A Photometer to Measure Light Scattering in Optical Glass

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A photometer is described for the measurement of light-scattering in high-quality, optical bulk glass. The photometer operates continuously over the wavelength range 0.5 to 1 μm and can measure scattering coefficients down to a level of 5×10^{-7} cm^{-1}. A photon counting technique is used for light measurement and calibration is by reflection of the incident beam from an ideal diffusing screen. Measurements with high-purity benzene are in good agreement with computed and previous experimental values. Results of scattering from two samples of optical glass are given.

1. Introduction

Interest in the development of cladded glass fibre optical waveguides for long-distance, wide-bandwidth communication systems has prompted the study of losses in high-quality optical glasses. An economic figure for the overall loss permissible in such a system has been quoted as 20 dB km^{-1}, equivalent to an attenuation of 4.6×10^{-5} cm^{-1}, and it is clear that the raw glass used in the manufacture of the glass fibre guide must have a bulk optical loss below this figure. Without considering the extra losses introduced in fibre drawing, the two sources of loss in the bulk glass may be divided into absorption loss due to ionic absorption bands, and scattering loss due to random density variations, concentration fluctuations characteristic of a frozen solution and inclusions of alien matter. The spectral range of interest in a fibre-optical communication system, where the bulk losses are generally at a minimum, extends from 500 nm to 1 μm. Since Rayleigh-type scattering from small particles decreases rapidly with increasing wavelength, an apparatus was required that could measure the angular scattering distribution within the above wavelength range down to a level equivalent to a scattering loss of 0.2 dB km^{-1} at the long wavelength end of this range. Previous work on light-scattering in glasses, in common with scattering measurements on liquids, appear to have been confined to the visible region of the spectrum, while commercial light-scattering photometers are normally restricted to a few visible emission lines from standard arc sources. The apparatus to be described employs a high-pressure xenon arc, with an output approximating to the emission from a black body at 6000° K, from which a particular narrow wavelength band in the required range can be selected using a multilayer dielectric interference filter. Measurements have also been made at a wavelength of 633 nm using a 1 mW He-Ne laser source.

2. Scattering Photometer

2.1. Optical System

The arrangement of the experimental apparatus used to measure the angular variation of light-scattering by optical glass samples is shown in fig. 1. The glass sample is in the form of a cuboid, 2 cm × 2 cm × 10 cm and is placed in a long rectangular trough. The inside of the trough is blackened, and provided with baffles, to reduce the intensity of stray light. Apertures in the baffles allow a light beam entering at one end of the trough to pass down it, close to one of the sides of the trough. The sample is placed against a rectangular slit 2 cm high by 5 mm wide in the side, through which a region in the centre of the sample is visible. Light scattered from the input beam emerges from the slit, and is received by a detector head rotating horizontally about the vertical axis of the slit, in a semicircular trough. The rectangular and semicircular troughs are

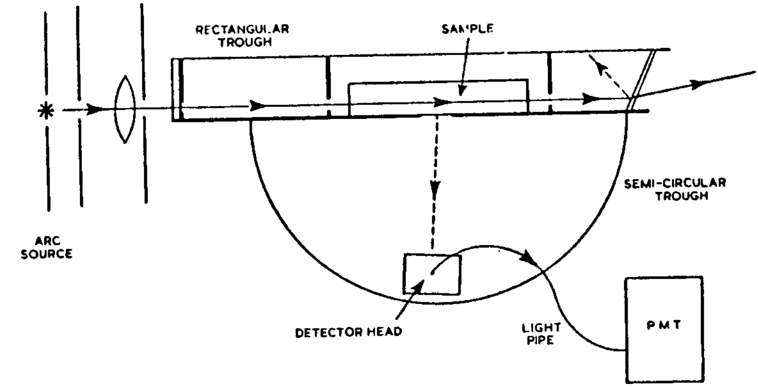

Figure 1 Schematic plan view of the optical parts of the light-scattering photometer.

