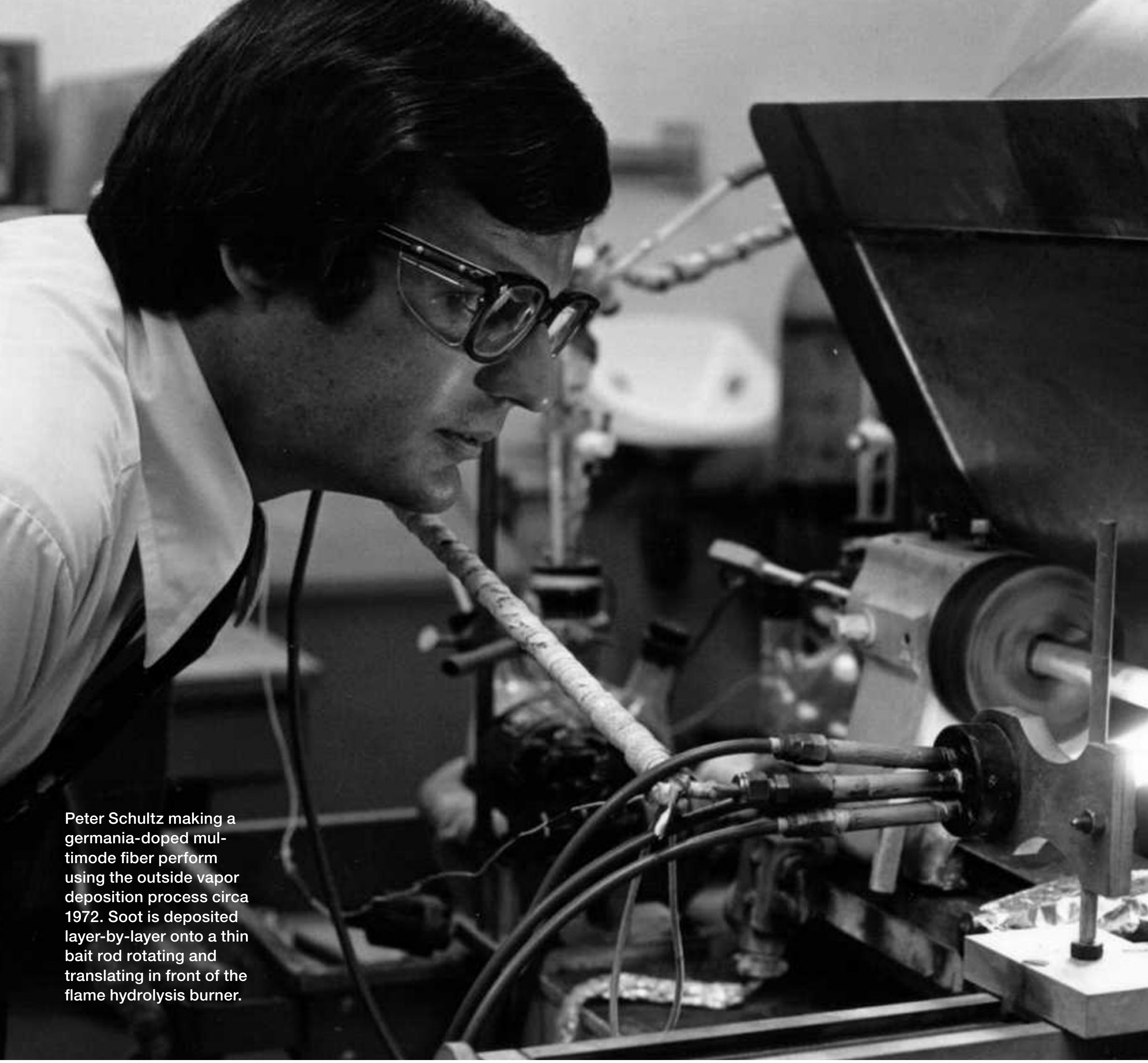

Peter Schultz making a germania-doped multimode fiber perform using the outside vapor deposition process circa 1972. Soot is deposited layer-by-layer onto a thin bait rod rotating and translating in front of the flame hydrolysis burner.

