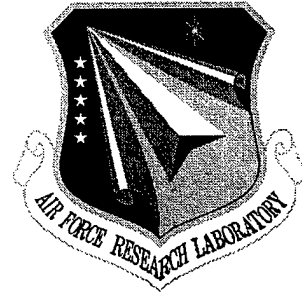


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SEMICONDUCTOR CYLINDER FIBER FABRICATION

Syracuse University

Philipp Kornreich

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13. ABSTRACT (Maximum 200 words) This report describes the fabrication of optical fiber that has an optically active material between the core and cladding. The materials used are CdTe, Cd ₃ P ₂ , and carbon. Processes are described for the deposition of these materials onto the glass rods used to form the fiber core. Also described is the collapsing process used to make the fiber perform. Absorption data for the fibers produced is given.				
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1 INTRODUCTION.

We finally, solved most of the problems of fabricating collapsed semiconductor ampoules. It took us longer than I thought. The ampoules form the inner part of a Semiconductor Cylinder Fiber (SCF) preform. In the past we worked mainly with the semiconductor CdTe. However, its energy gap corresponds at a wavelength that is too short for practical application in fiber optic communication. Therefore, we chose two other semiconductors Cd₃P₂ and GaSb. Both semiconductors can be made to work at wavelengths useful for fiber optic communication and are compatible with our fabricating process.

CdTe is compatible with our process of SCF fabricating. Indeed, we made CdTe cylinder fibers. We demonstrated that the energy gap of the thin, approximately 10 atom thick, semiconductor film is larger than the energy gap of the bulk semiconductor. The energy gap of bulk CdTe is 1.54 eV corresponding to a wavelength of 805 nm, while the energy gap of the CdTe film in the SCF had an energy gap of 1.823 eV corresponding to 680 nm. The energy gap expands due to the quantum size effect. CdTe has the following properties:

Melting point	1041 °C
Density	5810 kg per m ³
Formula Weight	240.01
Energy gap	1.54 eV, Direct gap
Crystal structure	Zincblende
Electron effective mass ratio	0.064
Heavy hole effective mass ratio	0.1

The change in the energy gap ΔE due to the quantum size effect is:

$$\Delta E = \frac{\hbar^2 \pi^2}{2m_0 e a^2} \left[\frac{1}{m_e^*} + \frac{1}{m_h^*} \right]$$

where $m_0 = 9.109534 \times 10^{-31}$ kg is the rest mass of an electron, and a is the thickness of the semiconductor film in the fiber. The expansion of the energy gap by 0.283 eV of the CdTe in the fiber requires a film thickness of 5.835 nm. This required an approximately 0.584 μm thick semiconductor film in the preform. This is very reasonable.

We calibrated our equipment for fabricating fibers with Cd_3P_2 semiconductor cylinders. We chose Cd_3P_2 since it can be thermally deposited in an evacuated ampoule with our traveling furnace and can be made to work at wavelength of 1550 nm and 1320 nm which are important for fiber optic communication. The traveling furnace deposition system is described in section 2 below. GaSb requires higher evaporation temperatures than are compatible with the traveling furnace deposition process. The fabrication of GaSb films requires either deposition with light in the visible wavelength range described below in section 4, or deposition in a Molecular Beam Epitaxy vacuum system still under construction.

Bulk Cd_3P_2 has an energy gap of 0.61 eV corresponding to a wavelength of 2033 nm. The energy gap of the very thin semiconductor layers surrounding the fiber core expand due to the quantum size effect. Depending on semiconductor film thickness the energy gap can be made to expand to 0.8 eV for operation at a wavelength of 1550 nm or to 0.939 eV for operation at 1320 nm. Cd_3P_2 has a melting point that is compatible with our fabricating process. Photoluminescence has been observed in this material.

Cd_3P_2 properties:

Melting point	700 °C
Density	5600 kg per m ³
Formula Weight	399.1476
Energy gap	0.61 eV, Direct gap
Crystal structure	Zincblende
Electron effective mass ratio	0.08
Heavy hole effective mass ratio	0.12

The vapor pressure of most semiconductors including Cd_3P_2 is given by

$$P_{\text{vapor}} = P_0 \exp\left[-\frac{E_A}{kT}\right]$$

where for Cd_3P_2 $P_0 = 3.02487 \times 10^{13}$ Pascal and $\frac{E_A}{k} = 17787.3774$ °K .

Let us, first, calculate the vapor pressure $P_v(25\text{ }^\circ\text{C})$ generated by the evaporation of Cd_3P_2 in the ampoule at "room temperature", at $25\text{ }^\circ\text{C}$ corresponding to $298\text{ }^\circ\text{K}$ using the above equation .

$$P_v(25\text{ }^\circ\text{C}) = 3.614287 \times 10^{-13} \text{ Pascal}$$

$$P_v(25\text{ }^\circ\text{C}) = 2.801016 \times 10^{-15} \text{ Torr.}$$

This is negligible compared to the residual pressure in the ampoule.

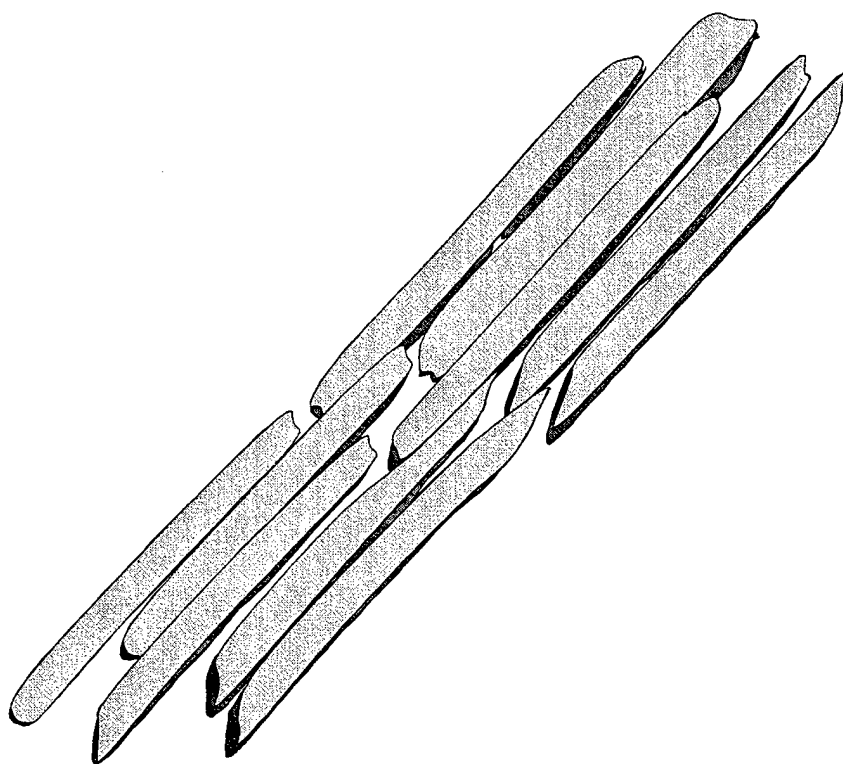


Fig. 1. Probable arrangement of semiconductor crystallites in polycrystalline film.

