Original Paper

Strength of glass influenced by shear stresses at the surface due to the forming process

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During the processing of glass melts shear stresses can arise within the contact layer with the shaping tools due to wall friction, which above a critical value may lead to defects on the product surface. These processes can be simulated and registered in a simple manner by the isothermally controlled extrusion process. For their investigation cylindric rods of an optical lead silicate glass have been extruded and subsequently their strength has been determined by means of static four-point bending tests. An influence of the die material on the flexure strength is observed. Glass rods extruded through stainless steel dies show a lower flexure strength than those being pressed through dies of electrographite. Moreover, their flexure strength decreases with increasing extrusion velocity. This can be explained by the formation of smallest imperfections on the glass surface during the flow of the glass melt along the die-channel wall. In contrast to this the flexure strength of the glass samples extruded through electrographite dies is independent of the extrusion rate.

The annealing of the extruded rods slightly above the transition temperature leads to an increase of the flexure strengths.

Einfluß oberflächennaher Scherbeanspruchungen während der Formgebung auf die Festigkeit von Glas

Während der Verarbeitung von Glasschmelzen können in der Berührungszone mit dem formgebenden Werkzeug infolge der Wandreibung Scherbeanspruchungen entstehen, die oberhalb eines kritischen Wertes zu Schädigungen der Produktoberfläche führen. Diese Vorgänge lassen sich in einfacher Weise durch den isotherm gesteuerten Strangpreßprozeß simulieren und meßtechnisch erfassen. Zu ihrer Untersuchung wurden kreiszylindrische Stäbe aus optischem Bleisilicatglas extrudiert und deren Festigkeit anschließend im statischen Vierpunktbiegeversuch ermittelt. Hierbei zeigt sich ein Einfluß des Matrizenwerkstoffes auf die Biegefestigkeit. Durch Cr–Ni-Stahlmatrizen extrudierte Stäbe weisen eine niedrigere Biegebruchspannung auf als solche, die durch Matrizen aus Elektrographit verpreßt wurden. Überdies sinkt ihre Biegefestigkeit mit zunehmender Strangpreßgeschwindigkeit ab. Dies wird auf die Entstehung kleinster Verletzungen der Glasoberfläche während der Strömung der Glasschmelze entlang der Matrizenkanalwand zurückgeführt. Bei den durch Elektrographitmatrizen extrudierten Glasproben ist die Biegefestigkeit demgegenüber unabhängig von der Strangaustrittsgeschwindigkeiten.

Eine Temperung der gepreßten Stäbe knapp oberhalb der Transformationstemperatur führt zu einem Anstieg der Biegefestigkeiten.

1. Introduction

Similar to the extrusion of polymers on extruding inorganic glass melts above a critical channel-flow velocity very noticeable defects will occur at the surface of the extruded rods, called melt fractures [1 and 2]. Assuming that already before the arising of those macroscopic faults smallest submicroscopic defects on the glass surface are formed, which have a strength-diminishing influence due to their notch effect and can also reduce the quality of the products in other respects, it will be obvious to use the indirect method for their proof, i.e. the determination of the flexure strength of the rods.

As the results for bending tests depend on the test conditions, in pretests with industrially drawn rods of alkaline earth-aluminosilicate glass (Supremax® by Schott Glaswerke, Mainz (FRG)) the most suitable test parameters have been determined first. These are the outer span and the kind of loading (three- or four-point bending). Additionally, the static flexure strengths of rods of an optical lead silicate glass (glass F 2 produced by Schott Glaswerke, Mainz (FRG)) extruded through dies of stainless steel and of electrographite (FE 52, SCHUNK Kohlenstofftechnik, Gießen (FRG)), respectively, were determined under the best test conditions.

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2. Bending equipment and defining factors

With the bending equipment shown in figure 1 and manufactured at the "Lehrstuhl für Werkstoffkunde und Mechanische Technologie" of the University of Kaiserslautern both three- and four-point bending tests can be carried out with rods of a maximum diameter D of 15 mm.

