Development of Resistive Glass Multi-Capillary Inlet Tubes for Enhanced Ion Transport

Paula Holmes, Ph.D. and Bruce N. Laprade, PHOTONIS USA, Sturbridge MA

PHOTONIS | INDUSTRY | SCIENCE | MEDICAL

Abstract:

Resistive glass tubes have been used successfully as reflectrons in TOF mass spectrometers and ion drift tubes in ion mobility spectrometers. This novel technology later enabled the exceptional performance of single capillary inlet tubes for ion transmission in atmospheric pressure ionization sources.

Uniform electric fields which guide sample ions are generated within the robust, single-piece glass tubes. An improvement in ion transfer efficiency by a factor of 100 compared to quartz inlet tubes has been reported. To further increase ion transmission, multi-capillary inlet tubes of resistive glass have been developed. These tubes have a circular array of six channels for the same outer diameter. Unique extrusion and drawing processes used to create these multi-channel tubes will be described and illustrated.

Introduction:

Resistive Glass

- Resistive glass tubes are designed to guide ions by generating a uniform electric field.
- Resistive glass tubes are composed of a proprietary lead silicate glass that has been hydrogen fired to create an integral semi-conductive layer, not simply a coating. (Figure 1)
- This resistive layer is typically several hundred angstroms thick.
- The resistivity can be varied over several orders of magnitude to suit the specific application.
- Resistive glass products are highly uniform.
- 4-point probe data indicates an overall variation in resistance of +/- 1.5% across a 75 mm plate. (Figure 2)
- Kelvin probe data indicates that the uniform resistance results in a uniform electric field. The resistive glass provides a uniform gradient with no significant anomalies. The smooth gradient indicates extremely uniform resistivity in the surface of the glass. (Figure 3)

Figure 1. Cross-section of resistive glass.

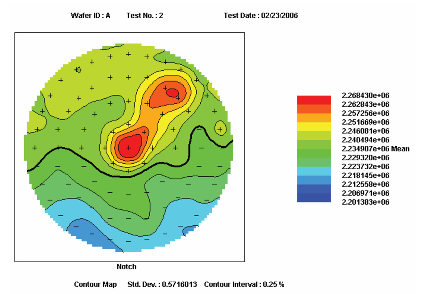
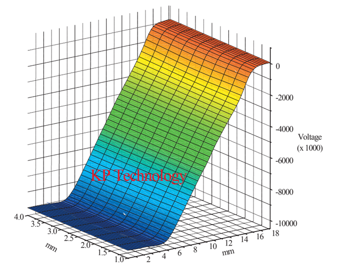


Figure 2 (above). 4-point probe data. (Data courtesy of Four Dimensions, Inc., Hayward CA.)

Figure 3 (right). Kelvin probe data. (Data courtesy of KP Technology, Caithness, Scotland.)



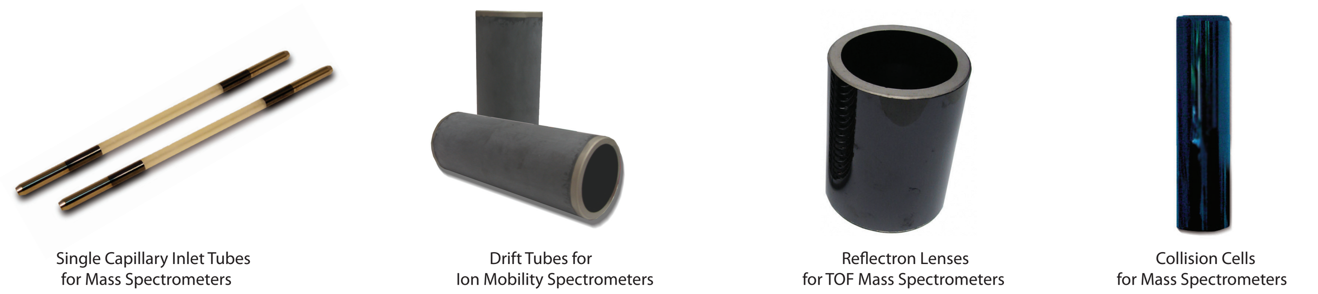


Figure 4. Examples of products made with Resistive Glass.

