Original Paper

Application of the Christiansen-Shelyubskii method to determine homogeneity and refractive index of industrial glasses[1)]

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The Christiansen-Shelyubskii method has been applied to determine the homogeneity of both colorless and colored technical glasses. It could be confirmed that this method is sufficiently sensitive to changes by the melting process of flat, container and special glasses. The homogeneity factor, which essentially is the standard deviation of the refractive index, can be obtained with a precision of about ±5 %. The measurement simultaneously delivers the mean refractive index with high accuracy. This property possibly could be used to substitute density measurements to control the constancy of glass composition. The Christiansen-Shelyubskii method can be standardized and highly automated. About 15 samples can be measured per day. Thus it shows all features of a method to be used for industrial quality control.

Anwendung der Christiansen-Shelyubskii-Methode zur Bestimmung von Homogenität und Brechzahl industrieller Gläser

Die Christiansen-Shelyubskii-Methode wurde zur Bestimmung der Homogenität sowohl von farblosen als auch von gefärbten technischen Gläsern angewandt. Diese Methode ist ausreichend empfindlich, um Veränderungen im Schmelzprozeß von Flach-, Hohl- und Spezialgläsern zu ermitteln. Der Homogenitätsfaktor, der im wesentlichen der Standardabweichung der Brechzahl entspricht, kann mit einer Genauigkeit von etwa ±5 % erfaßt werden. Gleichzeitig liefert die Messung auch die mittlere Brechzahl mit hoher Genauigkeit. Diese Eigenschaft könnte eventuell genutzt werden, um Dichtemessungen zur Kontrolle der Konstanz der Glaszusammensetzung zu ersetzen. Die Christiansen-Shelyubskii-Methode kann standardisiert und automatisiert werden, wobei pro Tag etwa 15 Proben gemessen werden können. Sie zeigt damit alle für eine industrielle Qualitätskontrolle notwendigen Merkmale.

1. Introduction

The technical glass melting process is determined by a variety of differing parameters on which many properties of the glass product depend. To provide a quantitative measure of the uniformity of the glass under production conditions, it is not sufficient to determine the density of large pieces only or to count the number of defects in a sample of the final product. Inhomogeneities may not be visible to the naked eye, but nevertheless may considerably effect, for example, the workability or the mechanical strength of the glass product. Several methods to characterize the homogeneity of glass, e.g. interferometry or schlieren methods, are known [1]. However, it still is a difficult task to measure the homogeneity quantitatively. Therefore, great attention was given to Shelyubskii [2], when he originated a new application of the Christiansen filter to determine glass uniformity. Shelyubskii's method is based on the application of Raman's theory [3] to explain the Christiansen filter phenomenon. In this method the transmission of light passing an optical cell, containing glass particles immersed in a liquid of comparable refractive index, can be varied either by temperature [4], wavelength [5], pressure [6] or by titration with another liquid [7].

Many authors worked on this method, both theoretically and experimentally [8]. Mostly measurements were made on laboratory glasses, and an early attempt to apply the Christiansen-Shelyubskii method to industrial glass quality control was not very successful [9 and 10]. There exist several mathematical models of the transmission curve, in which the light is partly handled as a wave, partly as a beam. The resulting values differ strongly. All models are based on ideal conditions, which are not fulfilled by most equipments.

The present work is based on an attempt by Högerl and Frischat [11], who modified the equipment in such a way that the transmission curves for the limiting cases of homogeneous and inhomogeneous glasses match closely those simulated by mathematical models. To be able to easily measure also colored glasses, a second laser was installed, together with a mirror system to switch between the two light sources. The preparation process of the glass grains was standardized and the measurement procedure highly automated. Thus, 15 samples can be measured per day under industrial conditions with an accuracy in the homogeneity factor of about ±5 %. Moreover, the Christiansen filter can be used to determine simultaneously the refractive index of the glasses

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Received November 17, 1995.

[1)] Presented at: Session of Technical Committee III of DGG on October 20, 1994 in Würzburg (Germany).

[2)] Now with: Deutsche Owens-Corning Glasswool GmbH, Taunusstein (Germany).