Making the First Low-Loss Optical Fibers

Peter C. Schultz

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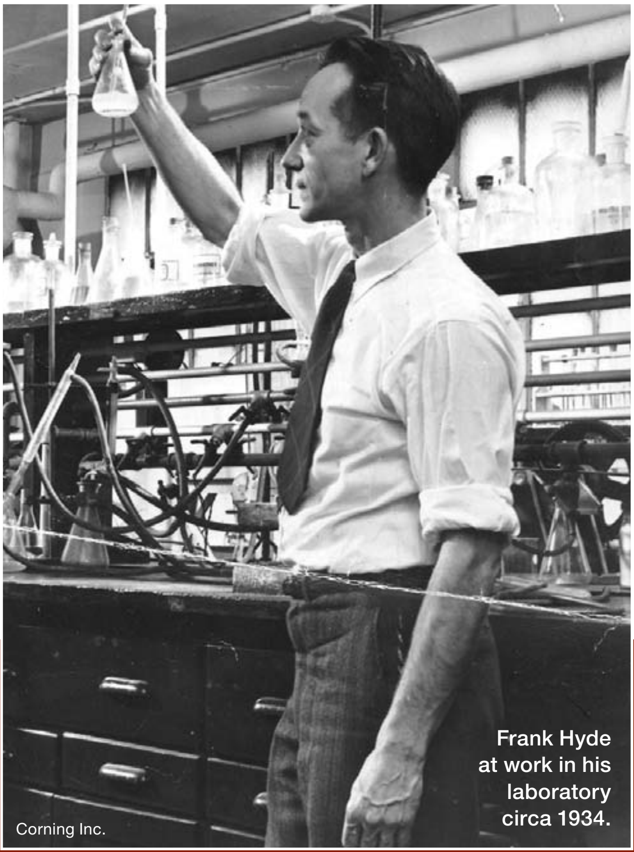
Thirty years before the laser was invented, researchers laid the groundwork for another huge breakthrough in optics—the development of low-loss fiber for communications.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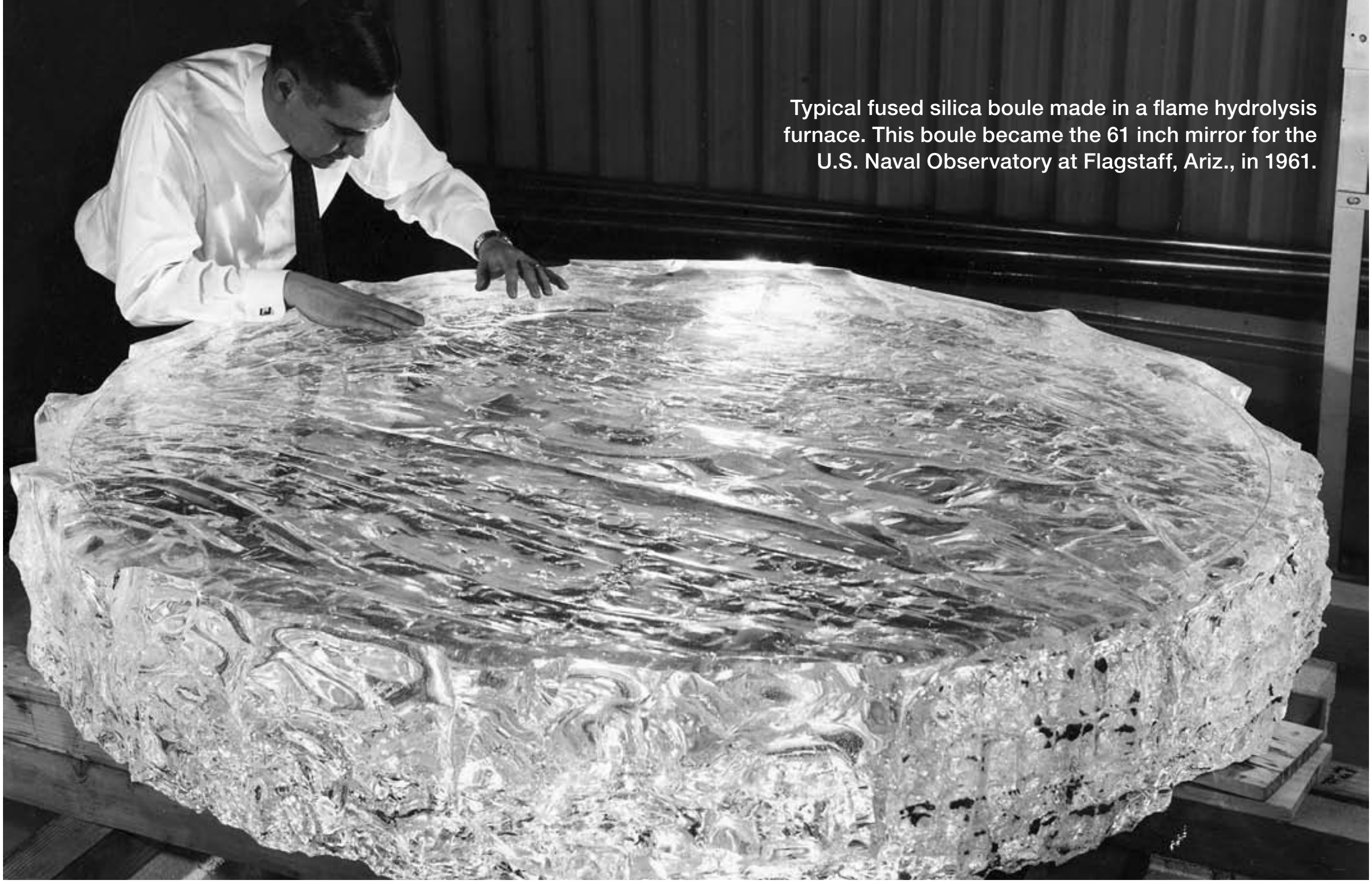
Frank Hyde at work in his laboratory circa 1934.

