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Transmission measurements in optical glass with an improved twin-beam spectrophotometer

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Improvements are described to a twin-beam spectrophotometer for measuring the bulk attenuation of optical quality glass over the wavelength range 0.4 to 1.1 μm. Results are presented for a number of glasses and a comparison is shown between the attenuation of a multimode fibre with a core of F7 glass and various samples of F7 rods.

1. Introduction

One of the major requirements of a glass or other suitable optically 'transparent' material, if it is to be used to make fibre waveguide for an optical communications system, is an adequately low absorption coefficient. For economic reasons transmission losses should be less than 10^{-4} cm^{-1} and perhaps lower depending on the particular application. The measurement of such losses in fibres of reasonable length is straightforward but in order to determine whether additional losses are introduced during the fibre-drawing process and to assess the quality of the starting material then measurements must also be made on the bulk glass itself. In general two types of technique have been used.

In the first of these, the so-called thermal methods [1-3], the temperature rise in the sample due to the absorption of energy from a transmitted beam is measured. In order to produce an adequate rate of temperature rise, particularly in low loss samples, a high-power optical source is required. Normally a laser is used [2, 3] with the result that measurements can only be made at spot wavelengths, none of which are at the wavelength of interest for optical communications [4], namely 0.9 μm.

The other technique [5-7] involves measuring the transmission loss of radiation passing through the sample. By using a white-light source continuous spectral measurements are possible, as will be reported below. The difficulty is that even with transmission paths as long as 30 cm, which require adequate uniformity of the sample, the attenuation to be measured, which may be as low as 0.1%, occurs in the presence of reflection and scattering losses at the entry and exit faces of 8% or more. In principle the effects of the end faces may be allowed for by comparing samples of two different lengths providing that the end surfaces have identical properties.

In a previous publication [7] a comparison was given of single-beam and double-beam

20

spectrophotometers together with a description of a particular version of the latter instrument and the results obtained with it. Since that time the instrument has been operating successfully and further improvements have been made which are described in the following notes. In addition results are reported on additional samples of glass and, for F7, a comparison is made between losses achieved in bulk and fibre forms.

2. System and improvements

The basic form of the spectrophotometer is similar to that described in detail earlier [7] and will not be repeated here. Briefly a collimated beam from a white-light source is split into two parts which are interrupted alternately by a chopper disc and pass through the two samples of unequal lengths. In the original version the beams were then recombined and allowed to fall on a single detector. In performing a measurement, attenuators consisting of contra-rotating silica flats in each beam were adjusted to give a zero output signal. The samples were then inserted and the out-of-balance square-wave signal on the detector was a measure of the differential loss between them. Subsequent improvements are as follows.

2.1. Spatial variation of detector sensitivity

When the samples are inserted, particularly the long one, the optical path length between the source and detector is increased. Since the beams have a finite, although small, divergence the spot sizes on the detector surface(s) change slightly. If the spatial variation of detector surface sensitivity is not zero then some measurement error results. This error has been reduced by a factor of ten as a result of an extensive search among various types and samples of detector involving the construction of an automatic scanning and recording equipment. With a particular EMI SPD1 detector the total spatial variation of sensitivity at a wavelength of 0.9 μm is 0.02 % over a distance of 3.5 mm in one direction and 0.05 % orthogonally over 4.5 mm for the 3 mm beam diameter. As can be seen from Fig. 1 the variation in sensitivity for smaller changes in beam position and diameter is negligible.

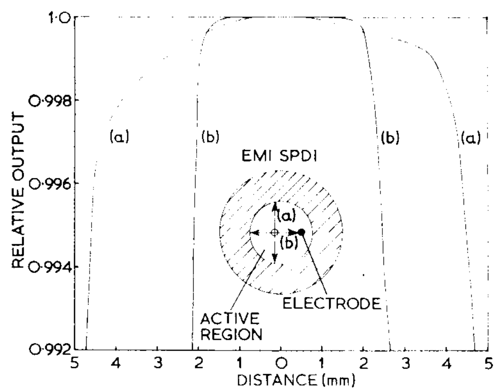

Figure 1 Variation in detector output as a 3 mm diameter spot is traversed across the surface.