Introduction (continued):

Single Capillary Inlet Tubes

- Resistive glass single capillary tubes (Figure 5) have been shown to significantly improve ion transfer efficiency compared to conventional quartz inlet tubes. The voltage applied across the inlet tube creates an electric field that preferentially attracts ions into the inlet tube and forces more ions into the mass spectrometer.
- It also prevents collisions with other ions and tube walls which can produce ion loss, resulting in more efficient sample transfer. (Figure 6)

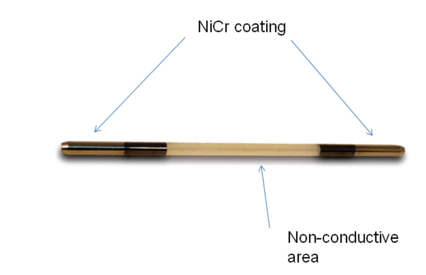


Figure 5. Typical single capillary resistive glass tube shown with an OD of 6.5 mm and an ID of 0.6 mm.

Nickel-Chromium metallization areas for applying voltage.

Non-conductive area designed to create a conductive path on inside of tube only.

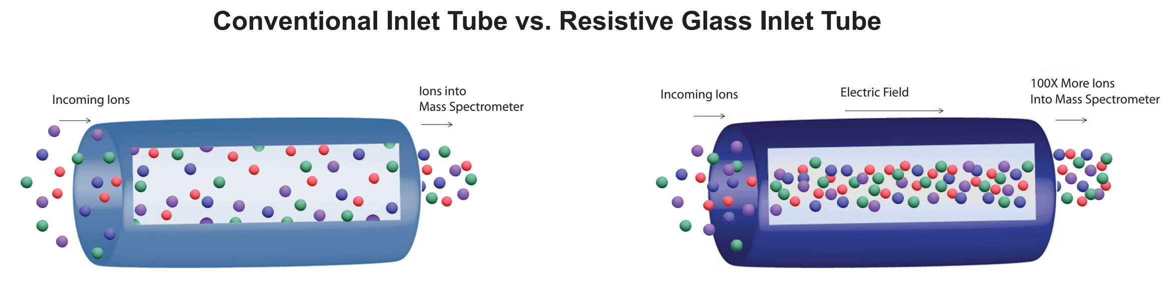


Figure 6. Comparison of sample transfer efficiency between a conventional inlet tube and a resistive glass inlet tube.

- Resistive glass inlet tubes also provide the unique ability to preferentially attract positive or negative ions.
- The rate of polarity switching can be accomplished more quickly with resistive glass tubes than standard quartz tubes.
- An increase in ion transfer efficiency by factor of 100 has been reported by a leading mass spectrometer manufacturer using resistive glass inlet tubes.
- Single capillary inlet tubes are fabricated by the simple extrusion and re-drawing of a glass tube. (Figure 7) Tubes are then hydrogen fired to create the resistive layer.
- In order to further improve ion transfer efficiency into the mass spectrometer, multi-capillary inlet tubes have been developed. These more complex tubes required a new approach to inlet tube processing.

Figure 7. The extrusion of a single capillary inlet tube.

Methods:

Multi-Capillary Tube Process Development

- The more intricate multi-capillary process required the drawing of a composite assembly consisting of small, thin-walled hollow tubes surrounding a solid rod center. (Figure 8)
- This 1.5" diameter assembly was then redrawn on a custom computer-controlled draw tower to the final 0.254" diameter.
- This process resulted in the formation of trapezoidal shaped capillaries as the spaces between the capillaries are filled during the drawing of the composite assembly. (Figure 9)
- Significant improvement in ion transmission over single capillary tubes was expected due to the increase in the number of capillaries per tube.
- Concerns however existed regarding potential disruption in ion flow due to the corners of the trapezoidal capillaries. This prompted development of a multi-bore extrusion process to create circular capillaries.
- The creation of more circular capillaries required a totally new technique whereby the multi-capillary structure is obtained directly from the extrusion process using a multi-bore die. (Figures 10,11)
- The 1.5" diameter tube with six parallel channels is then redrawn to the final, 0.254" diameter of the inlet tube.

Figure 8 . Photo of un-reduced multi-capillary assembly before redraw.

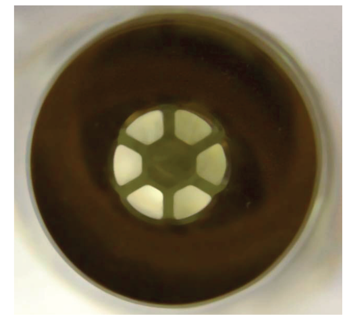


Figure 9. Photo of un-reduced multi-capillary assembly after redraw.

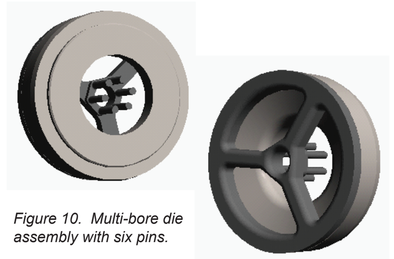


Figure 10. Multi-bore die assembly with six pins.

