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TIE-43: Optical Properties of ZERODUR®

0. Introduction

ZERODUR[®] is a glass-ceramic material exhibiting a very low coefficient of thermal expansion. The material is therefore used as a mirror substrate for astronomical telescopes or as a mechanical component when geometrical dimensions and shapes are required to be extremely stable against temperature changes. In such applications the optical properties of ZERODUR[®] are of negligible importance [1].

Nevertheless the trend to build larger telescopes like the planned ELT (extremely large telescope) projects also drives the need for large optical glass lenses and prisms [2]. Therefore the question sometimes arises if large ZERODUR[®] blanks can also be delivered in optical quality grade.

ZERODUR[®] is produced with methods similar to those used in modern optical glass technology, whereby suitable raw materials are molten, refined, homogenized and finally hot formed. Melting and machining methods are fully developed to match dimensions of a few millimeter up to several meters. The special measures for homogeneity optimization taken during ZERODUR[®] production are reflected by the excellent CTE homogeneity and internal quality as well as by the optical homogeneity in transmission of the material. With additional production effort involving an extensive selection it is possible to fulfil standard tolerances as given by the optical glass catalog [3] in geometries up to 300 mm in diameter and larger. The advantage of ZERODUR[®] in comparison to other optical glasses is that the material can be produced in very large dimensions and has a very low thermal expansion coefficient. Figure 1 shows a 1.33 m testing lens for the secondary mirror of GRANTECAN. In figure 7 a 1.5 m diameter ZERODUR[®] blank in optical standard grade is displayed.



Figure 1: 1.33 m diameter testing lens for the secondary mirror of GRANTECAN



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The following chapters give a summary of the optical properties of ZERODUR.

1. Refractive Index

The following table 1 displays the catalog refractive index values of ZERODUR[®] for different wavelengths. The refractive index of ZERODUR in general will only be measured on customer request. The applicable tolerances for the refractive index at the d-line is in the range of ± 0.0005 (equivalent to step 3 according to the optical glass catalog [3]). Tighter tolerances can be fulfilled on request implying a costy selection process. Unlike optical glass the refractive index of ZERODUR[®] can not be adjusted to tighter tolerances by a subsequent annealing process.

The standard measurement accuracy for the refractive index is $\pm 3*10^{-5}$ and covers the g- to C-line. Using a special spectrometer a refractive index measurement accuracy of $\pm 0.4*10^{-5}$ covering a wavelength range from 365 nm to 2325 nm can be achieved on request [4].

wavelength [µm]	Fraunhofer designation	refractive index measured	refractive index from Sellmeier dispersion
0,3650146	i		1,56685
0,4046561	h	1,55894	1,55894
0,4358343	g	1,55444	1,55444
0,45799914		1,54966	1,54966
0,4859975			1,54912
0,4861327	f	1,54911	1,54911
0,546074	е	1,54468	1,54468
0,5875618	d	1,54238	1,54238
0,5892938	D		1,54229
0,6328			1,54035
0,6438469	C'	1,53991	1,53991
0,6561			1,53945
0,6562725	С	1,53944	1,53944
0,7065188	r	1,53777	1,53777
0,85211	S	1,53422	1,53422
1,01398	t	1,53145	1,53145
1,06			1,53077
1,12864		1,52981	1,52981
1,395055		1,52639	1,52639
1,529582		1,52469	1,52469
1,81307		1,52092	1,52092
1,97009		1,51866	1,51866
2,24929		1,51423	1,51423
2.32542		1.51292	1.51292

Table 1: Refractive index of the ZERODUR catalog melt at different wavelengths (typical values, not guaranteed)



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The third column of table 1 lists the measured refractive index of the representative melt. These results were used to estimate the constants of the Sellmeier dispersion equation (formula 1 [4]) that can be determined to calculate the refractive index at wavelengths that are not directly accessible by the refractive index measurements.

$$\mathbf{n}(\lambda)^{2} - 1 = \frac{\mathbf{B}_{1} \cdot \lambda^{2}}{(\lambda^{2} - \mathbf{C}_{1})} + \frac{\mathbf{B}_{2} \cdot \lambda^{2}}{(\lambda^{2} - \mathbf{C}_{2})} + \frac{\mathbf{B}_{3} \cdot \lambda^{2}}{(\lambda^{2} - \mathbf{C}_{3})}$$
(1)

The coefficients of the representative melt are displayed in table 2

	1	2	3
B _x	1,3182408	2,44E-02	1,08915181
C _x	8,79E-03	6,09E-02	1,10E+02

Table 2: Sellmeier coefficients of the ZERODUR® catalog melt (typical, not guaranteed)

The fourth column of table 1 shows the results of the refractive indices calculated using the Sellmeier dispersion formulae.