For the four-point bending tests it is possible to adjust the distance, n, between both supporting cylinders within a range of 30 and 100 mm. These cylinders as well as the two outer supporting cylinders have a diameter of 12 mm. The outer span, L, will be adjusted by shifting the pilot blocks continuously between 40 and 300 mm. This equipment will be fixed between the lower and upper tension shackle of

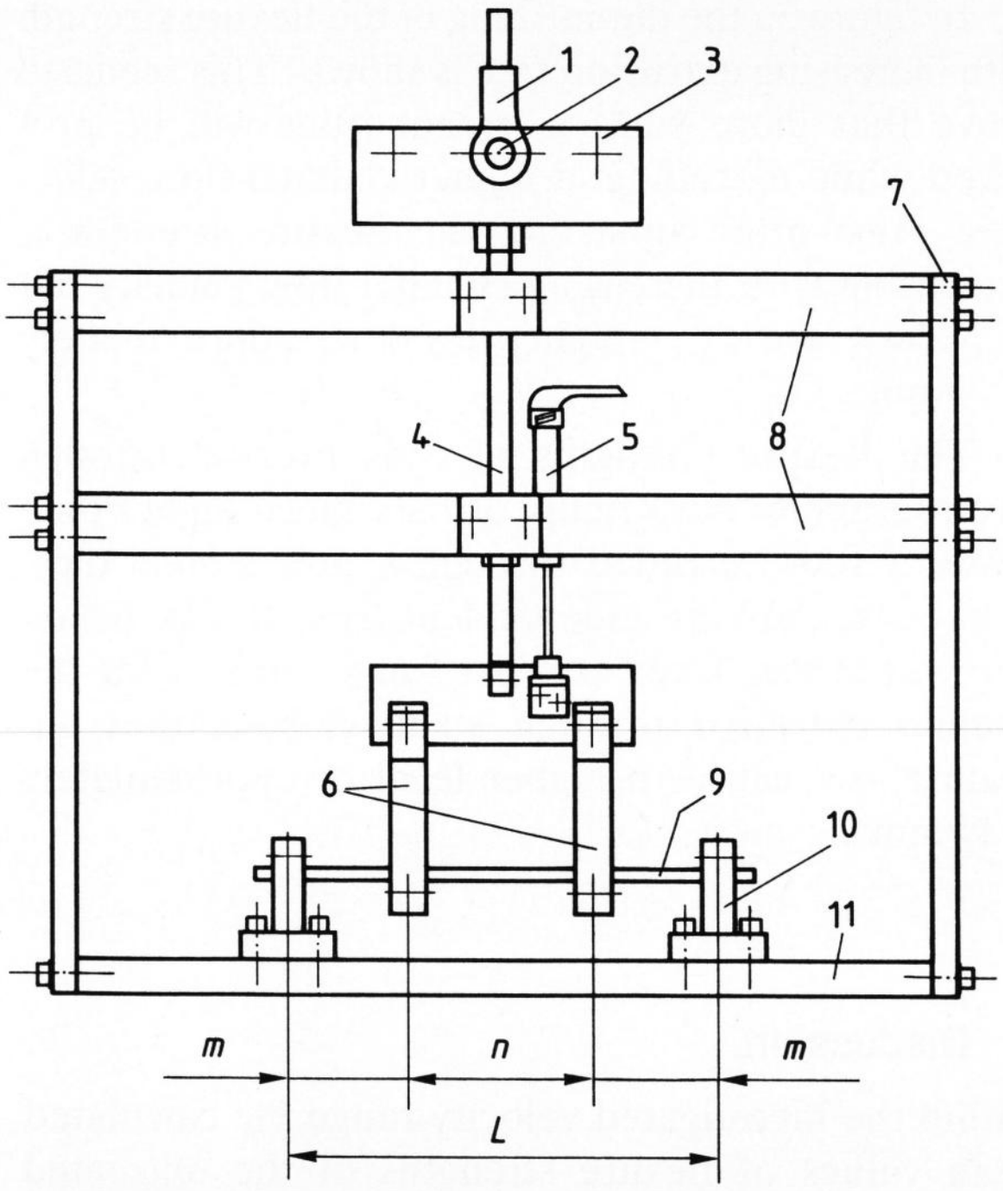


Figure 1. Bending equipment for extruded glass rods.
1: joint head, 2: hinge bolt, 3: swing support, 4: connecting rod, 5: inductive displacement transducer, 6: supporting cylinders, 7: side plate, 8: stretcher, 9: bending sample, 10: pilot block with supporting cylinder, 11: base plate.

an electromechanic universal testing machine (SCHENCK TREBEL RM 100, Ratingen (FRG)).