2.2. Effect of birefringence in the rods

The transmission of the beam splitter, and of the beam recombiner, is sensitive to the state of polarization of the transmitted light, as were the original attenuators since the contra-rotating silica flats were pivoted about parallel axes. Errors can thus be introduced if there is any birefringence in the rods. Initially all samples were thoroughly annealed before the ends were cut and polished but sometimes there could still be sufficient residual strain to cause concern. Since cutting and polishing is an expensive and time-consuming operation it was undesirable to carry out further annealing and thus the system was made less dependent on polarization.

Firstly only one attenuator is now used and it has been modified so that each flat of the pair rotates about orthogonal axes. This results in a small net displacement of the reference beam in which it is placed but this does not affect the measurements in any way or cause any inconvenience. Secondly, the beam recombiner has been dispensed with by using a separate detector in each beam and electronic comparison of the beam intensities. This also obviates the need for the two plane mirrors (see Fig. 1 of reference [7]). Finally the polarization in the two beams immediately following the beam splitter is, in effect, randomized by the insertion of calcite crystals with their axes at an angle of 45° to the sides of the beam-splitter cube. Thus whereas initially the split beam could be linearly polarized by as much as 60% the calcite crystals reduce the figure to only 0.5%. Together with the removal and modification of the other components the sensitivity of the instrument to birefringence has been reduced by a factor of 10^4 and the maximum measurement error for any sample is now < 1 dB km^{-1}. An additional bonus is that the alignment and setting-up problems are greatly eased.

2.3. Use of two detectors

A further advantage of the new arrangement is that the total light transmitted is increased threefold which results in an improved signal/noise ratio. Furthermore the long and short samples can now be placed sequentially in the same beam with the other retained as a reference. Hence, instead of having to select two detectors on the basis of a compromise between noise performance and spatial sensitivity variations one can be chosen solely on the former criterion and the other on the latter.

In using two detectors care has to be taken to ensure that there is no appreciable change in relative response over the period of the measurement. For the detectors chosen the output changes by less than 1 part in 10^4 for a differential temperature change of 0.1 °C and in practice no trouble has been experienced due to relative drift in detector performance although provision for fluid circulation through the brass detector mounts has been made. Obviously the detecting system has to be balanced at each wavelength of measurement but this must be done in any case because of the variation in the characteristics of the beam splitter with wavelength.

2.4. Phase-locked chopper motor

In the earlier system the synchronous chopper motor and the phase-sensitive detector were driven by a master oscillator. While the operation was satisfactory it required a certain time for phase stability between the two to be achieved due to temperature changes on warming up. A new circuit was therefore devised and installed by E. S. Radclyffe [8] in which the phase of the chopper motor is locked to the master oscillator. Any phase drift is thus quickly corrected by a feedback path via a power amplifier giving greater stability and with the result that the sensitivity of the system to imbalance is now equivalent to a loss of only 1 dB km^{-1} in a sample of length 30 cm.

2.5. New light source arrangement

It has been found that errors can occur due to slight wedge effects in the interference filters. These tend to misalign the beam by an amount which depends on variations in the various filters used. This is obviously unsatisfactory and the light source was therefore modified as shown in Fig. 2.

