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Microchip gas chromatography columns, interfacing and performance

Abhijit Ghosh[a], Carlos R. Vilorio[b], Aaron R. Hawkins[b], Milton L. Lee[a,*]

[a] Department of Chemistry and Biochemistry, Brigham Young University, Provo, UT 84602, USA
[b] Department of Electrical and Computer Engineering, Brigham Young University, Provo, UT 84602, USA

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ABSTRACT

Almost four decades of investigations have opened up many avenues to explore the production and utilization of planar (i.e., microchip) gas chromatographic columns. However, there remain many practical constraints that limit their widespread commercialization and use. The main challenges arise from non-ideal column geometries, dead volume issues and inadequate interfacing technologies, which all affect both column performance and range of applications. This review reflects back over the years on the extensive developments in the field, with the goal to stimulate future creative approaches and increased efforts to accelerate microchip gas chromatography development toward reaching its full potential.

1. Introduction

Microfabrication technology has ushered in a new era of miniature analytical instrumentation, which addresses many of the analytical needs of point-of-care, homeland security, drug testing, space exploration, and field applications. Microchip gas chromatography (GC) differs from conventional GC in that it typically is characterized by shorter, non-cylindrical columns that are microfabricated in planar substrates. Channel cross-sectional geometries can be rectangular, square, trapezoidal and semi-circular [1]. The theoretical promise of such technology was brought to light by both Golay and Giddings in the early 1980s, and was later re-emphasized by Spangler [2–4].

It is interesting that even though the first micro-analytical device made was a GC, and the last four decades have shown a significant growth in microchip GC research [5], the performance of microchip-based GC is still not on par with conventional bench-top GC. On the other hand, other microchip-based analytical systems, such as capillary electrophoresis (CE), have greatly benefited from miniaturization and found acceptance among analysts. This is largely because microchip CE does not involve intricate column geometries, challenging interfacing technology, stationary phase coatings, and temperature and pressure requirements, as does microchip GC [6,7].

Remarkable progress in microfabrication technology has allowed the etching of channels in a variety of planar substrates [8–13]; however, there is still no common consensus among researchers about the best geometry [14], ideal dimensions [15], choice of substrate and so on for the microchip GC column/channel. Furthermore, interfacing injectors and detectors to microchips has been a major challenge, and is still not satisfactorily resolved. In the majority of cases, adhesives such as epoxies or glue-based materials are used for attaching leads [16–21], which limits methods for deactivation [22,23] and prevents the use of microchips for analysis of non-volatile organic compounds [24]. The adhesives are either degraded at high temperatures or they shrink/crack and begin to leak after thermal cycling [25]. Finally, there continue to be issues with producing a desirable and uniform deposition of stationary phase film on the walls of microchip channels due to difficulties encountered in statically coating and maintaining uniformity of the stationary phase in the channel with its multiple turns and non-cylindrical geometry [6,7].

Most reports published in the area of microchip GC developments are dedicated to column technology [8–11,23]. However, it is well understood among GC researchers that to improve the performance of GC, both the column and interfacing technologies must be improved together [25]. This review, therefore, covers both fundamental and technical developments in microchip GC column technology and the challenges and advances made in interfacing GC microchips to injectors and detectors.

2. Column design and channel geometry

In the first microchip GC paper by Terry et al. [8], the authors reported that the cross-sectional profiles in silicon depend on its crystallographic orientation, as well as the etching process and conditions. For silicon, the most popular microchip substrate (Table 1), there can be two different types of cross-sectional geometries as determined by the fabrication process: isotropic and anisotropic (described in more

* Corresponding author.
E-mail address: milton_lee@byu.edu (M.L. Lee).

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Table 1
Microchip gas chromatography column specifications.

