POLYIMIDE COATED CAPILLARY TUBING: AN ANALYSIS OF END-FACE DEFECTS

BY JOE MACOMBER | MOLEX, LLC.

Polyimide coated fused silica capillary tubing is widely used in the separation sciences. This application note presents perspectives on the analysis of defects found on capillary end-faces and the application of those findings toward improved life time during usage.

INTRODUCTION

Fused silica capillary tubing used in Gas Chromatography (GC) is produced in long lengths and then provided to column manufacturers who employ a series of proprietary techniques to produce finished GC columns used in laboratories around the world. Whether one is manufacturing the column or using it in the laboratory for sample analysis, breakage of the capillary is never a welcomed event. Analysis of the end-face of a break can lead to valuable information in an effort to minimize the frequency of such occurrences. Elemental spectral analysis of any debris found near the surface flaw can provide added detail.

The primary objective of the end-face analysis is to determine the root cause of the material failure that lead to the breakage of the capillary tubing. Breakage is predominately traceable to a surface flaw on either the glass i.d. or o.d. Knowing which surface was damaged can be instrumental in the effort to find the root cause and take the appropriate corrective action.

EXPERIMENTAL

Images of end-face samples were collected by SEMTEC, Inc. (Phoenix, AZ) using a Jeol 6100 Scanning Electron Microscope (Jeol Ltd., Tokyo, Japan). The secondary electron mode of operation was employed. Although a number of samples have been examined over the years, those shown in Figure 1 were actual samples provided by Polymicro customers. Samples were readied for analysis by standard SEM preparation techniques, including gold sputtering of the capillary end-faces.

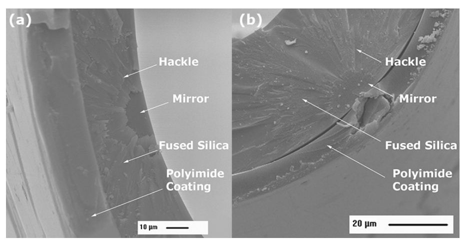

Figure 1: a) End-face of a break caused by an i.d. flaw; dark mirror pattern has a semicircular shape. b) End-face of a break caused by an o.d. flaw; debris that caused failure remains embedded within outer polyimide layer.

RESULTS

Surface flaws on both the glass i.d. and o.d. have been found on many occasions. Figure 1a presents the end-face of a break caused by a typical i.d. flaw. Figure 1b shows a corresponding image of an o.d. flaw. Of particular interest is the pattern of the flaw, which usually comprises two components referred to as the mirror and hackle. The mirror portion penetrates the surface to a depth of 5-10 µm and is semicircular in shape. It is smooth in appearance and is formed when the surface is initially damaged. When the capillary is subsequently stressed, typically by bending, this mirror-smooth flaw propagates outward in a series of jagged fractures referred to as the hackle. This fracturing ultimately results in breakage of the tubing. This mirrorhackle pattern can be seen in both figures.

The most common cause of i.d. flaws is glass debris from cleaving; the importance of which has been discussed previously (1). Introduction of debris from fittings, connection lines, or unfiltered solvents is less common, but has been verified on occasion. Debris is also the most common cause of o.d. flaws. The break shown in Figure 1b was caused when debris penetrated through the protective polyimide coating and contacted the glass surface. Elemental spectral analysis confirmed that the embedded debris was a piece of fused silica, most likely from a dirty work surface. Cleanliness of the work area and any equipment that contacts the capillary is critical (2). Determination of where the failure began greatly aids in taking the appropriate action to minimize breakage.

CONCLUSION

End-face analysis of damaged capillary is an important tool in determining the root cause of capillary breakage. Glass debris from poor cleaves or dirty work surfaces have been found to be the most common sources of breakage.

REFERENCES

(1) “Cleaving Procedure,” The Book on the Technologies of Polymicro, Polymicro Technologies LLC Publication, p.A-2, (2005)

(2) J. Macomber and L. Begay, LCGC Application Notebook, 72 (Sept 2003).

Originally Published in LCGC, The APPLICATION NOTEBOOK (February 2007)