The bending strength for fracture, σ_{bf}, will be calculated in the case of four-point bending according to

$$\sigma_{bf} = \frac{16\, F_{max} \cdot m}{\pi \cdot D^3} . \qquad (1)$$

In this formula F_{max} means the maximal bending load, D the diameter of the bending sample and m the inner span shown in figure 1.

3. Test conditions and realization

German standards (DIN) only describe bending tests at flat and profile glass samples [3 and 4]. For that reason the tests for this work have been carried out according to the American standard ASTM designation C-158-84 [5], in which also the testing of rods is determined.

Since the test conditions, especially the increase of tension speed, have a great influence on the measuring results, the constant rate of loading of the supporting cylinders should be chosen in such a way that during all tests the fracture will occur after the same time [6]. The rate of increase in bending stress lay within the limits required by the just mentioned ASTM standard (54 and 78 N/(mm^2 min)). The values noted there for the span and consequently for the length of the sample admit, however, a rather big clearance and therefore, a more detailed examination would be necessary for a selection of these test parameters. Therefore, industrially drawn glass rods of the alkaline earth-aluminosilicate glass Supremax have been used with a diameter of 5 ± 0.05 mm. Before starting the test each sample has been loaded with a prestress of 10 ± 0.2 N in order to fix it in the bending equipment. The bending force, F, measured by the load cell of the test machine and the displacements, w, of the load application points (w always smaller than D) found out by an inductive displacement transducer have been recorded with a x-y-coordinate plotter.

In order to avoid scratches at the surface the diameter of each sample has been measured after the test with a micrometer. The maximal bending load, F_{max}, has been taken from the diagram plotted by the x-y-recorder and used for calculating the bending strength for fracture, σ_{bf}, according to equation (1).

In the case of the four-point bending test, the highest bending moment extends over the distance, n, between the two inner loading points. Therefore, during this test it will be more likely to detect a strength-diminishing surface defect than at the three-point bending test having its maximum bending moment only in a single point.

In order to guarantee an optimum of material exploitation and of information from the values of flexure strength to be found out, four-point bending tests with an outer span of 120 mm and an inner span of 40 mm have been preferred.

4. Sample producing and heat treatment

For the investigations the lead silicate glass F 2 has been extruded at a temperature of (590 ± 2) °C corresponding to a viscosity, η, from $10^{7.74}$ to $10^{7.66}$ dPa s. The extrusion was carried out through stainless steel dies (made of X 15 CrNiSi 25 20 with 0.15 % C, 25 % Cr, 20 % Ni; material no. 1.4841) or through electrographite dies, each with a diameter of 4 and a length of 7 mm, at different channel-flow velocities. The extrusion process has been regulated by a constant ram speed in order to get reproducible constant channel-flow velocities. The upper limit has been fixed at the point where first macroscopic cracks at the sample surface (figure 2) appeared while using stainless steel dies.

A minimum of ten samples have been produced at each extrusion rate. These have been cut to the necessary length and then tested by bending.

In an additional series of tests the samples already cut were annealed at the transition temperature ($\eta = 10^{13}$ dPa s) for more than 1 h in order to eliminate possible residual stresses and finally they were cooled down with a rate of less than 1 K/min.

