Measurement of fictive temperature of silica glass optical fibers

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The fictive temperature, Tf, gives information about the structural state of glass and correlates both to the optical and the mechanical properties [1]. For instance, a low fictive temperature gives low Rayleigh scattering and thus low fiber attenuation[2]. A spectroscopic method [3], where the position of a reflection peak in the infrared spectrum around 1120 cm^{-1} is determined, has been improved and used. The reflection peak is due to the fundamental stretching vibration mode of Si–O bonds. An improved method for determining the peak position is suggested and the radial variation of Tf for some fibers are presented.

The Fourier transform infrared (FTIR)-spectrometer, Equinox 55 from Bruker, was equipped with an infrared microscope Hyperion 2000 having a special small area low noise detector, a motorized sample stage and an aperture for limiting the analyzed area. The fibers were cut perpendicular to the fiber axis using a standard fiber cleaver. The spectral resolution was varied from 0.25 to 4 cm^{-1} with the result that a low resolution (4 cm^{-1}) gives more than 10 times better repeatability in the peak position compared to the highest resolution. This is probably caused by the fact that there is a smaller mechanical movement of the scanning mirror at a lower resolution giving better wavelength stability. Different aperture sizes were tried. For a 10×10 μm aperture and 5 min measurement time the repeatability in the fictive temperature was approx. 1 °C. However, it should be noted that the measured spot on the sample surface is significantly larger than the aperture due to diffraction. The repeatability of the peak position was proportional to the square root of the aperture size and also the square root of the measurement time. For most measurements a resolution of 4 cm^{-1}, aperture size of 10×10 μm and averaging over 5 min were used.

The peak position was determined by making a least-squares polynomial fit and then calculating the point of zero derivative. First, it was found that the sensitivity to noise increases with increasing polynomial degree. Second, it was found that the choice of the discrete data points for the fit is very crucial. For instance, shifting the data points one step toward higher or lower wavelengths changed the fictive temperature as much as 50 °C using a second-order polynomial. As Tf, and thus the peak, shifts in wavelengths it is also necessary to change data points for the fit. Keeping a fixed set of data points is not possible, as it will not, in general, give the true peak position. Increasing the polynomial degree reduces the problem but at the expense of the noise sensitivity. Increasing the resolution gives more noise and results in a larger number of smaller steps. The reason for this problem is that the polynomial does not give a perfect fit to the reflection curve especially for points far from the peak. But by raising the measured reflectivity values to an exponent e, the curve gets a shape that makes a good fit to a low-order polynomial. First, e in Equation 1 is set to a value, then the coefficients A, B and C in Equation 1 is determined by a least-squares fit:

$$R^e = A + B \cdot w + C \cdot w^2 \quad (1)$$

where R is the measured reflectivity and w is the wavelength. The peak wavelength is the root of the derivative of the fitted curve. By changing the set of measurement points to higher and lower wavelengths and recalculating the peak position, the stability of the fit is verified. With an e-value around -3.5 the step was negligible for a wavelength range of ± 25 nm. The step can be both positive and negative and thus also exactly zero. Exponents between -3.5 and -4 all give small steps. By using Equation 1 the peak position can be determined accurately with a good repeatability. The peak position is allowed to vary over a wide range without any discontinuity in the calculated peak position and therefore in the calculated Tf. The repeatability of the peak position increases with the wavelength range, around the peak, up to about ± 25 nm. Larger ranges give only a very minor increase in the repeatability and some interference from a shoulder around 1200 cm^{-1} might occur.

The radial variation of Tf has been studied before with some divergent results [4]. One problem is that the reflection peak position varies not only with Tf but also with the material composition [5]. This is a problem as optical fiber has a different composition in the core and in the cladding surrounding the core. Therefore, homogeneous fibers (without any core) were drawn from a solid silica rod with the same drawing conditions as for the normal fibers drawn. The variation of the peak position along the cross section of the 125 μm-diameter fibers was studied by measuring at every 5 μm. As seen in Fig. 1, there is almost no variation along the diameter of the fiber without core but a strong variation for the normal fiber with a Ge-doped core.

The repeatability of the measurement was checked by performing several scans. Twenty-two points with a 5-μm separation were measured. Aperture size was 10×10 μm and the signal was averaged over 5 min. Thus, each diameter scan took about 2 hr and eight scans were made giving a total measurement time of 16 hr. The relation between Tf and peak position is obtained by measurement of samples heat-treated at well-controlled temperatures [4]. The error bars in Fig. 2

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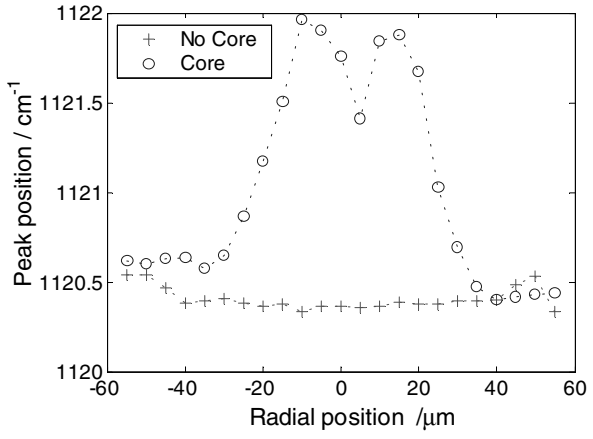

Figure 1 Peak position versus radial position for a fiber with core and a homogeneous fiber without core.