filled with a refractive-index-matching liquid, the particular liquid used depending on the refractive index of the glass being measured. The ends of the rectangular trough are provided with glass windows; the window remote from the light source is not perpendicular to the beam, so that light reflected from its surfaces does not return to the sample but is diverted and absorbed by the baffles. By using an index-matching liquid, corrections to the results because of refraction are much reduced, and a larger range of scattering angles may be used. Scattering from the ends of the sample is also reduced. The angular range that can be covered is from 20° to 160° to the optical axis and is limited by mechanical factors. The amount of light scattered at any given angle may be compared with the intensity of the transmitted beam, since the total optical loss through the sample will be small, and hence the fractional scattering loss per unit length which occurs at the selected angular direction can be obtained. It was decided not to rely on measurements from known scattering materials, such as benzene, to calibrate the transmitted beam, but to attenuate the beam by a known amount and to measure its intensity with the same detecting system as was used for the scattering measurements.

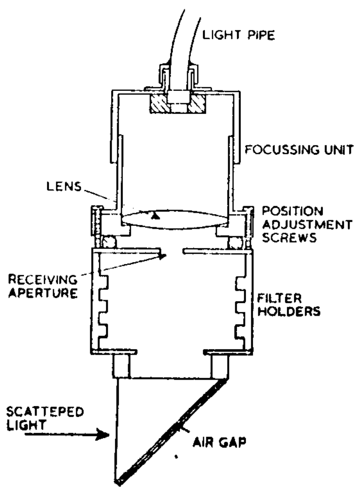

Figure 2 Detector head assembly.

A diagram of the detector head is shown in fig. 2. Light scattered horizontally is reflected upwards, by the prism, through a bank of filter holders and a collimating lens on to the end of a 4.5 mm diameter glass fibre light guide. The position of the input end of the light guide is adjustable, and is arranged so that an image of the circular end of the guide is focused on to, and completely covers, the rectangular slit through which light is scattered. Thus the image of the slit is focused on to the end of the light guide, and is smaller than the guide. This is easily accomplished by shining a light in reverse down the light guide, and observing its image. In practice two identical guides are used, one being

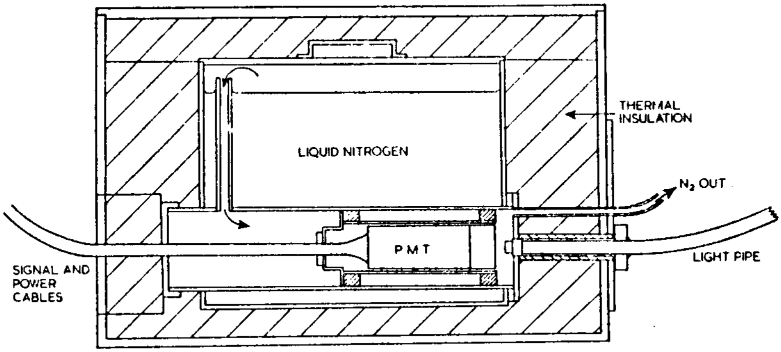

Figure 3 Photomultiplier housing showing cooling arrangements and fibre-optic light guide.

permanently sealed into the photodetector assembly. A machined socket locates the guides in the detector head.

A rectangular aperture in front of the lens determines the angular resolution of the system. The net angle of acceptance is 1° in the horizontal plane and about 4° in the vertical plane. Any divergence of the input light beam coming from the source lowers the system resolution. When a helium-neon laser is used the resolution is not affected. The light beam from the xenon arc is contained within a cylinder 0.5 cm diameter and 30 cm long, and thus diverges by up to about ± 1°.

