

TIE-26: Homogeneity of optical glass

0. Introduction

SCHOTT offers machined optical glasses with homogeneities up to H5 quality. The achievable homogeneity depends mainly on the glass type and dimension. Most optical glasses can be delivered from stock in homogeneities H2 or better.

The glass N-BK7 is an example of a high homogeneous glass that can be produced in high quantities with dimensions larger than 300 mm and homogeneities of H2 and better.

For N-BK7 parts with dimensions smaller than 150 mm even H5 quality can be achieved in large quantities.

1. Definition of 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. Short-range variations are variations over a distance of about 0,1 mm up to 2 mm (see TIE-25 for more information on striae). Whereas the spatially long range global homogeneity of refractive index covers the complete glass piece.

2. Generation of global inhomogeneities

There are three main reasons for the generation of global inhomogeneities:

- § The melting process: Optical glass is produced in a continuous melting process. Inhomogeneities of refractive index can be caused by gradients of the chemical composition during the melting process. This gradient is generated by surface evaporation of specific components and/or by reaction of the part of the melt that is in contact with the mold wall material. For process control during a continuous melting and casting process the refractive index is observed as a function of time. Glasses with highest homogeneities are extracted from castings in time frames where the refractive index was nearly constant in time.
- § Variations of the density due to thermal equilibrium: The density variations depend on the thermal history of the glass. At higher temperatures the equilibrium density is reached in a shorter time than at lower temperatures. The equilibrium density reached is different for different temperatures around the transformation temperature T_g . The refractive index homogeneity is a function of the density distribution in the glass. Uncontrolled cooling of the glass around temperatures near T_g will generate spatial refractive index inhomogeneities. In the production of optical glass subsequent fine annealing of the glass prevents such inhomogeneities.

The glass has to be cooled down slowly from temperatures slightly above T_g in order to prevent thermal gradients. The fine annealing of optical glasses with large dimensions to achieve high homogeneities is a very time consuming process.

§ Permanent stresses due to temperature gradients during cooling

3. Homogeneity grades

The availability of glasses with increased requirements for refractive index homogeneity comprises 5 classes in accordance with ISO standard 10110 part 4 [5]. The SCHOTT homogeneity grade H1 to H5 for single parts comprises ISO grades 1 to 5. SCHOTT uses classes 0 and also 1 of the ISO standard to describe the variation tolerances. The variation tolerance is the refractive index variations from piece to piece [4]. Table 1 gives an overview of the homogeneity grades.

ISO 10110 part 4 homogeneity grades	Maximum refractive index variation	SCHOTT homogeneity grades	Availability
0	$\pm 50 \cdot 10^{-6}$	S0	variation tolerance, homogeneity of single cut glasses is always better
1	$\pm 20 \cdot 10^{-6}$	S1	variation tolerance, for single cut glasses
	$\pm 20 \cdot 10^{-6}$	H1	for single cut glasses
2	$\pm 5 \cdot 10^{-6}$	H2	for single cut glasses
3	$\pm 2 \cdot 10^{-6}$	H3	for single cut glasses but not for all sizes
4	$\pm 1 \cdot 10^{-6}$	H4	for single cut glasses but not for all sizes and depending on glass type
5	$\pm 0,5 \cdot 10^{-6}$	H5	for single cut glasses but not for all sizes and depending on glass type

Table 1: Homogeneity grades.

A refractive index variation within an optical component leads to a deformation of the wavefront passing through the glass piece, according to the following formula:

$$\Delta s = d \cdot \Delta n$$

Δs is the wavefront deviation, d is the thickness of the glass and Δn is the peak to valley refractive index variation in the glass. For example: a plane wave passing once through a 50mm thick plane glass part with H2 quality will be deformed by a maximum of $50 \text{ mm} \cdot 10^{-6} = 500 \text{ nm}$. A H5 glass part of the same thickness leads to a wavefront deformation of 50 nm maximum.

4. Measurement methods

There are two different approaches to measure inhomogeneities in optical glass:

- § The integrating method by interferometry, preferably phase-measuring interferometry. This is the preferred procedure since the deformation of the entering wavefront is directly measured. The homogeneity is evaluated by integrating over the light path in the glass sample. Therefore a linear gradient of refractive index in the direction of the beam cannot be detected. In order to suppress surface irregularities the glass sample is either positioned between two oil-on-plates sandwiches, which are contacted by immersion oil or polished and measured in different orientations to eliminate the surface influence. Both methods are used by SCHOTT.
- § The statistical method. Several distributed samples will be cut from the glass plate to be inspected. The difference in refractive index between these samples is measured using a double slit interferometer. This technique is also used by SCHOTT and is explained in [6]

5. The Direct100 Fizeau Interferometer

SCHOTT in Mainz uses a DIRECT100 Fizeau Interferometer from ZEISS for homogeneity measurement with a maximum aperture of 508 mm (20 inch). Measurements with an aperture up to 600 mm are possible with a different interferometer and less accuracy. A schematic overview of the DIRECT100 setup is shown in figure 1.

