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Semipacked columns with atomic layer-deposited alumina as a stationary phase

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ABSTRACT

A new approach is described for utilizing atomic layer-deposited alumina as a gas–solid stationary phase medium for microfabricated gas chromatography columns. After atomic layer deposition (ALD) of aluminum oxide, a chloroalkylsilane is utilized to functionalize the oxide surface to improve peak symmetry and retention times. Semipacked columns (1 m-long, 190 μm-wide, 180 μm-deep with 20 μm-embedded circular micropillars) were utilized for validation of this newly developed approach. ALD-treated/silane-functionalized columns efficiently separated a multicomponent sample mixture and yielded 4200 plates per meter. The ALD-based stationary phase is found to be stable after multiple injections and at high operating temperatures. The proposed method facilitates a simple and wafer-level coating scheme that provides a highly controllable film thickness. The inherent properties of atomic layer deposition provide an easy route to coat very challenging microfabricated column designs.

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1. Introduction

Gas chromatography (GC) is a reliable chemical analysis technique that is used to separate and identify the constituents of complex gas mixtures. GC has applications in a vast range of areas such as environment monitoring, food processing, pharmaceutical industry, biomedical science, forensic and trace analysis. Traditional bench-top GC instruments, though widely used, are bulky, expensive, time- and energy-intensive. The progressive innovations in microfabrication techniques coupled with nanotechnology have prompted a renewed interest in miniaturizing key GC components over the last decade [1–16]. The development of new stationary phase coating techniques has been a continuous research area and the heart of micro gas chromatography (μGC) column performance [13,17–22]. Apart from the application of most common polymer-based gas–liquid stationary phases [6,13,23–26] for μGC separation columns, there has also been considerable research efforts toward the development of MEMS-compatible methods for the integration of solid-adsorbent materials (monolayer-protected gold [27], carbon nanotubes [17], silica nanoparticles [21], gold nanoparticles [28], silica [22] and graphite [29]) into μGC columns. Although these adsorbent/gas–solid stationary phases have shown attractive features for the separation of complex mixtures, the application of these coating schemes for very narrow-width (<20 μm) and deep (>150 μm) rectangular microchannels has not been straightforward as summarized in Table 1 [17,18,21,22]. The current adsorbent-based coating schemes for these particular column designs mostly suffer from factors such as low-yield, chip-level coating, high processing temperatures and requiring elaborate experimentation. Moreover, these methods present a major hindrance towards the monolithic integration of different μGC components on a single chip. This warrants the need for developing wafer-level facile complimentary metal-oxide-semiconductor (CMOS)-compatible coating techniques that produce mechanically and thermally stable, chemically inert and selective stationary phases.

ALD has emerged as a powerful tool for the growth of low-temperature thin films on a variety of substrates for myriad applications [30,31], generally as a gate oxide in CMOS technology. ALD enables conformal and homogeneous coatings of flat, very high-aspect-ratio (HAR) microchannels and even nanostructure surfaces with precision within a few angstroms. The use of ALD coatings for separation science has been very recently reported for ultra-thin-layer chromatography where the enhancement of separation capabilities of silica support media using different ALD coatings was demonstrated [32]. The use of ALD-based films as a standalone separation media for μGC has not been reported to the best of our knowledge.

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Table 1
Summary of microfabricated GC columns with gas–solid stationary phases.

Authors	Column geometry	Phase deposition	
		Method	Limitations
Reston (1994) [41]	Rectangular (30 μm-deep, 100 μm-wide, 0.9 m-long)	Evaporation of copper phthalocyanine film	Applicable to only shallow channel depth
Stadermann (2006) [17]	Rectangular (100 μm-deep, 100 μm-wide, 50 cm-long)	Chemical vapor deposition of carbon nanotubes	Incomplete coverage of the channel and high process temperature
Zareian-Jahromi (2010) [18] and Shakeel (2013) [27]	Rectangular (250 μm-deep, 25 μm-wide, 25 cm-long) Multicapillary (25 μm-wide, 200 μm-deep, 25 cm-long)	Self-assembly of thiol on electroplated gold surface	Extensive characterization, high-process temperature, and chip-level processing
Vial (2011) [22]	Rectangular and semipacked (100–125 μm-deep, 50–150 μm-wide, 1 m-long)	Wafer-level-sputtered silica film deposition	High stationary-phase film thickness variations and applicable to low channel depth
Wang and Shakeel (2013) [21]	Rectangular (250 μm-deep, 150 μm-wide, 1 m-long) Multicapillary (200 μm-deep, 20 μm-wide, 25 cm-long)	Silica nanoparticles using layer-by-layer technique	High process temperature and chip-level processing

