



Column Watch

Guest Author
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Although fused-silica capillary enables separation methods unimaginable prior to its introduction, most manufactured capillaries are highly variable in quality and less than optimal for any application. In this month's "Column Watch," guest author Steve Griffin identifies the fundamental problems in capillary manufacturing techniques that cause capillary defects and suggests some corrective actions.

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Fused-Silica Capillary — The Story behind the Technology

The advent of fused-silica capillary columns for gas chromatography (GC) was a major technological development in analytical sciences in the twentieth century. The story of how fused-silica capillary columns were discovered was the subject of an earlier *LCGC* article by the inventors (1). Not only have fused-silica capillary columns revolutionized GC, but the newer separation science technologies — capillary electrophoresis (CE), capillary LC, and capillary electrochromatography (CEC) — perhaps wouldn't have been explored without the availability of fused-silica capillaries. Most recently, nano-LC columns with inner diameters of less than 100- μm also have been fabricated from fused-silica tubing as well as from polyetheretherketone (PEEK) tubing.

Although individual end users might have little control over the coated capillary columns that they acquire from manufacturers, their understanding of the chemistry, physics, and mechanics of the underlying blank capillary can be beneficial in maximizing the performance of columns made of fused silica. In this installment of "Column Watch," I will discuss how capillary quality problems affect columns during manufacturing and chromatographic use. Specifically, I will cover the vital role of materials and methods in improving the quality and performance of the capillaries used in separation science columns.

Background

It has been almost 25 years since Ray Dandeneau and Ernie Zerenner's landmark paper announced the development of wall-coated open tubular (WCOT) fused-silica columns based upon synthetic fused-silica capillaries (2,3). Silica-based columns provided huge advantages over the preceding borosilicate technology and became the accepted standard much more rapidly than is characteristic of new equipment in analytical chemistry. Since then, researchers have made little progress in improving the base material — the blank silica capillary

— upon which most capillary GC columns, and essentially all columns for CE methods, are based.

The technology that permitted the production of fused-silica capillaries was adapted from two disparate emerging technologies: fiber optics and microelectronics. Although microelectronics and fiber optics have grown exponentially because of the rapidly expanding needs in various industries, fused-silica capillaries for chromatography continue to be made from the original materials and with the same basic equipment used almost three decades ago, but with continued advancement in production methods and materials.

Most synthetic silica tubing produced now is optimized chemically for telecommunications fiber production — a chemistry that has major drawbacks for use in chromatographic applications. Relative to telecommunication needs for optical fiber, the miniscule size and slow growth of the chromatography column industry has offered little incentive for investment in research and development since its introduction. The small improvements in capillary yields and overall quality are overshadowed by the fact that most of the adverse quality issues of two decades ago remain. Some of these problems include variations in capillary bore and outer dimensions, ovality, random brittleness, and surface activity. Even when chromatographers don't notice the actual problem, column prices are affected by lower yields caused by capillary rejection or column failure in the column production process.

Capillary Drawing Process

The types of manufacturing equipment that draw optical fiber and capillary tubing for chromatography share similar characteristics. Figure 1 shows two typical configurations of drawing and coating machines. Prepared synthetic silica tubes are fed into an inert, gas-shielded, open tubular, graphite furnace running at approximately 2000 °C and located approximately 10 m

above the factory floor. The silica melts in a precisely controlled heat zone and is drawn down to precise dimensions by a controlled-speed capstan located far from the furnace. Along the draw path, as many as six layers of polyamic acid solution are applied and dehydrated to form thin layers of polyimide on top of the glass. The bare glass outer diameter and cured coating diameters are monitored by laser micrometers in two orthogonal axes, and these meters have the ability to report and record minimum and maximum dimensions and ovality and to detect flaws, on-line and in real time. Currently, no commercially available means can monitor the capillary bore diameter on-line, although some researchers are working to fill this need. Limits in the commercially available sizes of the raw silica tube limit maximum continuous draw lengths to approximately 6 km for 0.53-mm capillaries or approximately 15 km for 0.25-mm capillaries. Drawing speed range is approximately 3–40 m/min, depending upon the material dimensions, equipment design, and individual manufacturer's philosophy.

