

## *Chapter 2: Sapphire Optical Fiber*

The potential of single-crystal fibers was recognized forty years ago, when the inherent strength and the perfection of the crystalline matrix of the fibers were determined. Despite this, sustained research into the production of such fibers was delayed until the nineteen seventies. Until then, control over the growth characteristics of the fibers was poor, and there were no well defined areas of utilization.[33] The first application that drove the production of single-crystal sapphire fibers was use as a reinforcing member for metal-matrix composites.[34] Sapphire was thought to be an attractive candidate to employ for this use because it possesses a high melting temperature of 2053 degrees Celsius, has a low solubility in water, is resistant to chemically hostile environments, and has adequate mechanical properties.[8] This application requires fibers possessing good mechanical characteristics, but makes no restriction on the optical qualities of the fibers.

Interest in developing optical-quality sapphire fibers emerged after the commercial effort to manufacture structural-grade sapphire fibers began. Researchers at Stanford, realizing that sapphire fibers were not likely to replace glass-based fibers for telecommunications applications, embarked on a program to investigate the potential applications and manufacture of optical-quality sapphire fibers. Sapphire fibers are not reasonable candidates for long-distance telecommunications lines because of the difficulties involved in manufacturing low-cost fibers having perfect crystalline structure and surfaces as smooth as conventional glass fiber. Their investigation indicated that fibers grown using the Laser Heated Pedestal Growth Method were optically superior to those grown using the Edge-Defined Film-Fed Growth method, which was used in the manufacture of the structural grade fiber [33]. Others concurred, and, with the possible

exception of Saphikon, Incorporated, the best optical-quality sapphire fibers have been grown by researches using the Laser Heated Pedestal Growth Method.[35, 36] Optical-quality sapphire fiber has found success as a means to deliver laser power in medical operations, in high-temperature thermometers, and in spectroscopy.[37] Efforts to develop sapphire-fiber-based strain and displacement sensors continue.

This chapter begins by describing the two most popular methods used to grow single-crystal sapphire fiber: the laser heated pedestal growth method and the edge-defined film-fed growth method. The positive and negative aspects of each are presented. Next, the optical and mechanical properties of sapphire fibers are described. The chapter concludes with a discussion of the possibilities that exist for cladding and overcoating the bare sapphire fiber. The difficulties associated with this procedure are addressed.

## **2.1 The Growth of Sapphire Fiber**

Single crystals of synthetic corundum, also known as  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> or white sapphire, may be grown according to a variety of methods.[38] The most common of these methods are: crystallization from a melt, solution growth, and condensation from the vapor phase. The second of these has attracted considerable interest because it permits the continuous and controlled growth of single-crystal fibers of uniform diameter. This method also permits the fibers to be grown along a specific crystallographic axis.[33] The two most popular implementations of this technique are the Edge-Defined Film-Fed Growth and the Laser Heated Pedestal Growth methods.

Laser Heated Pedestal Growth (LHPG), favored by university-based researchers, uses a float-zone technique to grow optical-quality sapphire fibers. Researchers at Stanford University[8, 33, 39], Rutgers University[40, 41, 42], and the University of South Florida[43, 44] are the most notable to use the LHPG method. This method has not been commercially embraced due to cost considerations: the LHPG process allows only a single fiber to be grown at a time. This method has been widely acknowledged to

produce better optical-quality sapphire fibers than the Edge-Defined Film-Fed Growth process,[41, 42] although recent product data claim that this may no longer be the case.[36]

Use of the LHPG method, schematically represented in Figure 2.1.1 and likened to the float-zone method of crystal growth, results in a single-crystal fiber being grown from the molten tip of a source crystal.[42] The tip of an  $\text{Al}_2\text{O}_3$  source rod is liquefied when the beam of a  $\text{CO}_2$  laser is split and focused onto it. Inserting a seed crystal into the liquefied bead creates a molten zone extending from the source rod to the seed crystal, with the shape of the molten zone determined by surface tension. The fiber is

