” *Appl. Opt.* **23**, 4237–4244 (1984).
20. M. Bougoin and J. Lavenac, “From HERSCHEL to GAIA, 3-meter class SiC space optics,” *Proc. SPIE* **8126**, 81260V (2011).
21. Heraeus, “Fused silica,” Data sheet Corning Company Heraeus, 2019, [https://www.heraeus.com/en/hqs/fused\\_silica\\_quartz\\_knowledge\\_base/properties/properties.aspx](https://www.heraeus.com/en/hqs/fused_silica_quartz_knowledge_base/properties/properties.aspx).
22. Corning, “ULE® corning code 7972 ultra low expansion glass,” Data sheet, 2008, <https://www.corning.com/media/worldwide/csm/documents/D20FD2EA-7264-43DD-B544-E1CA042B486A.pdf>.
23. R. Jedamzik, C. Kunisch, and T. Westerhoff, “ZERODUR®: progress in CTE characterization,” *Proc. SPIE* **8860**, 88600P (2013).
24. J. J. Shaffer and H. E. Bennett, “Effect of thermal cycling on dimensional stability of ZERODUR® and ULE®,” *Appl. Opt.* **23**(17), 2852–2853 (1984).
25. S. F. Jacobs and D. Bass, “Improved dimensional stability of Corning 9600 and SCHOTT ZERODUR® glass ceramics,” *Appl. Opt.* **28**(19), 4045–4046 (1989).

26. S. F. Jacobs et al., “Surface figure changes due to thermal cycling hysteresis,” *Appl. Opt.* **26**(20), 4438–4442 (1987).
27. R. Jedamzik, T. Johansson, and T. Westerhoff, “Modeling of the thermal expansion behaviour of ZERODUR® at arbitrary temperature profiles,” *Proc. SPIE* **7739**, 77390I (2010).
28. O. Lindig and W. Pannhorst, “Thermal expansion and length stability of ZERODUR® in dependence on temperature and time,” *Appl. Opt.* **24**(20), 3330–3334 (1985).
29. W. Vogel, *Glass Chemistry*, Springer Science & Business Media (2012).
30. R. Jedamzik, C. Kunisch, and T. Westerhoff, “ZERODUR thermo-mechanical modelling and advanced dilatometry for the ELT generation,” *Proc. SPIE* **9912**, 99120J (2016).
31. P. Hartmann, R. Jedamzik, and T. Westerhoff, “Zero-expansion glass ceramic ZERODUR®: recent developments reveal high potential,” *Proc. SPIE* **8450**, 845022 (2012).
32. R. Jedamzik et al., “Progress on glass ceramic ZERODUR® enabling nanometer precision,” *Proc. SPIE* **9780**, 97801K (2016).
33. T. Westerhoff, R. Jedamzik, and P. Hartmann, “Zero expansion glass ceramic ZERODUR® roadmap for advanced lithography,” *Proc. SPIE* **8683**, 86832H (2013).
34. R. Jedamzik and T. Westerhoff, “Advices for the use of ZERODUR® at higher temperatures,” *Proc. SPIE* **10706**, 1070634 (2018).
35. R. Jedamzik and T. Westerhoff, “ZERODUR® TAILORED for cryogenic application,” *Proc. SPIE* **9151**, 91512P (2014).
36. R. B. Roberts, R. J. Tainish, and G. K. White, “Thermal properties of ZERODUR® at low temperature,” *Cryogenics* **22**, 566 (1982).
37. S. J. Collocott and G. K. White, “Heat capacity and thermal expansion of ZERODUR® and ZERODUR® M at low temperatures,” *Cryogenics* **31**, 102 (1992).
38. J. Burge, T. Pepper, and S. Jacobs, “Thermal expansion of borosilicate glass, ZERODUR®, ZERODUR M. and unceramized ZERODUR® at low temperatures,” *Appl. Opt.* **38**, 7161 (1999).
39. A. Abusafieh et al., “Dimensional stability of CFRP composites for space based reflectors,” *Proc. SPIE* **4444**, 1–8 (2001).
40. R. Muller et al., “Ultraprecision dilatometer system for thermal expansion measurements on low expansion glasses,” in *Thermal Conductivity*, P. S. Gaal and D. E. Apostolescu, Eds., pp. 388–392, Technomic Publishing Company, Lancaster, Pennsylvania (1999).
41. R. Jedamzik et al. “CTE characterization of ZERODUR® for the ELT century,” *Proc. SPIE* **7425**, 742504 (2009).
42. W. A. Plummer and H. E. Hagy, “Precision thermal expansion measurements on low expansion optical materials,” *Appl. Opt.* **7**(5), 825–831 (1968).
43. R. Jedamzik et al., “Next generation dilatometer for highest accuracy thermal expansion measurement of ZERODUR®,” *Proc. SPIE* **9574**, 95740O (2015).
44. R. Schödel et al., “A new ultra precision interferometer for absolute length measurements down to cryogenic temperatures,” *Meas. Sci. Technol.* **23**(9), 094004 (2012).
45. A. Carré et al., “Review of space radiation interaction with ZERODUR®,” *Proc. SPIE* **10401**, 104010M (2017).
46. M. J. Davis and J. Fainberg, “Compaction effects of radiation on ZERODUR®,” *Proc. SPIE* **5179**, 38–49 (2003).
47. M. Rajaram, T. E. Tsai, and E. J. Friebele, “Radiation-induced surface deformation in low-thermal-expansion glasses and glass-ceramics,” *Adv. Ceram. Mater.* **3**(6), 598 (1988).
48. P. L. Higby et al. “Radiation effects on the physical properties of low-expansion-coefficient glasses and ceramics,” *J. Am. Ceram. Soc.* **71**(9), 796 (1988).
49. A. Carré, T. Westerhoff, and T. Hull, “Impact of ionizing radiations on ZERODUR®,” *Proc. SPIE* **10698**, 106981S (2018).
50. D. Doyle et al., “The Effect of electron irradiations on the radius of curvature of a ZERODUR® mirror,” *Proc. SPIE* **2775**, 166–188.
51. T. Döhring, R. Jedamzik, and P. Hartmann, “Mirrors for solar telescopes made from ZERODUR® glass ceramic,” *Proc. SPIE* **6689**, 66890X (2007).
52. T. Döhring, R. Jedamzik, and P. Hartmann, “ZERODUR® mirror substrates for solar telescopes,” in *Modern Solar Facilities-Adv. Solar Sci.*, Universitätsverlag, Göttingen, p. 77 (2007).