Capillary drawing is a bit more complex than optical fiber drawing for various reasons. An obvious difference in capillaries is the presence of an open bore that is absent in optical fiber. To prevent the formation of acids from atmospheric components within a silica tube during drawing, manufacturers generally use an inert gas purge or pressurization. Although they are of relatively high quality, the base silica tubes used for feeding the draw are not perfect in their dimensions, in the concentricity of the inner bore

to the outer diameter, or in circularity of the inner bore and outer diameter. Even low pressures cause these defects to be accentuated, and the most obvious result of these defects is glass ovality or noncircularity. Complicating the issue is the fact that the molten glass changes viscosity with temperature, and those viscosity changes alter the degree to which the internal pressure affects the capillary dimensions and geometry. As the glass becomes more oval, the polyimide coating becomes less uniform and circular (see "Control of Capillary Dimensions" section below).

Residual glass stress is an amazingly common column flaw that results from an improper design of the overall draw system. As an example, Figure 1b shows how capillaries must traverse several large wheels, numerous guide systems (used to adjust or maintain the capillary alignment with coating and cure equipment), and viscous resin buckets equipped with flexible silicone nipples before the capillaries reach the capstan that controls the draw speed. To maintain tension throughout this type of manufacturing system, the glass must be drawn cold. As any glassblower can attest, cold temperatures are a very bad idea and result in highly localized stresses in the glass produced. Silica is very forgiving in this respect, when tensile strength alone is the issue. Molten glass, even pure silica, is not terribly resistant to free flow, so tension is not an inherent requirement for pulling from a melt, and the resulting highly localized stresses cause major problems when cutting the capillaries and coiling them on cages.

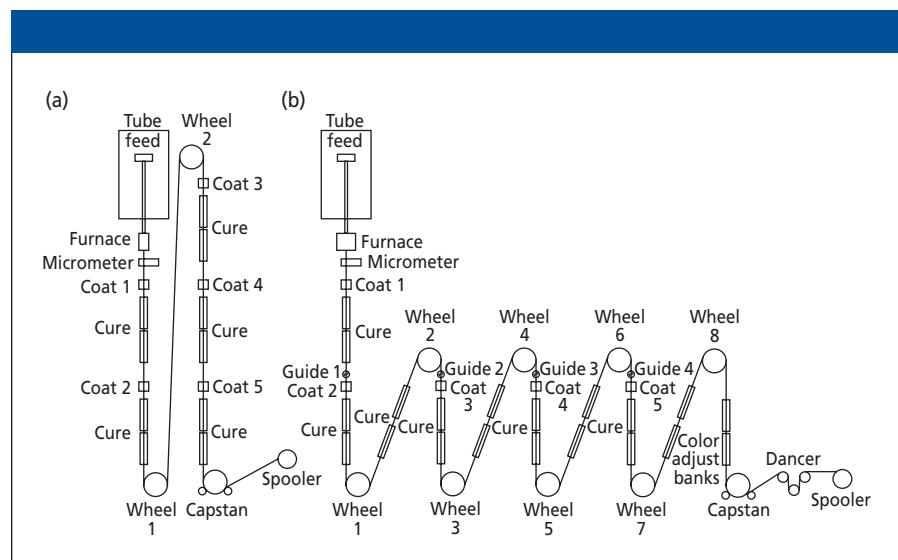


Figure 1: Two typical configurations of drawing and coating machines: (a) slow draw at low tension versus (b) fast draw at high tension.

Polyimide Coating

To produce more-rugged capillary columns, manufacturers use polyimide coatings to protect the outer fused-silica surface from abrasive damage and subsequent breakage. A tiny scratch or 1- μm pit on the silica glass surface can cause almost-immediate column failure, so manufacturers must exercise great care in processing fused-silica capillaries. Although polyimide-coated fused silica is much more rugged than bare glass, it also must be handled with care. The coating is a relatively high molecular weight polyimide derived from 3,3',4,4'-benzophenone tetracarboxylic acid dianhydride with 4,4'-oxydianiline and *m*-phenylene diamine, either alone or layered with another more thermally stable polyimide. Figure 2 depicts the chemical reaction used to prepare polyamic acid that, after dehydration, forms the typical yellow–orange or brown coating on the column exteriors.