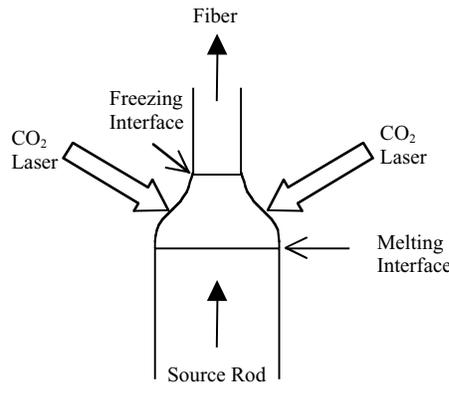


Figure 2.1.1: The Laser Heated Pedestal Growth Process [39]

grown by carefully controlling the speed at which the seed crystal is moved away from the source rod, and the speed at which the source rod is advanced into the molten zone. The longest length of fiber that can be grown using this method is determined by the dimensions of the source rod. The purity of the fiber should be determined by the purity of the source rod: the heating method is clean, and the molten zone never comes in contact with a crucible or a die.[39] Under optimal growth conditions, the fiber has a diameter of approximately one third of the source rod. Because mass is conserved during the growth process, the ratio

$$\frac{d_s}{d_f} = \sqrt{\frac{v_f}{v_s}} \quad (2.1)$$

where  $d_s$  is the diameter of the source rod,  $d_f$  is the diameter of the fiber,  $v_s$  is the velocity of the source rod, and  $v_f$  is the velocity of the fiber, is valid.[42]

While this description of LHPG is straightforward, it omits mention of the effort made to produce a fiber of uniform diameter and good optical quality. Surface irregularities in the fiber, including diameter fluctuations, contribute substantially to scattering losses, and the fiber diameter is directly dependent on the shape of the molten zone. The shape of the molten zone is affected by fluctuations in the diameter of the source rod, variations in the power of the CO<sub>2</sub> laser, and the velocities of the source rod and fiber.[8] In addition, the melt possesses a very low viscosity, and the molten zone, held in place only through surface tension, is easily perturbed by air currents and vibrations.[35] The detrimental effects of air currents are minimized by sealing the source rod and growing fiber end in an atmosphere controlled chamber.[33] This precaution also permits the growth of the sapphire fibers in an inert atmosphere: it is believed that losses arise when OH<sup>-</sup> and other gaseous species are incorporated into the sapphire fiber while it is growing. Researchers at Rutgers University used a computer controlled feedback system to stabilize the laser and to monitor, as well as to control, the fiber diameter. With this system, the authors of the paper were successful in producing sapphire fibers possessing a -0.5 percent diameter variation. Without the use of feedback, the diameter variations were a factor of ten higher. With this stable growth process and tight diameter control, the researchers produced fibers with losses of 0.3 decibels per meter at the 2.94 micron wavelength. This is very close to the theoretical minimum of 0.13 decibels per meter at the same wavelength.[35]

Edge-Defined Film-Fed Growth (EFG), patented by Saphikon Incorporated, permits the growth of many sapphire fibers simultaneously, unlike LHPG, making the commercial production of sapphire fiber economically viable. This method of sapphire fiber growth

was developed to meet the demand for sapphire fiber structural reinforcements for metal matrix composites. The mechanical strength, as opposed to the optical quality, of the fiber was the overriding consideration; the high loss in these early sapphire fibers attenuates nearly all injected light over a centimeter of propagation length. When the demand for structural filaments lagged, Saphikon Incorporated investigated the possibility of producing optical-grade sapphire fiber for use in high temperature fiber optics.[45] Currently, Saphikon Incorporated supplies sapphire fiber for this purpose and for medical and spectroscopy applications.[37]

The EFG method utilizes a reservoir, containing molten source material, to continuously supply the growth of a single-crystal sapphire structure. Unlike in the LHPG method, the melt is contained in a molybdenum crucible, and the cross section of the sapphire crystal is determined by the surface configuration of the molybdenum die anchored in the melt. During growth, the melt wicks into the dye, and the fiber growth, initiated by a seed crystal, occurs at the top of the dye. The use of an anchored die in the melt presents a stable platform from which to grow the crystal. Because the top of the die extends above the level of the melt in the crucible, thermal variations present in the melt are damped, as the melt travels the length of the tube. This contributes to the achievement of a stable melt temperature at the solidification interface. Molybdenum is considered to have good chemical compatibility with molten alumina: sapphire crystals grown using molybdenum crucibles and dies contain only 5 parts per million of molybdenum, and molten alumina is able to wet the molybdenum. When a material is wet by the liquid phase of another material, the contact angle is defined as being less than ninety degrees.[46] As in the LHPG method, it is necessary to isolate the growing crystal from vibrations and atmospheric currents, ensure a stable temperature at the growth interface, surround the growing crystal with an inert atmosphere, and carefully control the growth rate of the crystal to ensure optical-quality sapphire fibers.[45]