The setup consists of a He-Ne Laser light source and a large collimator that transforms the laser beam to the full aperture. The collimated, parallel beam travels through a partially reflecting Fizeau plate. Part of the light is reflected back by the Fizeau plate. The remaining light enters the cavity and travels through the sample for the first time. After passing the sample the light is reflected back by a plane mirror and passes the sample and the Fizeau plate and the collimator a second time before it interferes with the reflected light from the Fizeau plate on the CCD array where the interference fringes are recorded.

The Fizeau plate and the autocollimation mirror are made from ZERODUR. The interferometer employs the direct measuring interferometry method of Carl Zeiss [1]. This method is capable of providing interferograms and calculated wavefronts from the fringes in real time. The data for a single interferogram are taken within 2 ms. The complete wavefront data set is available after 40 ms. Thereby it is possible to average 4000 wavefront data sets in less than 3 minutes.

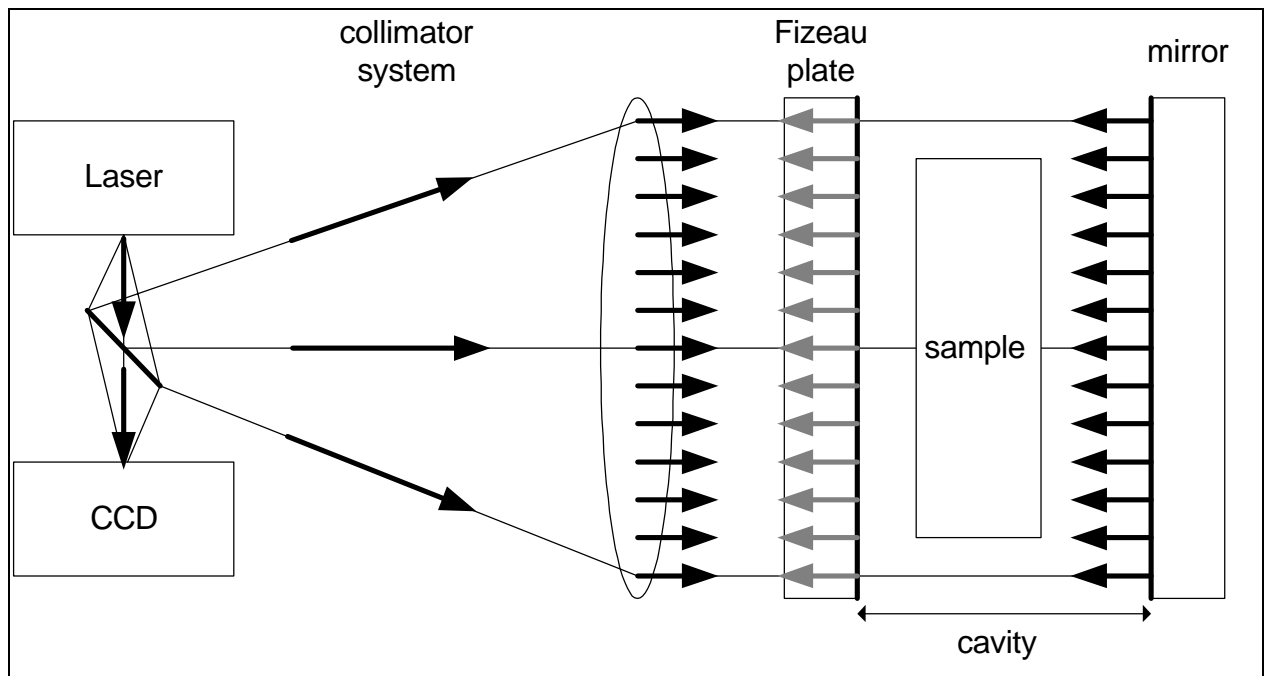


Figure 1: Schematic of the Interferometer setup.

The refractive index variation of the glass depends on the temperature due to the thermo-optical coefficient. Therefore temperature gradients within the glass have an impact on the measurement accuracy. Special measures have to be taken to reduce the temperature variations within the samples. The interferometer room is air-conditioned. The interferometer cavity is separated from the interferometer room by a special cabin (see figure 2). The temperature stability of this cabin is $\pm 0,05^{\circ}\text{C}$, the temperature stability in the room surrounding the cabin is $\pm 0,25^{\circ}\text{C}$. A special transfer system is used to move the prepared samples into the interferometer cavity.

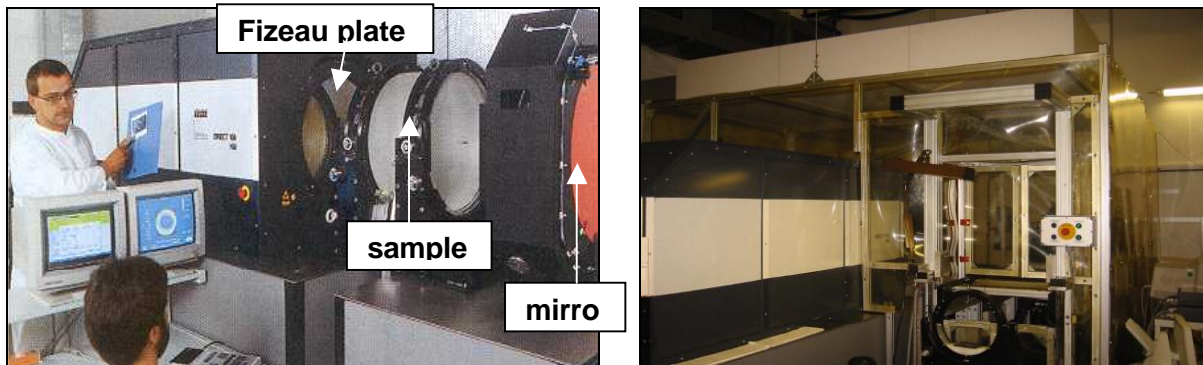


Figure 2: Left: DIRECT100 without cavity, right: inner climate cabin from the outside.