Note that these polyimide coatings usually cost more themselves than the synthetic silica.

Inconsistencies in applying and curing the polyimide coatings and high-temperature-draw-induced residual stress in the glass wall combine to make brittle capillaries. Furthermore, a basic lack of understanding of proper polyimide application and curing processes can cause lumps, particulate inclusions, and blisters in the finished coating, which further decreases the strength of the capillaries. Most brittle capillaries are scrapped during column manufacturing, but some causes of

brittleness delay the discovery of the flaw until after column production and quality assurance.

The coatings used on silica capillary are designed for casting dielectric films on the silicon substrates used in semiconductor chip manufacturing. Applied as relatively low solid content solutions of polyamic acids in *N*-methyl-2-pyrrolidone, the spin coating and slow thermal curing programs recommended by polymer manufacturers simply are impossible to duplicate, even in slow-speed capillary draw conditions. To complicate matters more, it generally is impractical to apply more than approximately 5–6 μm (cured film thickness) under continuous process conditions, so manufacturers must make multiple layers to achieve the desired product characteristics.

Compressing hours of thermal curing into a few seconds involves compromises. The window between unacceptably incomplete imidization or solvent removal and thermally damaged polyimide is extremely narrow. It becomes impossible to perform this delicate reaction if the thickness of the polyamic acid resin varies substantially around the capillary or along its length. Surface-tension effects alone can cause considerable unevenness in the film-thickness distribution, so readers can appreciate the problems introduced by any ovality of the substrate.

If an inadequately cured layer of polyimide is covered with a more fully cured layer, bubbles or blisters can form because of rapid vaporization of the trapped water

of imidization, residual solvent, and volatile monomer contaminants when heat is applied during the curing of succeeding layers. Larger coating defects of this type generally are caught on-line by blister monitors, which are laser micrometers in lump-detection mode, and defective capillaries are discarded. If the blisters are too small, they can be missed. Other sources of lumps, bubbles, and blisters exist, but their causes are obvious and completely avoidable with a well-controlled manufacturing process. These blisters are tiny areas of extremely thin polyimide that readily oxidize at GC column operating temperatures. The resulting voids on the column are zones of extreme susceptibility to physical damage.

This failure mode — oxidation of thin polyimide zones or voids produced by accelerated failure of the coatings around dust particles — limits the upper-temperature use limit and accumulated high-temperature exposure of capillary GC columns. Polyimides can withstand significantly higher temperatures than those to which they are exposed in GC applications, if oxygen is excluded. Often, the processes used in column manufacture consume much of raw capillaries' capacity for high-temperature exposure. Innova-Quartz (Phoenix, Arizona) recently introduced a gold-over-polyimide coating designed to reduce oxygen partial pressure at the polyimide surface and to provide expanded temperature ranges for GC columns.

High-precision, fixed-diameter, pressurized die coating application techniques have been extremely reliable for large-scale telecommunications fiber manufacturing. They also have been used successfully with polyimides but are not in current use by major fused-silica capillary manufacturers because of equipment or process limitations. Currently, open cup or flexible applicators and ceramic fiber-insulated, resistance coil curing ovens are the standards. This equipment causes continuous and increasing contamination of the polyamic acid resin reservoir throughout the draw through a combination of atmospheric water, dust, and debris. Inappropriate curing technology yields an imprecise and irreproducible polymer cure state within a single draw and even circumferentially around a capillary. These inconsistent curing states are demonstrated by the fact that microblisters almost always are found only on one side of a capillary, where the polymer was applied too thickly or the interior layers were undercured. Improvements in