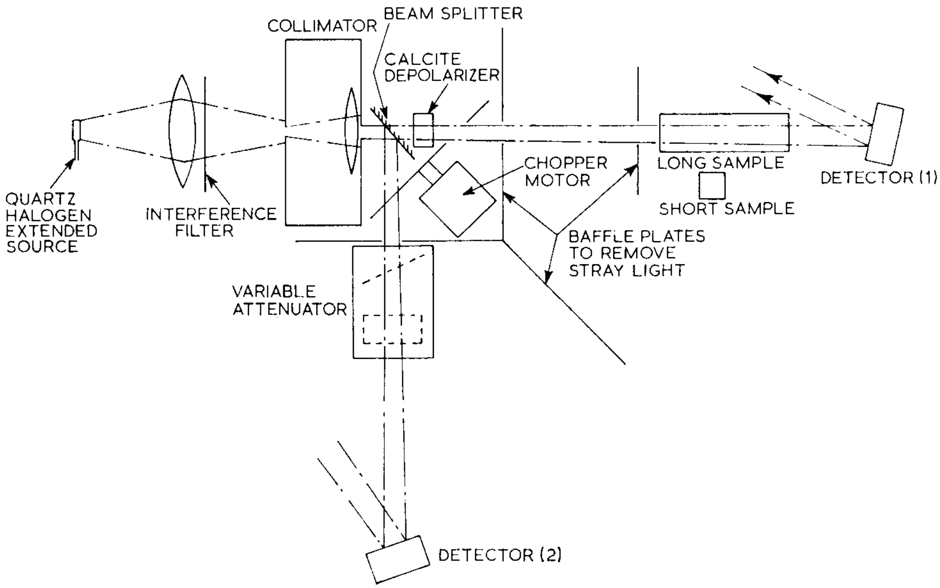

Figure 2 Schematic arrangement of optical system.

2.6. Forward-scattered stray light

Despite care being taken in collimating the beam there always tends to be some light at small angles to the axis caused by scattering at the various surfaces, especially in the beam splitter. To some extent the effect can be nullified by suitably-placed apertures. However, internal reflection of this scattered light from the surface of the rods, particularly the long one, can cause an anomalously high reading to be indicated on the detector. Although errors of up to 50 dB km^{-1} can be caused in this way the presence of the effect is easily observed by replacing the long rod by a tube of the same internal diameter and length when an apparent gain is observed. A simple and successful cure is to roughen the external surface of the rods and apply a matt black coating. An alternative technique would be to immerse the rods in index-matching liquid inside a surrounding blackened cylinder.

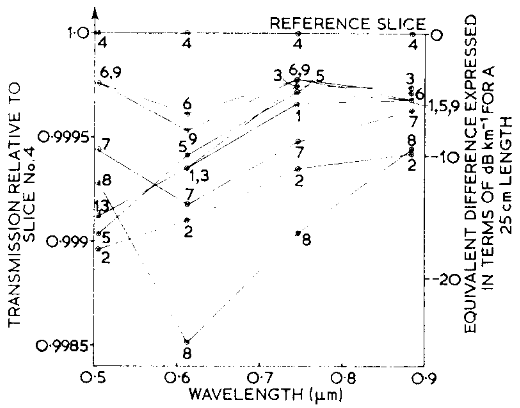

Figure 3 Relative transmission at four wavelengths of nine thin slices of F7 glass after pitch polishing.

2.7. Surface losses of samples

The accuracy of the method depends on the care with which the ends are polished and all the samples to be used in one set of measurements must be polished together in the same operation, see for example Fig. 6. Surface films of different refractive index can be studied by ellipsometry but surface absorption and scattering are more difficult to evaluate. We have therefore taken a batch of nine thin slices of one rod of glass (Schott F7) and, after pitch polishing, measured their relative transmissions at four wavelengths. The results are shown in Fig. 3 and are expressed as relative errors which would be incurred in a measurement with a differential length of 25 cm between long and short samples. The transmission is quoted relative to that of the best sample, (iv). It may be seen that the spread between the samples is relatively small and that near the semiconductor laser wavelength of 0.9 μm the total spread over six of the samples is equivalent to a loss error of only 1.8 dB km^{-1} while the total rms spread is 2.7 dB km^{-1}. The rms values at wavelengths 0.505, 0.61 and 0.746 μm are 6.1, 6.3 and 4.6 dB km^{-1} respectively, again expressed in terms of a difference in length between long and short samples of 25 cm. The polishing technique used at the time of the measurements was experimental and it is thought that even better results can now be achieved. These measurements therefore show that if samples are polished carefully, and in the same operation, then the errors due to differences in end reflectivities can be made small.