The semiconductor films surrounding the fiber core are, most likely, polycrystalline. However, similar to cold rolled steel, the crystallites are probably very elongated, as shown in Fig. 1. The materials in both the cold rolling process of steel and the fiber pulling process used here are substantially elongated. It is the contact surfaces between the semiconductor crystallites and the glass that might be responsible for

non-radiative recombination rather than the crystallites boundaries as is the case in bulk material. However, the surface area of the boundaries of the very thin and very elongated crystallites form only a small portion of the surface area.

2 THERMAL DEPOSITION PROCESS.

We can routinely now deposit high vapor pressure semiconductors such CdTe and Cd_3P_2 with our traveling furnace. We can deposit semiconductors with high vapor pressure that evaporate at temperatures below the temperature where the ampoule glass would deform. The following normally solid or liquid elements have vapor pressures greater than 10^{-2} Torr at temperatures below 460 °C:

As, Cd, Cs, Hg, I, K, Mg, Na, P_4 , Po, Rb, S, Se, Te, and Zn.

We use a temperature of about 460 °C corresponding to $(460^\circ + 273^\circ =) 733$ °K to deposit the film Cd_3P_2 . The thermal deposition process works as follows:

A 1.4 mm inside diameter, 2.8 mm outside diameter, 300 mm long type 7052 glass tube is closed at one tube. About 50 mg of Cd_3P_2 powder is placed near the closed end of the glass tube. A 1 mm diameter 100 mm long (type 7740) glass tube is placed at the open end in the glass tube. The open end is connected to a vacuum system as shown in Fig. 2. A traveling furnace is placed over the tube as shown in Fig. 2. The semiconductor powder evaporates in the glass tube in the furnace. It is too hot in the furnace for the semiconductor to deposit. The semiconductor deposits on the inside of the tube where the tube emerges from the furnace as shown in Fig. 2. As the furnace travels away from the vacuum system end of the tube the inside of the tube is coated. After the deposition is completed the tube is pinched off near its vacuum system end. The ampoule, containing a clear glass core rod, forms the inner part of a fiber preform.

The deposition rate is proportional to the surface area of the semiconductor powder in the glass tube. Thus, as the semiconductor material evaporates its surface area decreases. Indeed, we have experimentally found that the deposition decreases profoundly as the process progresses. To compensate for the decrease in the semiconductor powder surface area we increase the furnace temperature by approximately 0.65 % every 25 mm of furnace motion. This allows us to deposit about 100 mm long semiconductor layers in the tube.

The stepping motor requires 400 pulses per revolution and the furnace lead screw has 8 turns per inch (per 25.4 mm). The furnace speed is given by:

$$v_{\text{Furnace}} = \frac{25.4 \times 60}{400 \times 8 \times t_{\text{Period}}} \text{ mm per minute}$$

where t_{Period} is the period of the stepping motor driving voltage pulses.

$$v_{\text{Furnace}} = \frac{0.47625}{t_{\text{Period}}} \text{ mm per minute}$$

We use a stepping motor driving voltage pulse period t_{Period} of 0.228 seconds this gives a furnace velocity of 2.088816 mm per minute or 0.0822368 inches per minute (12 minutes 10 seconds per inch of travel).

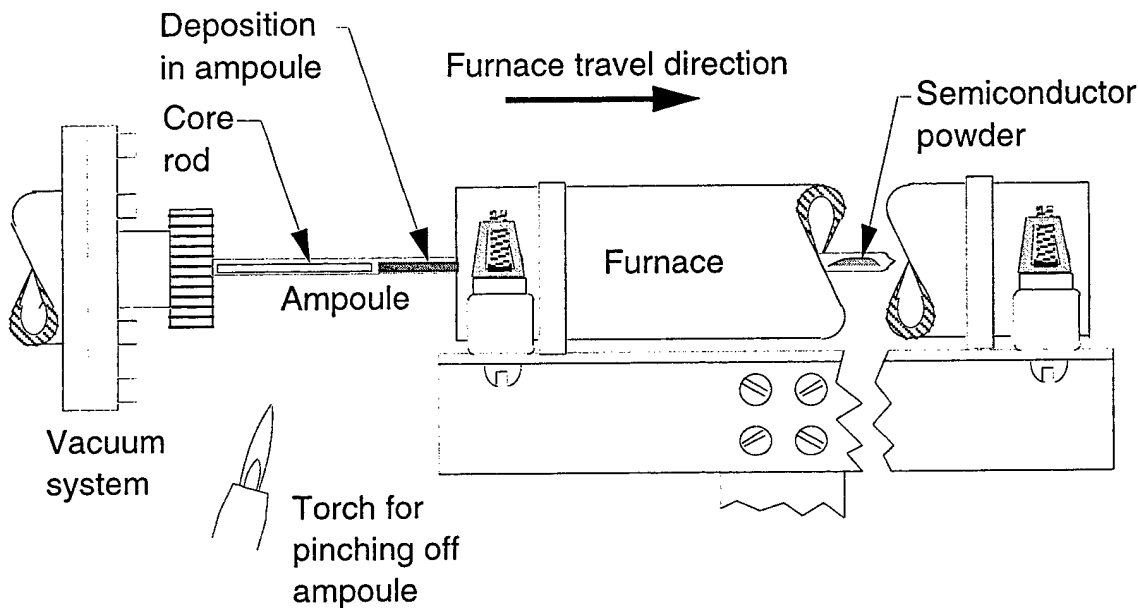


Fig. 2. Thermal semiconductor deposition with a traveling furnace.