6. Homogeneity measurement methods

The influence of the sample surfaces must be eliminated for homogeneity measurement. SCHOTT uses two methods for homogeneity measurement: The “oil-on-plates sandwich” method and the “polished sample” method.

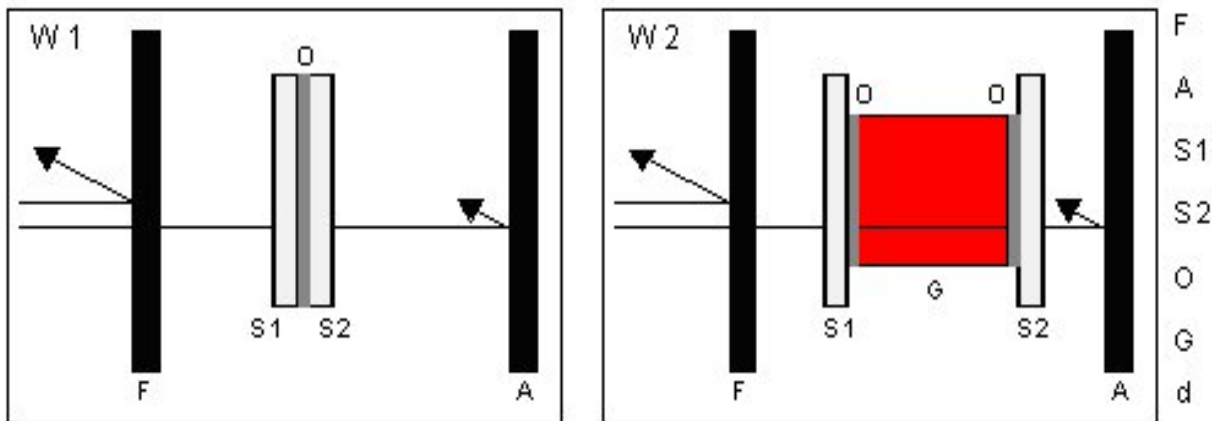


Figure 3: Oil on plate measurement setup.

For the “oil-on-plates sandwich” method the sample is placed between two glass plates (s1 and s2 in figure 3). These glass plates exhibit accurate polished surfaces. The glass plates are connected with the samples using an immersion oil liquid (O) that has the same refractive index as sample. For this method the sample (G) does not need to be polished for the measurement. Only lapped surfaces with a flatness of about $3\mu\text{m}$ are required. The measurement starts by measuring the oil-on-plates sandwich alone without sample (W1) and subtracting a measurement of the oil-on-plates with the sample (W2).

The result is a homogeneity plot of the sample. For the accuracy of this method it is very important that the immersion oil matches the refractive index of the sample very accurate (Δn should be less than $1 \cdot 10^{-4}$). To measure a wide variety of optical glasses with varying refractive indices mixtures of two immersion oils are used. Optical glasses with refractive indices from 1.473 to 1.651 can be measured using the “oil-on-plates sandwich” method.

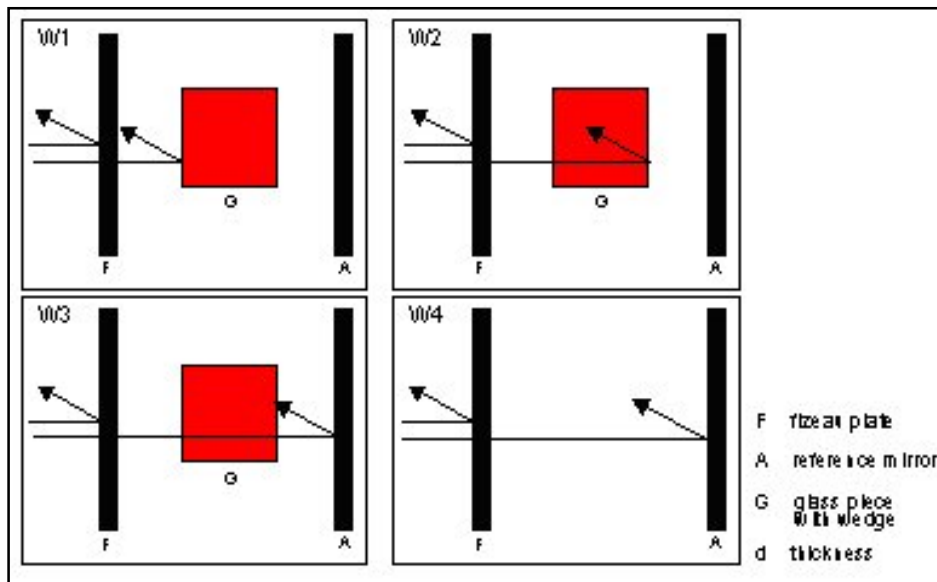


Figure 4: Polished sample measurement method.