The edges of the die are used to control the shape of the cross section of the sapphire crystal. When the die is shaped as a hollow capillary tube and the top of the die makes an

angle 75 degrees or greater with the inner edge of the die, the melt wicks up through the inside of the die and spreads over the top surface of the die. A fiber grown under these conditions will have a diameter equal to the outer diameter of the cylinder. Similarly, a die consisting of three capillary tubes fastened together can be used to grow three-bore tubing. In this case, the melt wicks up through the approximately triangular interstitial region created by the mating of the three tubes, and it spreads to cover the ringed surface formed by the ends of the three capillaries. Because the melt never extends over the inner diameter of any of the three conjoined tubes, three-bore tubing can be grown. The flexibility imparted to the growth process results in the ability to grow a sapphire crystal of nearly any cross-section, including ribbons, single filaments, single-bore tubing, and multiple-bore tubing.[46]

## **2.2 Optical and Mechanical Characteristics of Single-Crystal Sapphire Fiber**

The attractive features associated with using sapphire fiber as an optical waveguide is tempered by the troublesome optical and mechanical properties sapphire fiber possesses. Exploration of the optical uses of sapphire fiber began and is sustained because it is capable of operation in chemically harsh and high temperature environments, it has a large transparency window, and it is a strong and hard material. Sapphire fiber has not been adopted for a wide variety of uses because difficult to perfect growth processes result in sapphire fibers being more lossy than their glass counterparts, and because sapphire fibers will always be more expensive. Additional complications arise from sapphire fibers from being unclad and highly multimoded. Sapphire has also been observed to decrease in strength and flexibility at high temperatures.

Sapphire fiber is transparent over the 240 to 4000 nanometer range of wavelengths,[8] and has an unrealized theoretical loss minimum of 0.0034 decibels per kilometer at a wavelength of 1780 nanometers.[35] The measured transmission spectrum, shown in Figure 2.2.1, for bulk sapphire fiber, as published by Innocenzi *et al.* [47] and plotted as discrete squares, differs from the theoretical loss spectrum. The theoretical loss is

defined by the curves labeled Urbach , Brillouin , and Multiphonon .[42] The Urbach curve illustrates the increase of the value of the absorption coefficient as the frequency increases toward the band gap energy of sapphire.[48] Brillouin scattering occurs when incident light, consisting of photons, is scattered by a mode of vibration of the crystal lattice, composed of acoustic phonons.[49] Multiphonon refers to multiphonon absorption processes that occur in the sapphire crystal..

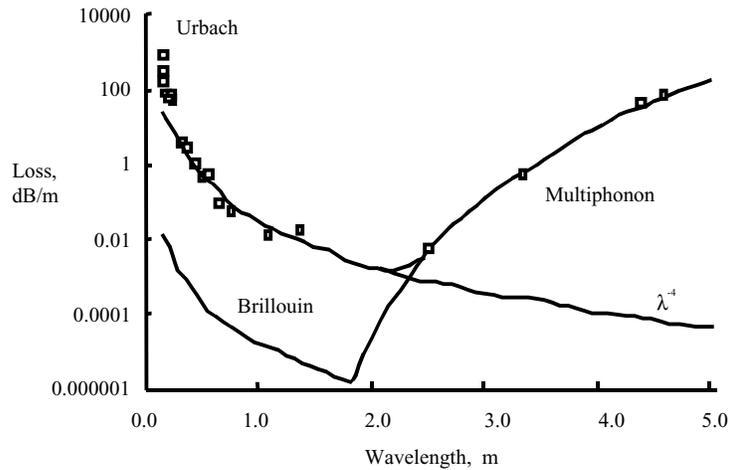


Figure 2.2.1: Measured and Theoretical Attenuation in Bulk Sapphire [42]