Figure 11. 1.5" diameter multi-capillary tube exiting furnace during extrusion process.

Results:

- The resulting capillaries are oval in shape (Figure 12). Efforts are underway to improve the roundness of the capillaries using specially designed dies.
- An increase in ion transmission of 6-10 X compared to single capillary inlet tubes has been realized by a leading mass spectrometer manufacturer, dramatically enhancing instrument sensitivity.
- Therefore, resistive glass multi-capillary tubes (Figure 13) provide an increase in ion transfer efficiency of up to 1000 X compared to conventional quartz tubes.

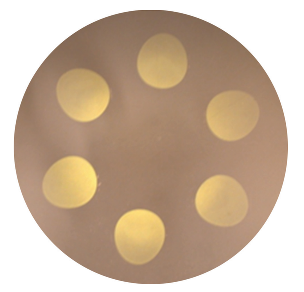


Figure 12. Close up photo of newly designed multi-capillary tube with oval channels.

Figure 13. Multi-capillary inlet tubes.

Results (continued):

- These tubes, like the single capillary inlet tubes help prevent collisions with other ions and the tube walls which can produce ion loss and provide the ability to preferentially attract positive or negative ions.
- In addition, new glass compositions were developed for high resistivity requirements and long length, 12" tubes.
- The resistivity of two multi-capillary tubes with starting values of 1.1 Gigohms and 120 Megohms was tracked for 5 months. The tubes were kept at ambient temperature and humidity for the duration of the test.
- The data presented in Figure 14 shows an increase in resistance of 6% for the higher resistance tube and an increase of 10% for the lower resistance tube indicating reasonable stability over time in an uncontrolled environment.
- Previous results published in 2006 by Mrotek, Laprade, Dunn and Ritzau[1] showed that resistive glass tubes are highly stable in vacuum and dry nitrogen with less than 1% increase in resistivity over a similar time frame.

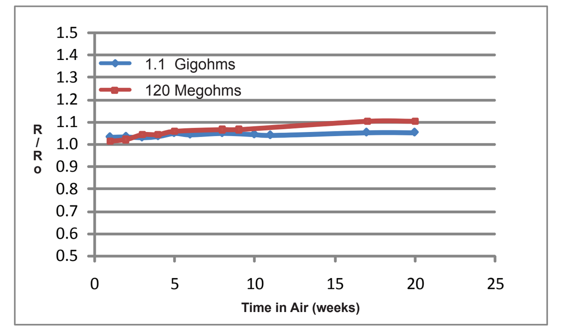


Figure 14. Resistivity of multi-capillary tubes tracked over five months shows reasonable stability over time in an uncontrolled environment.

Conclusions:

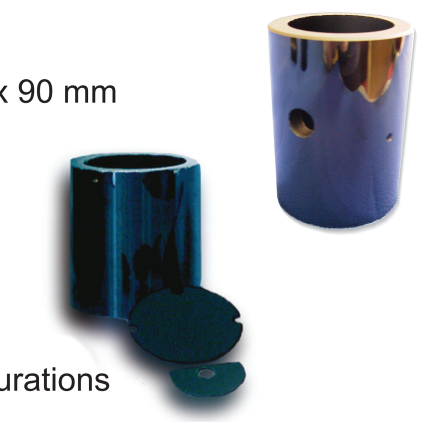
- A unique multi-bore extrusion process was developed to create resistive glass multi-capillary inlet tubes.
- An increase in ion transmission of 6-10 X has been realized by a leading mass spectrometer manufacturer through the successful combination of the multi-channel configuration with the unique resistive properties of the inlet tube.
- Uniformity of resistivity was demonstrated which leads to a highly uniform electric field.
- Good stability of resistivity was shown over 5 months in an uncontrolled environment for both low and high resistivity tubes.

Manufacturing Capability of Resistive Glass:

Tubes:
0.5 mm ID or greater
75 mm maximum OD
300 mm maximum tube length
1-6 mm wall thickness

Plates:
Sizes up to 90 mm x 90 mm
Flat within 3 fringes

Options:
Holes and/or slots drilled through glass
Custom metallization and sandblasting patterns
Custom end designs to accommodate specific cap configurations



Reference:

[1] Mrotek, Laprade, Dunn and Ritzau (2006). *Characterization of the Uniformity and Stability of Resistive Glass.* Paper 200-30P. Presented at the 2006 Pittsburgh Conference.