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The preparation of multimode glass- and liquid-core optical fibres

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A preform technique for drawing cladded-glass and hollow fibres suitable for application to optical communications is described. The parameters which need to be controlled are discussed and the preparation of the preforms is described. The resulting fibres have a high geometric uniformity and a probe beam remains largely at the same angle to the axis after more than 10^6 reflections at the core/cladding interface. Fibre attenuations of 150, 60 and 5.8 dB km^{-1} have been obtained with commercial glasses, preforms made from a special melt at Sheffield University [5], and a commercial liquid, respectively. The fibre drawing process does not appear to introduce any additional impurities and heat treatment has produced a significant reduction of transmission loss in glass fibres.

1. Introduction

The techniques for making fibres for optical communications can be divided into two broad classifications, namely those involving drawing directly from a melt and those in which the draw takes place from solid material in the form of a preform. The classical melt-type process is the concentric-crucible technique in which the inner crucible container is fed with the core glass and the outer one with cladding material. The method has the advantage of obviating the need for machining or polishing of the starting materials and thus avoids the possible introduction of impurities which could result from these processes. In addition, preforms for single-mode fibres are difficult to prepare. On the other hand instabilities can occur if care is not taken and the melt levels in the crucibles should ideally be kept reasonably constant. Thus the rate of introduction of new material must be well controlled and this is sometimes done by feeding in from preforms, thus depriving the method of one of its potential advantages. There is also the problem of avoiding the introduction of impurities from the crucible, particularly as the interface surfaces flow over both sides of the inner orifice. The technique is even more difficult to apply to silica fibres and tubes due to the high temperature involved and the necessity to avoid most transition metal impurities since these can give rise to a high transmission loss in the wavelength region of interest.

The preform methods include the rod-and-tube and early Selfoc [1] techniques and drawing from composite rods which themselves can be drawn directly from a two-layer melt. The two latter techniques involve fairly straightforward fibre-pulling which is discussed below but the rod-and-tube combination also involves machining or

re-drawing the two glasses into suitable shapes and sizes. Such operations can, in principle, introduce additional impurities over and above those already present in the bulk starting glasses but in practice any such contamination can be removed by suitable cleaning and etching. The length of fibre which can be drawn in one continuous length is limited by the length and diameter of the preform thereby resulting in a batch process. However, several kilometres of 100 μm fibre can be obtained from a preform 50 cm long and 1 cm diameter and this is likely to be adequate in practice. The advantages of the rod-and-tube method are its great versatility in allowing a wide range of fibre diameters and glass compositions to be used and the high degree of precision which can be achieved. It is more difficult to apply to single-mode fibres than to multimode ones because of the necessity to drill (and clean) long narrow holes in the tube material. The particular method to be used depends on the type of fibre to be produced, such as single-mode, multimode, graded index or liquid-core, and on the glass or glasses available. In this article the rod-and-tube method is discussed in detail and the results obtained are described.

2. Fibre-drawing machine

The method of making cladded fibres is to mount the rod-and-tube preform vertically in a gimballed chuck (to allow for slight bends in the tube) and to lower it slowly into a small resistance-heated furnace. The temperature is accurately controlled and is set at a value where both glass viscosities are below 10^5 poise. The fibre is drawn from the lower end of the furnace and is taken up on a precision-made aluminium winding drum of 1.3 m circumference having a smooth cylindrical surface accurately concentric with its axis of rotation. As the drum rotates it is traversed sideways so that the free drop of fibre remains vertical and lengths of several kilometres are wound in a single layer at speeds of several metres per second.

The equilibrium fibre diameter is determined only by the ratio of the preform feed rate to the fibre pulling speed. In order to maintain a fixed diameter over the full range of pulling speeds an electrical control circuit is used automatically to keep the ratio constant. The maximum pulling speed is governed by the glass viscosity, and therefore by the furnace temperature, since the fibre will obviously break when the tension becomes too high. In practice a careful selection of furnace temperature has to be made in order to achieve good fibre quality and a reasonable pulling speed. Changes or fluctuations in pulling speed and furnace temperature cause transient excursions from the equilibrium diameter (as set by the ratio of preform speed to drum speed). Unwanted fluctuations can be eliminated by suitable furnace design and accurate control. In our case the furnace temperature is constant to 0.1 °C and the pulling speed to better than 0.1 %.