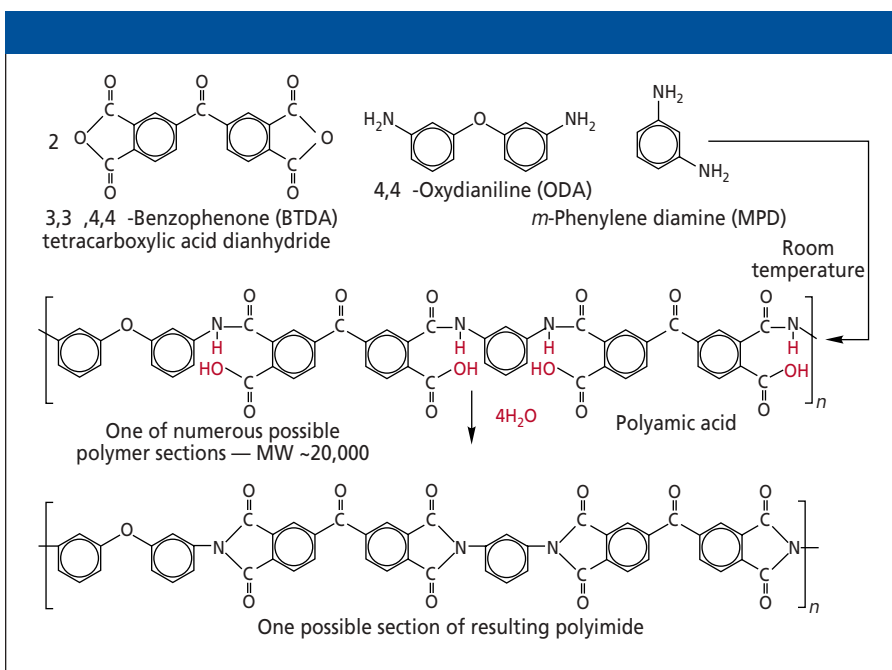


Figure 2: 350 °C continuous-use polyimide.

polyimide coating techniques would result in major improvements in the reliability of fused-silica capillaries.

Two remaining adverse affects of improperly applied and cured polyimide coatings plague column manufacturers and end users. Undercured polyimide is relatively soft and does not yield easily to most tools used for cutting capillary columns. Clean cuts (defined as cuts at approximately 90° to the column axis with only minor protrusions or voids of glass or polymer and no silica chip formation) are quite difficult to achieve with poorly cured polyimides. Without a clean cut, any connection within a GC system is subject to leakage and even complete column failure or fracture. In addition to the obvious problems that the projections of glass or polymer voids will cause for forming seals in press-fit-type glass unions, irregular glass edges can result and, inversely, can cause high-stress concentrations from which future fractures will propagate, particularly in the thermal cycling that occurs during repeated temperature-programmed runs. Polyimide projections and voids are symptomatic of delaminating coatings and will drastically shorten a column's life. In addition, any polyimide that got into a capillary's inner bore could cause uneven flow patterns or unfavorable interactions with certain polar analytes.

A more immediate problem is silica chip formation. Attracted by the electrostatic forces generated by wiping the column exterior clean before cutting it, tiny silica glass chips generated by bad cuts can adhere to a column's exterior or lodge deep inside the column bore. When a capillary is inserted into any type of connector and silica chips are present on the inner or outer surface, a great potential for column failure exists. If a chip on the exterior is forced through the protective polymer and scribes the capillary glass wall, any future

tensile stress likely will cause a fracture. Similarly, a chip lodged inside a capillary can be forced along the length of the column by carrier-gas flow. A column can be severely damaged through repeated impacts of the chip with the column's inside wall. All these coating problems can be avoided through proper design, the use of pressurized hard die coating systems, and appropriate cure oven design and use.

Raw Materials and Capillary Activity

Separation science applications of silica tubing represent a relatively insignificant market to manufacturers of the base tubing materials (primarily Heraeus Tenevo AG, Hanau, Germany). Fiber optic communications is the driving force in that industry, and all of the new, improved forms of pure silica are designed for those applications. In many respects, separation scientists are fortunate to have continuing access to the materials at all.

