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Abstract

SiO_2/Platinum Monolith Aerogels Realized in Closed µfluidic Channels †

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Abstract: Aerogels with noble metals have a wide range of applications such as sensing and catalysis, but research needs to be done to improve the integration of these materials in µ-channels. We realize silica aerogels without shrinkage and with high specific surface area (~600 m^2/g) inside of closed channels. Further, Pt nanoparticles are deposited via capillary forces, into the complete network.

Keywords: silica aerogels; noble metal nanoparticles; capillary impregnation; microfluidics

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1. Introduction

Silica aerogel is a nanostructured porous material with a wide range of applications thanks to, among other characteristics, high open porosity (up to 99.8%) and high specific surface area (SSA up to 1200 m^2/g) [1]. Additionally, other aerogels such as noble metal aerogels (NMAs), combine those characteristics with the conductive and catalytic properties of the nanoparticles, resulting in a final material with a range of applications such as sensing and catalysis [2].

Nevertheless, those materials present their own limitations. Silica aerogels are very fragile and present shrinkage during the forming process. Therefore, the use in a chip is limited to powder particles, thin films and single drops [3,4]. Additionally, NMAs have a significant lower specific surface area (~100 m^2/g) and/or longer gelation periods, lasting up to a month [2]. Nanoparticles can also be deposited by more complex methods such as supercritical impregnation, or CVD/PVD limited to open surfaces.

We report the synthesis and optimal integration of a monolith aerogel in closed µ-channels with minimum shrinkage and complete filling of the chip. Furthermore, Pt nanoparticles (PtNP) are deposited into the aerogel using capillary forces, filling the complete network.

2. Materials and Methods

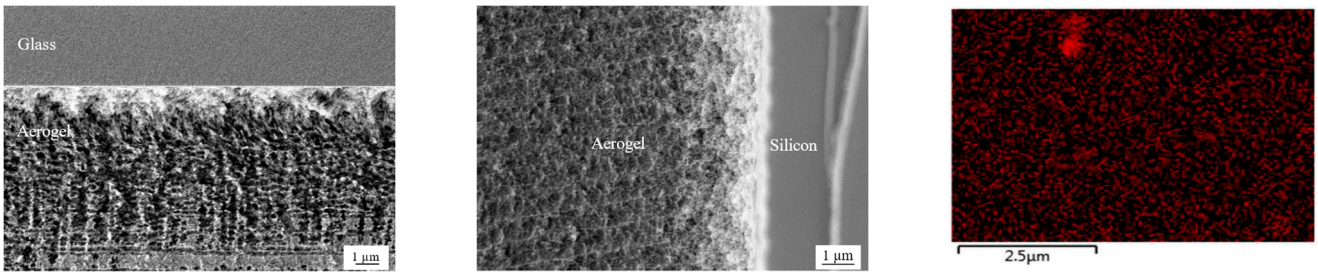
The synthesis process follows previous work [5]. It consists of a sol-gel technique with tetraethyl orthosilicate (TEOS), ethanol (C_2H_6OH) and an acid solution (H_2O/HCl). After 90 min. stirring at 60 °C, the basic solution (H_2O/NH_3) is added and the gelation occurs after 40 min. The µ-channels are filled with the gel during the gelation to avoid shrinkage inside the channel. When added before as a solution, shrinkage inside the channel and creation of cracks and opening happens. The aging is performed for a week to reinforce the network in TEOS and C_2H_6OH. Lastly, the gels are dried in a CO_2 supercritical drying machine (Leica CPD 300). The optimal parameters found are: mix of CO_2 and C_2H_6OH for 30 min, and 20 exchange cycles, to properly exchange the solvents and avoid leftovers inside the pores that lead to pore collapse and shrinkage. The µ-channel was realized in

the silicon layer of a glass/silicon/glass chip. Channel width, height, and length amount to 400 µm, 380 µm, and 8.2 mm, respectively.

First, PVD with Pt precursor was tested but only a thin film on the top of the aerogel was observed, therefore we focus on the deposition of the noble metals via capillary forces. The PtNP are synthesized following a reported method [6]. The synthesis uses H_2PtCl_6*xH_2O as the Pt precursor. The PtNP are dispersed in ethanol and led into the channel in a concentration of 10 wt%.

3. Discussion

The aerogel shows good adhesion to silicon and glass (Figure 1a,b). No shrinkage was observed inside the µ-channels, as shrinkage would make the aerogels detach from the walls. Nitrogen physisorption is performed, and an average pore size of 20 nm and a SSA of 600 m^2/g was measured. EDX is used to observe the dispersion of the PtNP through the aerogels network (Figure 1c). To analyze the penetration of PtNP into the network (not only staying on the surface) the samples were cut and the analyses were performed inside. Red dots are the PtNP, showing penetration and distribution. Powder XRD shows an amount of 7% of PtNP in the aerogels where most of them are Pt(0); the small loss is expected during dispersion and deposition. Testing with H_2 chemisorption presents that the capability of H_2 binding on the surface is increased by a factor of five compared to pure aerogel.



(**a**) (**b**) (**c**)

Figure 1. View of the chip filled with aerogel, (**a**) adhesion between glass and aerogel (**b**) between silicon and aerogel (**c**) EDX from the middle of the aerogel sample, red dots are Pt nanoparticles.

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References

1. Dorcheh, A.S.; Abbasi, M. Silica aerogel; synthesis, properties and characterization. *J. Am. Acad. Dermatol.* **2008**, *199*, 10–26. [CrossRef]
2. Wen, D.; Herrmann, A.-K.; Borchardt, L.; Simon, F.; Liu, W.; Kaskel, S.; Eychmüller, A. Controlling the Growth of Palladium Aerogels with High-Performance toward Bioelectrocatalytic Oxidation of Glucose. *J. Am. Chem. Soc.* **2014**, *136*, 2727–2730. [CrossRef] [PubMed]
3. Li, Y.K.; Chen, Y.-C.; Jiang, K.-J.; Wu, J.-C.; Chen-Yang, Y.W. Three-dimensional arrayed amino aerogel biochips for molecular recognition of antigens. *Biomaterials* **2011**, *32*, 7347–7354. [CrossRef] [PubMed]
4. Gaspar, A.; Nagy, A.; Lazar, I. Integration of ground aerogel particles as chromatographic stationary phase into microchip. *J. Chromatogr. A* **2011**, *1218*, 1011–1015. [CrossRef] [PubMed]

5. Reede, S.; Bunge, F.; Vellekoop, M.J. Integration of silica aerogels in microfluidic chips. *Proceedings* **2017**, *1*, 298. [CrossRef]
6. Loof, D.; Thüringer, O.; Schowalter, M.; Mahr, C.; Pranti, A.S.; Lang, W.; Rosenauer, A.; Zielasek, V.; Kunz, S.; Bäumer, M. Synthesis and Characterization of Ligand-Linked Pt Nanoparticles: Tunable, Three-Dimensional, Porous Networks for Catalytic Hydrogen Sensing. *ChemistryOpen* **2021**, *10*, 697–712. [CrossRef] [PubMed]