It all began in 1930, when a young organic chemist named Frank Hyde was hired by Corning Glass Works to explore ways to combine the newly discovered organic materials called plastics with inorganic glasses to form glass-like materials. In the course of his investigations, he received U.S. patent 2,272,342, which was filed in 1934; the patent disclosed how vapors of silicon tetrachloride, when passed through a flame, would hydrolyze to form a fine powder (which he called soot) of pure fused silica glass. This would become a fundamental step toward realizing glass-based optical fiber.

Fused silica is the most refractory, chemically resistant and thermally shock-resistant of all glasses. However, at the time it had limited uses because it could only be made by melting quartz crystals with an oxy-hydrogen torch or electric arc at temperatures exceeding 2000 °C. Even at those temperatures, it was an extremely viscous glass that was difficult to form into useful articles. The new method that Hyde discovered—called flame hydrolysis—showed promise as a way of providing an easier and lower-temperature method of making fused silica products such as tubes, crucibles and optical elements.

Hyde noted that the resultant glass had impurities in the range of 10 ppm. A few years later, Martin Nordberg—another Corning scientist—discovered that the addition of about 8 percent titania to the fused silica further reduced its already low



Typical fused silica boule made in a flame hydrolysis furnace. This boule became the 61 inch mirror for the U.S. Naval Observatory at Flagstaff, Ariz., in 1961.

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thermal expansion coefficient to essentially zero. In U.S. patent 2,326,059, he described how this ultra-low expansion glass could be made by Hyde's flame hydrolysis process using mixtures of titanium and silicon tetrachloride vapors. However, no commercial silica products were developed at the time.

In the 1950s, the flame hydrolysis process was resurrected at Corning to meet a growing demand for large clear blanks of pure fused silica that could be formed into low-attenuation acoustic delay lines for radar and large mirror blanks for a new generation of high-precision astronomical telescopes. In this new iteration of the process, multiple flame hydrolysis burners were arrayed on the top of a refractory brick furnace.

The burners created the silica soot streams and heated the furnace to >1700°C to simultaneously deposit and fuse the soot into a clear glass blank (called a boule), layer-by-layer in a rotating refractory cup. The resultant pure fused silica boule, called Code 7940 glass, was then shaped by grinding and polishing into delay lines, mirrors and even windows for space craft. This method was also employed to make Nordberg's ultralow expansion titania-doped silica glass boules (called ULE Code 7971), which were used for mirrors, including lightweight optics for spy satellites and the Hubble telescope.