For optical glasses that cannot be measured using the “oil-on-plates sandwich” method the “polished sample” method must be used [3]. For this method the sample must be polished on both surfaces to moderate optical quality. Additionally a small wedge of a few minutes angle must be introduced between front and back surface. The homogeneity measurement consists of a sequence of 4 individual measurements (see figure 4). First a measurement of the empty cavity is necessary. Then 3 measurements of the sample are performed. The sample will be measured in transmission, in reflection from the back surface and in reflection from the front surface. These 4 measurements are combined subsequently and the homogeneity distribution is evaluated. Much more time and effort is needed to carry out the “polished sample” method compared to the “oil-on-plate” method.

7. Measurement accuracy

As mentioned before the homogeneity of optical glass is measured by evaluating wavefront deviations using interferometric techniques. Therefore the measurement accuracy of the interferometer is given in nm wavefront deviation (peak to valley).

The accuracy of the interferometer can be evaluated by repeatability measurements of the empty cavity. The repeatability lies in the range of 3-4 nm peak to valley, which is the so called “noise” of the interferometer.

The overall accuracy of the wavefront measurement is influenced by the temperature homogeneity, the matching accuracy of the immersion oil liquid and the handling (oil-on-plate measurement setup, preparation of the samples for the polished method).

For the oil-on-plate measurement the standard deviation lies in the range of +/-10 nm wavefront deviation (peak to valley).

The practical meaning of +/-10 nm wavefront accuracy is, that for measuring homogeneity grade H5 ($\pm 5 \cdot 10^{-7}$) the sample needs to be at least 10-20 mm thick. The sensitivity of the measurement increases with increasing sample thickness.

8. Inspection certificate and interpretation of measurement results

For each homogeneity measurement the customer obtains a homogeneity inspection certificate. For the measurement with the DIRECT100 the inspection certificate contains a homogeneity map of the measured sample. This colored coded homogeneity map displays the refractive index variations within the measurement aperture. Different colors express different refractive index values. Color changes therefore express refractive index variations, in other words: in-homogeneities. The homogeneity is given as the peak to valley variation within this homogeneity map. Fig. 5 shows a typical homogeneity distribution color map and a 1D “height” profile along the direction of the arrow in the color map.

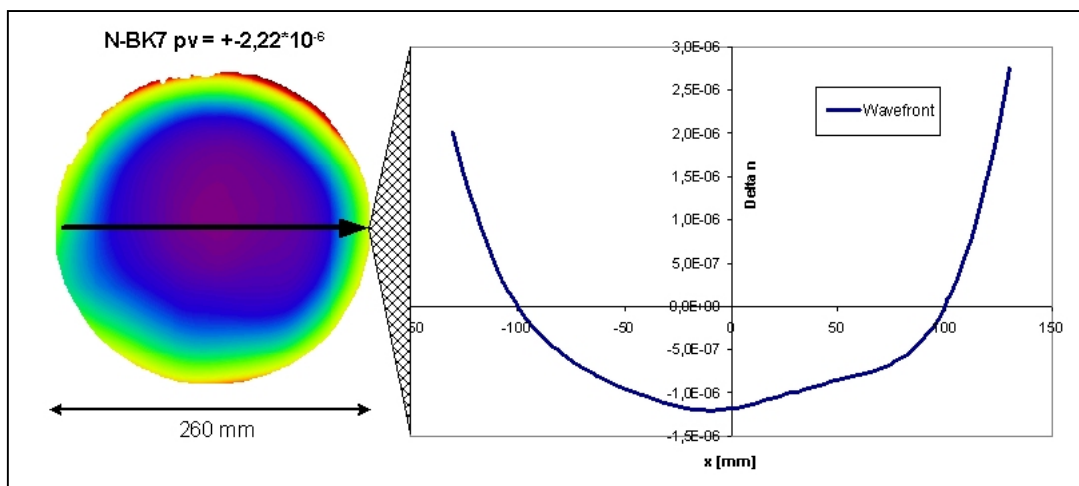


Figure 5: 2D homogeneity color map and 1D profile along arrow.

The shape of the wavefront deformation (and therefore the homogeneity distribution) can be mathematically described as a polynomial function being a summation of independent aberrations terms. These terms contain coefficients expressing the amount of focus, astigmatism, coma and spherical aberration within a wavefront. Piston and tilt deviations are subtracted from the wavefront in advance. SCHOTT uses for the decomposition of the wavefront the Zernike polynomial expansion [2].

The Zernike polynomial expansion of a given wavefront is only valid if the wavefront exhibits a circular aperture.

For certain applications it is important to know the Zernike coefficients to simulate the wavefront deformation due to the component in an optical setup, therefore the SCHOTT homogeneity testing certificate for circular apertures contains information on the Zernike coefficients.

Figure 6 shows the 1D homogeneity distribution with the appropriate main aberration polynomials. The picture on the right shows the 3D homogeneity distribution.