Unlike experimental data taken for fused silica, which typically coincide well with theoretical loss curves, crystal defects result in the loss measurements of crystalline materials normally being higher than those predicted by theory. The experimental data below 1.5 microns can be fitted by a power law varying as  $\lambda^{-4}$ , which implies the presence of Rayleigh scattering caused by inhomogeneities in the crystal.[42] Rayleigh scattering is due the presence of scatterers that are small compared to the wavelength.[50] Low loss sapphire fibers grown at Rutgers have a loss spectrum that is compared to the bulk data from Innocenzi *et al.* in Figure 2.2.2. The higher losses exhibited in the fibers are attributed to Rayleigh-Gans scattering, and are ascribed to crystal defects induced by subtle unrefinements in the mechanical system used to grow the fibers.[42] Rayleigh-

Gans scattering has been observed to occur in the presence of defects that, compared to the wavelength of light, are large in the transverse direction, but which induce small optical phase changes in the scattered rays.[50] Currently, published data from Saphikon Incorporated lists attenuation losses a few tenths of a decibel lower than those fabricated at Rutgers.[36]

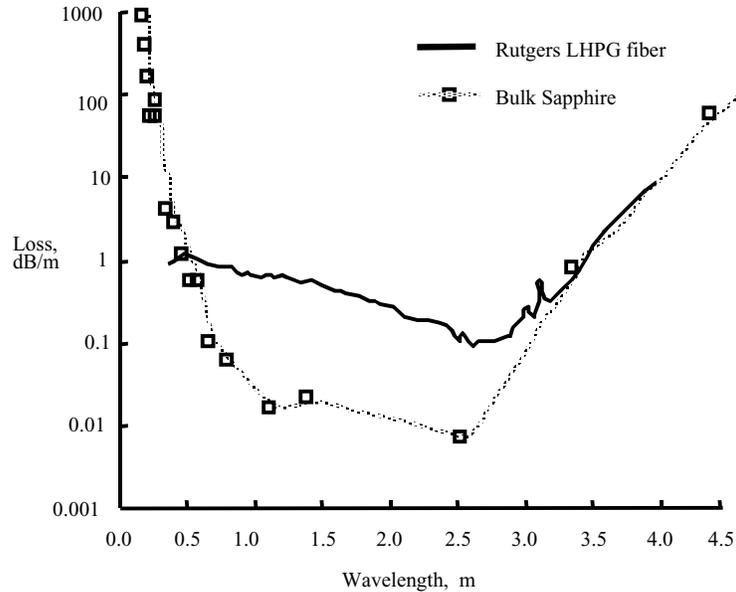


Figure 2.2.2: Measured Attenuation in Bulk Sapphire and in Sapphire Fiber [42]

Extrinsic loss in sapphire fibers, which should be minimized for optical applications, is largely due to scattering and absorption mechanisms. Impurities and color centers exacerbate extrinsic absorption, and voids, inhomogeneities, and surface perturbations contribute to scattering losses. Impurities in sapphire fibers may derive from the source material or gaseous species such as  $H_2O$ . [8] In fibers grown using the EFG method, the crucible and die may also be contributors. [46] Measurements suggest that fibers possess  $OH^-$  impurities and color centers, specifically V-type (hole) centers, that contribute to absorption losses at region around the wavelengths of 410 and 3000 nanometers. V-type centers occur when a hole is trapped in the vacancy of an ionic crystal. The absorption at 410 nanometers is attributed to a pair of holes trapped by an  $O^{2-}$  ion positioned near an

$\text{Al}^{3+}$  vacancy. There is also the probability of a OH impurities partially compensating V-centers. The presence of OH impurities is supported because the absorption corresponding to the stretching band for the OH impurity, occurring around 3000 nanometers, is observed. Also occurring around 3000 nanometers are three peaks that indicate the presence of transition metal impurities. The magnitude of the absorption in the bands around both 410 nanometers and 3000 nanometers may be greatly reduced by annealing the fibers in air or  $\text{O}_2$  at 1000 degrees Celsius.[42]

The majority of extrinsic scattering losses in sapphire fibers are attributed to voids internal to the fiber, inhomogeneities, and surface variations of the fiber. The number and type of scattering sites determine the attenuation of each fiber, and these are dependent on the quality of the growth process. It is well understood that any variations in the growth conditions of the fiber, including the temperature at the growth interface, changes in the speed of the growth, perturbations of the shape and size of the meniscus which feeds the growth of the crystal, will cause variations in the diameter of the fiber.[8] Unstable growth conditions are also responsible for the incorporation of inclusions into the fiber. It is noted in particular that some sapphire fibers grown using the EFG technique possessed bubbles that resulted from a growth rate that was too fast. Annealing does not appear to reduce these losses,[34] but it is believed that the refinements in the mechanics of the growth process can greatly reduce, if not eliminate these sources of loss.[42] Sapphire fibers generally do not suffer from twinning and dislocation defects, unlike bulk sapphire. This is because the diameter of sapphire fibers is small: unless a defect propagates parallel to the axis of the fiber, it will eventually grow out of the fiber. Dislocations in sapphire fibers will occur, but they will be of a lesser density than in bulk sapphire.[33]