[Flame hydrolysis boule furnace process]

Flame hydrolysis boule furnace process showing the flame soot stream depositing pure fused silica glass into a rotating refractory cup.

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Glass and fiber optics

During the 1950s, Robert Maurer, a physicist at Corning, carefully studied relative light scattering in glass. His results, published in 1956 and 1960, indicated that the boules of Code 7940 fused silica had the lowest Rayleigh scattering of all glasses he measured (roughly 10X lower than the best optical glasses).

Corning scientists first became aware of the need for low-loss optical fibers for communications on June 17, 1966. At Electrosil, a subsidiary of Corning in Sunderland, United Kingdom, William Shaver met with several representatives of the British government, including members of the ministries of aviation, radar and signal research and development who were looking for help to make fiber. Shaver was a roving scientist for Corning: He visited laboratories and companies around the world in search of new opportunities for its materials and products.

When Shaver returned to Corning, he told William Armistead, the head of Corning's R&D facilities, about the

My first assignment at Corning was to take a fresh look at Hyde's flame hydrolysis process and see what else could be done with this technology.

developing need for pure glass fiber optics. Armistead was interested, and he decided to pursue the idea further, assigning the task to Bob Maurer, in part due to Bob's earlier work on light scattering in glass.

Initial design requirements that had been given to Shaver in that meeting were for a single-mode fiber (100-µm-diameter with 0.75 µm core) having a total attenuation of roughly 20 dB/km. To put this attenuation goal into perspective, keep in mind that the very best *bulk* optical glasses of the day had attenuations of about 1,000 dB/km—and these were not in this special fiber form. This meant that an improvement in transparency of 10^{98} was needed to reach the 20 dB/km goal! Obviously, a process was needed to make such glass in this difficult fiber geometry. It was hardly obvious at the time that these goals could ever be reached.

Bob Maurer was familiar with the work of Eli Snitzer of American Optical Corporation, published in 1961, in which short single-mode glass fibers were first demonstrated. Bob began his work with a summer intern in 1967, drawing short lengths of single-mode fibers from conventional glass fiber cores placed in capillary tubes (a rod-in-tube process). Although the losses were very high as measured with HeNe laser light, the results suggested that better glasses and processes might yield better results.

The fused silica approach

Influenced by his earlier work on light scattering, he decided to look at fused silica. A furnace capable of drawing this high-temperature glass was available in the laboratory and thus—again using the rod-in-tube approach—he made fibers using commercially available Code 7940 fused silica as the cladding and ULE® Code 7971 titania-doped fused silica as the core (since its refractive index is higher than pure fused silica). Losses were still very high (about 10 dB/meter), but Bob was encouraged enough to continue, and Bill Armistead agreed. He approved the addition of two scientists to this low-key effort in late 1967.

I was one of those new additions. I joined Corning as a scientist in July 1967, direct from Rutgers University, where I had earned a Ph.D. in glass science. My first assignment was to take a fresh look at Hyde's flame hydrolysis process and see what else could be done with this technology. I built a small boule furnace in my lab and began making various doped fused silicas and measuring their properties.

In January 1968, Don Keck, a Ph.D. physicist, joined Corning and began working full-time on the fiber project, having been recruited from Michigan State by Bob. Based on Bob's earlier results, we focused our efforts exclusively on fused silica fibers made by flame hydrolysis. These were the purest glasses made that we were aware of and we had some expertise to process them into fiber. However, our approach was a bit counterintuitive in that we were essentially adding an "impurity" to the pure fused silica to raise the refractive index in order to create the fiber core.

Again, it was not at all clear that this "doping" technique would work. Much later, we learned that we were the only group pursuing this high silica approach at the time. Our initial results were no better than Bob's. We tried various methods of cleaning and polishing the rods of ULE® and tubes of fused silica, but still the losses remained far too high.