A wide range of fibres has been produced limited only by the maximum furnace temperature of 1500 °C and the availability of suitable preforms. Fibres with overall diameters between 20 and 500 μm, and core diameters between 0.5 and 450 μm, have been made using mainly Schott F7 glass as core material. The range of cladding materials has included Pilkington PWL84 and ME1, Pyrex, soda glass and Schott LF5. The machine has also been used to redraw F7 rod for making preforms. The overall diameter is monitored continuously by projecting a shadow of the fibre on a slit and

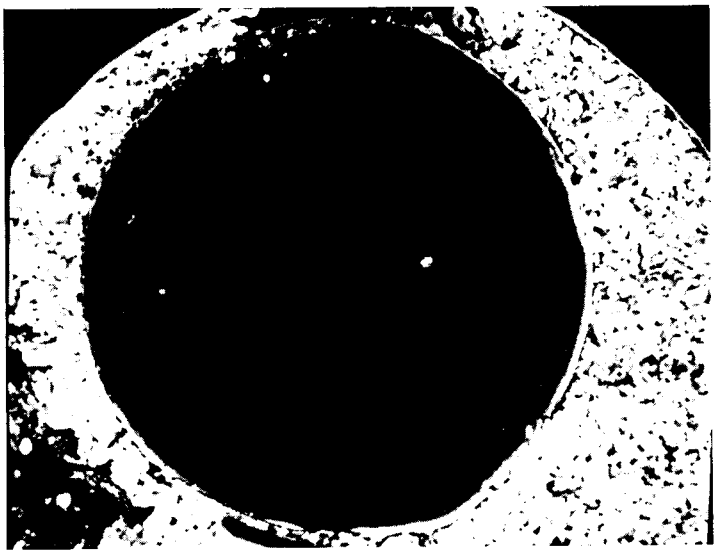

Figure 1 Scanning electron microscope view of multimode fibre cross-section for core diameter of 85 μm and cladding thickness 10 μm.

measuring the amount of transmitted light. Over lengths of a few hundred metres the diameter varies by less than 1 μm and is generally due to taper in the preform. A typical 150 μm fibre taper is ± 2 μm over 1 km and could easily be corrected for during pulling, if necessary, by introducing a control system to vary the ratio of drum to preform speed. The circularity and concentricity of core and cladding are also excellent.

During the pulling process it is possible for the more volatile of the glass components to be evaporated in the hot zone of the furnace and to condense on the cooler glass higher up in the preform. This sometimes shows up as a white deposit on the boule remaining at the end of the pull. The interface is the part most badly affected by this volatilization which results in a higher fibre attenuation. Care must therefore be taken not to pull at too high a temperature which can also cause interdiffusion between the two glasses. The amount of diffusion in our case is less than 1 μm as shown by the electron micrograph in Fig. 1.

Degradation of the fibre can also arise due to devitrification but this can be minimized by operating at a low temperature and designing the temperature profile so that the fibre is cooled rapidly on leaving the hot zone.

Hollow fibres for the production of liquid-core fibres, require only a hollow tube preform and in this case, for a given furnace temperature, there is a minimum pulling speed below which the hole size falls to zero and a solid fibre results. Above this speed, which depends on a number of factors including type of glass and dimensions of preforms, the area of the fibre bore increases linearly with speed giving a gradually increasing ratio of bore to overall diameter. This ratio is an important parameter and can be optimized for minimum filling time as described in Appendix 1.

3. Preparation of preforms

Originally it was thought that in order to obtain a good interface between core and cladding which was smooth and free of scattering it would be necessary for the rod and inner tube surfaces to be highly polished. It seemed to the authors that this would be a very difficult condition to meet without introducing impurities at the interface. In fact the opposite approach has been taken of primarily ensuring cleanliness and, contrary to general belief, the surface finish of the preform interface is not important as each irregularity is smoothed by surface-tension-induced viscous flow and stretched by $\sim 10^4$ during the drawing process. By delaying the point of closure between core and cladding the surface tension smoothing effect may be increased resulting in an improved interface. For this reason and for ease of cleaning, as well as to avoid introducing scratches when making up the preform, a large gap is left between rod and tube. A suitable cleaning process has been evolved with various stages of detergent, solvent and acid soaks. This is followed by careful drying and baking to remove adsorbed gases. A similar process is carried out with tube preforms.

4. Results

4.1. Cladded-glass multimode fibres

We have pulled two main types of cladded fibre, namely those made from (i) commercially-available glass materials and (ii) preforms drawn from a special glass melt and supplied by the University of Sheffield [5]. In the former case a range of combinations has been investigated but the most interesting results have been obtained with F7 core glass and ME1 cladding. We have reduced the attenuation with this combination to 150 dB km^{-1} which is the lowest yet reported for commercially-available materials and we believe it to be limited by losses in the F7 starting material.