2.2. Detection System

The scattered light collected by the detector head and focused on to the light pipe is directed on to the cathode of a photomultiplier. A diagram of this arrangement and of the cooled housing is shown in fig. 3. To cover the wavelength range required, an EMI 9684 photomultiplier with an S1 type of photocathode is used, cooled in a liquid nitrogen enclosure to about 100° K to reduce the dark current. The use of a sealed-in light pipe has obviated problems of the misting-up of input windows. Care must be taken to use only the most sensitive part of the photocathode [1]. In view of the low light intensities to be measured, and the small (order of 0.2%) quantum efficiency of the S1 photocathode, it was decided to use a photoelectron-counting technique in preference to the more usual phase-sensitive or synchronous detection. The signal/noise ratio of counting techniques at very low light levels has been found to be as good as, or superior to, the phase-sensitive detection method [2, 3], while long integration times present no problems. The counting system, shown in fig. 4, is simple and relatively inexpensive and the numerous readings of the digital output are easy to record.

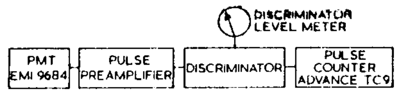

Figure 4 Block diagram of counting system.

The photomultiplier dynode chain is fed from a stabilised EHT supply, and the dynodes give an electron multiplication of about 10^6. Thus a photoelectron gives rise to a charge pulse of 1.6×10^{-13} C at the anode, which has a load resistance of 4.7 kΩ. A valve pre-amplifier is used which has a voltage gain of 15 and has a total input capacitance, including the photomultiplier, of 24 pF so that an average photoelectron produces a 7 mV pulse at the anode, and about 100 mV at the pre-amplifier output.

The pre-amplifier is followed by further amplification and a pulse-height discriminator circuit. Smaller-than-average pulses, possibly originating from field emission in the dynode structure, are gated out by the discriminator, which has a variable gating level. Originally provision was made for the removal of large-amplitude pulses also, which, it has been suggested, might be produced by cosmic rays or natural radioactivity [4], but the frequency of such pulses was so low that the upper-level discrimination was discontinued.

The dark pulse count of the photomultiplier when cooled is expected to be of the order of 100 pulses per second. The discriminator gating level was adjusted, with a small illumination level, for optimum signal/noise ratio when the dark current actually measured was about 15 per second. A count is usually taken over a 10 sec period and is measured by a crystal-controlled oscillator. The amplifying, discriminating and counting circuits together are limited to about 2 MHz maximum rate, and thus about 1% of pulses might be under-recorded, due to co-incidence, at a count rate of about 20 KHz.

3. Sensitivity of Scattering Apparatus

The output beam from the xenon arc is collimated and filtered to pass only a narrow wavelength band about 10 nm wide. The brightest part of the arc is extremely small, and did not completely fill the aperture in front of the arc. On inspecting the magnified image of this aperture, it was seen that no more than a tenth of the source aperture was filled by the bright spot of the arc. Assuming this spot to be a black body radiator at 6000° K, the power in the beam was calculated to be 2×10^{-5} W. Of the scattered light, the detector accepts a solid angle of 1.5×10^{-3} steradian, scattered from an incident beam length of 0.5 cm. A count of 10 photoelectrons per second corresponds to about 2×10^{-16} W incident on the detector head, or 1.3×10^{-13} W scattered per steradian. An isotropic Rayleigh scatterer would direct $3/16\pi$ per steradian of the total scattered power at 90° to an unpolarised input beam, so that in the above example the total scattered power is about 2×10^{-12} W, equivalent to an attenuation by scattering of 2×10^{-7} cm^{-1}, or 0.1 dB km^{-1}.