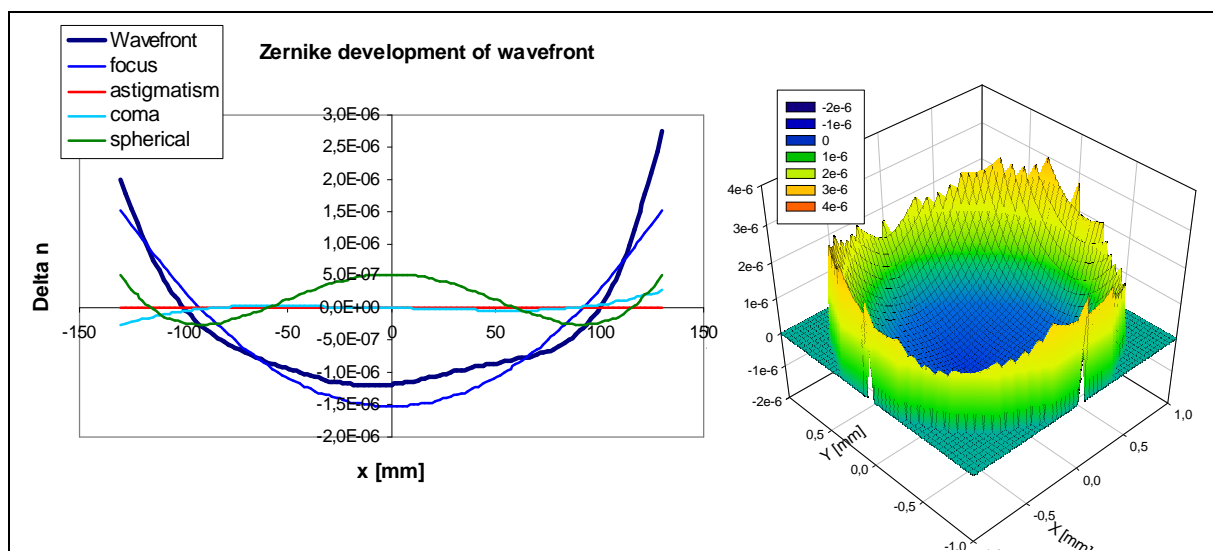


Figure 6: Example of the Zernike polynomial expansion.

For most applications of optical glass the focus term can be compensated in lens design by refocusing by the adjustment of lens distances. The peak to valley homogeneity after subtracting the focal term from the complete wavefront is in most cases much lower than the initial value (see figure 7).

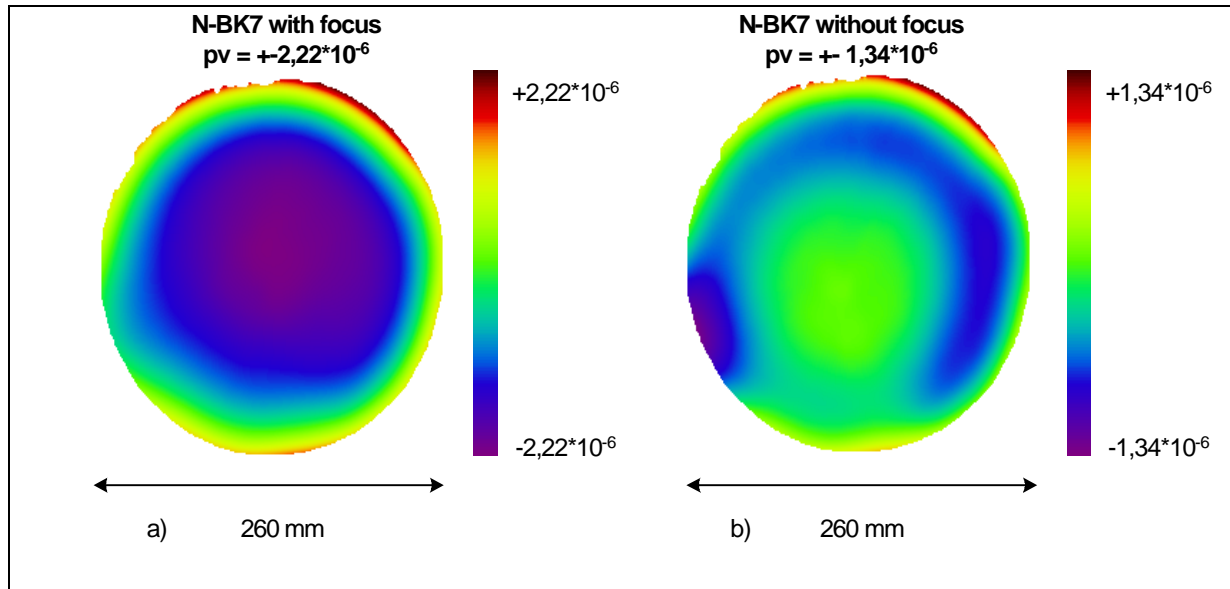


Figure 7: N-BK7 blank with and without focal aberration.

9. Material selection/Implications

There are several criteria for the selection of homogeneous material from a melt campaign. The plot of the refractive index versus time during a melting process is used to evaluate if glass was produced in higher homogeneities. The best results are achieved if the refractive index remains constant with time, which means that for high homogeneities glass should be taken from areas where the slope of the plot is close to zero.