A measure of the amount of low-angle (5°) scattering from the surface of the samples was obtained by placing a detector of 1 cm diameter at a fixed position 20 cm from each slice in turn. The variations in the intensity of scattering between the samples correlated well with the differences in transmission indicating that the transmission loss is probably dominated by surface scattering. It turns out, conveniently, that the poorer samples can be easily detected by the naked eye.

3. Assessment of spectrophotometer

The linearity of the system has been checked by inserting first one, then two, silica flats into the measuring beam. This corresponds to a differential loss between beams of

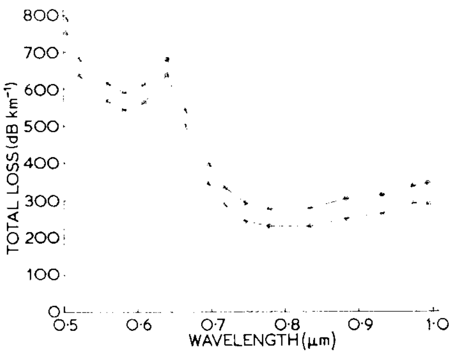

Figure 4 Attenuation of F7 rods (sample i) showing the effect of roughening and blackening of the cylindrical surfaces. The rods were of 1 cm diameter and of lengths 4.75 and 32.3 cm. The solid and dashed lines are after, and before, blackening respectively.

about 6.7 and 13%, the precise values depending on the wavelength. Over this range the system was linear to 2%. The fact that consistent results were obtained with the long and short rods, (Fig. 6), confirms as expected, that the linearity is satisfactory at low levels also.

A typical measurement for a particular pair of Schott F7 rods, sample (i), of diameter 1 cm and lengths 4.75 and 32.3 cm is shown in Fig. 4. The lower curve was obtained before the rods were roughened and blackened and the effect of reflection of forward-scattered light at the internal surfaces can be clearly seen. The repeatability of the measurements is indicated by Fig. 5 where the three solid curves are for another pair of

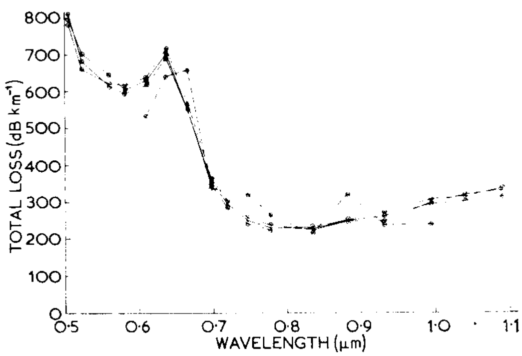

Figure 5 Attenuation of F7 rods (sample iv). The three solid curves were obtained (a) after initial polishing; (b) after removal and replacement in the instrument; (c) after removal, re-polishing and replacing in the instrument. The dashed curve represents preliminary measurements with a thermal loss method for the same sample of F7 glass.

F7 rods, sample (iv). For the three sets of measurements the rods were measured; then removed, re-inserted and re-aligned; then removed, the ends re-polished and re-inserted. It is seen that the shapes of the curves are identical and the maximum deviation from the mean is about 8%. The dashed curve is a preliminary result obtained with a thermal

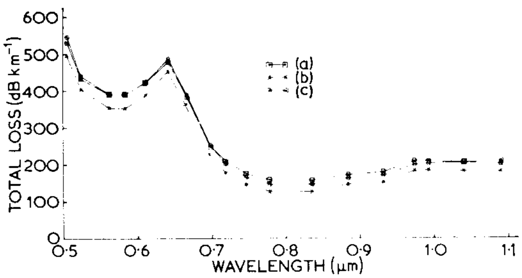

Figure 6 Attenuation of F7 rods (sample iii) of 1 cm diameter. The lengths are as follows: (a) 1.85 and 30.2 cm; (b) 1.85 and 20.8 cm; (c) 1.85 and 12.2 cm. The 20.8 cm rod was polished separately from the others.

loss method. It will not be discussed in detail since the apparatus is still being evaluated but the agreement with the spectrophotometer results is encouraging.

