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The Effect of Annealing on the Properties of Optical Waveguides

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Abstract

The effect of annealing on the optical waveguide characteristics of glass coverslips was studied by investigating the change in Rayleigh scatter intensity between annealed and unannealed samples. It was determined that the annealing process improved the overall quality of the waveguide by reducing the variation of the refractive index along the length of samples.

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Theory

The purpose of this experiment was to study how the optical properties of glass coverslips were affected by annealing. A change in the amount of Rayleigh scatter along the length of the coverslips was chosen as a means of investigating this process.

The reduction of intensity of a light beam as it propagates through in absorbing medium J of thickness dx can be written as:

$$dI = -\alpha [J] / dx, \text{ or } d \ln I = -\alpha [J] dx$$
(1)

where I is intensity, [J] is the concentration of J, and α is the proportionality coefficient. To obtain the intensity that emerges from a sample of thickness x when the incident intensity is I_i one can sum up all of the changes in thickness:

$$\int_{1}^{1} d \ln I = - \int_{0}^{x} a[J] dx$$
 (2)

If the sample is assumed to be homogeneous, then the above equation integrates to the Beer-Lambert Law:

$$I_{f} = I_{i}e^{-a[J]x}$$
(3)

It can be seen that intensity will decrease exponentially with the sample thickness. A more useful form of the Beer-Lambert Law is:

$$\ln \left(I_{\rm f} / I_{\rm j} \right) = -\alpha [{\rm J}] \mathbf{x} \tag{4}$$

A plot of in I_f vs. x with give a linear plot with a slope = $-\alpha[J]$, which is called the molar absorption coefficient, and the y-intercept will equal the initial intensity (1). These relationships can be applied to glass media for they are fairly homogeneous in nature.

The behavior of a light wave as it propagates through the interface between two media with different refractive indices can be described by Snell's Law:

$$n_1 \sin \theta_1 = n_2 \sin \theta_2 \tag{5}$$

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The angle of light propagating inside an optical waveguide can be determined by using Snell's Law after knowing the angle with which the light enters the guide, and its refractive index (2).

The process of annealing glass is an important industrial practice. This process involves a heating followed by a slow cooling of the glass. There are many reasons why glass is annealed. An important one concerning optical samples is the reduction of the spatial variation in the refractive index (3). This will create a more homogeneous sample, and thus a better waveguide. Since light tends to scatter as it encounters a change in refractive index, a sample that has been annealed should show less scatter along its length than a sample that is unannealed.

Experimental

Reagents. Corning glass coverslips were used as the waveguiding medium. These are made of a BK7 type glass, and are approximately 150 microns thick.

Sample Preparation. Before annealing, the samples were cut in half using a diamond tipped cutter. The cut was made so that each sample was of approximately the same length. One of the coverstip halves was annealed in a Thermolye Model 21100 tube furnace, the other was left unannealed as a control. A constant flow of nitrogen gas was passed through the furnace chamber to create an inert atmosphere during the annealing process. Different conditions for annealing were created by varying the temperature and length of time in the furnace. After removal from the furnace, all samples were allowed to cool to room temperature before storage. All of the samples were annealed while standing on edge in the sample tray except for two sets of three. These six were laid flat on top of the sample tray. Upon their removal from the tube furnace, it was noted that the edges of each sample had bowed up. Apparently, some stress had been introduced into the samples due to uneven heating. Therefore, these six were not analyzed. After being annealed, all samples were stored in a dry, nitrogen cabinet until needed.

Analytical. Each sample was mounted and exposed to a 632.8 nm light beam from a Meiles-Griot helium-neon laser. The laser was rated to a maximum output of 10 mW, but a typical beam intensity for this experiment was approximately 4 mW. Before reaching the sample, the beam was passed through a 1/20 focusing iens with a 500 mm focal length. The sample holder itself was mounted on three stages: X and Y transitional and rotational. This allowed the experiment to be repeated easily for all the samples. Each sample was mounted with the incoupling edge at a distance of approximately 1 mm from the inner edge of the sample holder. An optical fiber, with a total exterior diameter of 6 mm, was mounted on a separate transitional stage to detect the scattered light. This fiber was traced along the distance of the sample from 4 mm away from the incoupling edge, to the exit coupling point. The physical limitations of the sample holder's design prevented the movement of the fiber any closer to the incoupling point. Light emitted form the other end of the fiber was passed directly into a Pacific Model 3150 photomultiplier tube. The tube was powered with 500 V from a Fluke Model 409A power supply. The signal detected was passed through a current-to-voltage converter with a sensitivity setting of 5 V output per 1 μ A input. The amplified output went to a Fluke Model 8010A digital multimeter and the intensity of the scattered light was read and recorded in volts.

Each coverslip was mounted at the same point, so the beam entry location was at approximately the same height. Data was recorded starting at 4 mm from the incoupling edge to the exit edge, a total distance of 25 mm, with a collection frequency of every half mm.

Results and Discussion

The angle of propagation of light inside the waveguide can be determined by using equation (5) from above, and simple geometry. Given that $n_{air} = 1.000$, $n_{coverslip} = 1.515$ for i = 632.8 nm, and $\theta_{air} = 23.0^{\circ}$, then $\theta_{in} = 14.9^{\circ}$. Thus the angle of internal reflection inside the guide = $\theta_{guide} = 90^{\circ} - \theta_{in} = 75.1^{\circ}$. This is the angle of propagation of the beam as it travels through the guide. This geometry was held constant for all analyses.

Plots of $\ln I$ vs. x were made for each data set. Figure 1 contains typical plots for a sample that was annealed at 400° C for 10 minutes. The unannealed sample is on top, while the annealed is on the bottom. Linear linefits were used to determine the slopes for each graph, and the resultant equations are included on each. Note the change in the slope between the two samples. The annealed sample shows a 24% decrease in slope when compared to its unannealed half.