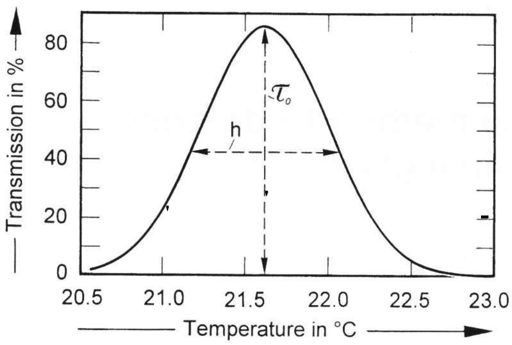


Figure 1. Example of a temperature-transmission curve of a Christiansen filter with maximum transmission, τ_0, and half-width, h.

investigated. It was checked whether the accuracy of this measurement is sufficient to control the constancy of the glass composition, too.

2. Method and theory

2.1. Homogeneity

An optical cell filled with grained glass and an immersion liquid is penetrated ly light. Light can traverse the cell only within a narrow region in which the matching of the indices between glass and liquid is satisfactory. Variation of temperature yields a change in the refractive index of the liquid and results in a transmission curve (figure 1), which is correlated with the homogeneity factor, σ. It is the standard deviation of the refractive indices related to the individual glass grains. Only for relatively homogeneous glasses the transmission curve is of Gaussian shape [11].

Raman [3], using an interference model for the Christiansen filter, derived an equation for the transmission, τ, of a Christiansen filter with an absolutely homogeneous solid phase

$$\tau = \exp(-k^2 \pi^2 \lambda^{-2} d l \Delta n^2) \tag{1}$$

where d = average diameter of the particles, l = filter length, λ = wavelength of the light, Δn = difference of refractive indices of liquid and solid phase, k = constant which depends on the geometrical properties of the powder.

Shelyubskii applied Raman's equation to a Christiansen filter containing glass particles with normally distributed refractive indices. The particles were hypothetically arranged in such a way that their refractive indices changed gradually along the optical axis, but did not change perpendicular to it. For the transmission of the Christiansen filter the following equation was developed

$$\tau = \exp(-k^2 \pi^2 \lambda^{-2} d l (\Delta n^2 + \sigma^2)) \,. \tag{2}$$

Using the parameters of the temperature-transmission curve, half-width, h, and maximum transmission, τ_0, the equation for the homogeneity factor can be described as follows

$$\sigma = \frac{h}{2} (\mathrm{d}n/\mathrm{d}T) \sqrt{\frac{-\ln(\tau_0)}{\ln 2}} \tag{3}$$

where $\mathrm{d}n/\mathrm{d}T$ is the temperature gradient of the refractive index of the immersion liquid. Högerl and Frischat [11] developed an equation based on a statistical model of a Christiansen filter with 0.5 volume fraction of glass cubes and immersion liquid each. The transmission of the filter results in

$$\tau = \exp(-\pi^2 \lambda^{-2} d l (\Delta n^2 + 2\sigma^2)) \tag{4}$$

and the standard deviation of the refractive indices is

$$\sigma = \frac{h(\mathrm{d}n/\mathrm{d}T)}{2} \sqrt{\frac{-\ln(\tau_0)}{\ln 4}} \,. \tag{5}$$

These equations can be applied to colorless glasses only. Further details concerning other transmission models can be found e.g. in [12 and 13].

In the case of colored glass the transmission of the Christiansen filter must be corrected with respect to the absorption at a given wavelength. For the assumption that the absorption of the glass grains does not depend on the homogeneity, the absorption coefficient $\alpha = \alpha\ (\lambda)$ can be introduced into the mathematical model for the transmission of the Christiansen filter as follows

$$\tau = \exp(-\pi^2 \lambda^{-2} d l (\Delta n^2 + 2\sigma^2)) \exp(-\alpha d') \,. \tag{6}$$

A similar proposal was made already by Henry [14] for a filter with homogeneous particles. The homogeneity factor is described then by

$$\sigma = \frac{h(\mathrm{d}n/\mathrm{d}T)}{2} \sqrt{\frac{(-\ln(\tau_0) - \alpha \cdot d')}{\ln 4}} \,. \tag{8}$$

Equations (5 and 7) can be used now to compare the homogeneity factors of colorless and colored glasses, however, for practical cases it is difficult to estimate correctly the effective thickness, d', of the packing of the glass grains in the case of the colored glass (equation (7)). A solution to this problem can be obtained by measuring the transmission at two dissimilar wavelengths λ_i, with α_i, τ_{0i}, and h_i (i = 1,2). One obtains then

$$\sigma = \frac{h_1 \cdot h_2 (\mathrm{d}n/\mathrm{d}T)}{2} \sqrt{\frac{(\alpha_1 \cdot \ln(\tau_{02}) - \alpha_2 \cdot \ln(\tau_{01}))}{\ln 4 \cdot (h_2^2 \cdot \alpha_2 - h_1^2 \cdot \alpha_1)}} \,. \tag{8}$$