Sapphire fibers are not clad, which results in a number of undesirable characteristics. Single crystal sapphire fibers cannot be grown as a core-clad structures from rod-in-tube source rods, because convective currents in the molten zone quickly act to destroy the geometrical structure of the source rod.[41] Other techniques, discussed in Section 2.3,

have been proposed to clad sapphire fibers, but none represent a universally acceptable solution. The fiber cladding exists to mechanically support the core, to act as a barrier separating the core from environmental contaminants, and to reduce scattering losses by lessening the difference in dielectric constants at the surface of the core.[51] Scattering can result in power from the scattered mode being coupled into other modes or being ejected from the fiber as radiation losses.[50] Contaminants on the surface of the sapphire fiber can attenuate the intensity of the guided light through a few mechanisms. Any substance in contact with the surface of the sapphire fiber possessing an index of refraction higher than that of sapphire will, by defeating total internal reflection, cause loss at all points of contact. Similarly, a material, which has an index of refraction lower than that of sapphire and different than that of the operating atmosphere, that is in sporadic contact with the surface of the sapphire fiber will cause loss: scattering will occur at the points of discontinuity between dielectric constants.[45] Sapphire fibers have also been observed to incorporate impurities into their structure, particularly at elevated temperatures, which causes losses through scattering.[19]

Sapphire fibers are not currently singlemoded for operation in the 250 to 4000 nanometer window of transparency, and are not likely to be in the near future. Without a physical cladding, the entire volume of the sapphire fiber may be considered to be the core, and the surrounding atmosphere the cladding. Sapphire fibers typically have diameters between 100 and 300 microns. For singlemode operation, the diameter of such a fiber is given by

$$d = \frac{\lambda(2.405)}{\pi n_s} \quad (2.2)$$

where  $d$  is the diameter of the fiber,  $n_s$  is the index of refraction of sapphire, and  $\lambda$  is the wavelength of operation.[51] This specifies fiber diameters ranging from approximately 1 to 0.1 microns for singlemode operation over the transparency window for sapphire. It is claimed that 40 micron fibers can be grown without modification to existing LHPG

machinery, and that 5 micron diameter fibers could be grown, but that the cost would be prohibitive.[52] Successfully handling such a fiber without breaking it would also be extremely difficult. Sapphire fiber is also a birefringent waveguide that is traditionally grown so that the c plane [0001] coincides with the optical axis of the fiber.[53]

Sapphire is a brittle material, and the flexibility and strength of sapphire fiber decreases at high temperatures. Sapphire is among the hardest of natural minerals and is rated as a 9 on the Moh s scale.[38] Despite this, because of the brittle nature of sapphire, handling will result in surface damage and consequent reduction in strength.[54] Fibers tumbled against one another have been observed to suffer a tensile strength reduction of 30 percent.[55] Saphikon Incorporated has combated this problem by presenting the option of coating the surface of each fiber with an organic polymer, to protect the fiber, before shipping. While this protection is recognized as necessary for strength preservation, the process used to remove the sizing can both damage and fail to remove all of the impurities on the surface of the fiber.[54] Sapphire also suffers a severe degradation in strength as the temperature is increased, as is shown in Figure 2.2.3.

This property impacts the minimum diameter bend that the fibers can survive at any given temperature. The bend survival strength  $\sigma_{bs}$ , also shown in Figure 2.2.3, is determined by

$$\sigma_{bs} = \frac{d_f E}{2R_s} \quad (2.3)$$

where  $d_f$  is the fiber diameter, E is the elastic modulus (450 gigaPascals), and  $R_s$  is the bend survival radius. Fibers fail when plastic deformation permits the growth of fractures in the fibers. Bend tests conducted at temperatures above 1650 degrees Celsius result visible plastic deformation of the fibers: this occurs due to dislocation (pyramidal plane) slip that is activated at temperatures exceeding 1600 degrees Celsius. The bend behavior

is reported to be dependent on surface flaws and the presence of inclusions in the fiber.[53]