Another criteria for selecting the glass with higher homogeneity is the striae inspection. As a rule glass with a very good global homogeneity does not exhibit striae. Figure 8a shows the homogeneity measurement of a N-BAK1 glass block without striae, figure 8b shows the homogeneity measurement of a N-BAK1 glass block from the same melt with striae. The homogeneity of the striae free block is two times better than the block exhibiting striae. The spatial resolution of the DIRECT100 interferometer is too small to make the striae itself visible.

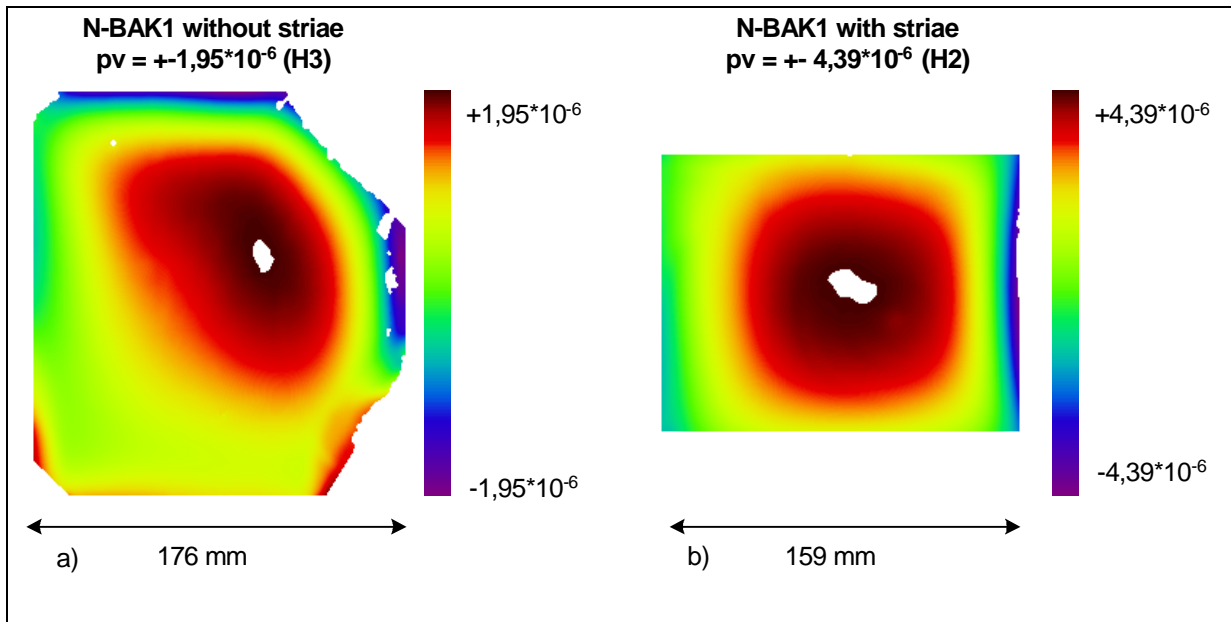


Figure 8: Striae content as selection criteria for homogeneous glasses.

Either if the glass format is circular or block shaped, most homogeneity distributions exhibit a rotational symmetry. Therefore if the diameter of a large casting is reduced by cutting and grinding the homogeneity increases.

Figure 9 shows the homogeneity of a disc shaped N-BK7 part (with starting diameter 260 mm) as a function of the diameter of the part. At 260 mm diameter the disc exhibits H2 quality, at 250 mm H3, at 210 mm H4 and at 170 mm H5 quality. In general it can be observed that the homogeneity increases with decreasing diameter.