When silica capillary was first made, synthetic silica possessed a relatively high bulk water content (reported as $[\text{OH}^-]$ at 400–600 ppm), captured in the glass during the consolidation of the hydrolysis product of silicon tetrachloride (some chlorine also remains at approximately 125 ppm). This contaminant water limited the efficiency of light transmission in the near-infrared wavelengths used in telecommunications. Consequently, manufacturers expended massive efforts to reduce the contaminant levels. Today, most fused-silica tubing has a water content of less than 0.2 ppm. Although this improvement might seem advantageous to GC — because the reduction in bulk $[\text{OH}^-]$ also lowers the inherent surface activity in the capillary — higher levels of chlorine (approximately 1500 ppm) replace the water when the freshly formed silica is dried with chlorine gas. This chlorine pre-

sents no problem in optical applications, but it is extremely difficult to deal with when making GC capillary columns.

Earlier, I discussed the need to draw capillaries in an inert internal atmosphere. If the ends of the silica tube being drawn are open to the atmosphere outside of the furnace's inert gas curtain, acids of nitrogen will form on the silica surface. In combination with atmospheric water, the surface siloxane will be hydrolyzed to silanol (Figure 3).

On extremely humid days, capillary drawn from open tubes can contain fairly surprising volumes of acid solutions. Accordingly, tubes with bulk silanol content in the 400–600 ppm range (standard high-OH silica) can produce capillaries with surface silanol concentrations that are considerably greater than the bulk concentration. Worse yet, the extent to which this surface modification occurs is irreproducible, because it is dependent upon several uncontrolled variables. An obvious moderation of this unintended, acid-catalyzed surface activation, if not a complete solution, is to fill the tube being drawn with inert gas to displace the atmosphere. In fact, beginning more than a decade ago, most GC capillaries have been drawn with some grade of inert gas in the bore. As currently practiced in the industry, this solution has been incomplete and has its own undesirable consequences. Tight control of the gas quality and pressure is necessary for this technique to provide any benefit.

I do not believe that high inner-bore surface activity is a problem in all cases. In fact, CE applications rely upon surface silanols to establish conditions that permit electroosmotic flow. In GC column production, high-activity silica might be helpful for some separations or stationary phase-coating processes. However, unknown and continuously changing surface activity is problematic when predicting the stationary phase-to-glass surface interactions that greatly affect the thickness and uniformity of applied stationary phases.

Anthony and co-workers (4) showed that the modified chemical vapor deposition process can deposit low-activity silica within the bore of standard, high $[\text{OH}^-]$ silica tubes. These tubes, drawn into capillaries, are superior because reversible and irreversible retention and degradation of susceptible compounds is almost eliminated on bare silica capillaries without deactivation. With current capillary production yields, this interior coating process

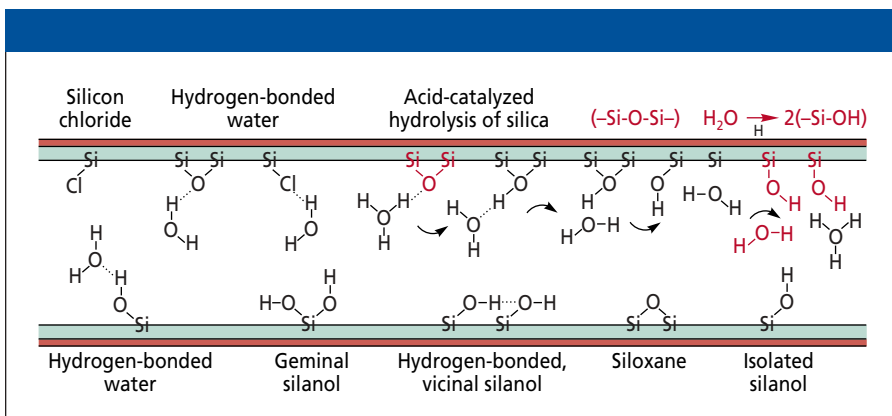


Figure 3: Silica surface chemistry.