3 COLLAPSING PROCESS.

We, next, discuss the ampoule collapsing process which works very well. The viscosity of most glasses is given by:

$$\mu = \mu_0 \exp\left[\frac{E_V}{k(T - T_0)}\right]$$

The activation viscosity amplitude μ_0 , the energy E_V , and the temperature T_0 can be determined from the annealing point temperature T_A , the softening point temperature T_S and the working temperature T_W of the glass. Here T_{ST} is the strain point temperature. The viscosity at the three temperatures are defined as follows. The viscosity at the annealing point is equal to 10^{12} Pascal seconds. The viscosity at the softening point is equal to $10^{6.6}$ Pascal seconds, and the viscosity at the working point is equal to 10^3 Pascal seconds. We have

Glass type	T_{ST} °K	T_A °K	T_S °K	T_W °K
7052	708.15	753.15	981.15	1388.15
7720	758.15	798.15	1028.15	1383.15
7740	793.15	838.15	1093.15	1493.15
7760	753.15	798.15	1053.15	1483.15

Glass type	μ_0 Pascal-sec.	$\frac{E_V}{k}$ °K	T_0 °K
7052	$4.318114139928 \times 10^{-3}$	12519.574236654	374.640196087
7720	$1.435571386738 \times 10^{-4}$	16225.973201020	353.356611581
7740	$2.227542955647 \times 10^{-4}$	17448.284694635	354.019565229
7760	$1.304321387373 \times 10^{-3}$	15350.391299607	350.265384626

Let us return to the properties of the Cd_3P_2 film that we deposit inside the ampoule. It is easiest to collapse the ampoule at the softening point of the type 7052 ampoule glass of 708 °C. This is above the melting point of 700 °C of the Cd_3P_2 film. When the film melts it forms little round pellets. To avoid this we collapse the Cd_3P_2 ampoules at a lower temperature. Also, at 708 °C (981 °K) the vapor pressure of Cd_3P_2 is 403766.1465 Pascal or 4.117269 Atm. (1 Atm. = 98066.5 Pascal). Actually, the pressure generated in the ampoule is less than the vapor pressure since there is not

enough Cd_3P_2 material. The pressure at 708 °C in a typical ampoule is 1.7 Atm. Indeed, we made an experiment trying to collapse a Cd_3P_2 ampoule at atmospheric pressure at 708 °C. It did not collapse, it expanded as expected. We can readily generate a pressure of 300 lb. per square inch, or 20.4 Atm. in the collapsing chamber. We next calculate the temperature needed to collapse the ampoule at a pressure of 300 lb. per square inch.

In one dimension the pressure P is equal to the viscosity μ times the change of the velocity v of an incremental volume of the glass with position.

$$P = \mu \frac{dv}{dx}$$

As we have seen above, the viscosity is temperature dependent. For the case when the change of the velocity v of an incremental volume of the glass with position is kept constant, the viscosity $\mu(T_n)$ at temperatures T_n is proportional to the pressure at that temperature.

$$\frac{P_1}{P_2} = \frac{\mu(T_1)}{\mu(T_2)}$$

By substituting the expression for the temperature dependence of the mobility into the above equation and taking the logarithm of the resulting expression we obtain:

$$\ln \frac{P_2}{P_1} = \frac{E}{k} \left[\frac{1}{T_2 - T_0} - \frac{1}{T_1 - T_0} \right]$$

In particular we are interested to calculate the temperature T_2 at a pressure of 300 lb. per square inch that will give the same change of the velocity v of an incremental volume of the glass with position as a 1 Atm. (14.7 lb. per square inch) will produce at the softening temperature of the glass. For the four types of glass we have:

Glass type	T_s °K	T_2 °K	T_2 °C
7052	981.15	903.831575	630.681575
7720	1028.15	952.946764	679.796764

7740	1093.15	1009.417272	736.267272
7760	1053.15	967.861389	694.711389

At 630 °C (903 °K) the vapor pressure of Cd_3P_2 is 84320.64096 Pascal or 0.859831 Atm. (1 Atm. = 98066.5 Pascal) However, the pressure in the stainless steel pressure chamber is 20.4 Atm. Thus, the ampoule will readily collapse. Indeed we have collapsed many ampoules using this process.

We have built a pressure chamber for collapsing which is shown in Fig. 3. It consists of a stainless steel pipe, a cap, pressure supplying plumbing, as well as a thermocouple. The pressure seal is provided by copper gaskets at both ends of the stainless steel tube. The stainless steel tube and inserts in the cap and nitrogen gas pressure connection have knife edges that cut into copper gaskets to provide a pressure seal, see Fig. 4. The ampoule floats on Sn solder in a stainless steel boat in the pressure chamber during collapse as shown in Fig. 3. The Sn solder melts at 420 °F or 215.556 °C The liquid Sn solder heats the ampoule uniformly as well as keeping it straight during the collapsing process.

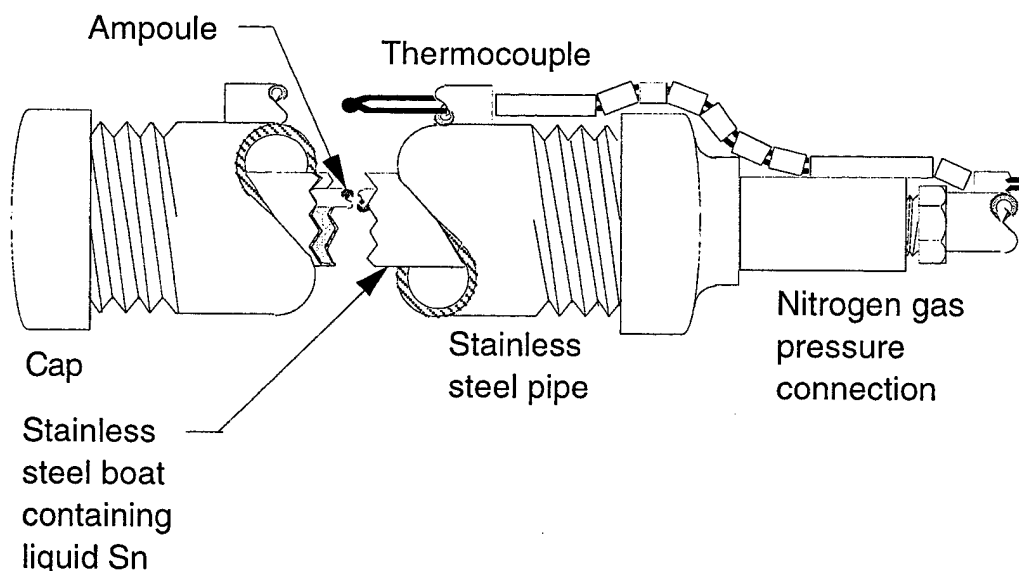


Fig. 3. Arrangement for collapsing ampoules and the outer tubes forming the cladding of the preform. The pressure seals are achieved by the use of copper gaskets. The ampoule floats in liquid Sn solder while being pressurized during the collapsing process.

The collapsing pressure uses two furnaces as shown in Fig. 5. The ampoule in the pressure chamber is collapsed in the high pressure chamber in the high temperature furnace at 620 °C. After the ampoule is collapsed the pressure chamber is moved to the low temperature furnace which is kept at about 260 °C. At this temperature the ampoule hardens while still floating on the liquid solder.

