close

Microscopy and Microanalysis (2004), 10: 484-485 Cambridge University Press

doi:10.1017/S1431927604886161

Login Subscribe to journal Email abstract Save citation Content alerts

Volume 10
Supplement S02 - Aug 2004
Add to basket £14.00 / $20.00
Cited by Articles (CrossRef)
Cited by Articles (Google Scholar)

Contributed Paper

Physical Sciences Symposia - Microscopy and Microanalysis in Catalysis

Magnetization and Composition Studies of Alumina and Silica Sol-gel Encapsulated Co/Fe/Ru Catalysts in Micro-reactors for Conversion of Syngas to Higher Alkanes

Naidu V. Seetala [a1], **J. Leonard** [a1], **V. S. Nagineni** [a2], **S. Zhao** [a2], **D. Kuila** [a2], **J. Fang** [a2] **and U. Siriwardane** [a2]

[a1] Grambling State University, Louisiana
[a2] Louisiana Tech University

Extended abstract of a paper presented at Microscopy and Microanalysis 2004 in Savannah, Georgia, USA, August 1–5, 2004.

back to top

Magnetization and Composition Studies of Co/Fe/Ru Encapsulated Al_2O_3 or SiO_2 Sol-gel Micro-reactors*

N. V. Seetala[a], J. Leonard[a], V. S. Nagineni[b] , S. Zhao, D. Kuila, J. Fang, and U. Siriwardane[c]

[a] Department of Physics, Grambling State University, Grambling, LA 71245.
[b] Institute for Micromanufracturing, Louisiana Tech University, Ruston, LA 71272.
[c] Department of Chemistry, Louisiana Tech University, Ruston, LA 71272.

Most heterogeneous catalysts are comprised of transition elements, which have incomplete d-electron shell and unpaired electron spins. These features are responsible for their specific magnetic as well as their valuable catalytic properties [1,2]. Here we studied the magnetic characters of Co (12%), Fe (12%), and Ru (1%) encapsulated Al_2O_3 or SiO_2 sol-gel deposited micro-reactors as deposited, reduced and exposed to the syn-gas ($CO+H_2$) using Vibrating Sample Magnetometer (VSM). SEM and EDX are used to study the coating uniformity and elemental composition in order to optimize the sol-gel preparation and deposition processes.

The SEM view graphs of micro-channel reactors are shown in Figures 1-3. It is observed that the 5μm spacing between the channels (Figure 1) is too narrow for sol-gel to go in. We tried to improve the spreading of the sol-gel throughout the channels by considering 25μm column-channels (Figure 2). By widening the channels/columns to 25μm, the sol-gel is easily going to the bottom of the channels, but the Al_2O_3 sol-gel is not sticking to the walls (Figure 2). If we use SiO_2 sol-gel instead of Al_2O_3, the adhesion has improved (Figure 3). The catalyst studies using GC revealed [3] that the syn-gas ($CO+H_2$) conversion rate is improved in the case of SiO_3 sol-gel, may be due to increased surface area as it is spread throughout the channels evenly.

TABLE 1 Sample Type	Intended Metal Loading	EDX Results (% Metal Loading)		
		Fe	Co	Ru
Al_2O_3 Sol-gel	Fe12%, Co12%	2.5	3.7	-
SiO_2 Sol-gel (from nitrates)	Fe12%, Co12%	2.6	2.7	-
SiO_2 Sol-gel (from oxide nano-particles)	Fe12%, Co12%	3.3	3.9	-
SiO_2 Sol-gel (with Ru)	Fe12%, Co12%, Ru1%	1.9	1.8	0.2

Table 1 shows the results of EDX elemental analysis for 25 μm channel reactors coated with Fe/Co/Ru encapsulated Al_2O_3 or SiO_2 sol-gel prepared from metal nitrate solutions or from metal oxide nano-particles. The metal oxide nano-particle sol-gel showed higher metal loading compared to the sol-gel prepared from metal nitrates. The GC results showed that the syn-gas conversion has improved from 50% to 78% by adding small %Ru [3], even though all metal loadings are less in these coatings. So it clearly proves that Ru is acting as a good promoter.

The magnetization characters of these sol-gel micro-reactors are studied using VSM to understand the reduction efficiency during hydrogenation and the level of poising of catalysts after syn-gas conversion, depending on the ferromagnetic component of the curve. During the hydrogenation the

1. S. V. Naidu, et al., Microsc. Microanal., Vol. 9 (2003) 408.
2. M.A Akundi, et al., IEEE Trans. on Magnetics, Vol. 37 (2001) 2929.
3. D. Kuila, et al., to be published elsewhere.

* Work supported by grants from NSF-EPSCoR and DOE.

