Module 5: Manufacture and Physical Properties of Optical Fibers

August 31, 1995

1 Overview of Optical Fiber Manufacture Processes

The primary material in optical fibers is silica glass ($SiO_2$). Impurities called dopants are purposely added to modify the physical properties of the fiber, primarily the index of refraction. Recall the index of refraction of the core has a slightly higher index than the cladding. The nominal value for pure silica is $n = 1.458$. In what follows, let $n_1$, $n_2$ denote the refractive indices for the core and cladding, respectively, with $\Delta$ defined as:

$$\Delta = \frac{n_1 - n_2}{n_1} \times 100\%$$

In the case of step-index fibers, $n_1$ is constant in the core. For graded-index (GI) fibers, the refractive index is maximum at the center; in this case, $n_1$ denotes this peak value. Single mode (SM) fibers have a core radius of $4-10 \ \mu m$, and multi-mode (MM) step-index or graded-index fibers have core diameter of about $50 \ \mu m$. The cladding diameter is typically up to ten times the core diameter.

Current technology permits optical fibers with loss as low as $0.2 dB/km$ in the 1.1-1.6$\mu m$ range (with the nominal center wavelength usually 1.55$\mu m$). This loss is near the theoretical lower limit for silica glass, so current research is focusing on developing optical systems (sources and detectors as well as fibers) that operate in the mid-infrared (2-5$\mu m$) and far-infrared (8-12$\mu m$) ranges. For example, losses as low as $0.01 dB/km$ have been reported at 2.55$\mu m$ in fluoride glass. Here, however, we shall discuss conventional modern silica fibers operating in the 1.1-1.6$\mu m$ range.

The index of refraction is typically lowered by adding germanium ($Ge$) or phosphorus ($P$) in glass form, that is $GeO_2$ and $P_2O_5$, respectively, and is typically raised by adding fluorine ($F$) in elemental form or boron ($B$) in glass form as $B_2O_3$. Glass is a noncrystalline material that is usually modeled as a very viscous liquid. Refer to Figure 1. As a liquid is cooled, its volume decreases at a relatively rapid rate. When the melting temperature ($T_m$) is reached, a sudden drop in volume occurs as the liquid crystallizes. The atoms or molecules get locked into precise, repetitive symmetrical arrangements; removal of heat does not decrease the temperature but instead increases the extent of crystallization. After the liquid is solidified, further drops in temperature cause significantly smaller changes in volume. If crystallization does not occur at $T_m$, then removal of heat causes further contraction of the constituents atoms or molecules, resulting in a supercooled liquid. At the glass transition temperature ($T_g$), a sudden change in the slope of the volume versus temperature curve, called the coefficient of thermal expansion, occurs. Further reduction in temperature affects the volume very slightly. The liquid flows very slowly, and behaves much more like a solid than a liquid. There is no long range crystalline order, but there is short range order. The thermal agitation of the constituents atoms or molecules is sufficiently low that attractive forces bind atoms or molecules across several atomic diameters, thus accounting for the high viscosity. The process of forming a glass is called vitrification.
The impurities in the silica are chemicals in solution, and so the chemical formulas as denoted $SiO_2 \cdot GeO_2$, or $SiO_2 \cdot F$, and so forth. This notation means that $GeO_2$ or $F$, respectively, are interspersed with $SiO_2$, and substitute for $SiO_2$ uniformly through the material, much like dopants in a semiconductor or other crystalline solid either substitute for substrate atoms or occupy interstitial sites between substrate atoms, but do not fundamentally change the crystalline structure. Table 1 summarizes the maximum change in index of refraction, \[ \Delta = \frac{n-n_0}{n_0} \times 100\% \] where $n_0$ is the index of refraction for pure $SiO_2$, which can be obtained by adding various dopants.
<table>
<thead>
<tr>
<th>Material</th>
<th>Max. Δ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GeO₂ · SiO₂</td>
<td>3.5</td>
</tr>
<tr>
<td>P₂O₅ · SiO₂</td>
<td>1.2</td>
</tr>
<tr>
<td>B₂O₃ · SiO₂</td>
<td>-0.5</td>
</tr>
<tr>
<td>F · SiO₂</td>
<td>-0.7</td>
</tr>
</tbody>
</table>

Table 1: Change in Index of Refraction for Various Dopants

The most common methods for manufacturing optical fibers are classified as vapor deposition (VD). The raw materials are liquids such as SiCl₄, GeCl₄, POCl₃, SF₆, or SiF₄ which are heated to gasform (vapors). The hot vapors combine with oxygen gas O₂ and solidify into the glass compounds. In most cases, vitrification does not occur directly from the vapor form. Instead, the silica and other glasses are deposited on a surface as a fine powder or particulate form, called soot. The soot is heated and cooled to vitrify to a glass called the preform rod. The preform rod is very thick (perhaps by several orders of magnitude) compared to the final fiber. The process of converting the preform rod to the final optical fiber is called drawing. The preform rod is suspended and one end is heated to cause a thin glass stream to flow under gravity. This stream is cooled and becomes the final fiber. The fiber is spooled by wrapping it around a drum for storage.