We tried depositing the fused silica cladding onto a core rod inside my small boule furnace. That didn't work either. We found that one source of the high loss was the formation of reduced titanium (Ti^{3+}) color centers during the high- temperature fiber-drawing step.

We learned to anneal these away by heat-treating the fibers at 800 to 1000 °C, but this in turn drastically weakened the fibers due to surface crystallization. This necessitated an additional process step wherein the fibers were passed through a hydrofluoric acid bath to remove these surface flaws and thus regain some of the fiber strength lost during heat treatment. We experimented with lower levels of titania. Although we made some progress, losses were still very high—not much better than conventional glasses.

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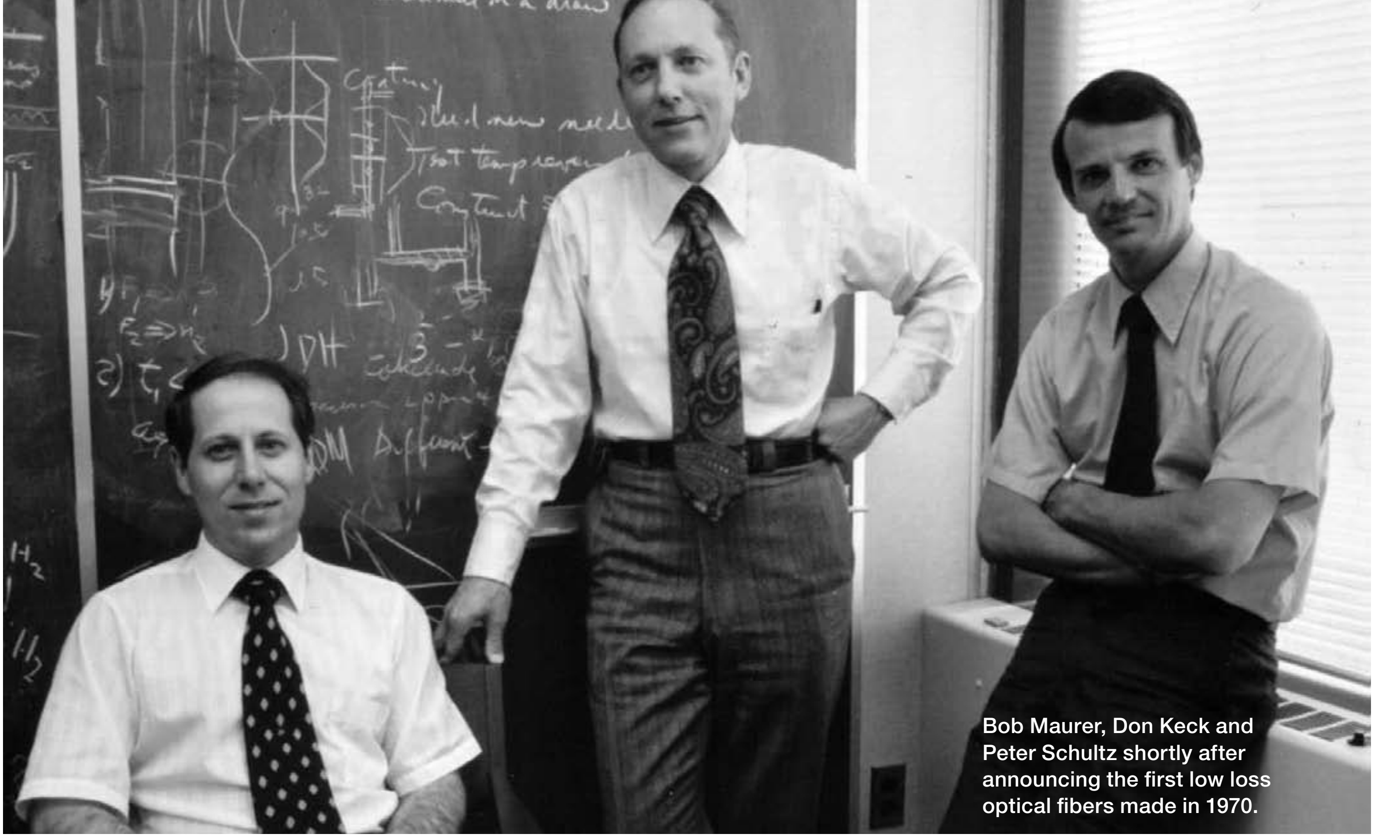
4-Actual laboratory equipment used to make the first sub-20 dB/km optical fibers. A burner emits core glass soot which is deposited onto the inside surface of a rotating silica glass cladding tube held by a lathe head. Deposition of the soot is assisted by a vacuum cleaner attached to the exit end of the tube (not shown).