2. Internal Transmittance

The catalog reference transmittance values for ZERODUR[®] are given in table 3. Due to laws of economics slight variations in the purity of the raw materials must be taken into account. Therefore also the internal transmittance (transmittance curve corrected for reflection losses) might vary from batch to batch. Depending on the specified value the material has to be selected in advance. Therefore transmission specifications can only be treated on request.

Figure 2 shows an internal transmittance curve of ZERODUR[®] batch measured with a wavelength resolution of 1 nm. Additionally the catalog reference values are displayed. It can be seen that the transmittance values of the measured batch are slightly higher compared to the catalog values. It can also be seen that the transmittance curve is not linear between the reference wavelengths but influenced by several absorption bands (similar to optical glass [5]). The sample melt displayed is slightly better than the catalog reference melt, reflecting normal variations from batch to batch.

Compared to other optical glasses ZERODUR[®] has an improved transmittance at wavelength >2000 nm. Figure 3 displays the transmittance of a 0.6 mm thick ZERODUR[®] at wavelengths >2500 nm. It can be clearly seen that there is a residual transmittance between 3000 nm and 4000 nm which will not be recognizable at larger thicknesses of the material.



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	internal	internal	internal		
wavelength	transmittance	transmittance	transmittance		
[nm]	at 5 mm thickness	at 10 mm thickness	at 25 mm thickness		
2500	0,859	0,737	0,467		
2325	0,961	0,924	0,820		
1970	0,971	0,943	0,864		
1530	0,976	0,952	0,884		
1060	0,981	0,962	0,907		
800	0,984	0,969	0,924		
700	0,977	0,955	0,890		
680	0,974	0,949	0,877		
660	0,971	0,942	0,861		
640	0,966	0,934	0,842		
620	0,962	0,925	0,823		
600	0,955	0,913	0,796		
580	0,948	0,899	0,766		
560	0,940	0,884	0,734		
546	0,933	0,870	0,706		
540	0,929	0,863	0,692		
520	0,916	0,839	0,644		
500	0,901	0,812	0,593		
480	0,881	0,776	0,530		
460	0,856	0,734	0,461		
440	0,825	0,681	0,382		
436	0,818	0,669	0,367		
420	0,781	0,611	0,291		
405	0,712	0,506	0,182		
400	0,650	0,422	0,116		
390	0,370	0,137	0,007		
380	0,076	0,006	0,000		

Table 3: Typical internal transmittance values of ZERODUR[®] at 5 mm, 10 mm and 25 mm thickness.



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Figure 2: Internal Transmittance curve of a ZERODUR[®] sample at 25 mm thickness compared to the catalog reference values.



Figure 3: Transmittance curve of ZERODUR[®] at 0.6 mm thickness.



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2.1 Rayleigh Scattering

The reason for the low transmittance of ZERODUR[®] in the visible and near UV spectral range is the Rayleigh scattering due to the crystal phase within the material. This scattering behavior is wavelength depended. Table 4 displays the fraction of light scattered in 90° direction per path length of the observed primary ray as a function of the selected wavelength [8].

wavelength [nm]	R90 [10 ⁻³ /cm]
404.7	23
435.8	16
546.1	6
578.1	5

 Table 4: Rayleigh-scattering of ZERODUR[®] at different wavelength [8].

The strong decline of the scattering with increasing wavelength leads to high transmittance of ZERODUR[®] in the far red and near infrared. This has been exploited for the internal quality inspection of the huge and bulky cylindrical boules for the AXAF/CHANDRA Projects (figure 4). Even today this method is frequently used for the inspection of up to 1 m thick ZERODUR[®] boules.



Figure 4: Focal scanning of large thickness ZERODUR[®] boules with an IR sensitive CCD camera (for AXAF/ Chandra).