It is thought that this improvement has been brought about by paying attention to the following three factors in particular. Firstly, the high degree of stability in the drawing process and the prevention of gross defects such as air bubbles, has reduced scattering, particularly in the interface, to a very low level. Measurements have shown [2] that the total fibre scattering loss is less than 8 dB km^{-1} at 0.9 μm and is comparable with that in the bulk glass. Secondly, adequate cleaning has greatly reduced the amount of interface contamination. Thirdly, by drawing at a low temperature, volatilization of glass components and diffusion of impurities between core and cladding, and from the interface, has been minimized.

An attenuation curve for a fibre with a core of F7 is given in Fig. 2 and shows the typical characteristic of a broad minimum from 0.75 μm to longer wavelengths and a rise towards shorter wavelengths. Superimposed on this general shape are two peaks, a strong broad one centred at about 0.64 μm and a smaller one at 0.97 μm. The former is attributed to chromium ion impurity and the latter to the OH radical. Other common impurities which can be troublesome in glass are copper, iron, nickel, manganese and platinum and it is likely that a mixture of these, and others, gives rise to the broad minimum in the curves. The rise at short wavelengths may be due to the u.v. absorption edge arising from transitions associated with the Pb^{2+} ions in this lead-based glass, although a similar effect can be produced by Fe^{3+} ions. Using a Perkin-Elmer, type

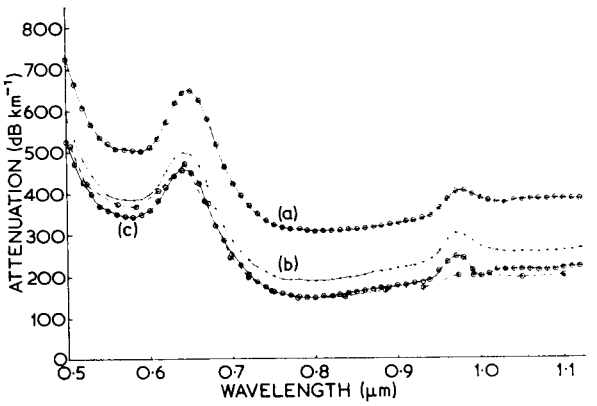

Figure 2 Comparison of attenuation of F7 multimode fibre with that of bulk F7 glass. Curves (a), (b) and (c) are for the fibre with heat treatment at 460° C for (a) 0 h, (b) 2.5 h, (c) 8.5 h while the dashed curve is for bulk F7 glass.

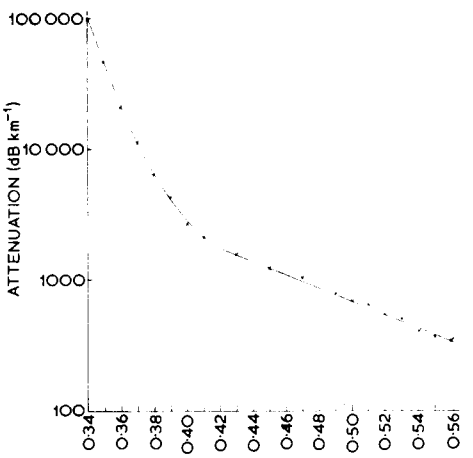

Figure 3 Attenuation of F2 glass over the wavelength range 0.34 to 0.56 μm.

EPS-3T, spectrophotometer the measurements have been extended to a wavelength of 0.34 μm with a sample of Schott F2 glass, which is very similar to F7, as shown in Fig. 3. This result agrees closely with that obtained by Stroud [3] for a glass of composition $Na_2O.SiO_2.2PbO$ and who claims that the short wavelength absorption is characteristic of the glass and not of impurities. Further, a similar rise is present in the Sheffield University glass which has been produced under pure conditions [5] and has a very low iron content.