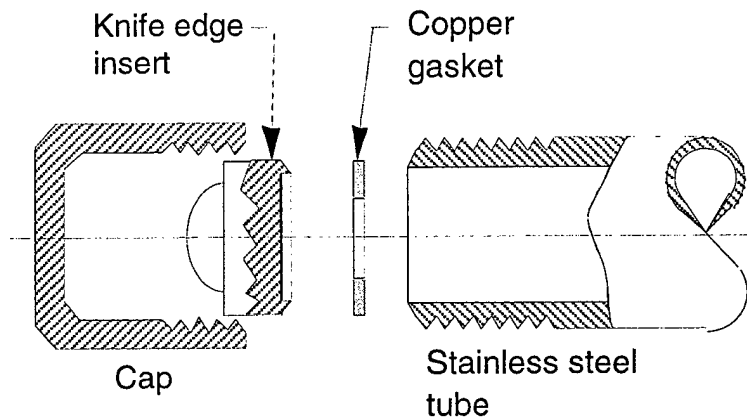


Fig. 4. Knife edges on the stainless steel pipe and the cap insert bite into a copper gasket to provide the pressure seal. A similar arrangement is used at the pressurizing end of the stainless steel tube.

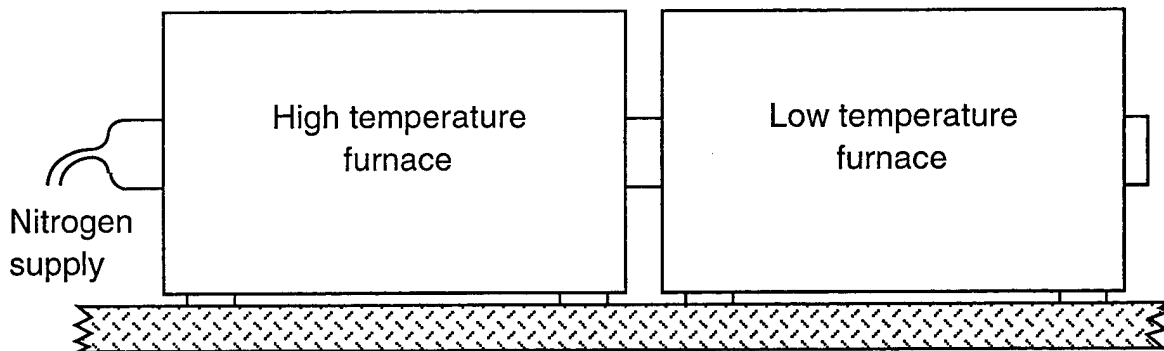


Fig. 5. Collapsing furnace arrangement.

We use a temperature of about 460 °C corresponding to $(460^\circ + 273^\circ =) 733$ °K to deposit the film. Thus, since we collapse the ampoules at 620 °C corresponding to 893 °K some of the Cd_3P_2 film should reevaporate. The evaporated portion of the Cd_3P_2 film will redeposit over the complete inside surface of the ampoule. However, such a thin layer might not deposit as a uniform film. We, now, routinely deposit about 100 mm

long films in ampoules. However, we assume a worst case of an only 2 inch long deposited film in the ampoule for the calculation of the loss in film thickness due to reevaporation.

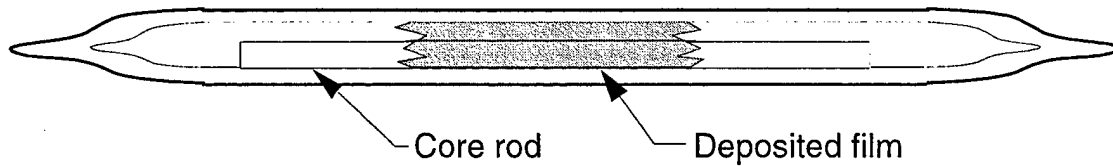


Fig. 6. Schematic representation of the ampoule. Drawing not to scale.

We collapse the ampoules at 620 °C corresponding to 893 °K. By putting these numbers into the equation for the vapor pressure we obtain for the pressure P_v generated by the evaporating Cd_3P_2 in the ampoule:

$$P_v(620 \text{ }^\circ\text{C}) = 67629.43691 \text{ Pascal}$$

$$P_v(620 \text{ }^\circ\text{C}) = 0.689628 \text{ Atmosphere}$$

$$P_v(620 \text{ }^\circ\text{C}) = 524.117533 \text{ Torr.}$$

We, next, calculate the amount of Cd_3P_2 material evaporated. We assume the ampoule, shown schematically in Fig. 6, is 100 mm long and has an inside diameter of 1.4 mm. The core rod is 95 mm long and has a diameter of 1 mm. We assume the worst case where the deposition is only 50 mm long on the inside surface of the ampoule tube. The "ideal gas" law is:

$$P = nkT$$

where n is the density of Cd_3P_2 molecules per cubic meter in the ampoule and Boltzmann's constant $k = 1.380662 \times 10^{-23}$ joules per °K. The higher the gas temperature the better approximation this is of the behavior of the gas. At 620 °C corresponding to 893 °K this approximation is fairly good. The density n of Cd_3P_2 molecules in the ampoule at 620 °C corresponding to 893 °K is:

$$n = 5.4852556 \times 10^{24} \text{ per m}^3.$$

The ampoule has a volume V of $8.325221 \times 10^{-8} \text{ m}^3$. By multiplying the volume V by the density n of Cd_3P_2 molecules per unit volume we obtain:

$$nV = 4.566597 \times 10^{17} \text{ Cd}_3\text{P}_2 \text{ molecules in the ampoule.}$$

The "formula weight" (FW) of Cd_3P_2 is = 399.1476. The unit atomic or molecular weight is equal to $1.6605402 \times 10^{-27} \text{ kg}$. Thus, each Cd_3P_2 molecule weighs $6.628006 \times 10^{-26} \text{ kg}$. The $4.566597 \times 10^{17} \text{ Cd}_3\text{P}_2$ molecules weigh $3.026743 \times 10^{-7} \text{ kg}$. Solid Cd_3P_2 has a density of 5600 kg per m^3 . Thus the $4.566597 \times 10^{17} \text{ Cd}_3\text{P}_2$ molecules when condensed into a solid have a volume $V_s = 5.404899 \times 10^{-11} \text{ m}^3$. We assume, a worst case, that only 2 inches or approximately 50 mm of Cd_3P_2 film was deposited as shown in Fig. 3. The deposited film here has an area A_f of $2.199115 \times 10^{-4} \text{ m}^2$. By dividing the volume V_s of the condensed solid form of the Cd_3P_2 gas in the ampoule by the area A_f of the film we obtain the thickness t_f of the portion of the film that will evaporate:

$$t_f = 0.2457761 \mu\text{m}$$

We now calculate the thickness of the film in the ampoule assuming the Cd_3P_2 redeposits uniformly when the ampoule collapses. The surface area of the inside of the ampoule is $7.225663 \times 10^{-4} \text{ m}^2$. The redeposited film in the ampoule will have a thickness t_R of

$$t_R = 0.0748014 \mu\text{m}$$

Thus the resulting film in the deposited region will be $0.2457761 \mu\text{m} - 0.0748014 \mu\text{m} = 0.1709747 \mu\text{m}$ less thick than the original deposited film.