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3. Refractive Index Homogeneity

One of the most important properties of optical glass is the excellent spatial homogeneity of the refractive index of the material. In general one can distinguish between the global or long-range homogeneity of refractive index in the material and short-range deviations from glass homogeneity. Striae are spatially short-range variations of the homogeneity in a glass, typical extending over a distance of about 0,1 mm up to 2 mm [7]. Whereas the global homogeneity of refractive index denotes long range variations extending from the cm range to the full cross section of the optical element [6].

3.1 Global Refractive Index Homogeneity

The availability of optical glasses with increased requirements for refractive index homogeneity comprises 5 classes in accordance with ISO standard 10110 part 4 [6]. The SCHOTT homogeneity grade H1 to H5 for single parts comprises ISO grades 1 to 5. Table 5 gives an overview of the homogeneity grades.

class	H1	H2	H3	H4	H5
maximum variation of refractive index	± 2*10 ⁻⁵	± 5*10 ⁻⁶	± 2*10 ⁻⁶	± 1*10 ⁻⁶	± 5*10 ⁻⁷

Table 5: Refractive index homogeneity classes of optical glasses according to the SCHOTT pocket catalogue

Figure 5 shows the homogeneity map of a 250 mm diameter ZERODUR blank. The homogeneity is within H3 class. In smaller areas of the center of the blank the homogeneity in general is much higher. The homogeneity map was derived from a measurement using a Zeiss Direct 100 interferometer with a maximum aperture of 500 mm [6].

Refractive index homogeneity up to H3 quality could be achieved on dimensions up to 350 mm, H4 quality on dimensions up to 250 mm. The achievable global refractive index homogeneity quality strongly depends on the size of the part. Nevertheless, special production measures including an intensive selection process are necessary, therefore refractive index homogeneity specifications are treated on special request only.



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Figure 5: Homogeneity map of a 250 mm diameter ZERODUR[®] disc

3.2 Striae

Striae in optical glass are inspected and characterized using the shadowgraph method [7]. SCHOTT standard striae quality for optical glass fulfills ISO 10110 part 5, class 5 exhibiting a wavefront deformation of 30 nm maximum.

In contrast to optical glass, striae in ZERODUR[®] are evaluated according to the mechanical stress they exhibit to the surrounding material by measuring the stress birefringence. These values can not be compared to the results from the shadowgraph measurement that is used to characterize the striae quality in optical glass.

Nevertheless the striae inspection by stress birefringence measurement is a necessary tool for the preselection of ZERODUR[®] material for optical application. After preselection of material with a low amount of stress birefringence caused by striae the ZERODUR[®] will be polished on both surfaces for the optical stirae testing using the shadowgraph method.

Therefore on special request and with additional effort in stress birefringence preselection and shadowgraph qualification (including polishing of the blanks) it is possible, depending on the geometry, to select ZERODUR[®] material with optical striae quality sufficient to fulfill ISO 10110 part 4 classes 1 to 5 !



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The following figure 6 shows a shadowgraph of a ZERODUR blank. The shadowgraph measurement is a very sensitive method to detect striae in optical glasses. Even striae wavefront deviations as faint as 10 nm can be detected using this method.



Figure 6: Shadowgraph of a specially selected and produced 250 mm diameter ZERODUR[®] blank. No striae above 10 nm wavefront deviation are visible in this setup.

As indicated before, the achievable striae quality also strongly depends on the size of the blank and the selected part of a blank. The following picture shows a 1.5 m diameter ZERODUR[®] blank. This blank has been selected out of a number of blanks by starting with a measurement of the stress birefringence caused by striae. After this preselection the blanks with the lowest stress birefringence caused by striae have been polished for optical striae testing to finally select the displayed blank for the application. Within this blank optical strandard grade striae quality could be achieved. Additionally this blank also contains a very low amount of bubbles and inclusions.



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Figure 7: 1.53 m diameter and 176 mm thick, 819 kg heavy ZERODUR[®] blank with optical standard grade striae quality and excellent bubbles and inclusion quality.

4. Temperature Coefficients of Refractive Index

Even if ZERODUR has a very low thermal expansion coefficient the temperature has a recognizable influence on the refractive index. The dependence of the refractive index on temperature is as high as that of those optical glasses that are most sensitive in this respect. The change of refractive index with temperature depends on the wavelength λ , the temperature T and the air pressure p. There are two possibilities to express these relations. For the relative temperature coefficient $\Delta n_{relativ}/\Delta T$, the material and the surrounding air have the same temperature. The information pertains to air pressure p=0.10133 MPA.