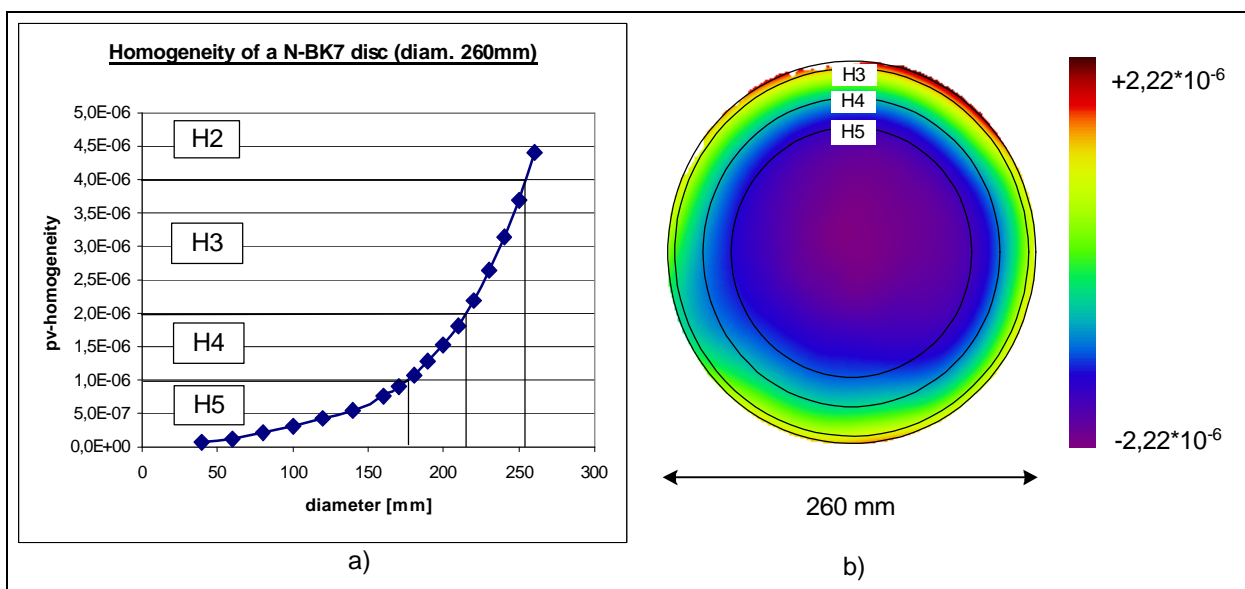


Figure 9: Homogeneity as a function of diameter.

The strongest deviations from homogeneity can be found near the edge of the glass disc. The reason for the decrease in homogeneity towards the mould wall can be found in the casting process. The glass flow during casting forms specific convection patterns. The crucible is filled from the bottom to the top and from the center to the outer part. Slight changes in the refractive index during the time needed to fill up the crucible will be reflected in the refractive index distribution later on. This phenomenon will especially have an influence in the production of large blanks. Additional reactions with the refractory wall materials of the crucible can worsen the homogeneity at the outer part of the glass.

Figure 10 shows the homogeneity measurement result of an 840 mm diameter N-BK7 disc. Within an aperture of 464 mm H4 quality was achieved.

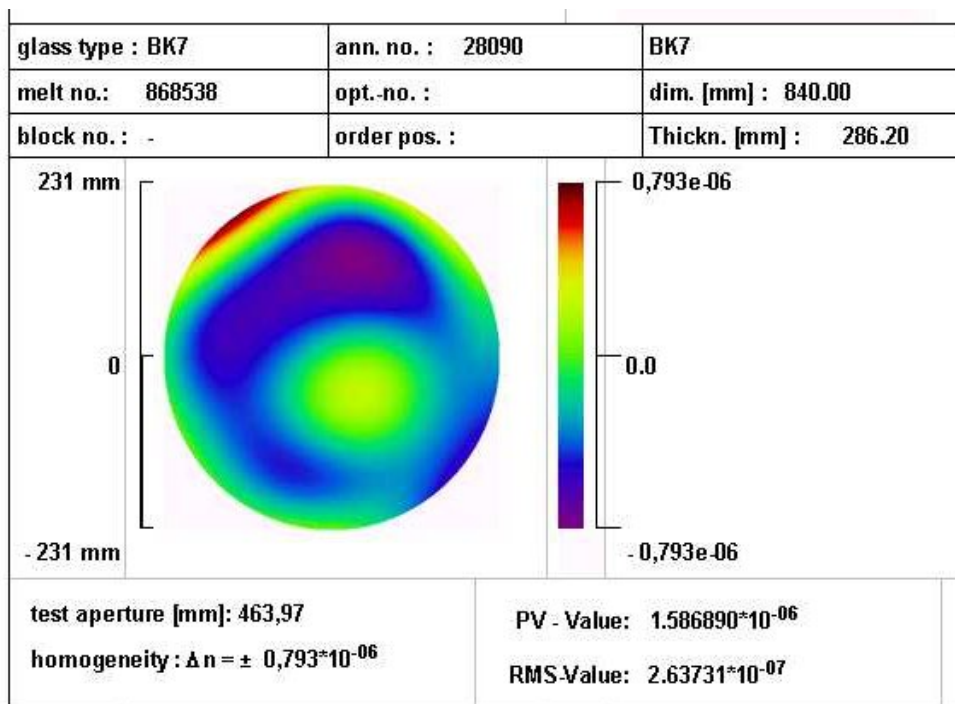


Figure 10: Homogeneity of an 840 mm diameter N-BK7 disc.

The slight differences in the refractive index of the melt during casting time will be more prominent in the homogeneity measurement in edge/edge direction than in the top/bottom direction measurement direction. Figure 11 shows the homogeneity distribution of a N-BK7 glass block in top/bottom and edge/edge direction. The homogeneity in edge/edge direction is lower than in top/bottom direction. This observation may be important for the selection of material for extreme quality prism applications.