Author	Substrate	Geomerty (Column type)	Length (m) x Width (μm) x Depth (μm)	Column etching	Microcolumn device	Wafer bonding	Interfacing	Stationary phase	Column coating	Theoretical plates	Application temp range
Terry et al. [8]	silicon	cir. sp. (OC)	1.5-3 x 200 x 30	wet	silicon-Pyrex	anodic	pneumatic/ compression	PDMS	dynamic	385-2,300 plates	NR
Reston & Kolesar [16]	silicon glass	cir. sp. (OC)	0.9 x 300 x 10	wet	silicon-Tempax	anodic	epoxy	CuPc	sublimation evaporation	NR	70–90
Kolesar & Reston [128]	silicon glass	cir. sp. (OC)	0.9 x 300 x 10	wet	silicon-Pyrex	anodic	epoxy	CuPc	sublimation evaporation	NR	55–80
Wiranto et al. [17]	silicon	sqr. sp. (OC)	1.25 x 100 x 20	wet	silicon-Pyrex	anodic	epoxy	NR	NR	18,200 plates	NR
Yu et al. [30]	silicon	cir. sp. (OC)	5.6 x 100 μm i.d.	wet	silicon	fusion	NR	PDMS	dynamic	40,000 plates	65
Wiranto et al. [33]	silicon	sqr. sp. (OC)	1.25 x 100 x 20	wet	silicon-Pyrex	anodic	epoxy	PDMS	dynamic	862-2,192 plates	150
Lehmann et al. [110]	silicon	cir. sp. (OC)	2.5 x 210 x 100	dry	silicon-Pyrex	anodic	epoxy	PDMS like	CVD	NR	35–140
Lehmann et al. [18]	silicon	serp. (OC)	2 x 70 x 27	dry	silicon-Pyrex	anodic	epoxy	PDMS like	CVD	NR	10–82C
Frye-Mason et al. [78]	silicon	sp. (OC)	1 x 40 x 250	dry	silicon-Pyrex	anodic	NR	PDMS	NR	NR	40
Frye-Mason et al. [87]	silicon	cir. sp. (PC)	0.2 x 300 x 300	dry	silicon-Pyrex	anodic	NR	carbon packed	packing	NR	60–150
Dziuban et al. [88]	silicon	cir. sp. (OC)	5.9 x 300 x 150	isotropic (NS)	silicon-Borofloat	anodic	ferrules	squalan	NR	NR	NR
Noh et al. [19]	parylene	cir. sp. (OC)	1.0 x 100 x 350	dry	parylene	compression	epoxy	parylene	PVD	NR	100
Briscoe et al. [9]	ceramic	cir. sp. (MC)	0.10-1.0 x 10-40 x 80-250	laser punch emboss	ceramic	sintering	NR/NS	PDMS	porous plug, NS	NR	NR
Lambertus et al. [20]	silicon	sqr. sp. (OC)	3 x 150 x 240	dry	silicon-Pyrex	anodic	epoxy	PDMS, TFPMPS	dynamic	4,620-8,240 plates 3,530-5,550 plates	22–200
Bhushan et al. [10]	nickel	NR (OC)	2.0 x 50 x 600	LIGA	nickel	electrodeposition	NR	NR	NR	NR	NR
Agah et al. [132]	silicon	sqr. sp. (OC)	3 x 150 x 250	dry	silicon-Pyrex	anodic	NS	PDMS	dynamic	8,000 plates	room temp-55
Lorenzelli et al. [92]	silicon	rect. sp. (OC)	1.3 x 200 x 20	dry	silicon-Pyrex	anodic	flip-chip	5% Phenyl-PDMS	static	NR	25–200
Lambertus & Sacks [159]	silicon	rect. sp. (OC)	3.0 x 150 x 240	dry	silicon-Pyrex	anodic	epoxy	PDMS, TFPMPS	dynamic	5,400-6,000 plates	22–70
Zampolli et al. [124]	silicon	cir. sp. (PC)	0.50-0.75 x NR X 620-800	dry	silicon-Pyrex	anodic	NR	carbograph carbowax	packing	NR	22–60
Lambertus et al. [160]	silicon	sqr. sp. (OC)	3.0 x 150 x 240	dry	silicon-Pyrex	anodic	epoxy	PDMS	dynamic	5,500 plates	30–120
Lu et al. [162]	silicon	sqr. sp. (OC)	3.0 x 150 x 240	dry	silicon-Pyrex	anodic	epoxy	PDMS	dynamic	6,500 plates	25–100
Lewis & Wheeler [67]	nickel	serp.	1 x 250 i.d.	LIGA	Nickel	NS	NS	PDMS	NS	NR	80
Reidy et al. [82]	silicon	sqr. sp. (OC)	3.0 x 150 x 240	dry	silicon-Pyrex	anodic	epoxy	PDMS	static	12,500 plates	30–180
Kendler et al. [104]	silicon	sqr. sp. (OC)	1.0 x 150 x 240	dry	silicon-Pyrex	anodic	epoxy	PDMS	static	4,000 plates/m	room temp-250
Stadermann et al. [114]	silicon	serp. (OC)	0.5 x 100 x 100	dry	silicon-glass (NS)	anodic	epoxy	SWNT	CVD	NR	90–150
Sanchez et al. [31]	silicon	rect. sp., radiator (OC)	2.0 x 50 x 30	dry	silicon-Pyrex	anodic	epoxy	TEOS + F13-TEOS	sol-gel	NR	75
Agah et al. [144]	silicon	sqr. sp. (OC)	0.25-3 x 150 x 250	dry	silicon-glass (NS)	anodic	epoxy	PDMS	dynamic	625 (0.25 m)	25–130
Bhushan et al. [22]	silicon	serp. (OC)	0.5-2.0 x 50 x 600	LIGA	nickel	electrodeposition	epoxy	PDMS	static	17,500 (2 m) plates/m	70

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