3.1. Measurement of Intensity of Incident Beam

In order to obtain absolute scattering measurements the power in the input beam must be compared with the scattered power. Since the light passes through a considerable length, about 30 cm, of index-matching liquid, which may absorb 50% of the light, the attenuation of the liquid must be taken into account. The input beam is therefore passed through a trough of the same liquid, the length corresponding to the path length in the apparatus. Corrections for window and sample reflections must be made. The beam is too intense for a direct photoelectron count so known attenuators must be used. So-called neutral density filters had to be discarded, since their attenuation in the infra-red is much less than in the visible. Some attenuation was carried out by reducing the aperture size in the detector head with specially measured apertures, but the beam was mainly attenuated by shining it normally on to a perfect diffusing screen, and viewing the spot on the screen with the detector head from a distance of about 70 cm. Thus it was not necessary to determine additionally the sensitivity of the detection system. The screen used could be coated with a fresh layer of smoked magnesium oxide or a thick coating of barium sulphate paint, or replaced by a disc of pressed barium sulphate powder. Over the wavelength range of interest, the last two types of diffusing screen have an absolute reflectance greater than 0.99 [5] and the angular reflectance of barium sulphate powder is nearly perfect. Fig. 5 shows

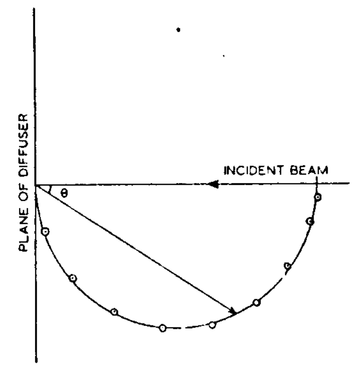

Figure 5 Variation of scattered intensity with θ for pressed barium sulphate diffuser illuminated normally.

the variation of reflected power with angle for barium sulphate. It may be seen that the deviation from a cosine law for an ideal surface is not greater than 2%. After some initial experiments with the other two types of diffuser, the pressed barium sulphate powder diffuser was used for incident beam attenuation.

3.2. Geometrical Factors

The light beam on its passage through the photometer encounters many boundaries between media of different refractive index. Account has to be taken of the effect of these boundaries for two reasons. Firstly a small reflection loss occurs at each boundary, and secondly the geometry of the optical system is altered, affecting acceptance angles and scattering angles.

Rayleigh's ratio $R(\theta)$ may be calculated from the scattering measurements, $k(\theta)$ being defined as the scattered power per unit solid angle per unit sample length viewed at an angle θ to the input beam axis when the sample is illuminated with unit intensity unpolarised light. For a true scattering angle in the sample θ, the detector will in general be set at some different angle θ', and will measure a scattered light count $p(\theta')$. The

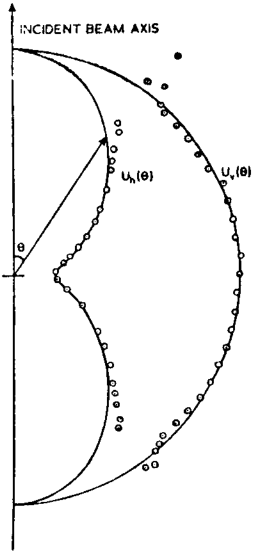

Figure 6 Polar scattering diagram for F7 glass at 633 nm. The points show experimental measurements and the solid curves are theoretical values of $U_v(\theta)$ and $U_h(\theta)$.

reflection losses for scattered light and incident beam measurements may be calculated from the known refractive indices of the various media, and may be combined in a transmission ratio $T(\theta')$. With an incident beam count of P_I, $R(\theta)$ may be written as:

$$R(\theta) = \frac{p(\theta') \, . \, a \, . \, \Delta}{P_I \, . \, \delta\Omega_1 \, . \, l \, . \, T(\theta')}$$

where $\delta\Omega_1$ = acceptance solid angle of scattered

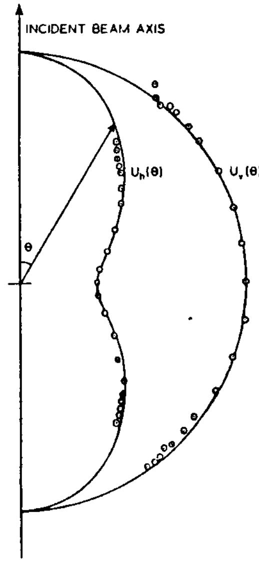

Figure 7 Polar scattering diagram for SSK2 glass at 633 nm. The points show experimental measurements and the solid curves are theoretical values of $U_v(\theta)$ and $U_h(\theta)$.