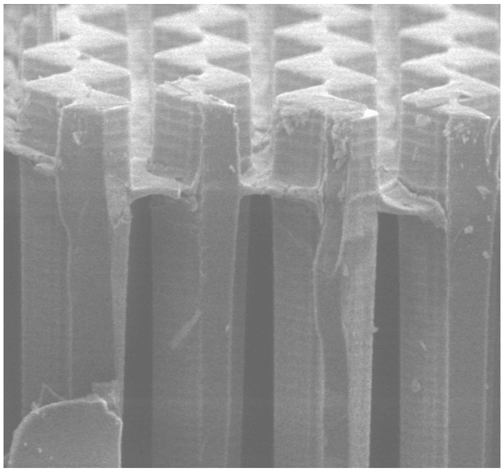


Fig. 1: SEM view graph of Al_2O_3 sol-gel deposited 5µm zigzag channels reactor. It shows that the sol-gel is mostly spread on the surface and not able to go in the 5 µm-channels of 100µm depth.

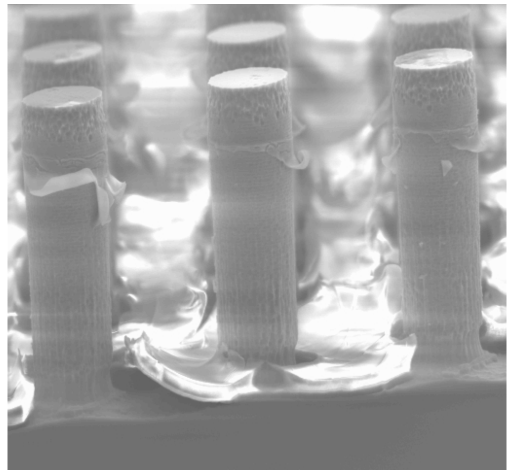


Fig. 2: SEM view graph of Al_2O_3 sol-gel deposited 25µm column- reactor. It shows that most of the Al_2O_3 sol-gel is going to the bottom and not sticking to the walls.

a b

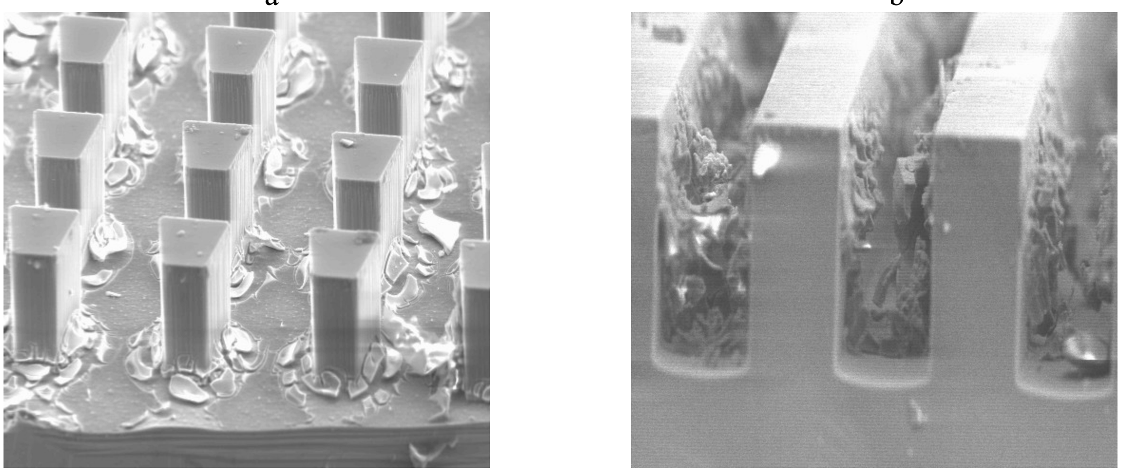
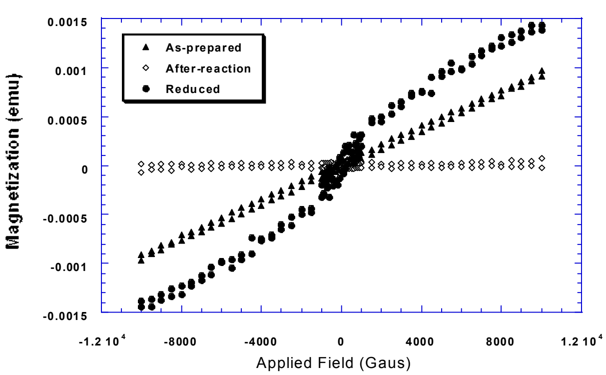


Fig. 3: SEM view graph of SiO_2 sol-gel deposited 25µm a) column and b) straight channel reactors. It shows that the sol-gel is more evenly distributed and sticking to walls in both these reactors compared to Al_2O_3 sol-gel.

Figure 4: Typical magnetization curves for as-deposited (▲), hydrogenated (● reduced), and after catalytic reaction (○) sol-gel coated Fe/Co micro-reactors.



Fe and Co oxides are reduced to pure metals as evidenced by the ferromagnetic nature of the curve for the reduced sample (Fig. 4). The ferromagnetic nature disappears in the post-catalytic reaction sample, indicate that Fe and Co are forming carbides or carbonyls during the reaction. The magnetization results indicated more reduced metal catalysts in the nano-particle incorporated sol-gel. Due to the catalytic reaction about 85% catalyst was poisoned, probably by forming carbides of Fe and Co.