Semipacked separation columns, having an ordered micropillar array in the rectangular separation channel, are shown to reduce band-broadening, achieve high separation efficiency [22,25,26,33] and improve sample capacity [34]. Previously semipacked columns have been coated with both polymeric [25,26,33] and adsorbent-based stationary phases [22]. The gas–solid/adsorbent-based coating of semipacked columns was limited by the 100 μm column depth [22] due to the poor step-coverage afforded by a sputtering process for the high-aspect-ratio microstructures. The novel wafer-level application presented here utilizes a silane-functionalized aluminum oxide thin film as a stationary phase medium for semipacked columns. ALD uses self-limiting gas-phase chemical reactions for deposition, thus affording an alumina film with very high step-coverage of semipacked columns having aspect ratios (depth:width) of 10:1. Since ALD has been successfully employed for coating micro/nanostructures with aspect ratios of 1000 and beyond [35], our proposed coating technique could easily be integrated to more challenging μGC columns with very narrow channel widths (5 μm or less). The detailed fabrication process and chromatographic analysis of our proposed scheme is explained below.

2. Experimental procedures

The fabrication of microfabricated separation columns employs standard microelectromechanical systems (MEMS) processes for

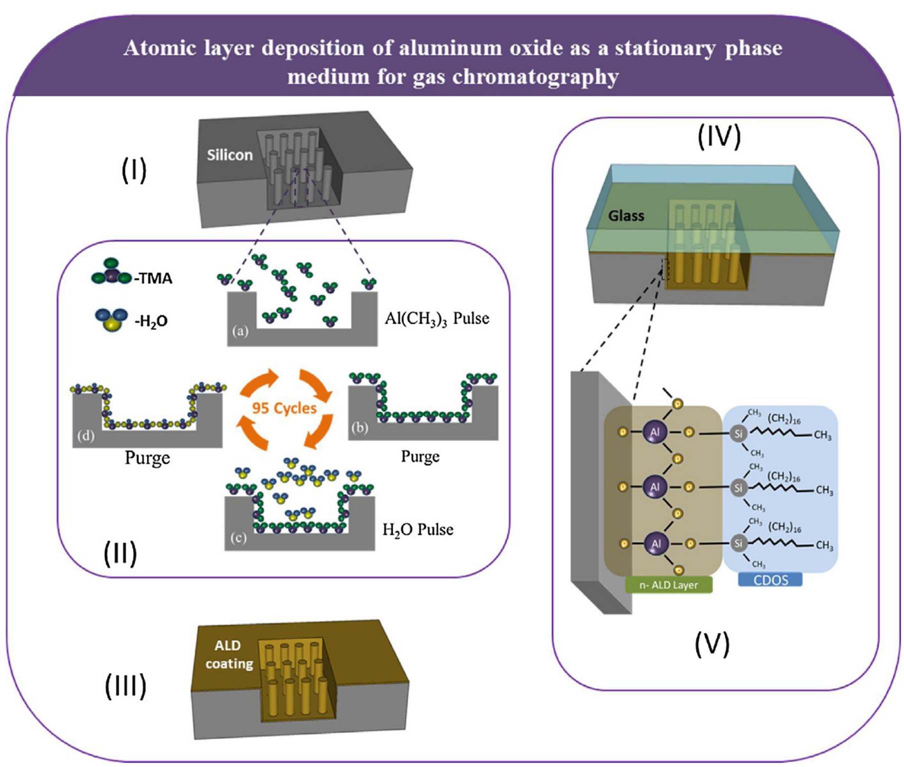

Fig. 1. Schematic representation of ALD-based stationary phase coating. (I) Anisotropically etched semipacked column; (II, III) atomic layer deposition of 10 nm alumina film and (II) 95 cycles with each cycle having four steps (a) TMA exposure (b) purge (c) H2O exposure (d) purge; (IV) anodic bonding of ALD-coated wafer with glass substrate; (V) silane deactivation for 24 h.

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