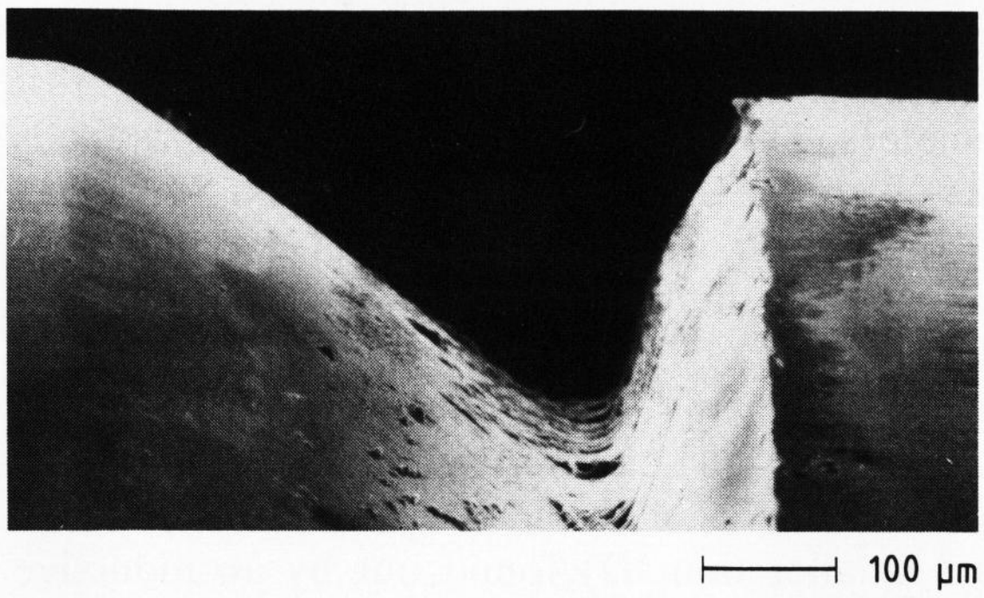
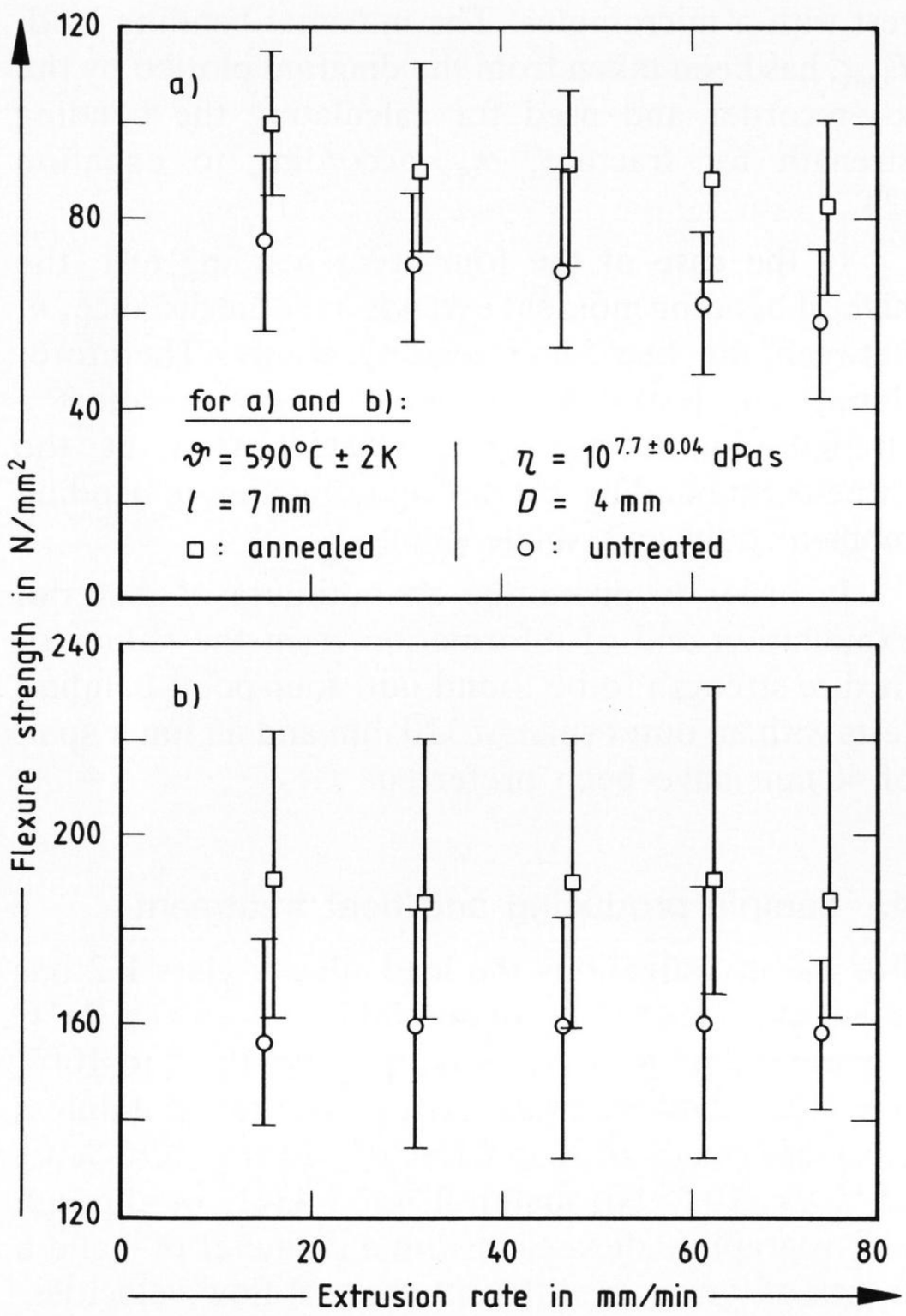