light, measured in the sample; a = aperture attenuation for incident beam measurements; l = scattering slit width; Δ = diffuser attenuation = $\frac{\delta\Omega_2 \, . \, \epsilon \, . \, \cos\phi}{\pi}$; $\delta\Omega_2$ = acceptance solid angle of detector head, for incident beam measurements; ϵ = reflectance of diffuser; ϕ = angle between viewing direction and normal to diffuser surface.

The attenuation coefficient α due to scattering may be determined by integration of the polar scattering diagram. From a medium whose irregularities are small compared with the wavelength of light, the shape of the scattering diagram may be computed theoretically [6]. Polarising the input beam either vertically or horizontally results in detected scattered intensity components $U_v(\theta)$ and $U_h(\theta)$, where U denotes that no analyser was used at the detector head. It is easily shown that

$$U_h(\theta) = U_h(90) + [U_v(90) - U_h(90)] \cos^2\theta$$

and

$$U_v(\theta) = U_v(90) \, .$$

The depolarisation $\rho(90)$ may be expressed as

$$\frac{U_h(90)}{U_v(90)},$$

and hence α may be expressed [6] as a function of the Rayleigh ratio and the depolarisation perpendicular to the input beam:

$$\alpha = R(90) \cdot \frac{8\pi}{3} \cdot \frac{2 + \rho(90)}{1 + \rho(90)} \cdot$$

4. Results

Initial experiments have been carried out on two samples of optical glass supplied by Schott and Co: a lead flint glass, type F7, and a dense barium crown glass, type SSK2. The polar scattering diagrams for $U_h(\theta)$ and $U_v(\theta)$ were obtained at 633 nm using the He-Ne laser source, and are shown for the respective glasses in figs. 6 and 7. Superimposed on the experimental points are theoretical curves of $U_v(\theta)$ and $U_h(\theta)$ calculated from the depolarisation

$$\rho(90) = \frac{U_h(90)}{U_v(90)}$$

measured perpendicularly to the input beam. The "peaks" at about 37° and 143° are attributable to light scattered from the entry and exit faces of the sample being reflected into the detector from the polished back face of the sample. This is confirmed by the fact that the "peak" angle agrees with geometrical predictions and varies as the sample is moved in the longitudinal direction. In order to eliminate these reflections in later measurements, the backs of the cuboids are to be cut at a suitable angle, leaving rods of approximately triangular section. Nevertheless throughout the angular range over which the photometer can be used the scattering in our samples of F7 and SSK2 glass are within 3% of the ideal Rayleigh curves. Curves of similar shape have also been obtained over the whole wavelength range. It is therefore not unreasonable to compute the total scattering loss by integrating, on the basis of the experimental results at $\theta = 90°$, over a solid angle of 4π assuming a Rayleigh scattering law. Thus the scattered light has been measured at $\theta = 90°$ over the whole wavelength range and the computed variation of scattered power per unit length with wavelength is shown in figs. 8 and 9. It can be seen that for both glass samples the scattered power varies as $\lambda^{-4.5}$. The wavelength of particular interest for fibre optical communication applications is that corresponding to the output of a gallium arsenide semiconductor laser operated at room temperature, namely 900 nm. In this region the bulk scattering loss of the F7 sample is 4.3 dB km^{-1}, and that of the SSK2 sample is 5.0 dB km^{-1}. These figures are considerably less than the total allowable loss. At shorter wavelengths the loss by scattering increases to about 20 dB km^{-1} at 630 nm for the F7 sample.