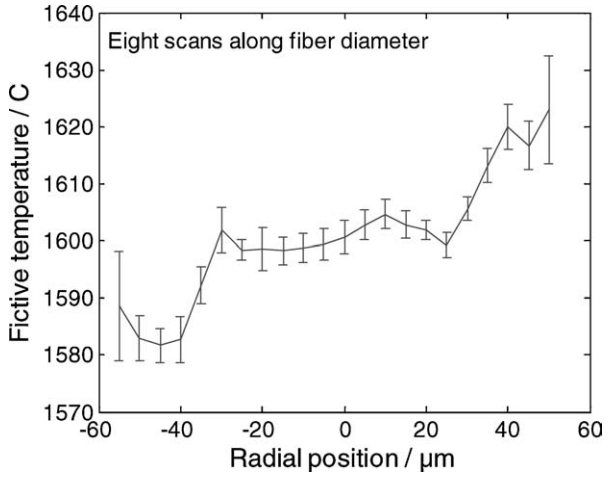

Figure 2 Fictive temperature for a homogeneous fiber along one diameter with error bars calculated from eight measurements at each position.

give one standard deviation calculated for each point. The signal amplitude is smaller and the standard deviation is larger at the periphery of the fiber. There is no significant radial variation but there is a variation from left to right indicating an asymmetry of the fiber. This may be due to an asymmetry during the drawing of the fiber such as an imperfect centering of the fiber in the heating zone. The repeatability was about 10 °C as compared to 1 °C for consecutive measurements of one single spot. Scanning along the diameter means that each point is measured eight times during a 14-hr period and this long duration is probably causing the poorer (but still rather good) repeatability. The conclusion is that there is no detectable radial variation of the fictive temperature.

Fibers were drawn at three different speeds 22, 44 and 82 m/min with and without core, and the fictive temperature was calculated using the results from the outer (undoped) part of the fibers. The results are given in Table I and show an increase in the Tf at increasing drawing speed caused by more rapid cooling of the glass.

The peak width was also calculated and is shown in Fig. 3. The width is almost constant for the homogeneous sample but varies for the normal fiber with variation in composition. This information could thus be used to distinguish between variations due to compositional variation and fictive temperature.

TABLE I Measured fictive temperatures for different drawing speeds

Drawing speed	22 m/min	44 m/min	82 m/min
Normal fibers	1503 °C	1531 °C	1541 °C
No core fibers	1514 °C	1555 °C	1562 °C

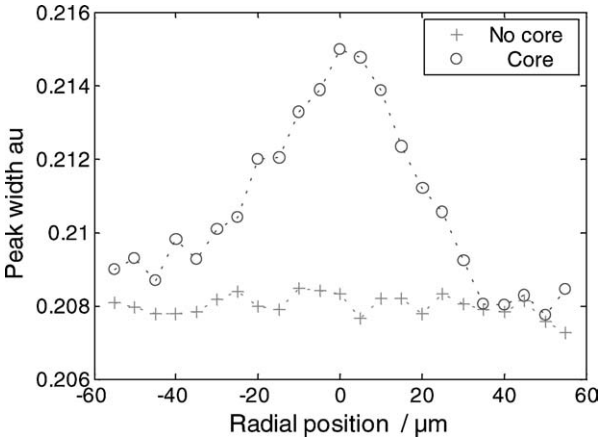

Figure 3 Peak width versus position for a fiber with core and a homogeneous fiber.

The present measurements show that the signal-to-noise ratio is sufficiently high allowing direct measurement on the cleaved fiber without any other sample preparation. The method of cleaving the fiber is simpler than the earlier published method [5] where the fiber was cut at a small angle. An aperture size as small as $10 \times 10\ \mu$m was used giving a repeatability of about 1 °C for consecutive measurements and 5 min of averaging time. Over a 14-hr interval the repeatability was about 10 °C. An improved method for the calculation of the peak position was used. This circumvents the problem with a discontinuity in the peak position as the data points used for the calculation are changed. The radial variation of the peak position was found to be due to the chemical composition of the sample and no radial variation in Tf along the radius was found. The width of the reflection peak was calculated and seems to depend primarily on the material composition of the sample.

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