2 Vapor Deposition

A typical optical fiber requires metal ion impurities of less than 1ppb (part per billion) and hydroxyl ion (OH⁻) of less than 10ppb. Whereas “ordinary methods” can result in impurities of less than 1ppm (part per million), chemical vapor deposition (CVD) is a relatively simple, common method for achieving commercial quality purity. The purity achieved by exploiting the phenomenon of vapor pressure.

When a pure liquid is exposed to an ambient environment, it partially vaporizes to the gaseous state so that the material in gas form maintains a constant pressure, called the vapor pressure $P_v$, above the surface of the liquid. Equilibrium is maintained if the ambient pressure $P$ equals the liquid's vapor pressure. If $P < P_v$, the liquid will evaporate (additional molecules will vaporize to gas form). If $P > P_v$, the vapor will condense (molecules in the vapor will convert to liquid state). If a liquid is comprised of different materials in solution, then a pure vapor can be extracted by properly adjusting the ambient pressure, or, equivalently, by heating the liquid to increase $P_v$ above the ambient pressure.

The main criteria when selecting the dopants and the liquid compounds used as raw materials are their availability, high purity, ease to liquefy, vapor pressure significantly different from transition metals ($Cu^{2+}, Fe^{2+}, Ni^{2+}, V^{3+}, Cr^{3+}, Mn^{3+}$), ability to vitrify with silica, stability when vitrified, and a coefficient of thermal expansion close to that of $SiO_2$. If the coefficient of thermal expansion of the dopants and $SiO_2$ do not match closely, then cracks and stresses will occur in the fiber under ordinary thermal variations.

In this section, we summarize the principle features of several vapor deposition methods: modified CVD (MCVD), plasma CVD (PCVD), plasma-enhanced CVD (PM CVD), outside vapor deposition (OVD) and vapor axial deposition (VAD).
MCVD is illustrated in Figure 2. Various liquids which provide the source for Si and dopants are heated in the presence in oxygen gas. Chemical reactions called oxidizing reactions occur in the vapor phase, as listed below:

\[
\begin{align*}
\text{GeCl}_4 + O_2 & \rightarrow \text{GeO}_2 + 2\text{Cl}_2 \\
\text{SiCl}_4 + O_2 & \rightarrow \text{SiO}_2 + 2\text{Cl}_2 \\
4\text{POCl}_3 + 3O_2 & \rightarrow 2\text{P}_2\text{O}_5 + 6\text{Cl}_2
\end{align*}
\]

The flow of oxygen is controlled by mixing valves, and the vapors are blown into a heated tube where the oxidizing takes place. The chlorine gas $\text{Cl}_2$ is blown out of the tube, but the oxide compounds are deposited in the tube in the form of soot. The concentration of iron and copper impurity is reduced from about 10ppb in the raw liquids to less than 1ppb in the soot.

The tube is heated and continually rotated to vitrify the soot into a glass. By adjusting the relative flow of the various vapors, several layers with different indices of refraction can be obtained, for example core versus cladding or variable core index profile for GI fibers. After the layering is completed, the tube is heated and collapsed into a rod with a round, solid cross-section, called the preform rod. In this step it is essential that center of the rod be completely filled with material and not hollow. The preform rod is then put into a furnace for drawing, as will be described in the next section.
The main advantage of MCVD is that the reactions and deposition occur in a closed space, so it is harder for impurities to enter. The index profile of the fiber is easy to control, and the precision necessary for SM fibers can be achieved relatively easily. The equipment is simple to construct and control. A potentially significant limitation of the method is that the rod size is limited essentially by the dimensions of the tube. Thus, this technique can be used to form fibers typically of 3-5 km in length, or 20-40 km at most. In addition, impurities in the silica tube, primarily $H_2$ and $OH^-$, tend to diffuse into the fiber. Also, the process of melting the deposit to eliminate the hollow center of the preform rod can cause a depression of the index of refraction in the core. In terms of cost and expense, the main disadvantage of the method is that the deposition rate is relative slow because it employs indirect heating—the tube is heated, not the vapors directly, to initiate the oxidizing reactions and to vitrify the soot. The deposition rate is typically 0.5 to 2 g/min.

PCVD is illustrated in Figure 3. A plasma is a high energy ionized gas. It is used as a source of direct heat. A more conventional heat source is an oxyhydrogen flame, one which contains high quantities of oxygen, hydrogen and/or water vapor. By contrast, the plasma is inert with respect to fiber formation. The plasma is formed by using a microwave cavity resonator to generate strong electromagnetic fields to ionize and energize it. Typically, the resonator is operated at 200W, 2.45GHz. This plasma is confined to the silica tube, and provides sufficient heat to oxidize the vapors directly into glass layer, not soot. The cavity resonator is slid back and forth along the length of the tube to form uniform glass layers. Each layer, formed by a single pass, is very thin, so up to 100 or more passes must be made.

The main advantage of this method is the very high deposition rate, up to 2.5 g/min, and nearly 100% deposition efficiency. That is, the exhaust gas is almost completely depleted of the Si and dopant material. The plasma heat is easily controlled, so the index profile can be varied precisely. Note that this technique involves direct heating, not indirect heating. This method is well-suited for SM fibers.