The absolute temperature coefficient $\Delta n_{absolut}/\Delta T$ applies to vacuum. Both coefficients are listed for different temperature ranges and wavelengths in table. More detailed information on how to calculate these parameters can be found in [4].



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emperature		Aprelat	./ AT [10-6/KJ			Anaba	TA/ tut	[10 ⁻⁶ /k]	
°۲	C'	d		F'	0	C	d	ρ	F'	0
100/ 80	12.2	12 /	12.5	12.9	12.2	96	9.7	0.0	01	9
-100/-80	12.2	12.4	12.5	12.0	12.4	0.0	0.7	0.0	10.0	10.2
-60/-40	12.4	12.0	13.0	13.4	13.8	10.3	10.4	10.5	10.0	11.2
-40/-20	13.1	13.3	13.4	13.8	14.2	11.0	11.2	11.3	11.7	12.1
-20/0	13.5	13.7	13.9	14.3	14.7	11.8	11.9	12.1	12.5	12.9
0/20	14.0	14.1	14.3	14.7	15.2	12.4	12.6	12.8	13.2	13.6
20/40	14.4	14.6	14.8	15.2	15.7	13.1	13.2	13.4	13.9	14.3
40/60	14.8	15.0	15.2	15.7	16.0	13.6	13.8	14.0	14.5	14.9
60/80	15.2	15.4	15.6	16.1	16.6	14.2	14.4	14.6	15.0	15.5
80/100	15.6	15.8	16.0	16.5	17.0	14.6	14.9	15.1	15.6	16.1
100/120	15.9	16.1	16.3	16.9	17.4	15.4	15.3	15.5	16.0	16.6
120/140	16.2	16.7	16.7	17.2	17.8	15.4	15.7	15.9	16.4	17.0

Table 5: Relative and absolute temperature coefficients of refractive index according to the ZERODUR[®] catalog [8].

5. Stress Birefringence

The stress birefringence in ZERODUR[®] depends on the diameter to thickness ratio of the part. The thicker the part is in relation to its diameter the higher the remaining internal stress will be. Nevertheless precision annealing quality (SSK, [3]) with stress birefringence values below 4 nm/cm can also be achieved for ZERODUR[®] blanks with sizes in the 1.5 m range.

6. Bubbles and Inclusions

For general ZERODUR[®] applications only bubbles and inclusions of a diameter > 0.3 mm are taken into account [8]. The bubbles and inclusion specification of optical glass is in general much tighter. The evaluation starts at bubble and inclusions diameters of > 0.03 mm [3]. For smaller and thinner parts with a maximum thickness of up to ~100 mm optical grade inclusion quality for ZERODUR can be achieved by precise selection. For large and thick ZERODUR[®] blanks (> 800 mm in diameter and > 100 mm in thickness) such a selection process is not applicable due to the low visibility of very small inclusions inside a big ZERODUR[®] blank. In this case the actual inclusion specification has to be fixed in close cooperation with the customer. In general for the selection of ZERODUR[®] based on optical quality bubbles and inclusion grades, it is mandatory to polish the inspection surfaces of the material, therefore optical grade bubbles and inclusions specifications are treated on special request only.



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7. Conclusion

It has been shown that ZERODUR[®] can be produced in optical quality for the use in transmittance optics. Standard optical quality grades have been achieved for 300 mm diameter blanks if defined measures along the process chain are carefully applied to individual process steps. By an intensive selection process and additional process optimization efforts it is also possible to select 1.5 m diameter large blanks in standard optical quality grade. To ease the selection process it is necessary to specify the optical requirements as precisely as possible in advance in close cooperation between the customer and SCHOTT.

8. Literature

[1] SCHOTT Technical Information TIE-37 "Thermal expansion of ZERODUR®"

[2] SCHOTT Technical Information TIE-41 "Large optical glass blanks"

[3] Current SCHOTT Optical Glass Pocket Catalog

[4] SCHOTT Technical Information TIE-29 "Refractive index and dispersion"

[5] SCHOTT Technical Information TIE-35 "Transmittance of optical glass"

[6] SCHOTT Technical Information TIE-25 "Homogeneity of optical glass"

[7] SCHOTT Technical Information TIE-26 "Striae in optical glass"

[8] ZERODUR[®] catalog

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