2.2. Refractive index

Any determination of the homogeneity factor using the Christiansen-Shelyubskii method includes the simultaneous measurement of the refractive index of the corresponding glass. This measurement is very simple since for ϑ_{max}, the temperature of maximum transmission, τ_0, the refractive index of the glass, n_G, and the refractive index of the liquid, n_L, are equal (figure 1). To prevent experimental influences, e.g. differences in heating rates, affecting the measurement of the refractive index, the system has to be calibrated with optical glasses having suitable and precisely known refractive indices. As will be shown later, an accuracy of $\pm 5 \cdot 10^{-5}$ can be reached for the refractive index determination. This is sufficient to use this kind of measurement for the control of the constancy of technical glass compositions [15].

3. Experimental

3.1. Apparatus

The measurement system is shown in figure 2. Two HeNe laser light sources were used (543 nm, 0.5 mW, model OEMG 05P, and 633 nm, 5 mW, model 105 SF, Aerotech, Pittsburgh, PA (USA)). The laser beam runs from the mirror system through the beam-expanding system and the filter to the detector. The beam diameter is 3 mm. Switching from one laser to the other one is possible within a few minutes. Cylindrical Suprasil cuvettes (Thermo type 165, Hellma, Mühlheim/Baden (Germany)) with a length of 5 mm were used for the filter. They allowed a sufficiently rapid temperature equilibration of the granulated glass material. The test cell was thermostated by a water circulation system (F 10 HC, Julabo, Seelbach (Germany)), possible temperature range 3 to 80 °C. The temperature measurement was done by Pt-100 temperature sensors (Heraeus, Hanau (Germany)) in the water behind the filter with a resolution of 0.01 K. Temperature measurements in the filter cell itself resulted in differences < 0.05 K. The whole measurement system was computer-controlled and every filter was measured two times, during heating and during cooling. Equation (5) was used to calculate the homogeneity factor for colorless glass. For colored glass the modified equation (7) was used. The absorption coefficients needed to apply this equation were measured on bulk glass pieces by means of a two-beam spectrophotometer (Omega, Bruins, München (Germany)).

3.2. Glass preparation

The test glasses were ground and sieved and several particle sizes ranging from 0.1 to 0.2, 0.2 to 0.35, and 0.35 to 0.5 mm were collected. To remove dust the grains were washed several times with acetone. After drying ferromagnetic metallic particles were removed by a magnet. Immediately after that handling the grains were filled into the cells, together with the immersion liquid. For soda–lime–silica glasses (flat glass, container glass) with a refractive index near 1.52 chlorobenzene was used ($n_D^{20} = 1.5241$, $-dn/dT = 5.3 \cdot 10^{-4}\,K^{-1}$). For special glasses and glass-ceramic materials other liquids had to be used, e.g. bromobenzene for the glass-ceramic Zerodur (Schott Glaswerke, Mainz (Germany)) (1.5585 and $-5.3 \cdot 10^{-4}\,K^{-1}$). Bubbles had to be removed and the grains were compacted by a frequent tapping of the cell. This kind of handling the preparation process comes close to a standardization.

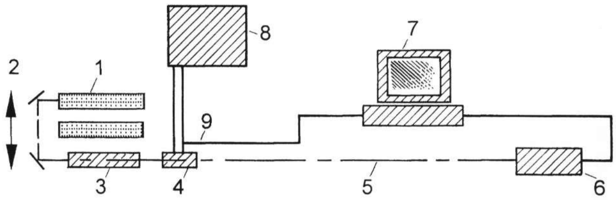


Figure 2. Experimental set-up of the Christiansen-Shelyubskii method (schematic).
1: laser, 2: mirror system, 3: beam-expanding system, 4: cuvette, 5: reference and direct beam, 6: detectors, 7: computer, 8: thermostate, 9: temperature sensor.