Figure 2. Typical melt fracture of the lead silicate glass F 2. Die material: stainless steel, die diameter: 5.5 mm, die length: 5 mm, ϑ: 592 °C, viscosity: $10^{7.66}$ dPa s, extrusion rate: 200 mm/min.



Figures 3a and b. Flexure strength of the lead silicate glass rods F 2 extruded through dies of different materials versus the extrusion rate, a) stainless-steel dies (X 15 CrNiSi 25 20 with 0.15 % C, 25 % Cr, 20 % Ni; material no. 1.4841), b) electrographite dies.

5. Results

The courses of the obtained four-point flexure strengths of untreated and annealed samples are shown in figures 3a and b, respectively.

While figure 3a shows the results of the rods being pressed through stainless steel dies, figure 3b shows those for rods extruded through electrographite dies.

In figure 3a the diminishing of the flexure strength with increasing extrusion rate is shown. This seems to prove that more surface irregularities will be produced while extruding at higher channel-flow velocities. Also after annealing the flexure strength is decreasing with increasing channel-flow velocity but all values are at a higher level of approximately 25 N/mm^2.

The flexure strengths of rods pressed through electrographite dies (figure 3b) are much higher than those of rods extruded through stainless steel dies. Moreover, they are independent from the extrusion rate within the tested velocity range. Just as for the samples extruded through stainless steel dies annealing also causes a higher level of approximately 30 N/mm^2.

6. Discussion

Within the investigated velocity range the calculated mean values of flexure strengths of the untreated samples extruded through stainless steel dies are between 58 and 75 N/mm^2. They are considerably lower than the results from glass samples pressed through electrographite dies. These average values of flexure strengths lie between 157 and 160 N/mm^2. Consequently, the samples extruded through electrographite dies show a more than double as high flexure strength in comparison to rods pressed through stainless steel dies.

The scattering of the average values of the flexure strength proved standard deviations from 15 to 26 % for the untreated as well as for the annealed samples. These values are not unusual for the material glass. Spauszus [7] obtained standard deviations from 12.4 to 25 %, Woelk and Elsenheimer [6] state deviations up to 30 %.

The diminishing of flexure strengths with increasing extrusion rate while using stainless steel dies is regarded to be based on the increase of the shear stresses in the flowing glass melt along the die-channel wall. These shear stresses probably lead to submicroscopic strength-diminishing surface irregularities of the extruded glass rod. The quantity and depths of these defects will increase with the channel-flow velocity and consequently decrease the flexure strengths of the corresponding samples.

For the case of complete wall sticking of the flow, the shear stresses at the wall can be determined by means of the Hagen-Poiseuille law. These are increasing up to $\tau = 10.15$ N/mm^2 within the tested velocity range. This means that already the existing slight shear stresses at small flow velocities will be sufficient to produce smallest defects at the surface of the extruded glass rod, being proved by the strength decrease of the samples with rising channel-flow velocities.

Another possibility seems to be the entering of smallest metal pieces of the die material into the surface of the glass melt during extrusion which would lead to local elevated tensions by mechanical load resulting in a reduction of the strength. Until now there exists no direct proof of this assumption, not even on analyzing the sample surface by Auger electron spectroscopy (PHI SAM 660, PERKIN ELMER, Eden Prairie, MN (USA)) and scanning electron microscopy (JSM-T 300, JEOL, Tokyo (Japan)). Nevertheless, observations of an oxide layer on glass rods extruded through stainless steel dies lead to assuming this possibility.