Bob Maurer, Don Keck and Peter Schultz shortly after announcing the first low loss optical fibers made in 1970.

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Whoopee!

Then we hit on an idea that proved to be the key: Since the core of the single-mode design was only one-tenth the diameter of the fiber, why not try depositing a thin layer of core glass soot onto the inside surface of a flame-polished thick-walled cladding tube? This might greatly reduce light scattering at the core-clad interface, which by now we knew to be a major loss source. After a series of experiments to develop the process, we made our first fiber preform.

The equipment we used was crude but effective. A lathe headstock from a large ball-bearing held the rotating cladding tube in front of a flame hydrolysis burner. The burner produced a soot stream containing 1.5 percent titania-doped silica. When at first our soot-stream didn't go into our tube, we attached a lab vacuum cleaner to the tail end of the tube and sucked the soot from the flame, depositing a thin layer onto the inside tube surface. We then placed this coated tube in the fiber draw furnace where the soot sintered into a clear glass layer, and the hole collapsed to form a solid rod containing the doped core—which was then drawn down into fiber. After heat treatment, we measured the fiber loss.

To our great surprise and delight, after several attempts, the attenuation was reduced to 17dB/km. We had done it! After making this now-famous measurement, Don Keck wrote in his lab notebook: "Whoopee!" That first fiber was very short, and it was difficult to measure such low loss, so we went to work making more fibers by this approach (wearing out several vacuum cleaners in the process).

We confirmed our results, reaching a low value of 16dB/km on an almost 1-km fiber. This time Don wrote in his notebook the more reserved response "Q.E.D.," an abbreviation of the Latin *quod erat demonstrandum*, which literally means "that which was to be demonstrated," and indicated that we had established our proof.

In May 1970, we filed patent applications disclosing the use of doped fused silicas and the method of making these fibers by depositing the core glass on the inside of a cladding tube. U.S. patents 3,659,915 and 3,711,262 would prove to be very valuable to Corning in future years.

In September 1970, Bob attended an Institution of Electrical Engineers conference in London and publicly announced that we had made a fiber with a total attenuation of only 16dB/km, and we published an article in *Applied Physics Letters* shortly afterward. Ironically, a review article published in the same month in the *Proceedings of IEEE*, titled "Optical Communications—A Decade of Preparations" stated: "At the present time, the glass used in fiber optics is very lossy, amounting to a decibel per meter at the very best…..which makes the material clearly unsuitable for long-distance transmission."

Making it to manufacturing

We were careful not to discuss the details of our fiber compositions and fabrication methods outside of the lab, since we soon realized we were far ahead of our competition, including the British Post Office, STL and AT&T. At their request, Bob brought samples of our fragile fibers to these companies so they could verify our measurements. He tried to carefully gather up every scrap of fiber that broke off during handling in these tests, but he did not always succeed. Once the attenuation results were verified, the labs' efforts to catch up to us intensified, since their earlier work focused primarily on millimeter waveguide technology.

Despite the fact that our breakthrough fiber solution was not exactly robust—since we could only make small preforms and

To our great surprise and delight, after several attempts, the attenuation was reduced to 17dB/km. We had done it!

the troublesome heat treatment required to achieve low attenuation made the fibers brittle—Corning management decided to move the project into the development stage in 1971.

Engineers were assigned to look for ways to make the fiber manufacturable and to supply samples to potential customers and joint development partners that the business team was lining up. Meanwhile, I focused my attention on evaluating other dopants to try to eliminate the troublesome fiber heat treatment step required with titania doping. We also learned that a preferred fiber design was now multi-mode rather than single-mode, in order to take advantage of commercially available LED light sources that could be more readily coupled into the large core of multimode fiber.

