Supplementary Material (ESI) for Chemical Communications

Mesoporous Silicon Oxynitride Thin Films

Jiacheng Wang,[a,b] and Qian Liu*[a]

[a] *State key Laboratory of High Performance Ceramics and Superfine Microstructure, Shanghai Institute of Ceramics, 1295 Dingxi Road, Shanghai 200050 P. R. China.*

[b] *Graduate School of the Chinese Academy of Sciences, 19A Yuquan Road, Beijing 100039 P. R. China*

*Correspondence should be addressed to Prof. Qian Liu (E-mail: qianliu@sunm.shcnc.ac.cn).

Experimental section

Synthesis: Mesoporous silicon oxynitride thin films (MSONTFs) were prepared as following by two steps. Firstly, mesoporous silica thin films (MSTFs) were synthesized by dip-coating. Specifically, 7.7 mL of tetraethyl orthosilicate (TEOS) were prehydrolyzed in a solution containing 3.7 g of dilute hydrochloric acid (pH ≈ 2) to 10 mL of tetrahydrofuran (THF) under vigorous stirring at room temperature. Following 2 h of stirring, this prehydrolyzed silica solution was mixed with a solution containing 1.8 g of the block copolymer $EO_{20}PO_{70}EO_{20}$ (Pluronic P123, BASF Corporation) dissolved in 30 mL of THF. Then, the solution was stirred for 15 min, after which films were prepared by dip-coating (at a speed of 70~75 mm/min) onto the carefully cleaned quartz substrates. Then, the as-synthesized MSTFs were stored in an ambient atmosphere for 24 h.

Secondly, MSONTFs were prepared by heating the as-synthesized MSTFs in flowing ammonia of 400 mL min^{-1} in a quartz tube furnace. The quartz tube was evacuated several times and flushed with N_2 to remove air before the run was started. The precursors were heated at 5 ºC min^{-1} to the final temperatures (T =900, 950, 1000, 1050 ºC) and then held for 12 h. After cooling, the products were purged with N_2 for several minutes. According to the treatment temperatures, we defined the ordered MSONTFs as MSONTFs-T. For example, MSONTFs-1000 means the MSTFs was treated at 1000 ºC.

Characterization:

X-ray powder diffraction patterns were collected on a Rigaku D/ MAX-c β instrument using CuKa (λ = 0.15406 nm) radiation at 40 kV and 60 mA.

High-resolution transmission electron microscope (HRTEM) image was obtained using a JEOL 2010CX TEM.

Infrared spectroscopic investigation of the samples at room temperature was carried out on a Bruker Vector 22 spectrometer using the KBr pellet method. IR spectrum of a dehydrated material (the MSONTFs) was recorded using a Nexus-470 FTIR

spectrometer (made in NICOLE Company, USA) on self-supporting wafers in a glass vacuum IR cell. Prior to the collection of spectrum, the sample was heated in the cell for 4 hours at 300 ºC.

X-ray photoelectron spectroscopy data were obtained with an ESCALab220i-XL electron spectrometer from VG Scientific using 300W AlKα radiation. The binding energies were referenced to the C1s line at 284.8 eV from adventitious carbon.