While using electrographite dies there will be no relevant shear stresses at the channel wall due to the favourable slip conditions. Therefore, almost no strength-diminishing surface irregularities produced by high shear stresses are expected. For that reason no influence of the channel-flow velocity on the flexure strength within the investigated range could be observed.

The annealing of the bending samples before testing causes an increase of the strength from 20 to 30 N/mm^2 independent of the die material. If this treatment produces a diminishing of surface compressive stress after the extrusion, induced by the natural cooling of the glass rods, this should lead to a strength diminution. Consequently, a lowering of the notch effect of the surface defects produced by the shear stresses should be responsible for the increase of strength by annealing. The heat treatment causes a decrease of the acuity and dimension of the notch root, resulting in a diminution of the overload of stress induced there. Therefore, the fracture appears only at higher loads, i.e. the flexure strength increases. This conclusion has been confirmed by tests of Manns and Brückner [8 and 9].

The irregularities are mainly produced by the extrusion process itself – this results from the continued diminution of flexure strengths with increasing channel-flow velocities while using stainless steel dies. However, they can also arise on guiding the glass rod at the extrusion equipment, after it has left the die, and during the subsequent handling until the rupture of the sample at the bending test. This explains the increasing of the strength by annealing the glass rods pressed through electrographite dies.

According to all the tests, electrographite seems to be the considerably better die material, because it does not show any strength-diminishing surface irregularities. On the other hand, the tool life of these dies seems to be shorter than the durability of stainless steel dies.

The results obtained by the present investigations show that for glass manufacturing processes shaping tools with favourable glide properties for glass melts should be preferred. In addition possible mechanical damages should be prevented during all processes after shaping.

7. Summary

Before the occurrence of melt fracture, already slight shear stresses will be sufficient to produce smallest invisible defects at the surface of glass rods extruded through stainless steel dies. For their investigation bending tests were carried out. The results show that no strength-diminishing surface irregularities can be observed by using electrographite dies. Therefore, during the forming of glass, non-wettable shaping tools should be preferred. Moreover, mechanical damages by handling of glassware should be prevented.

8. References

[1] Roeder, E.: Flow behaviour of glass during extrusion. J. Non-Cryst. Solids **7** (1972) p. 203–220.

[2] Roeder, E.; Dengel, A.: Experimentelle Untersuchungen zum Auftreten von Schmelzbrüchen bei der Formgebung von Glasschmelzen. Glastech. Ber. **61** (1988) no. 3, p. N14.

[3] German standard DIN 52 303, T. 1 (Aug. 1984): Prüfverfahren für Flachglas im Bauwesen; Bestimmung der Biegefestigkeit, Prüfung bei zweiseitiger Auflagerung. Berlin: DIN 1984.

[4] German standard DIN 52 303, T. 2 (Mar. 1983): Prüfung von Glas; Bestimmung der Biegefestigkeit, Prüfung von Profilbauglas. Berlin: DIN 1983.

[5] USA standard ASTM C 158-84: Standard methods of "Flexural testing of glass (Determination of modulus of rupture)". Philadelphia, PA (USA): Am. Soc. Test. Mater. 1984.

[6] Woelk, H.; Elsenheimer, K.: Biegebruchspannung von Floatglas und thermisch vorgespanntem Floatglas. Beschreibung der Prüfmethoden und statistische Berechnung der Gebrauchsfestigkeit. Glastech. Ber. **52** (1979) no. 1, p. 14–24.

[7] Spauszus, S.: Zur Festigkeitsbestimmung von Glas. Silikattechnik **24** (1973) no. 8/9, p. 285–287.

[8] Manns, P.; Brückner, R.: Biegefestigkeit von Kalk-Natron- und Borosilicatglas von Raumtemperatur bis zur Littleton-Temperatur. Glastech. Ber. **56** (1983) no. 6/7, p. 155–164.

[9] Manns, P.; Brückner, R.: Einige Besonderheiten bei der Bestimmung der Heißbiegefestigkeit hochviskoser Glasschmelzen. Glastech. Ber. **59** (1986) no. 5, p. 139–150.

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