To make such fibers (both step- and graded-index), we developed another flame hydrolysis approach, later dubbed "outside vapor deposition" (OVD). In this method, first the core and then the cladding soot were deposited onto a rotating bait rod to build up a porous soot perform. This preform (containing both the core and cladding of the fiber) was then removed from the bait rod and sintered into a bubble-free glass blank in a gradient furnace at 1350 °C in a helium atmosphere.

Introducing dopants

By keeping the core glass at these lower temperatures, we could now incorporate dopants that vaporized in the higher temperature boule process. One of these was germania, a glass-former similar to silica. In June 1972, we drew the first multimode preform, incorporating 9 percent germania in the core. We immediately knew that we were on the right track when we could still see the bright light of the draw furnace shining through the end of a kilometer of fiber on the wind-up drum. Don measured only 4 dB/km without the need for any heat treatment. Fiber strength was excellent. The first truly practical low-loss fiber had been made.

The OVD process and germania-doped fibers were quickly transferred to the development stage and scaled up to make larger and larger performs of multi-mode graded index fiber. In early 1973, we published a paper in *Applied Physics Letters* predicting that the fiber loss could reach as low as 2 dB/km beyond 800 nm in these multi-mode fibers. Incidentally, today it stands at 2.3 dB/km at 850 nm and 0.6 dB/km at 1,300 nm in multi-mode fibers and 0.18 dB/km in single-mode fiber at 1,550 nm. In 1975, Corning began building a pilot production facility that came on line in 1976. The commercialization of our inventions was underway.

However, Bob, Don and I had a new and unexpected career awaiting us: that of witnesses in patent litigation. To protect its position, Corning aggressively defended its fiber patents. The first suits were filed in July 1976 against IT&T and the U.S. government. Ironically, it was during the IT&T depositions that Don and I became aware of Charles Kao's articles proposing optical fiber for communication. These lawsuits were quickly followed by other suits against Canada Wire and Cable, Philips/Valtec and, finally, Sumitomo.

We three spent countless hours in depositions and trials. However, in all cases, the patents were held valid, enforceable and infringed. These actions helped shape the industry and put Corning in the role as a leading fiber supplier for many years to come. In one of the last trials, decided in October 1987, Federal Judge William Conner of the U.S. District Court of southern New York wrote in his opinion:

"Here, the validity of the Corning patents is confirmed by the presence and strength of all these objective criteria of non-obviousness…After years of unsuccessful search, when industry experts had all but despaired of reaching the goal, Corning had an unexpected "breakthrough" which was immediately recognized as the long sought solution and enthusiastically acclaimed. It literally created a new industry of substantial size…Pioneer patents, like the '915 patent, are entitled to liberal construction and a broad range of equivalents."

Today, 40 years and 1.3 billion km of fiber later, virtually every communication fiber is still made by soot-deposition processes and using glass compositions that can be traced back to these original inventions.

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[References and Resources]

- >> R.D. Maurer. "Light scattering by glasses," J. Chem. Physics **25**(6), 1206-9 (1956).
- >> R.D. Maurer. "Light scattering by neutron-irradiated silica," J. Phys. Chem. Solids **17**(1/2), 44-51 (1960).
- >> E. Snitzer and H. Osterberg. "Observed Dielectric Waveguide Modes in the Visible Spectrum," J. Opt. Soc. America **51**(5), 499-505 (1961).
- >> M.W. Jones and K.C. Kao. "Spectrophotometric studies of ultra-low-loss optical glasses II: double beam method," J. Sci. Instruments series **2**(2), 331-5 (1969).
- >> F.P. Kapron et al. "Radiation Losses in Glass Optical Waveguides," Appl. Phys. Lett. **17**, 423-5 (1970).
- >> D.B. Keck et al. "On the Ultimate Lower Limit of Attenuation in Glass Optical Waveguides," Appl. Pys. Lett. **22**(7), 307-9 (1973).
- >> J. Hecht. *City of Light—The Story of Fiber Optics*, Oxford Univ. Press (1999).
- >> M.B.W. Graham and A.T. Shuldiner. *Corning and the Craft of Innovation*, Oxford Univ. Press (2001).