As well as impurities the thermal history of a fibre can have an important influence on the attenuation as indicated in Fig. 2. The upper curve shows that when first drawn a particular fibre had a minimum attenuation of 300 dB km^{-1} but after soaking for 2.5 h at a temperature of 460° C the attenuation fell to 150 dB km^{-1}. What is interesting is that more or less the same drop in attenuation of 150 dB km^{-1} occurred over the

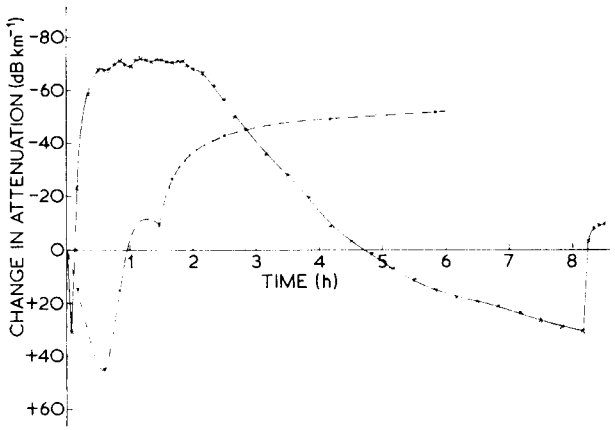

Figure 4 Change of attenuation at 0.8 μm for an F7 core fibre at a temperature of 460° C. The dashed curve is for a Sheffield University [5] fibre at 390° C and the time scale has been expanded by a factor of 10, i.e. the total time occupied by the dashed curve is 35 min.

entire spectral range of 0.5 to 1.1 μm. Further heat treatment for another 6 h caused an increase in attenuation. Also shown in Fig. 2 is an absorption curve for the best sample of bulk F7 glass rod measured in these laboratories by J. P. Dakin [6], which coincides very closely with the best fibre curve.

Two conclusions can be drawn from this result. Firstly that it is possible to draw fibre with the same attenuation as the bulk glass from which it is made, implying in turn that the interface between core and cladding is good and that there is little diffusion of the lossy (in our case) cladding into the core. Secondly, since heat treatment does not affect the general shape, but mainly the level, of the attenuation curves that the similar variation in the corresponding curves for different samples of F7 glass* may be, in part, due to varying thermal histories. If this is so then it may be possible to obtain lower loss glass by more careful thermal processing.

We have investigated this effect further by monitoring continuously the attenuation of various fibres while they were undergoing treatment and a typical result is shown in Fig. 4. This shows that when a particular fibre with an F7 core was put in a furnace at 460° C the attenuation, measured at a wavelength of 0.8 μm, first increased by 30 dB km^{-1} over a period of 5 min, then fell by 100 dB km^{-1} over a further 30 min. The attenuation remained constant for a further hour or so and then gradually increased over a period of 6 h by 100 dB km^{-1}. When, after a total period of 8 h, the test was terminated, the attenuation fell by 40 dB km^{-1}. Experiments with a range of temperatures, times and fibres have shown that the initial increase in attenuation is reversible and corresponds to that recovered when the fibre is removed from the furnace. The intermediate change is irreversible and comprises initially a fall in attenuation followed by a subsequent rise. Obviously in order to obtain a minimum final fibre attenuation it is necessary to monitor the change in loss and to remove the fibre from the furnace after the appropriate interval. This has been done for the fibre represented by the dashed curve in Fig. 4

*The fibre and bulk glass attenuations were measured by quite different techniques.

which again shows all the features described above but in this case the fibre was removed from the furnace before any appreciable permanent degradation was allowed to occur.

The three effects involved, namely the reversible increase in loss, the irreversible decrease and the irreversible increase in loss can be commented on as follows. The initial, relatively rapid and reversible, increase may be attributed simply to the change in temperature of the fibre which causes a broadening, and an increase in intensity, of the absorption bands of the impurity ions [4]. This will cause an increase in attenuation if the u.v. and infra-red bands are broadened towards the visible and also if the 'background' loss is due to a mixture of impurities. The reverse effect has been observed by cooling the fibre to liquid nitrogen temperature. The time constant of the process is simply related to the thermal mass of the fibre.

The irreversible *decrease* in fibre attenuation is superimposed on the reversible change and has a time constant which is strongly dependent on the temperature. It probably results from a stabilization of the glass structure to a form corresponding to a lower temperature than that frozen in by rapid fibre cooling (to a lower fictive temperature). Two effects may occur, namely a change in the ionic environment in the glass and a change in the oxidation state of an impurity ion. A well-known illustration of the first effect is that the colour of glass may be changed by heating [4]. The colour change can often be frozen in by quench cooling when the apparent high-temperature environment, as seen by the impurity ion, is preserved. The high-temperature structure can then be released by annealing and this could well be happening when the fibres are heat treated. The second effect, namely a change in oxidation state, may result from the high-temperature equilibrium ratio of two oxidation states of an impurity ion being frozen in by rapid cooling and relaxing to a lower temperature equilibrium during annealing.