Table I: Published capillary dimensions and tolerances by manufacturer

| IQinc | | MicroQuartz* | | | Polymicro Technologies† | |
|-----------------------------|---------------------|-------------------------------|---------------------|-------------------------------|-------------------------|-------------------------------|
| Nominal Inner Diameter (mm) | Inner Diameter (mm) | Polyimide Outer Diameter (mm) | Inner Diameter (mm) | Polyimide Outer Diameter (mm) | Inner Diameter (mm) | Polyimide Outer Diameter (mm) |
| 0.18 | 0.180 ± 0.005 | 0.363 ± 0.006 | 0.180 ± 0.010 | 0.370 ± 0.035 | 0.180 ± 0.010 | 0.340 ± 0.010 |
| 0.25 | 0.250 ± 0.006 | 0.363 ± 0.006 | 0.250 ± 0.012 | 0.370 ± 0.035 | 0.250 ± 0.012 | 0.360 ± 0.015 |
| 0.32 | 0.320 ± 0.007 | 0.440 ± 0.008 | 0.320 ± 0.012 | 0.450 ± 0.040 | 0.320 ± 0.012 | 0.435 ± 0.015 |
| 0.45 | 0.450 ± 0.007 | 0.670 ± 0.012 | 0.450 ± 0.012 | 0.690 ± 0.050 | N/A‡ | N/A |
| 0.53 | 0.540 ± 0.008 | 0.670 ± 0.012 | 0.525 ± 0.017 | 0.690 ± 0.050 | 0.542 ± 0.012 | 0.673 ± 0.025 |
| Diameter ± (%) | 2.1% | 1.7% | 4% | 8.5% | 4.1% | 3.6% |

*MicroQuartz GmbH (Munich, Germany), div. of Siemens AG. The MicroQuartz data were converted to a standard ± format for ease of comparison.
†Polymicro Technologies LLC (Phoenix, Arizona).
‡N/A = not available.

is too expensive for general applications. By adopting at least some of the advances in automated manufacturing that have been developed by the telecommunications industry, capillary manufacturers could gain higher yields and a lower labor component for enough savings to permit interior surface modification with low-activity silica without significant price increases.

Control of Capillary Dimensions

Another challenge in the production of capillaries is maintaining control over the capillary dimensions, not only of the outer and inner diameters but of the ovality as well. It is obvious from the practice of good chromatography that the inner bore of a capillary should be as consistent as possible throughout its entire length. The determination of stationary-phase film thickness, phase ratio, and carrier-gas linear velocity is dependent upon the inner bore consistency, and the use of retention indices and retention-time measurements assumes a constant bore from column to column. Dimensional variations result in unnecessary nonuniformities in stationary-phase thickness and variations in height equivalent to a theoretical plate (HETP) throughout the column lengths. Although techniques such as multiple internal standards and retention-time locking (5) will compensate for some variations, maintaining a consistent inner bore is good chromatographic practice.

Most nominal capillary dimensions are similar among capillary manufacturers, but dimensional tolerances differ substantially. In the case of 0.53-mm capillaries, even the nominal dimensions differ considerably. As bore size decreases, the traditional tolerances become a significant percentage of the nominal dimensions. For simplicity, I will consider only large-bore GC columns in this "Column Watch."

Table I summarizes published dimensions and tolerances for independent GC capillary manufacturers. In the 0.53 row, the 0.53-mm-bore columns are shown to be within specifications with a bore as small as 0.508 mm or as large as 0.554 mm. Although 46 µm of diameter range may seem trivial, it represents approximately 10% variance in the optimum linear gas velocity of the mobile phase in columns that are supposed to be identical and almost 20% difference in volume for columns of identical length.

In terms of capillary outer diameter, gas-tight connections in compression fittings and in press-fit connections require a consistent diameter and minimal ovality. Better control of capillary outer dimensions not only is possible but also is absolutely necessary to exploit the advantages of pressurized hard die coating systems, which are inherently intolerant of wide variations in glass diameter. Although small variations in the silica feed-tube dimensions often are cited for limiting the precision to which dimensions can be maintained, this claim is unsupported based upon simple geometric ratios. Feed rates for silica tubes into draw furnaces are on the order of millimeters per minute, and draw speeds are on the order of meters per minute. If a trend in diameter change is detected online — and only gentle trends should be detected for processes that are in control — computer feedback control of the tube feed rate easily can compensate. It is a vast oversimplification to compare diameter control of pressurized bore capillary draws with telecommunications optical fiber production; however, the fundamentals remain the same. In telecommunications, anticipatory controls of this type permit maintenance of glass diameters to a precision of +1 µm/−0 µm. This level of precision should be approachable in fused-silica capillary drawing.