3.3. Accuracy and reproducibility

There is a number of random and systematic errors which may affect the measurements. Adhering glass dust and impurities may influence the transmission, the same is true if gas bubbles have not been removed carefully or have been formed during heating. Moreover, the constancy of the laser intensity has to be checked from time to time. A filter cell with an optical glass was measured ten times. The maximum transmission amounted to $\tau_0 = 92.1 \pm 0.4\,\%$, the half-width was $h = 0.84 \pm 0.02$ K, and the homogeneity factor $\sigma = (5.4 \pm 0.2) \cdot 10^{-5}$. The reproducibility of the whole method was checked by repeating the measurement on a container glass from the very beginning. The results are $\tau_0 = 81.1 \pm 1.9\,\%$, $h = 0.83 \pm 0.04$ K, and $\sigma = (8.5 \pm 0.5) \cdot 10^{-5}$. From these data it was concluded that the present equipment is sufficiently precise and accurate to be applied to the control of technical glasses.

4. Results and discussion

4.1. General

Figure 3 displays the transmission curves obtained for the optical glass K5 (Schott Glaswerke, Mainz (Germany)) using the two lasers. Differences in temperature for τ_{0i} originate from the dissimilar dispersions of glass and immersion liquid. The theoretical h_i values can be calculated to be 0.45 K (at 543 nm) and 0.53 K (at 633 nm), respectively. The experimental lines are still broadened. Reasons for that may be seen in a nonideal form of the grains and the grain fractions. Moreover, the finite length of the optical path may also cause an

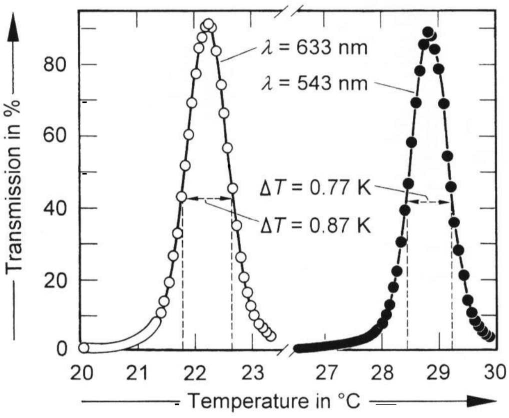


Figure 3. Temperature-transmission curves for the K5 glass measured at two wavelengths (chlorobenzene, d = 0.35 to 0.50 mm, l = 5 mm).

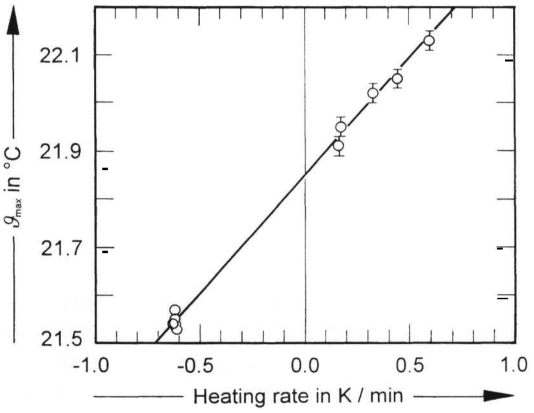


Figure 4. Dependence of ϑ_{max} of maximum transmission, τ_0, on heating (cooling) rate (K5 glass, chlorobenzene, λ = 633 nm, d = 0.35 to 0.50 mm, l = 5 mm).

additional broadening. The little difference in maximum intensities of the two lines is a consequence of theory, too.

Maximum transmission and half-width are also dependent on the length of the optical cell. Measurements have shown that a length of 5 mm is optimal. Furthermore, τ_0 and h are dependent on the grain size of the glass, too. Increasing grain size decreases h and increases τ_0. Finally, it could be shown that heating or cooling rates >1 K/min decreased τ_0 and increased h, obviously due to temperature gradients in the cuvettes. Therefore, the present measurements were performed at <0.8 K/min. As figure 4 shows, ϑ_{max} and the heating rate are linearly related in this case. Such a dependence is important for the determination of the mean refractive index of the glasses.

To obtain comparable results the measurements have to be performed under constant experimental conditions, for further details see [13].

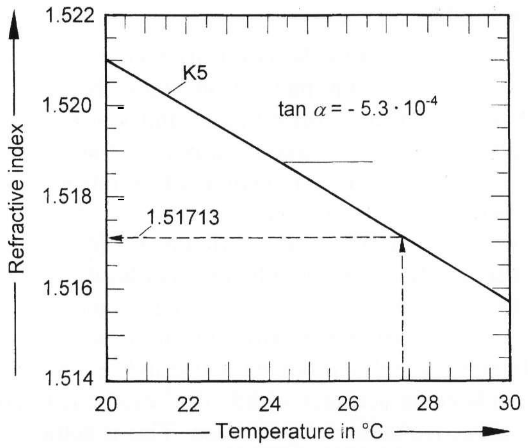


Figure 5. Example for the determination of the refractive index of a float glass (Flachglas AG, Weiherhammer (Germany)), cooling rate −0.6 K/min.