We have collapsed a number of ampoules with deposited films in the ampoule ranging from 50 to 100 mm in length. The semiconductor layers in these ampoules survived the collapsing process. The transmission spectrum of one of these ampoules is currently being analyzed by the Optical Science Center at the University of Arizona.

Subsequently more type 7052 clear glass tubes are collapsed onto the collapsed ampoules until a fiber preform with an outside diameter of 9 mm is obtained. A fiber is then drawn from this preform. It is true that the preform is heated to about $700 \text{ }^\circ\text{C}$

during the fiber pulling process where the Cd_3P_2 film would melt. However, at this point the film is trapped between the core glass and the cladding glass layers.

4 LIGHT DEPOSITION PROCESS.

We are in the process of developing a process that uses light with wavelengths in the visible range to deposit semiconductor layers in a closed evacuated ampoule. The advantage of using visible wavelength light is that the ampoule glass is transparent to this radiation while the semiconductor material absorbs light in the visible wavelength range.

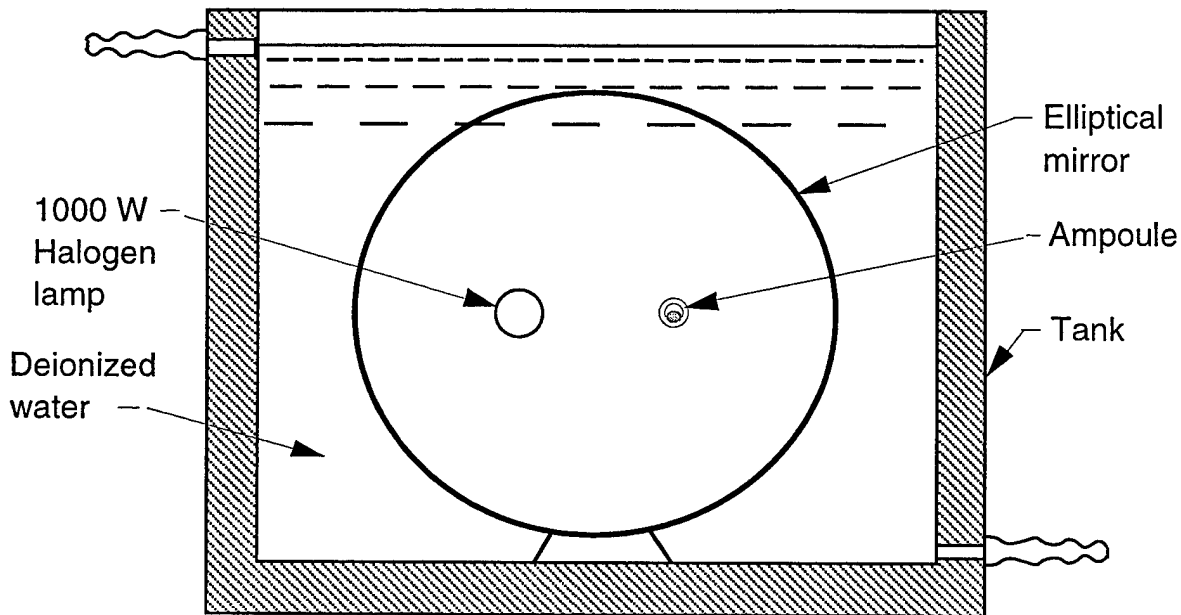


Fig. 7. Light deposition system. Both the ampoule containing a few mg of semiconductor powder and a cylindrical 1000 W tungsten halogen lamp are located in the focal lines of an elliptical mirror.

We have built a tank trough which deionized water is circulated. An elliptical mirror is located in this water tank as shown in Fig. 7. The ampoule containing a few grams of semiconductor powder and a cylindrical 1000 W tungsten halogen lamp are located in the focal lines of the elliptical mirror. This arrangement is similar to the pumping arrangement of large lasers. The focusing of the light from the tungsten halogen lamp onto the ampoule is very critical.

The water serves both to cool the 1000 W tungsten halogen lamp and to filter out the longer wavelength of the light from reaching the ampoule. Water transmits light with wavelengths shorter than 1100 nm and absorbs light with wavelength larger than 1100 nm.

The ampoules used in this experiment were made by closing a glass tube at one end and connecting it to the vacuum system similar to the ampoules described in the thermal evaporation process. A few mg of semiconductor powder was placed near the closed end of the ampoule. A pill of getter material was placed in the tube near its open end which is connected to the vacuum system. The getter pill was heated to evaporate its protective coating and to outgas it. After the outgasing is complete the ampoule was sealed. The getter absorbs gas in the ampoule improving the vacuum in the ampoule.

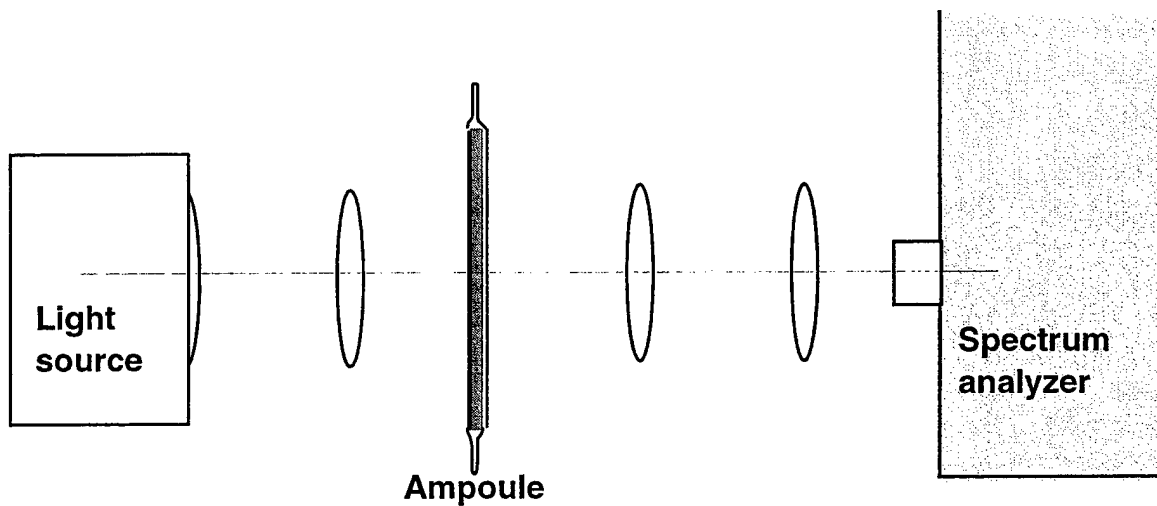


Fig. 8. Arrangement for measuring the transmission spectrum of the light deposited ampoules.