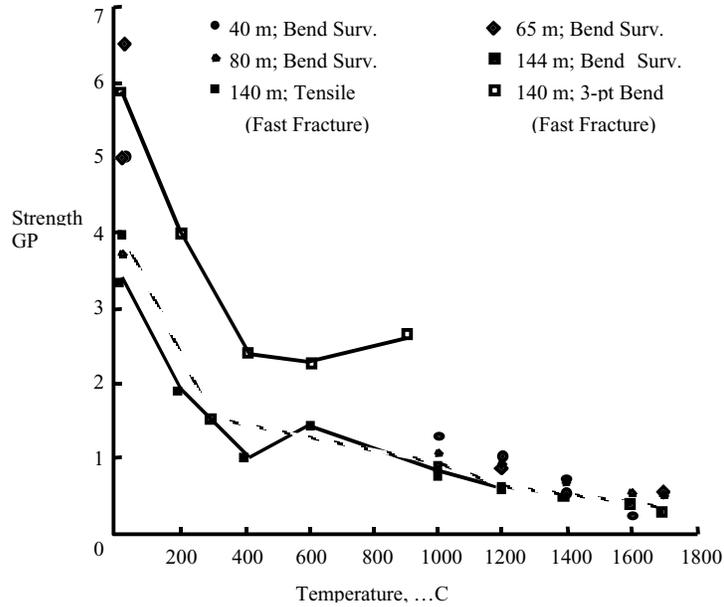


Figure 2.2.3: One Hour Bend Survival Strengths, Fast-Fracture Tensile Strengths, and Bend Strengths Versus Temperature [53]

### 2.3 Cladding and Coating Sapphire Fiber

Identifying suitable materials to act as coating and cladding materials for optical-grade sapphire fibers is difficult because of the number of requirements they must meet. Ideal claddings should enhance the optical characteristics, without limiting the useful operation, of the sapphire fibers. Ideal coatings should act as a barrier between chemically harsh environments and sapphire, and the coatings should be capable of operation over at least the same temperature range as sapphire alone. Obstacles to this goal include the need to match the coefficient of thermal expansion of the materials and sapphire, the need for a good bond between the fiber and the material, the (so far) insurmountable problem of growing a core-clad sapphire fiber in one process, and the

inability to identify materials suitable for a wide range of applications. The benefits of cladding and coating sapphire optical fibers is addressed in Section 2.2.

Claddings will potentially permit singlemode operation, improve the waveguiding properties of the sapphire fiber core, prevent radiation losses, and provide mechanical support to the core of the fiber. The cladding must have a lower index of refraction than sapphire, possess a coefficient of thermal expansion compatible with that of sapphire, form a layer with low intrinsic stress, and be able to survive large fluctuations in temperature. Because sapphire fibers cannot be clad as they are grown, as explained in Section 2.2, claddings must be applied after the growth of the fiber. Sapphire fibers can be clad using either ion exchange techniques or the direct deposition of materials. Few reports exist in the literature to describe the effects dopants have on the index of refraction of sapphire fiber; it is not a method that is being actively investigated. Direct deposition is a more viable method.[56]

The direct deposition of a cladding on the surface of sapphire fiber can be effected by metallorganic chemical vapor deposition (MOCVD), physical vapor deposition, or solution based methods. During physical deposition methods, such as evaporation and sputtering, the fiber must be rotated to assure a uniform coating. Solution based methods, such as sol-gel, frequently require an additional process to densify the porous coating. This densification can result in considerable stress between the cladding and the sapphire fiber. MOCVD is presented as a superior technique because the deposited layer is typically dense, homogenous, and pure. A wide variety of materials may be deposited using this method, and the composition and thickness of the deposited layer may be controlled to within tight tolerances. Suggested cladding materials to investigate for cladding deposition include  $\text{SiO}_x\text{N}_y$ ,  $\text{Mg}_x\text{SiO}_y$ , and  $\text{Ti}_x\text{Si}_y\text{O}$ , whose x/y ratio may be varied to give a range of refractive indices.[56] In general, adequate cladding materials that are both transparent and capable of surviving temperatures over 1000 degrees Celsius are considered so rare as to not exist. There has been some success with polycrystalline alumina ( $\text{Al}_2\text{O}_3$ ), but these coatings are considered prohibitively expensive.[10, 52]