The disadvantages are that the equipment is more complex and larger than that used in MCVD. The cavity resonator can be large, a low inner pressure (10-30 torr) must be sustained in the tube, thus making the system harder to maintain, and the whole tube must be preheated. In addition, the preform is limited in size, as with MCVD. This technique was developed primarily at AT&T.

PMCVD, illustrated in Figure 4, is similar to PCVD, except that the plasma is formed under normal pressure. The power source is more powerful, typically 10-20 kW, and is at 1-10 MHz. The oxidized gas turns into soot, with a very high deposition rate of 6 g/min up to as much as 20 g/min.
This technique provides for very high precision profile control. In addition to the high deposition rate and efficiency, this technique can be used to manufacture a very long fiber, up to $50\text{km}$, since the Si tube can have a larger inner diameter, $50-60\text{mm}$, compared with MCVD and PCVD. In addition, since normal pressure is maintained in the tube, MCVD equipment can be employed. The main disadvantage is the only one thick, potentially uneven layer is formed, so it is not appropriate for GI fibers. It is thus used for SM fibers. This method was developed at Philips-Holland.

![PMCVD Method](image)

**Figure 4: PMCVD Method**

In OVD, illustrated in **Figure 5**, the oxidizing reactions are replaced by hydrolysis:

$$GeCl_4 + O_2 \rightarrow GeO_2 + 4HCl$$
$$SiCl_4 + O_2 + 2H_2 \rightarrow SiO_2 + 4HCl$$

These reactions occur in the vapor phase, and in particular HCl is in the form of hydrochlorine gas in the exhaust. The deposit is in the form of soot. Unlike the methods described previously, the soot is deposited on the outside of a silica substrate, not on the inner surface of a tube. After deposition is completed, the starting rod is pulled off and is dehydrated and vitrified in a furnace to yield the preform rod. The process of drying and vitrifying a wet soot is called sintering.
In this method, the sooting, hydrating and sintering processes are separated. The soot is formed quickly, up to 5g/min, because of the direct heating. There is no limit to the preform rod size, so long continuous fibers, 50-100 km, are possible.

The main disadvantage is that the reactions and deposition occurs over an open space, so is more susceptible to the introduction of impurities. Therefore, a clean space (a special, impurity-free ambient environment) must be maintained. In addition, the process of pulling off can cause structural irregularities and tensile stress at the core center. This is particularly critical when the coefficient of thermal expansion between the core and cladding differs significantly, which in turn is more severe for high numerical aperture (NA) fibers. Recall that NA is proportional to $\Delta$, and a large $\Delta$ is achieved with high dopant concentrations, which in turn corresponds to larger variations in the coefficient of thermal expansion. OVD is used primarily by Corning.

The VAD process is illustrated in Figure 6. Unlike the methods described previously, VAD permits a continuous length endless silica fiber to be produced. It was developed in Japan, and is used primarily for SM fibers. The process is similar to OVD in that $SiCl_4$, $GeCl_4$ and other dopant vapors are blown with $O_2$ and $H_2$ into the lower end of a rotating silica boule. A flame causes the same hydrolysis reactions as in OVD. A porous soot rod of $SiO_2 \cdot GeO_2$ is formed, which is pulled up while being rotated. The porous rod is dehydrated and consolidated in a furnace.
The index of refraction in the preform is controlled by precise design of the flames. The temperature distribution on the lower surface of the soot preform rod, the angle at which the flames blow, the number of torches, the flow rate of the SiCl$_4$ and GeCl$_4$ vapors, and the distribution of gas in the flames are the main parameters which determine the index of refraction.

In addition to the possibility to have a continuous, endless length preform, this technique has several other advantages. The torch does not move, the gas flow rates are constant, a high deposition rate of up to 6g/min can be achieved, and a SM fiber can be manufactured. The disadvantage is that the flame control is very hard, and it is difficult to form high bandwidth fibers.

3 Drawing and Spooling

Converting the preform rod to the final fiber involves drawing, in which a long, uniform extremely thin stream is pulled out from one end of the rod. Since compression and microbends and kinks in the fiber are extremely undesirable, it is important to wrap the fiber around a drum by a precisely controlled process called spooling. These two processes are performed at the same time by a single apparatus, as shown in Figure 7.
One end of the preform rod is heated so that a thin stream falls by gravitational pull. As this stream cools, it becomes the thin optical fiber, which is wrapped around a drum with a capstan motor. Fiber drawing is typically performed at 2100 °C, with the melting temperature of silica approximately 1900 °C. A primary plastic coating is applied to the fiber immediately after drawing. The coating prevents dust and moisture from adhering to the fiber, and reduces the effect of microbending loss. Additional coatings may be applied later.

The flow of hot gas, the furnace temperature, and the rotation of the capstan motor must be controlled very precisely so that the diameter and tension in the fiber is constant during the drawing and spooling process. Closed-loop feedback is necessary, so tension must be measured by detecting the resistance (torque) of the capstan motor and the fiber diameter is measured with LED sensors. This information is processed by computer to adjust the operation of the system.

References