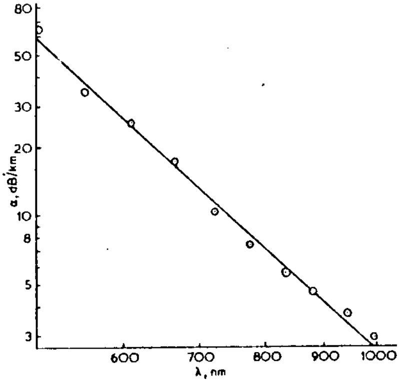

Figure 8 **Measured value of scattering loss coefficient for F7 glass as a function of wavelength.**

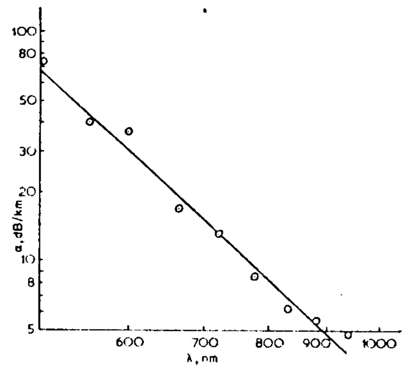

Figure 9 **Measured value of scattering loss coefficient for SSK2 glass as a function of wavelength.**

The performance of the instrument has also been investigated by measurements on two

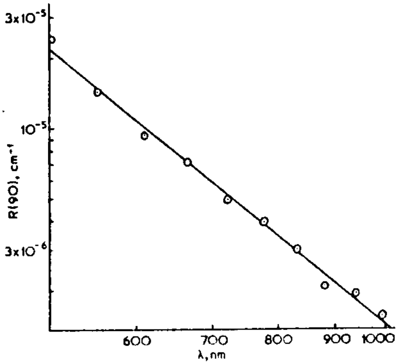

Figure 10 Experimental values of Rayleigh ratio $R(90)$ as a function of wavelength for benzene.

samples of commercial high-purity benzene. Rayleigh's ratio $R(90)$ for benzene is shown as a function of wavelength in fig. 10 and varies as $\lambda^{-4.0}$ which corresponds to ideal Rayleigh-type scattering as might be expected. $R(90)$ has been found by interpolation at 546 nm and compared with some previous calculated and experimental values [7]. For our two samples $R(90) \times 10^5\,\mathrm{cm}^{-1}$ was found to be 1.67 and 1.81 respectively. Values calculated [7] for pure benzene by two different methods are 1.55 and 1.64, while selected previous experimental values [7] are 1.54, 1.58 and 1.60. In view of the measured wavelength-dependence and the difficulty of obtaining sufficiently pure benzene the agreement is satisfactory.

5. Conclusions

A light-scattering photometer has been constructed to measure the scattering loss of optical glass, in the visible and near-infrared region of the spectrum. The instrument has a high sensitivity and the measured signal counts at different wavelengths are in good agreement with those predicted. The accuracy of the values obtained for Rayleigh's ratio in benzene is satisfactory. The main advantage of the instrument is its ability to measure small scattering losses, down to $\sim 5 \times 10^{-7}\,\mathrm{cm}^{-1}$ (0.2 dB km^{-1}), out to 1 μm wavelength without the need for a calibration standard. Measurements on samples of F7 and SSK2 glass indicate Rayleigh-type scattering at a level corresponding to 4.3 and 5.0 dB km^{-1}, respectively, at 900 nm.

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References

1. W. A. GAMBLING and H. EDELS, *Brit. J. Appl. Phys.* 8 (1957) 481-482.
2. J. K. NAKAMURA and S. E. SCHWARZ, *Appl. Optics* 7 (1968) 1073-1078.
3. R. R. ALFANO and N. OCKMAN, *J. Opt. Soc. Am.* 58 (1968) 90-95.
4. G. A. MORTON, *Appl. Optics* 7 (1968) 1-10.
5. F. GRUM and G. W. LUCKEY, *ibid* 7 (1968) 2289-2294.
6. W. H. MARTIN, *Trans. Roy. Soc. Canada* 17 III (1923) 151-155.
7. G. DEŽELIĆ, *J. Chem. Phys.* 45 (1966) 185-191.