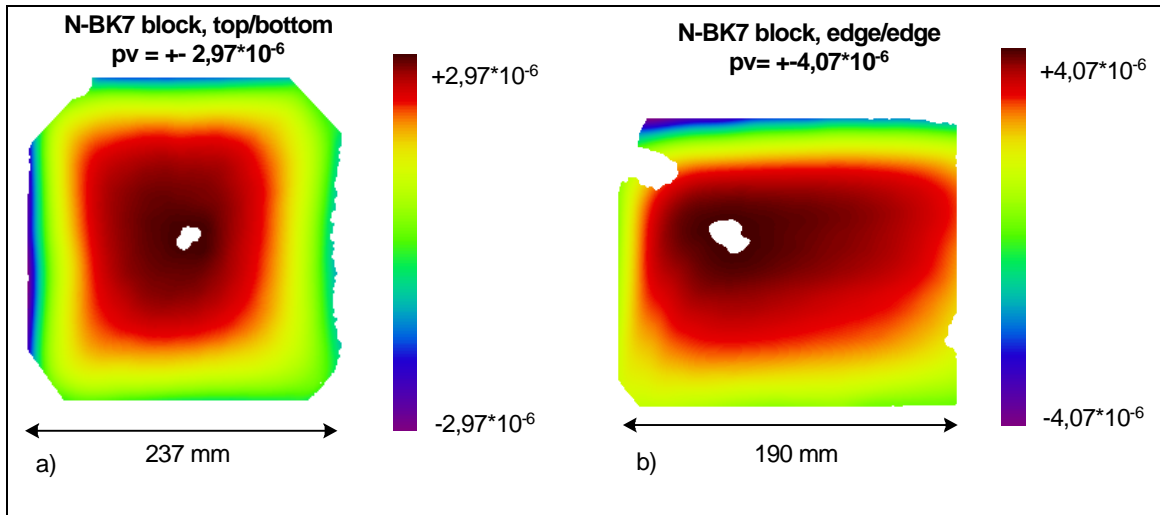


Figure 11: Homogeneity depending on view direction.

The achievable homogeneity also depends strongly on the glass type and production process. The most common optical glass that can be produced with excellent optical homogeneity in large diameters and high quantities is N-BK7. Figure 12 shows the frequency distribution of about 280 homogeneity measurements on N-BK7 of different diameters from normal production without special measures for very high homogeneity. It can be seen that in the diameter category of 300 mm almost 90% of all tested blanks fulfill H2 quality. 83% of all N-BK7 blanks with diameters equal or less than 150 mm even exhibit H5 quality.

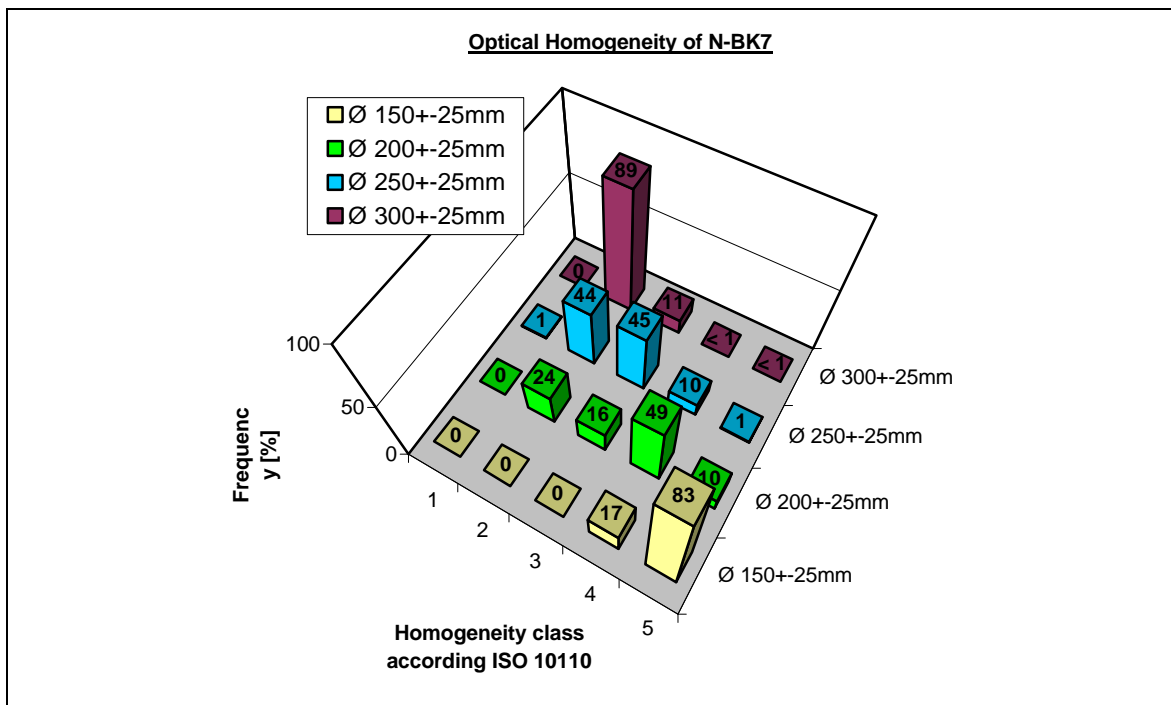


Figure 12: Homogeneity distribution of N-BK7 measurements.

10. Conclusion

In general the global refractive index homogeneity of optical glasses is better than $\pm 20 \cdot 10^{-6}$ (comprising ISO 10110 part 4 homogeneity grade 1).

Most machined optical glasses can be delivered in homogeneities H2 or better. SCHOTT can supply optical glasses with homogeneities up to H5 quality. The achievable homogeneity depends on the glass type and the size. For special applications SCHOTT also offers good homogeneity in two perpendicular directions. The most common glass that can be fabricated in big sizes and very high homogeneity is N-BK7.

11. Literature

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