Another dimensional parameter that warrants careful control is the ovality of the fused-silica capillary. The raw tubing material sometimes is not circular and presents excessive ovality in the glass and polyimide coatings. This ovality characteristic is especially important if chromatographers use press-fit silica connectors to join capillaries or to split flows into dual columns. Press-fit silica connectors have improved to a point at which they provide extremely consistent performance. Previous versions were made by collapsing a silica tube bore with a torch to form a curving taper. Highly precise, constant-angle tapers that are formed by computer numerical control laser machining largely have replaced these early devices. For example, InnovaQuartz's laser production process yields glass connectors that provide reproducible performance with greater than 6σ probability or less than one failure per 1 million units. Although these modern glass press-fit connectors are extremely forgiving for generalized dimensional errors, it is unreasonable to expect them to form good seals on highly oval capillary; it would be like fitting an oval peg in a round hole.

Under normal insertion pressures achievable manually, I would predict that a competent, gas-tight seal cannot be formed on capillary that differs in dimensions, in two axes, by more than approximately 10 µm. A definition of ovality is the difference in minimum and maximum diameters, divided by the nominal diameter, and expressed as a percentage, so a 0.53-mm capillary with ovality greater than approximately 1.5% wouldn't seal well in press-fit type devices. My colleagues and I have measured helium-leakage rates of less than 10^{−9} mL/min with capillaries that have twice this ovality, which is a testament to the versatility of function and fault tolerance of these simple devices. Unfortun-

nately, even higher ovality values routinely are found in the current capillary supply.

In an open tube draw that uses some basic care to ensure proper tube positioning in the draw furnace, the maximum ovality of capillary would be that of the base silica tube from which it was made. For the most common silica tube materials (F-110 Suprasil from Heraeus Tenevo), this maximum is 0.5%. Add pressure to the draw conditions, and this number rises. Current manufacturers seem able to control either the inertness of the bore or the ovality of capillaries — not both. Grob (6) recently observed that he finds it best to use capillaries from different manufacturers, depending upon whether he needs an inert bore or a good seal with a press-fit type connector.

It is possible to eliminate acid-catalyzed hydrolysis of surface siloxane in capillary draw and maintain ovality of less than 1%. It follows that it also is possible to produce low-activity capillary by using low-activity base silica (alternatives are becoming available) or by depositing tuned-activity interior coatings through modified chemical vapor deposition or other methods, without sacrificing compatibility with connectors.

Leaking connectors are not limited to press-fit type glass devices. Current capillary materials can be difficult to seal even in swaged ferrule-type devices. The glass and polymer ovality occasionally can be so high that any reasonable efforts will fail to form a truly gas-tight seal.

No discussion of ovality would be current or complete without addressing recent research describing the critical importance of a uniform column cut in cold on-column injections. Many chromatographers have adopted the Shortix capillary cutter (Scientific Glass Technology, SGT, Middelburg, The Netherlands), which uses a rotating diamond to scribe the capillary uniformly around the entire diameter, rather than to rely upon a fracture propagating uniformly from a single side of the column. For clean cuts to be produced by this capillary cutter, capillary ovality must be minimized.

Conclusion

Although the technology of fiber optics has made great strides in recent years, capillary manufacturers have made only marginal improvements in quality and in manufacturing efficiency for fused-silica capillary production since the late 1970s. Capillary column manufacturers are becoming

increasingly aware of the need for improvements in the quality of fused-silica capillary. As stationary phases have become increasingly sensitive to variable capillary chemistry and performance requirements of applications have become more rigorous, the conditioning, deactivating, and coating application regimens have become increasingly complex and costly.

Separation scientists using capillary zone electrophoresis and CEC, and especially those who purchase blank capillaries, are well aware of the subtle — and sometimes not-so-subtle — differences in fused-silica capillary obtained at different times from their suppliers. With this “Column Watch” installment, I hoped to challenge manufacturers of chromatography-grade fused-silica tubing to adopt some of the improvements of the fiber optic industry to improve the quality of tubing used to manufacture capillary columns, thereby benefiting chromatography as a whole.

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