4.2. Mean refractive index

To determine the mean refractive indices of glasses with $n \approx 1.52$ (flat and container glasses) chlorobenzene and two reference glasses (Schott Glaswerke, Mainz; K5: n = 1.52437 for λ = 543 nm, n = 1.52027 for 633 nm; KF9: n = 1.52605 for 543 nm, n = 1.52131 for 633 nm) were used. Figure 5 displays an example for the wavelength 633 nm. The glass K5 has a peak temperature of ϑ_{max} = 21.53 °C ±0.01 K (cooling rate −0.6 K/min), a distinct float glass sample shows ϑ_{max} = 27.32 °C ± 0.02 K for the same cooling rate. Thus, $\Delta\vartheta_{max}$ = 5.79 K which yields $\Delta n = -3.07 \cdot 10^{-3}$. The float glass has $n = 1.52020 - 0.00307 = 1.51713 \pm 4 \cdot 10^{-5}$. Another possibility to determine the refractive index is to extrapolate to the heating rate 0 K/min. Doing this yields ϑ_{max} =21.85 °C ± 0.02 K for the K5 glass and 27.60 °C ± 0.02 K for the float glass already mentioned. This gives: $\Delta\vartheta_{max}$ = 5.75 K and $\Delta n = 3.05 \cdot 10^{-3}$. The refractive index of the float glass is obtained then as $n = 1.51715 \pm 6 \cdot 10^{-5}$.

This method to determine the mean refractive index is very accurate, more accurate than e.g. the measurement with a refractometer ($\pm 1 \cdot 10^{-4}$). However, in order to be able to reach this high precision, it is necessary to have a suitable reference glass with precisely known n values for calibration. If the Pt-100 temperature sensor is exchanged, the calibration has to be repeated since the characteristics of such sensors may be different.

4.3. Flat glass

Glass homogeneity obviously depends on melting history. Therefore, it is interesting to ask for the influence different melting treatments may have. Figure 6 shows examples of differently prepared float glasses of technical origins. Although all glasses, except the glass taken from a period of change-over from optifloat white to optifloat green, are very homogeneous with $\sigma \leq 1 \cdot 10^{-4}$, a bubbling treatment is more efficient than stirring. This

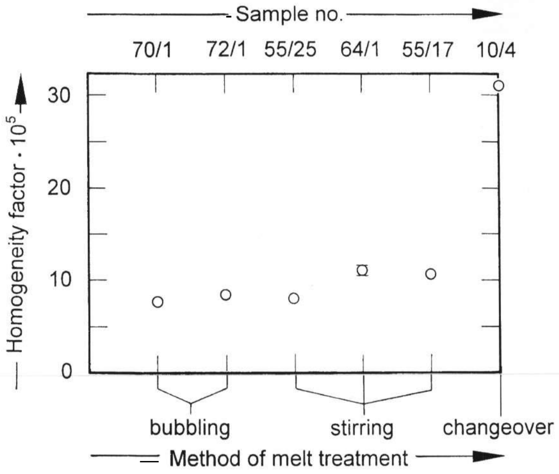


Figure 6. Homogeneity factor, σ, of differently treated float glasses (Flachglas AG, Weiherhammer (Germany)), $\lambda = 633$ nm.

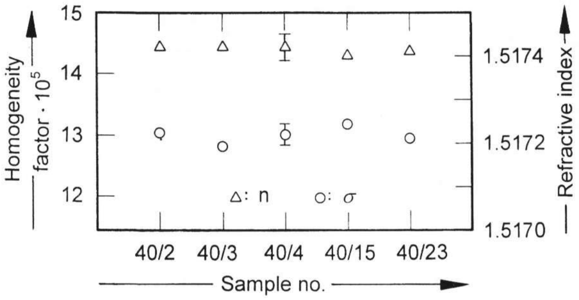


Figure 7. Homogeneity factor, σ, and refractive index, n, of an optifloat white glass (Flachglas AG, Weiherhammer (Germany)), $\lambda = 633$ nm.

is in line with earlier findings [16]. The relatively low homogeneity of the sample taken from the changeover period is caused by a change not only in batch composition but also in furnace conditions.