The irreversible *increase* in attenuation appears to be due to some other mechanism such as diffusion of impurities from the cladding, phase separation or devitrification, etc. Again the time constant is strongly dependent on temperature and, in particular, at a sufficiently low annealing temperature the effect is absent, i.e. the heat treatment can be continued indefinitely after maximum improvement has been obtained without such degradation occurring. A careful choice of annealing temperature is therefore necessary.

The second type of preform we have used has been supplied by H. Rawson and B. Scott of the Department of Glass Technology, University of Sheffield. They avoid the

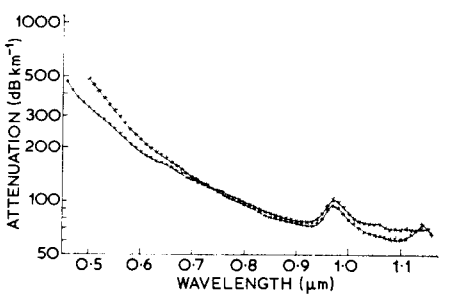

Figure 5 Attenuation of fibre made from Sheffield University glass [5]. The lower curve was obtained after heat treatment.

possibility of introducing impurities by drawing the preform directly from a two-layer melt [5] in which pure glass raw material is heated by R.F. induction thereby greatly reducing the transfer of impurities from the crucible into the glass. Fibres have been pulled from the preform as described above and typical results are shown in Fig. 5. The minimum attenuation achieved is 60 dB km^{-1} near 1.1 μm and again the effect of heat treatment is important. The only distinct absorption peak is that due to OH at 0.97 μm and the rise towards shorter wavelengths may possibly, as indicated above, be due to the tail of the u.v. absorption of the lead-based core glass. Considering the fact that the preform was one of the first prepared by this new method the results are very encouraging and even better results should soon be forthcoming.

4.2. Liquid-core multimode fibres

Using the techniques described in Sections 2 and 3 we have drawn fibre tubes of ME1 glass which have subsequently been filled with hexachlorobuta-1,3-diene. The detailed properties of this combination have been described elsewhere [7] and we are concerned here only with those properties which reflect on the quality of the fibre. By using simple purification techniques the minimum attenuation has now been nearly halved to 5.8 dB km^{-1} and Fig. 6 shows some small residual absorption peaks which may be attributed to

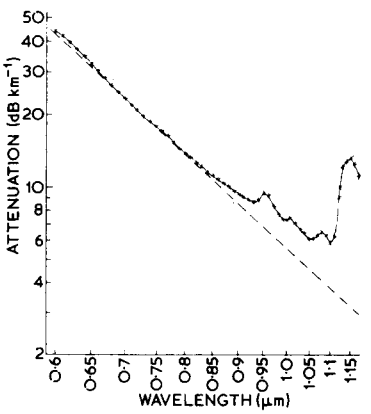

Figure 6 Attenuation of liquid-core fibre of length 600 m and core diameter 95 μm. The straight line has a slope of λ^{-4}.

overtones and combinations of C-H and O-H impurity vibrations. Over most of the wavelength range the attenuation follows a λ^{-4} dependence on wavelength as shown and thus may be assumed to arise mainly from Rayleigh scattering. The excess loss for wavelengths in the region 0.85 to 1.15 μm is due to impurities together with a contribution of between 1 and 2 dB km^{-1} from the lossy cladding (ME1 tubing). By purifying the liquid and using low-loss cladding, such as silica, it should be possible to reduce the loss at, say, 1.06 μm to 4.5 dB km^{-1} and at 0.9 μm to 8.5 dB km^{-1}.

The interface in these fibres, i.e. the inner surface of the fibre tube, seems to be excellent despite the high pressures (up to 1400 atmospheres) [7] under which they are filled and we have not yet observed any effects which can be attributed to interface

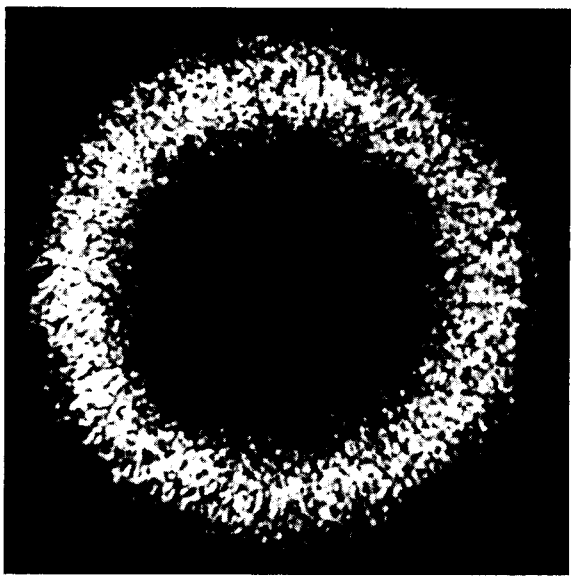

Figure 7 Far-field output distribution of 400 m liquid-core fibre for a narrow input beam at an angle of incidence of 20°. The core diameter was 96 μm and the fibre was wound on a drum of radius 25 cm.