Coatings isolate the fiber from environmental contaminants and protect the surface from abrasion. This protection will enable wider use of sapphire fiber by extending the useful lifetime of the fiber and increasing the number of potential operating environments. There is no one candidate material that can serve as a sapphire fiber coating in any environment; the coating must be chosen, in part, according to the application.[56] It is also desirable that elements do not inter-diffuse between the coating and the fiber materials. Commonly considered materials include polycrystalline alumina ( $\text{Al}_2\text{O}_3$ ), metal niobium, silicon carbide (SiC), and zirconia ( $\text{ZrO}_2$ ). Coating techniques include chemical coating, vapor deposition, plasma spraying, and electrochemical plating.[57]

The suggested sapphire fiber coatings have different strengths and weaknesses. Alumina and sapphire share the same chemical composition, and the only stable phase of alumina above 1199 degrees Celsius is  $\alpha\text{-Al}_2\text{O}_3$ ; single crystal  $\alpha\text{-Al}_2\text{O}_3$  is sapphire. Alpha alumina has a coefficient of thermal expansion (CTE) of  $8.1 \times 10^{-6}$  per degree Celsius at 1000 degrees Celsius, and a melting temperature of 2053 degrees Celsius. Other properties of polycrystalline alumina, such as the tensile strength and hardness, are dependent on the grain size, porosity and purity of the alumina. Alumina possesses high chemical stability, but, in a sodium atmosphere above 1100 degrees Celsius, sodium diffusion will occur. This leads directly to chemical reactions and microcracking.[57]

Silicon carbide has a CTE of  $5.8 \times 10^{-6}$  per degree Celsius at 1000 degrees Celsius, and a melting temperature of 2830 degrees Celsius. It is strong, dense, and thermally shock resistant. While it readily oxidizes to form an outer layer of silica ( $\text{SiO}_2$ ), this layer will protect the material from further oxidation. Silicon carbide is used to best effect in a reducing atmosphere.[57]

Zirconia is a less attractive option, because pure, or unstabilized, zirconia undergoes a change in structure, from monoclinic to tetragonal, at 950 degrees Celsius. This results in an approximately 9 percent increase in volume.[57] Sapphire fibers coated with

unstabilized zirconia experience large strains when the zirconia changes structure, and the coating suffers microcracks.[58] It is recommended that zirconia be stabilized with yttria ( $Y_2O_3$ ) to force a stable cubic structure.[57] The monoclinic form of zirconia has a CTE of  $6.5 \times 10^{-6}$  per degree Celsius at 1000 degrees Celsius, and the stabilized cubic form of  $10.5 \times 10^{-6}$  per degree Celsius at 1000 degrees Celsius.[59] When zirconia is stabilized, the strain is reduced, but residual strain results from the difference in CTEs between the sapphire and stabilized zirconia. This will likely result in a strength degradation.[58] Zirconia will react with hydrochloric and nitric acids, and is stable in oxidizing and moderately reducing atmospheres. At 2000 degrees Celsius, zirconia will react with refractory carbides and stabilized zirconia will begin to vaporize.[57]

Niobium has a CTE of  $7.1 \times 10^{-6}$  per degree Celsius, which makes it an attractive option for a sapphire coating. It resists reactions with sodium, mercury, and other at elevated temperatures, but a high sodium vapor atmosphere at temperatures exceeding 1000 degrees Celsius will result in reactions between the niobium and alumina.[57]

A research group at Drexel University has clad sapphire fiber with alumina and overcoated the clad fiber with silicon carbide. The fibers are intended for use as sensors embedded in ceramic matrix composites. The authors of the paper use a novel chemical deposition technique to ensure good adhesion between the sapphire and the polycrystalline alumina. The technique involves combining alumina particles and a polymerizable monomer carrier. The monomer is allowed to polymerize on the sapphire surface, the binder is removed, and the layer is sintered. This process is repeated several times to achieve a cladding layer 20 microns thick.[10] A 0.25 micron layer of silicon carbide is applied, via plasma enhanced chemical vapor deposition, as an outer coating to protect the embedded fiber from the surrounding environment.[32] After thermal cycling tests, performed at 900 and 1300 degrees Celsius, evidence of microcracks, debonding, or damage to the fiber was observed.[60]