Figure 7 displays both the homogeneity and the refractive index data measured at different positions of a 2.1 mm thick optifloat white glass ribbon. Both data reveal that the glass is very homogeneous over the total width of the ribbon. An example of much less homogeneous glasses shows figure 8. Again an optifloat ribbon taken in the changeover period from white to green was investigated. It could be shown further that the homogeneity is position-dependent over the width of the ribbon. Obviously the additional striae occurring during this changeover period vary, too. In another series of experiments samples were taken every day (figure 9). The changeover from white to green is represented by a jump in the refractive index, however, the values of σ and n demonstrate that prior to and after the changeover nearly all the glasses are relatively homogeneous on this day-by-day control scale.

The Christiansen-Shelyubskii method is sufficiently sensitive to control the homogeneity development during the float glass production process. Thus, bubbling,

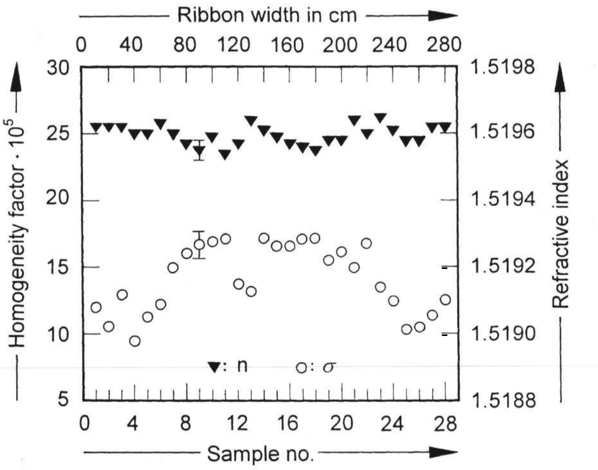


Figure 8. Homogeneity factor, σ, and refractive index, n, of an optifloat glass ribbon taken in a color change-over period from white to green (Flachglas AG, Weiherhammer (Germany)), $\lambda = 633$ nm.

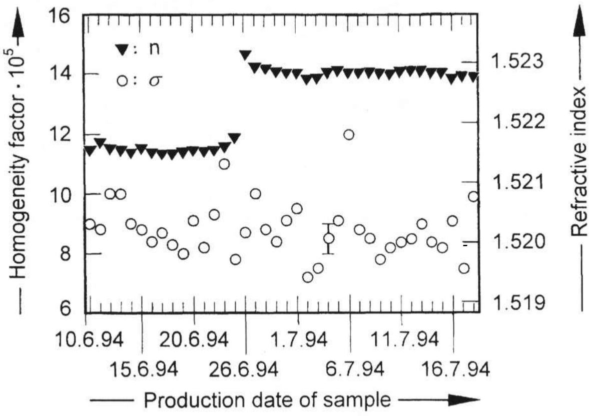


Figure 9. Homogeneity factor, σ, and refractive index, n, of an optifloat glass taken on a day-by-day control scale (Flachglas AG, Weiherhammer (Germany)), $\lambda = 543$ nm.

stirring and color changeover processes can be monitored. However, a problem may arise from the fact that all flat glasses display nearly macroscopic striae-like layers parallel to their surfaces [17]. As long as these striae are undisturbed, they do not influence the optical performance greatly. The homogeneity measured by the present method depends on the number and strength of defects, including these striae, irrespective of their orientation. Thus, there is no direct connection between the Christiansen-Shelyubskii homogeneity and the optical properties of float glass determined by these perfectly arranged layers. Following the homogeneity provides a means to monitor overall changes caused by changes in the production process.

4.4. Container glass

Homogeneity has been suggested to be a parameter affecting the workability of container glasses, too [18]. In

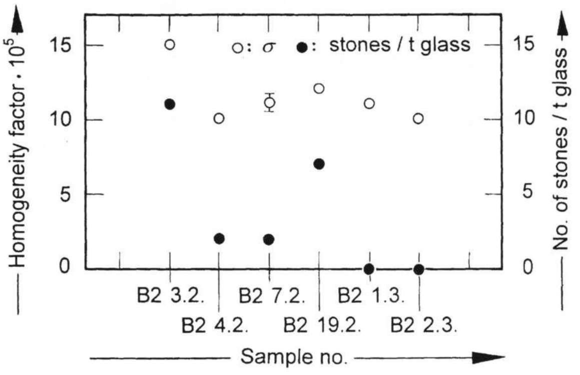


Figure 10. Homogeneity factor, σ, and number of stones/t glass [20] of flint container glasses (Heye-Glas, Obernkirchen (Germany)), $\lambda = 633$ nm.