imperfections. Thus the mode conversion (ray scattering) which has been demonstrated [8] seems to be a function only of the bend radius of a fibre and we have observed no limiting amount of mode conversion such as would be expected from scattering or inhomogeneities. This is a valid conclusion to draw despite the fact that the lossy cladding also produces [9] a mode filtering effect (i.e. attenuates preferentially the higher angle rays). A more direct demonstration of this aspect of fibre quality is given in Fig. 7 which shows the far-field distribution of light from a 400 m length where a 'probe' beam of solid semi-angle 0.3° was launched at an angle of 20° (in air) to the axis. Over this length the probe beam makes $\sim 10^6$ reflections with the interface but nevertheless the mean angle of the output rays was still 20°. Some mode conversion occurred since the angular width of the ring is greater than 0.3° but this seems to be due to the curvature of the fibre on the supporting drum. The absence of light at small angles to the axis at the output is a clear indication of good fibre quality. For the shorter lengths of glass core (F7) fibre we have been able to measure, a geometric ray analysis also holds [10] indicating good homogeneity in this case as well.

5. Conclusions

We may conclude that the preform technique can be used to draw fibres of high geometric quality and without necessarily introducing any impurities additional to those already present in the preform. In the case of glass-core fibres the transmission loss can sometimes be reduced by appropriate heat treatment.

Acknowledgements

We are indebted to Mr H. Rawson and Dr B. Scott of the Department of Glass Tech-

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Appendix 1. Filling of liquid-core fibres

Fluid flow in a hollow glass fibre is described by the classical equations for laminar flow in small-bore pipes. The volume rate of flow Q of liquid in a fibre of internal diameter d is given by

$$Q = \frac{\pi(d/2)^2 P}{8l\mu} , \tag{1}$$

where P is the applied pressure, μ is the coefficient of viscosity of the liquid and l is the length of the fibre containing liquid.

The mean velocity v of fluid flow is

$$v = \frac{Q}{\pi(d/2)^2} = \frac{P d^2}{32\mu l} . \tag{2}$$

It can be seen that the velocity varies inversely with the length of the fibre, so that the liquid movement becomes progressively slower, as expected, as the fibre is filled.

The time T taken to fill a length of fibre L is

$$T = \int_0^L \left(\frac{1}{v}\right) dl = \int_0^L \frac{32\mu l}{P d^2} \, \mathrm{d}l = \frac{16\mu}{P}\left(\frac{L}{d}\right)^2 . \tag{3}$$

In order to reduce the filling time for a given applied pressure the internal fibre diameter d should be as large as possible. A kilometre of fibre of 100 μm bore could be filled with hexachlorobutadiene (viscosity 32×10^{-3} poise at 25°C) in 29 h using a pressure of 5×10^7 N m^{-2} – approximately 500 atmospheres – whereas a single-mode fibre of 1 μm bore would take 10^4 times longer.

The maximum applicable pressure is limited by rupture of the fibre. This pressure is given approximately by

$$P_{\max} = \frac{S(D - d)}{D} , \tag{4}$$

where D is the overall diameter, and S is the tensile strength, of the glass. S has a value of 10^8 to 10^9 N m^{-2}, depending on the glass and the surface condition of the fibre. Substitution of $P_{\max}$ into Equation 3 gives the shortest possible filling time for a fibre of given dimensions.

$$T_{\min} = \frac{16\mu L^2 D}{S d^2(D - d)} . \tag{5}$$

However, in practice, the overall diameter of the fibre is limited by flexibility considerations, while the internal diameter may be varied by the fibre-pulling technique outlined earlier. In this case there is an optimum bore diameter for minimum filling time, found by differentiating [5], namely

$$d = 2D/3 .$$

If this requirement is met by suitable choice of pulling conditions, then a pressure equal to one-third of the tensile strength of the glass may be applied before rupture occurs, and the filling time will be given by

$$T_{\mathrm{OPT}} = \frac{108\mu}{S} (L/D)^2 .$$

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