We made ampoules containing CdTe, Cd_3P_2 , and GaSb semiconductor layers. We could only measure the transmission spectrum of a CdTe ampoule since the energy gap of Cd_3P_2 and GaSb is 0.7 eV corresponding to 1771 nm which is beyond the range of our test equipment.

We measure the transmission spectrum of our ampoules by illuminating them at an angle of 90° to the long axis of the ampoule as shown in Fig. 8. The normalized transmission spectrum of the CdTe film deposited with light in an ampoule is shown in Fig. 9.

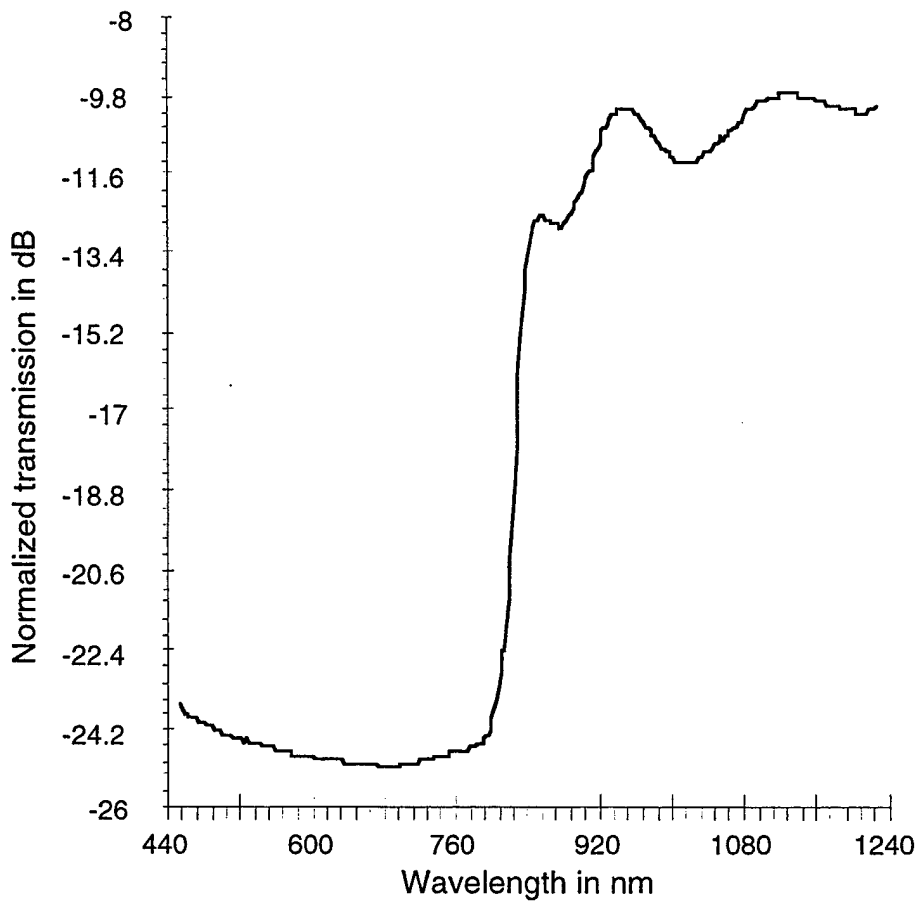


Fig. 9. The normalized transmission spectrum of the CdTe film deposited with light in an ampoule.

We replotted the transmission spectrum of the light deposited CdTe film on a linear scale and as a function of energy as shown in Fig 9. We note the exciton energy minima at 1.0243, 1.2217, 1.4164 eV. We previously measured the transmission spectrum of thermally deposited CdTe ampoules with similar results.