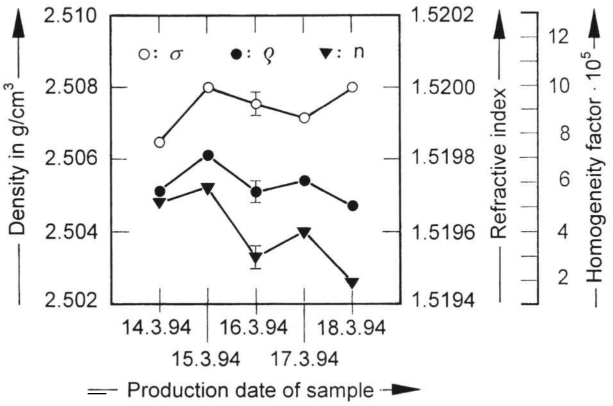


Figure 11. Homogeneity factor, σ, refractive index, n, and density [20] of glasses taken from a flint container glass tank (Heye-Glas, Obernkirchen (Germany)), $\lambda = 633$ nm.

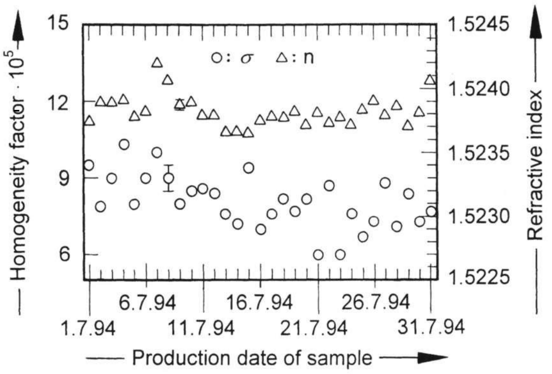


Figure 12. Homogeneity factor, σ, and refractive index, n, of flint container glasses taken on a day-by-day control scale (Heye-Glas, Obernkirchen (Germany)), $\lambda = 543$ nm.

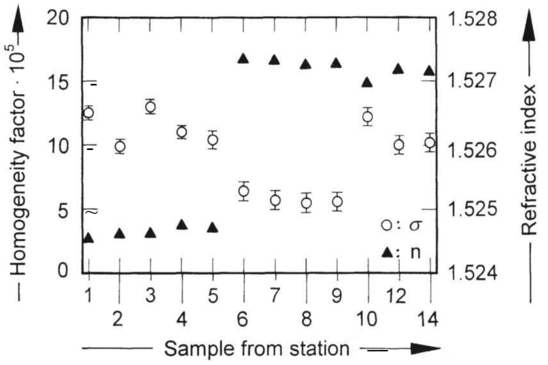


Figure 13. Homogeneity factor, σ, and refractive index, n, of flint and green container glasses taken from different stations (Nienburger Glas GmbH, Nienburg (Germany)), $\lambda = 543$ nm.

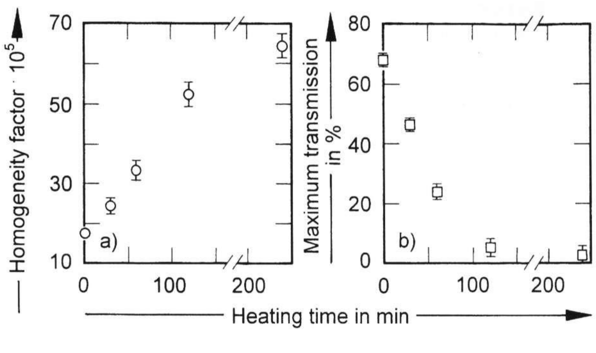
order to verify this statement, different industrial samples were investigated. First two amber glasses, one of "poor" (sample I) and one of "good" (sample II) quality, were measured. Sample I displayed $h = 0.97 \pm 0.02$ K and $\sigma = (2.6 \pm 0.2) \cdot 10^{-4}$, whereas sample II gave $h = 0.82 \pm 0.02$ K and $\sigma = (1.2 \pm 0.1) \cdot 10^{-4}$. Sample II is thus much more homogeneous than sample I, which is in line with Brückner and Yue's [19] finding that melt II shows a "brittle" fracture only at higher deformation rates than sample I.