Fig. 6 is for another batch of four F7 rods, sample (iii), of lengths 1.85, 12.25, 20.25 and 30.25 cm. The losses obtained with the longest and shortest pair combinations are in good agreement (maximum deviation from the mean is 3%) but unfortunately the 20.85 cm rod was polished separately from the other three (the polishing is not under our control) and therefore does not give a fair comparison.

Nevertheless the results described show that consistent values of loss can be obtained and that the main remaining source of error, namely variation of end surface losses, can be controlled with care. Relative spectral variations can be accurately obtained simply by changing filters and in this sense the spectrophotometer is complementary to the thermal loss methods using lasers.

It is difficult to give a figure for the absolute accuracy of the instrument, as a comparison with a low-loss sample measured by other workers has not yet been possible. However, from a consideration of the various factors discussed above, the estimated expected errors are indicated in Table I. As with any loss-measuring technique care has to be

TABLE I Estimated accuracy

Wavelength range (μm)	Expected error (dB km^{-1})	Comments
0.4 to 0.5	Up to ± 5% ± 30	Noise limited due to falling light level and sensitivity at short wavelengths.
0.5 to 0.65	± 2% ± 10	Mainly limited to end-loss variations.
0.65 to 0.95	± 2% ± 5	
0.95 to 1.1	± 2% ± 5 rising to ± 2% ± 30 at 1.1 μm	Increasingly dominated by detector spatial variations at long wavelengths.

taken in handling the sample. In the above method, for example, scratching or contamination of the end surfaces could cause errors in single measurements. Ideally, therefore, three readings should be made with each set of samples, including one re-cleaning and one re-polishing (see Fig. 5).

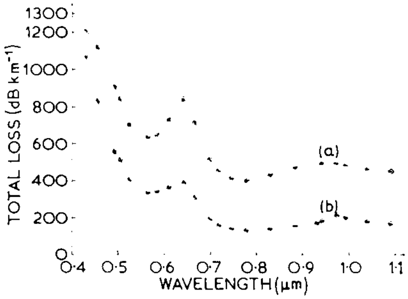

Figure 7 Attenuation of glass rods of lengths as follows: (a) LLF1, 1 cm diameter, lengths 2.0 and 15.0 cm; (b) F2, 1 cm diameter, lengths 4.55 and 42.6 cm.

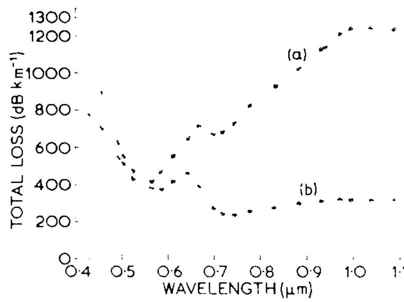

Figure 8 Attenuation of glass rods of lengths as follows: (a) BAK1, 3 cm diameter, lengths 2.06 and 17.4 cm; (b) LF5, 1 cm diameter, lengths 4.55 and 42.9 cm.

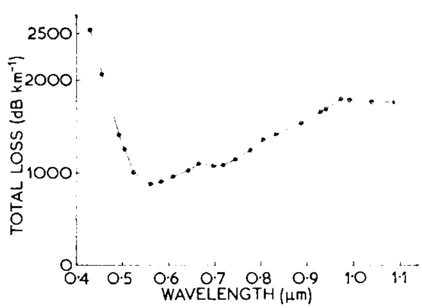

Figure 9 Attenuation of glass rods of SK7 glass of lengths 3.0 and 16.6 cm.