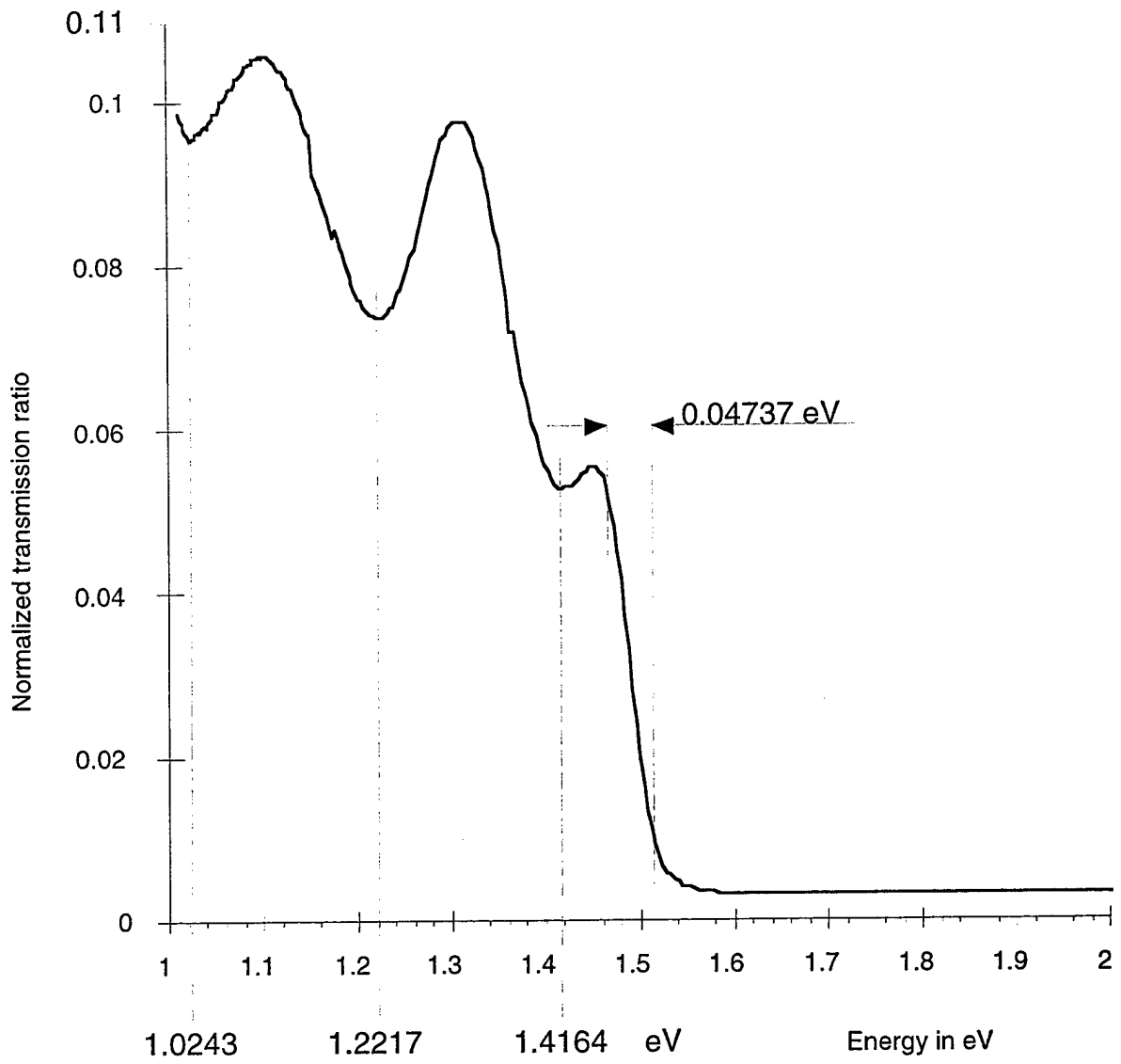


Fig. 10. Normalized transmission spectrum of light deposited CdTe ampoule. The data has been replotted as a function of energy. Note the exciton energy levels at 1.0243, 1.2217, and 1.4164 eV.

5 NANO PARTICLE CYLINDER FIBER FABRICATION.

We have fabricated fibers with a carbon nano particle cylinder surrounding the core. This fabrication method was suggested by professor Douglas Keller. The fibers were made by using a flame to coat a 1 mm Pyrex rod with carbon black. Unfortunately since we did not have core rods and cladding tubes with different indices of refraction both the core and cladding of any resulting fiber had the same index of refraction. The rod was inserted into a 3 mm O. D. 1.8 mm I. D. glass tube that was closed at one end.

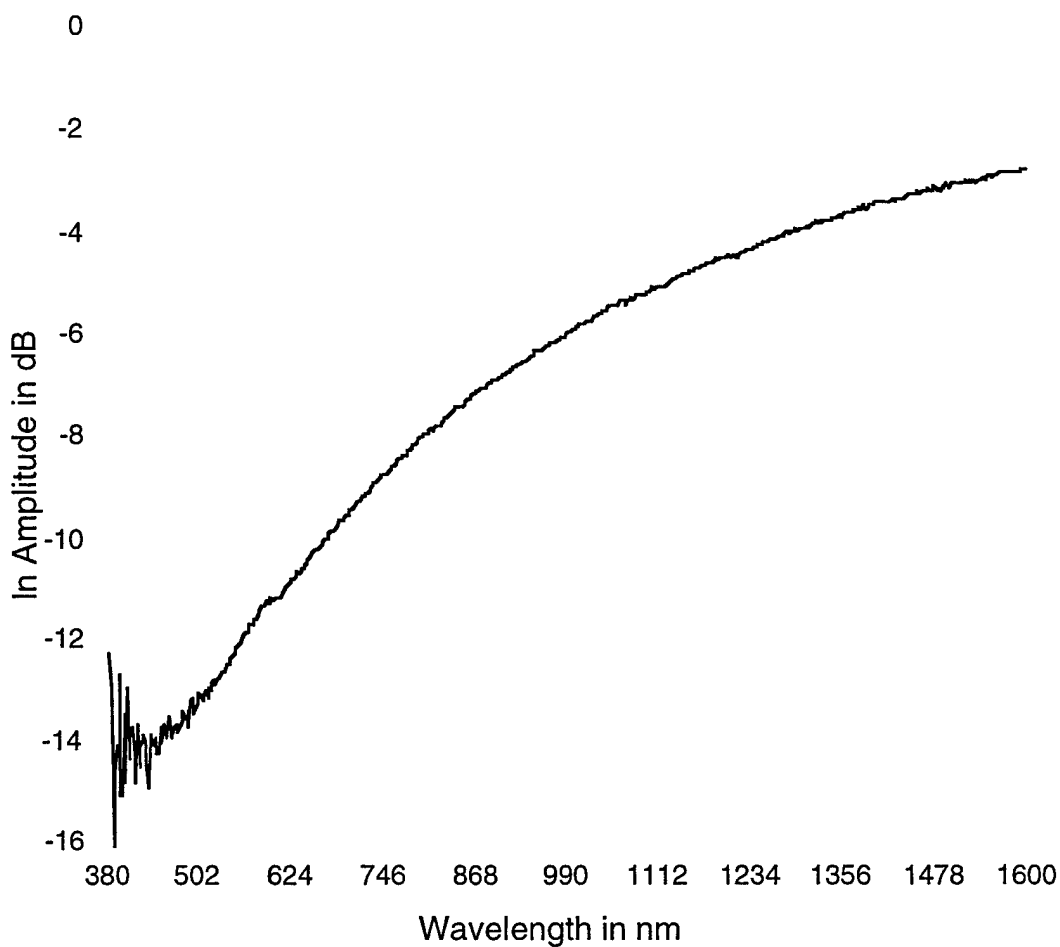


Fig. 11. Normalized transmission spectrum of carbon nano particle cylinder preform. A 1 mm diameter uncoated core rod was used as reference.

The tube was evacuated to 10^{-7} Torr., heated at about 250° C for two hours, and sealed at the vacuum pump end to form a closed ampoule. The ampoule was collapsed under pressure at about 650° C. Two more Pyrex tubes were collapsed onto the collapsed ampoule. We measured the transmission spectrum of the preform. The normalized transmission spectrum of the carbon nano particle cylinder preform is shown in Fig. 11. A 1 mm diameter uncoated core rod was used as reference. The normalized transmission spectrum exhibits a large absorption in the visible and UV.