53. T. Westerhoff et al., “Manufacturing of the ZERODUR® 1.5-m primary mirror for the solar telescope GREGOR as preparation of light weighting of blanks up to 4-m diameter,” *Proc. SPIE* **7739**, 77390M (2010).
54. R. Jedamzik, T. Werner, and T. Westerhoff, “Production of the 4.26 m ZERODUR® mirror blank for the Advanced Technology Solar telescope (ATST),” *Proc. SPIE* **9151**, 915131 (2014).
55. M. Warner et al., “Construction update of the Daniel K. Inouye Solar Telescope project,” *Proc. SPIE* **10700**, 107000V (2018).
56. M. Cayrel et al., “ELT optomechanics: construction status,” *Proc. SPIE* **10700**, 1070018 (2018).
57. E. D. Fitzsimons et al., “Elisa technology consolidation study overview,” *Proc. SPIE* **10563**, 105632K (2017).
58. D. I. Robertson et al., “Construction and testing of the optical bench for LISA pathfinder,” *Class. Quantum Gravity* **30**, 085006 (2013).
59. P. Hartmann, “Minimum lifetime of ZERODUR® structures based on the breakage stress threshold model: a review,” *Opt. Eng.* **58**(2), 020902 (2019).
60. T. Westerhoff and T. Werner, “Advances in ZERODUR® manufacturing for space and ground based telescopes,” *Proc. SPIE* **10706**, 107060Q (2018).
61. K. U. Schreiber and J.-P. R. Wells, “Large ring lasers for rotation sensing,” *Rev. of Sci. Instrum.* **84**(4), 041101 (2013).
62. R. B. Hurst et al., “High-accuracy absolute rotation rate measurements with a large ring laser gyro: establishing the scale factor,” *Appl. Opt.* **56**(4), 1124–1130 (2017).
63. C. Halbgewachs et al., “Qualification of HEIDENHAIN linear encoders for picometer resolution metrology in VTF Etalons,” *Proc. SPIE* **9908**, 99084H (2016).

**Peter Hartmann** received his doctorate degree in physics from the University of Mainz, Germany, in 1984. From 1985–2018, he has held different positions at Advanced Optics of SCHOTT AG, Mainz. He served on the SPIE Board of Directors in 2011–2013, on the Board of Trustees of the Max-Planck-Institute for Astronomy, Germany, and as convener of ISO and DIN working groups. He is a fellow of SPIE and a senior member of OSA.

**Ralf Jedamzik** received a diploma degree in physics from the University Düsseldorf and a doctorate in material science from the Technical University Darmstadt. In 1999, he joined SCHOTT as quality technology expert. Since 2013, he has been the principal scientist and leader of ZERODUR® and optical glass development projects. As application manager, he works as an international technical consultant for numerous industrial projects. He has authored more than 50 papers and is a senior member of SPIE.

**Antoine Carré** has graduated as a chemical engineer and theoretical chemical physicist. He obtained an international PhD in theoretical chemistry related to the atomistic description of glass material. Since 2010, he has worked in the development department of the optical business unit of the glass manufacturer Schott. His main focus areas are process optimization, support to production, and material properties investigation under extreme conditions.

**Janina Krieg** received her master’s degree in physics from the University of Stuttgart, Germany, and her doctorate degree in material science from the Technical University of Darmstadt, Germany, in 2017. In the same year, she joined the Advanced Optics Unit of SCHOTT AG, Mainz. She is the responsible product manager for ZERODUR® glass ceramics for aerospace applications.

**Thomas Westerhoff** received his doctorate degree in physics from the University of Mainz. He joined the Optics Business at SCHOTT as product manager in 1996. He was promoted to director of sales and marketing SCHOTT Lithotec in 1999. In 2007, he took responsibility for the SCHOTT product group ZERODUR®. In 2018, he was promoted to the position vice president of strategic business field ZERODUR® responsible for Astronomy & Space, IC and FPD Lithography, and Industrial Application.