4. Results

Figs. 7, 8 and 9 show loss measurements for three lead-based glasses, namely Schott F2, LF5 and LLF1 together with the barium glass BAK1 and the barium/boron glass SK7. The result for F2 is very similar to that obtained previously [7] while the other curves are new. All the lead-based glasses have a peak at 0.64 μm which is generally attributed to chromium but is not inconsistent with a nickel impurity. None of these glasses has a sufficiently low loss to be suitable for making into optical fibre waveguide without purification but initial results [9] with similar lead-based glasses are certainly

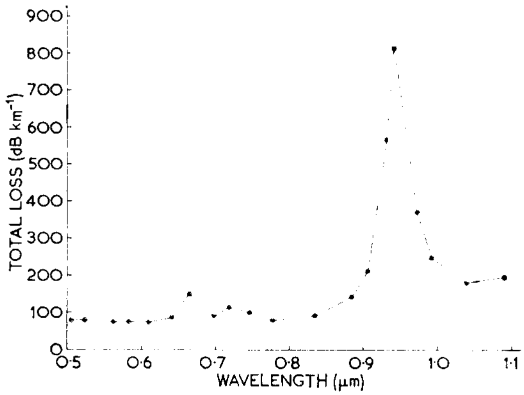

Figure 10 Attenuation of Spectrosil A.

encouraging. A fibre loss of 60 dB km^{-1} has already been achieved and there are sound hopes for further improvement.

The curve for Spectrosil A in Fig. 10 indicates a minimum loss of 75 dB km^{-1} although a much lower attenuation of 4 db km^{-1} is possible in silica [2, 3, 10]. The characteristic peak due to OH has a high attenuation and the wavelength of the maximum is not at the true value since an insufficient number of filters were available in this region.

The variations which are possible between the losses in different samples of ostensibly the same glass is illustrated by the measurements on the three sets of samples of F7 glass. Thus batches (i), (iii) and (iv) have minimum attenuations of 225, 150 and 225 dB km^{-1}, respectively, although the spectral distributions are the same. Fig. 11 shows a comparison

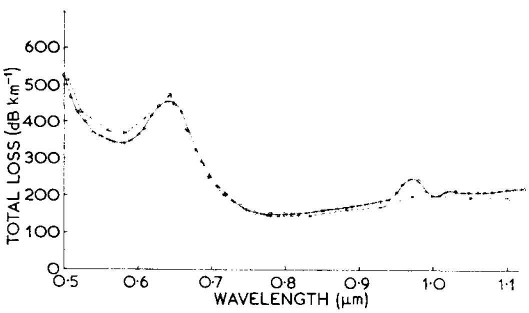

Figure 11 Attenuation of multimode fibre comprising F7 core with ME1 cladding (solid line) and F7 glass rod (sample iii) (dashed line).

between sample (iii) and a fibre drawn in these laboratories comprising an F7 core (although not of batch (iii)) of diameter 60 μm and a cladding of ME1. The F7 rod from which the fibre was drawn was not measured but the spectral distribution of loss, and its value, in bulk glass and fibre are very similar. This indicates that no great additional loss or impurities are introduced during the fibre-drawing process. This conclusion is confirmed by measurements of the scattering loss in bulk glass [11] and fibres [12].

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References

1. H. N. DAGLISH and J. C. NORTH, *New Scientist* **49** (1970) 14-15.
2. T. C. RICH and D. A. PINNOW, *Appl. Phys. Lett.* **20** (1970) 264-266.
3. K. I. WHITE and J. E. MIDWINTER, *Opto-electronics* **5** (1973) 321-332.
4. M. RAMSAY, *ibid* **5** (1973) 261-274.
5. K. C. KAO and T. W. DAVIES, *J. Sci. Instr.* **1** (1968) 1063-1068.
6. M. W. JONES and K. C. KAO, *ibid* **44** (1969) 331-335.
7. P. J. R. LAYBOURN, W. A. GAMBLING, and D. T. JONES, *Opto-electronics* **3** (1971) 137-144.
8. E. S. RADCLYFFE, M.Sc. Project Report, Department of Electronics, University of Southampton, (1971).
9. B. SCOTT and H. RAWSON, *Opto-electronics* **5** (1973) 285-288.
10. R. D. MAURER, paper presented at the 1st European Electro-Optics Conference and Exhibition, Geneva (1972).
11. P. J. R. LAYBOURN, J. P. DAKIN, and W. A. GAMBLING, *Opto-electronics* **2** (1970) 36-42.
12. J. P. DAKIN and W. A. GAMBLING, *Optics Communications* **6** (1972) 235-238.