A summary of results of all of the samples is given below in Table I. The first column describes the conditions under which the samples were annealed. This includes the temperature and length of heating. Three samples were prepared for each condition, and the mean change in slope between the unannealed and annealed samples is given in column two. This was calculated by taking the slope of the annealed sample and subtracting off the slope of the corresponding unannealed control sample. The average of the three measurements is reported along with the standard deviation.

It can be seen that for every annealing that took place above 400° C, a negative change in slope occured, indicating an improvement in the waveguiding characteristics of the samples. Also, as temperature increases, there is a decreasing trend in the standard deviation. The samples unnealed at 450° C, taken as a group, show the lowest standard deviation of all.



Figure 1. Typical plots constructed for each sample. A linear linefit was used in each case.

Above graph represents an unannealed, control sample. The graph below is of a sample annealed for 10 minutes at 400 C.



Annealing Conditions	Mean Change in Stope(mm ⁺)	Standard Deviation(mm ⁻¹)
450° C for 10 minutes	-0.032	0.039
450° C for 20 minutes	-0.005	0.021
450° C for 30 minutes	-0.014	0.028
400° C for 10 minutes	-0.026	0.059
400° C for 20 minutes	-0.036	0.073
400° C for 30 minutes	-0.012	0.034
350° C for 10 minutes	0.089	0.094
300° C for 10 minutes	0.025	0.028
250° C for 10 minutes	0.022	0.079
200° C for 10 minutes	0.003	0.044

Table I. Summary of Annealing Conditions Used and Results Obtained.

A study was also done to see if the samples were fairly homogeneous across their width. A sample that was annealed at 450° C was chosen and analyzed. Data was collected at different incoupling locations along the edge and the results are found below in Table II.

The results show that there is a fairly large variance of the waveguiding characteristics across the width of both samples. The standard deviation of the slopes, however, is less for the annealed case. It appears that annealed sample should exhibit more reproducible results when compared to the unannealed, control sample. Also, since the standard deviation is less for the annealed case, the annealing process is improving the overall quality of the guiding medium.

Unannealed Half	Annealed Half
Slopes(mm ⁻¹)	Slopes(mm ⁻¹)
-0.498	-0.438
-0.362	-0.408
-0.491	-0,499
-0.532	-0.432
-0.485	-0.443
Mean = -0.474	Mean = -0.444
SD = 0.065	SD = 0.034

Table II. Study of the Homoseneity of a Typical Sample

The change in the quality of the waveguide after annealing is best seen by the eye, so photographs were taken as a means of comparison. Figure 2 contains these prints.

The top photograph is of an unannealed, control sample. The sample holder has been masked off with black, electrical tape in an attempt to improve the contrast of the photograph. The sample can be seen in the center of the photograph. The bright streak of light is the beam propagating through the length of the waveguide. Note the strong intensity of the beam, indicating a high degree of scatter. Also notice the small, bright dots near the top of the waveguide. These are dust particles which have settled on the surface of the sample. The exposure time for this photograph was approximately seven seconds.

The second photograph is of a sample which had been annealed at 450° C for ten minutes. Once again the streak in the center of the guide is the beam propagating through the sample, however the intensity of the scatter has been reduced dramatically. All of the bright spots are once again dust particles on the surface of the guide. They are more prominent in this photograph because the exposure time was increased to approximately thirty seconds. This lengthening in exposure time was necessary, otherwise the faint streak of the beam as it was propagating through the waveguide would never have been apparent.

A comparison of the two photographs shows the degree to which the amount of scatter is reduced by annealing. A majority of the incoupled beam is passed through the annealed waveguide, while a great deal more of the beam is scattered by the unannealed, control waveguide.

Figure 2. Comparison photographs of unannealed and annealed waveguides

Photograph of an unannealed waveguide taken with a 35 mm focal length, zoom lens, with an aperture setting of 3.5, and an exposure time of seven seconds. Note the very intense scatter coming from the beam as it propagates through the waveguide.

Photograph of a waveguide that had been annealed at 450° C for ten minutes. The exposure conditions were the same except that the exposure time had been lengthened to thirty seconds. Note the reduction in the amount of scatter indicating that a majority of the beam was being propagated through the waveguide.

Conclusion

Annealing of glass coverslips improves their ability to act as optical waveguides by reducing the variation in the refractive index along the sample. This improvement manifests itself on'y when a temperature of above 400° C is used for annealing. This experiment, however, can not conclude any relevant information concerning the magnitude of the improvement, only that there is one.

Literature Cited

- (1) Atkins, P.W. <u>Physical Chemistry Third Edition</u>. W.H. Freeman and Company, New York, 1986. p. 464.
- (2) Sears, Francis W., Mark W. Zemansky, and Hugh D. Young. <u>University Physics</u> Seventh Edition. Addison-Wesley Publishing Company, Reading, MA, 1987. p. 843.
- (3) Uhlmann, D.R. and N.J. Kreidl. <u>Glass: Science and Technology. Volume 3:</u> <u>Viscosity and Relaxation.</u> Academic Press, Inc. New York, 1986. p. 276.

Bibliography

- Atkins, P.W. <u>Physical Chemistry Third Edition</u>. W.H. Freeman and Company, New York, 1986.
- Sears, Francis W., Mark W. Zemansky, and Hugh D. Young. <u>University Physics</u> Seventh Edition. Addison-Wesley Publishing Company, Reading, MA, 1987.
- Uhlmann, D.R. and N.J. Kreidl. <u>Glass: Science and Technology, Volume 3: Viscosity</u> and Relaxation. Academic Press, Inc. New York, 1986.