Samples were taken from a flint glass tank in a certain time interval. Except for one sample the homogeneity of the glasses is good and correlates well with the number of stones [20] (figure 10).

The container glass industry uses density measurements for process control. Figure 11 confirms that the refractive index measurement by the Christiansen-Shelyubskii method can be a means to substitute this porcedure. All three data, σ, n, and density, clearly correlate, and the refractive index seems to be even more sensitive to changes than the density. Similar results were obtained for amber and flint glasses taken from other tanks [13].

Figure 12 demonstrates a measurement of both the homogeneity and the refractive index on glasses taken from a flint glass tank. Despite some small scattering over a one month period, the σ values $< 1 \cdot 10^{-4}$ display the high glass quality. On the other hand, amber glasses are much less homogeneous. Their σ values are situated between $(2$ to $3) \cdot 10^{-4}$ and the scattering both in σ and in n is much stronger than for flint glasses. Obviously the higher infrared absorption causes a strong temperature difference between top and bottom in the amber glass tank with the consequence of less perfect homogenization during melting.

Figure 13 shows a comparison of the σ and n values of different container glass production stations. At stations 1 to 5 flint glass was produced. There is a little scattering in the homogeneity data, however, the homogeneities are sufficiently high. At stations 7 to 14 green glasses were produced, with extremely high homogeneities of the glasses made at the stations 7 to 10. At stations 12 and 14 green glasses were produced, too, how-



Figures 14a and b. Homogeneity factor, σ (figure a), and maximum transmission, τ_0 (figure b), of the Zerodur glass-ceramic as a function of heating time at 950 °C, $\lambda = 633$ nm.

ever, another glass furnace was used. The homogeneity values of $\approx 1 \cdot 10^{-4}$ are still high, but the glass quality from stations 7 to 10 is much better.

As a consequence of the results described, the Christiansen method is equally well-suited to control the homogeneity development during the production of container glass. Moreover, the measurement of the mean refractive index delivers a further quality criterion and could substitute the density measurement.

4.5. Special glasses and glass-ceramics

The Christiansen-Shelyubskii method is applicable also to determine the homogeneities of special glasses. A limit of the present equipment is reached for extremely homogeneous glasses with σ values $\leqslant 5 \cdot 10^{-5}$. Differences in the homogeneity of such glasses cannot be determined any more. However, for example, television tube glasses and the Schott Zerodur glass-ceramic can be measured as well. Figures 14a and b show that annealing of this glass-ceramic at 950 °C causes a strong increase in the homogeneity factor, combined with a decrease of maximum transmission from ≈ 70 to $< 10\%$ after a 100 min treatment. X-ray diffraction measurements showed no change in crystal phases, however, the annealing obviously changed the microstructure of this glass-ceramic with a coarsening of the crystal grains. This has a drastic effect on the measurement, but the influence of crystal growth cannot simply be separated from the homogeneity of the material.

5. Conclusions

The present work clearly confirms that the Christiansen-Shelyubskii method is a very useful means to determine the homogeneity of most technical glasses, both colorless and colored. In the case of flat glass melt treatments like bubbling, stirring or color changeover processes can be monitored. In the case of container glass samples of different workability can be differentiated and the method is also suitable to be used for a day-by-day control. Special glasses and transparent glass-ceramics can be measured, too. Homogeneous float and container glasses display σ values $\approx 1 \cdot 10^{-4}$. There the method has its highest sensitivity. The measurement delivers simultaneously the mean refractive index with high precision. This possibly could substitute the density measurement used in the container glass industry to control the constancy of the glass composition.

The Christiansen-Shelyubskii method has been standardized and highly automated. About 15 samples can be measured per day with high accuracy. It shows all features to be used as a method for industrial quality control.

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These investigations were conducted with the kind support of the Arbeitsgemeinschaft industrieller Forschungsvereinigungen (AiF), Köln (AiF-No. 8971) under the auspices of the Hüttentechnische Vereinigung der Deutschen Glasindustrie (HVG), Frankfurt/M., utilizing resources provided by the Bundesminister für Wirtschaft, Bonn. Thanks are due to all these institutions. The authors also acknowledge helpful suggestions by an industrial consulting group, especially by Dr. J. Bretschneider, Flachglas AG, Weiherhammer; Prof. Dr. A. Lenhart, FH Nürnberg; and H. P. Williams, M. Sc., Heye-Glas, Obernkirchen.

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