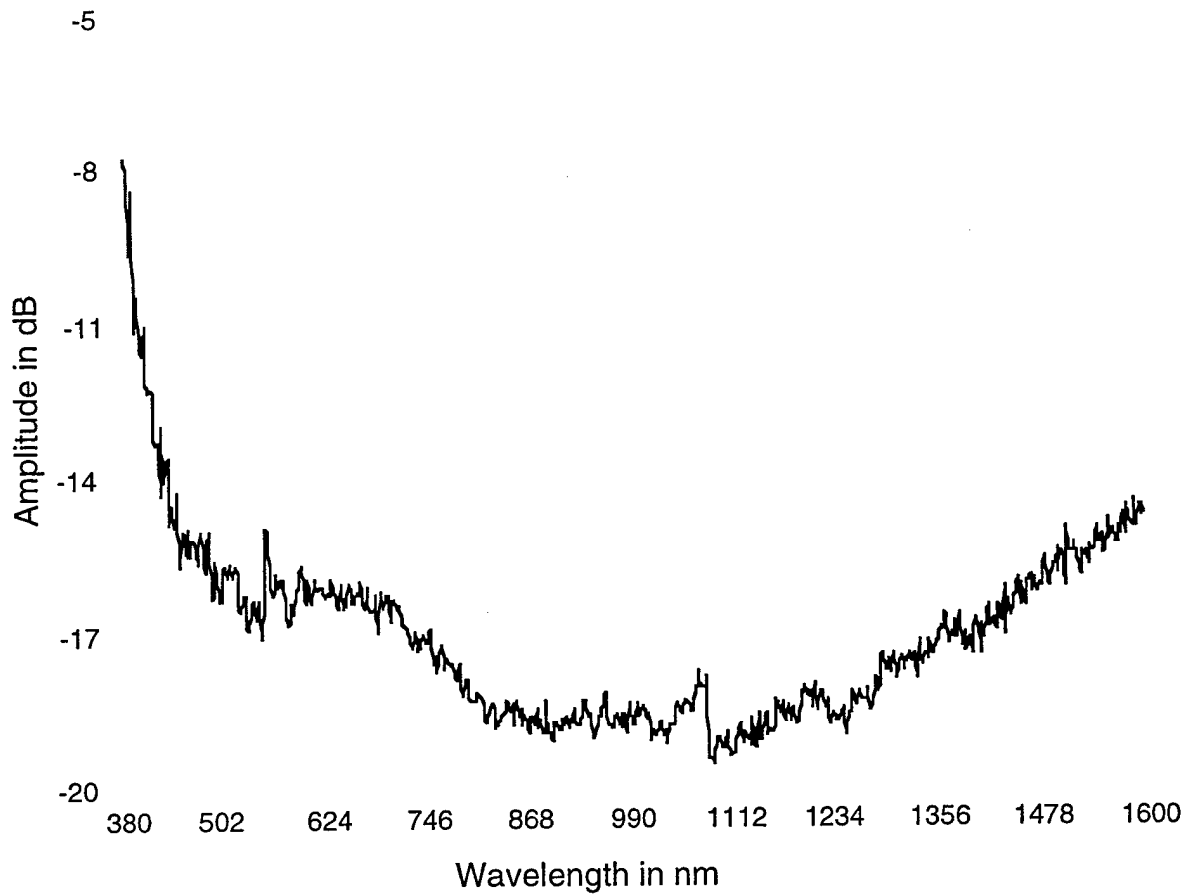


Fig. 12. Normalized transmission spectrum of a Carbon Nano Particle Cylinder Fiber. A 1 mm diameter uncoated core rod was used as reference.

A fiber was pulled from this preform. The fiber pulled exceedingly smoothly. The carbon flowed smoothly in the preform taper during the fiber pulling process.

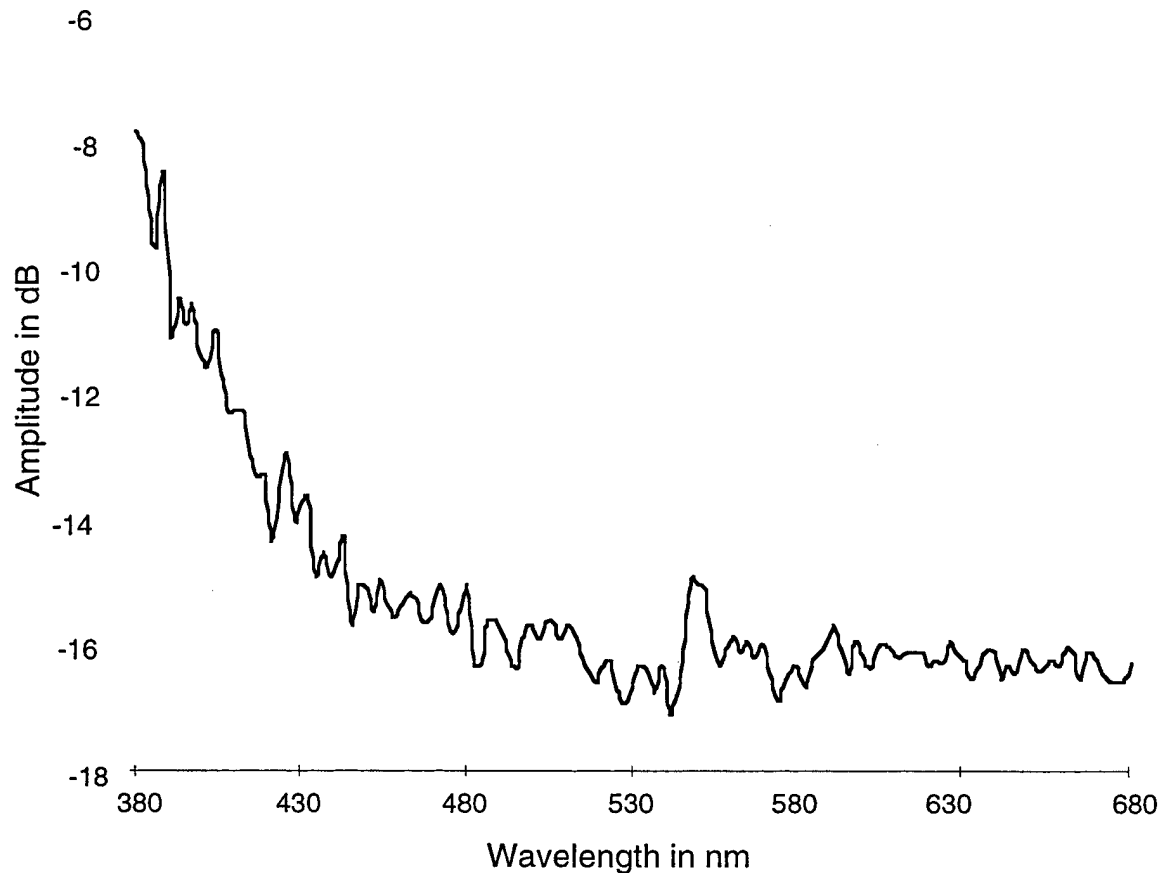


Fig. 13. Short wavelength portion of the normalized transmission spectrum of a Carbon Nano Particle Cylinder Fiber. A 1 mm diameter uncoated core rod was used as reference.

We measured the transmission spectrum of a Carbon Nano Particle Cylinder Fiber. The fiber had a core diameter of 25 μm , an outside diameter of 200 μm , and a length of 60 mm. As stated above the core and cladding had the same index of refraction. The normalized transmission spectrum of the Carbon Nano Particle Cylinder Fiber is shown in Fig's. 12. and 13. We note the large transmission in the UV. The fiber only guides where the carbon nano particle cylinder interacts with the light. This occurs in the UV. A similar phenomena was observed in our Semiconductor Cylinder Fiber.

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