Electronic Supplementary Information (ESI) for Dalton Transactions

Monolithic porous magnesium silicide

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Table ESI 1: N₂ sorption and PXRD data of different Mg_2Si samples at synthesis conditions as indicated with porous Si as precursor (samples with "Si44 μ " labeled derive from commercial Si powder)

	SA _{BET} (m²g⁻¹)	Crystallite size of Mg₂Si* (2 2 0) [nm]	Crystallite size of Si [*] (1 1 1) [nm]
Magnesium silicide_650 °C_30min	<50	100	
Magnesium silicide_650 °C_2 h	<50	116	
Magnesium silicide_650 °C_5 h	<50	140	
Magnesium silicide_600 °C_2 h	<50	128	25**
Magnesium silicide_600 °C_5 h	<50	130	
Magnesium silicide_700 °C_30min	<50	101	
Magnesium silicide_700 °C_2 h	<50	151	
Magnesium silicide_Si44µ_650 °C_30min	<50	544	442***
Magnesium silicide Si44µ 650 °C 2 h	<50	248	1800****

* Rietveld refinement; ** 7% silicon, 93 % Mg₂Si; *** 7% silicon, 93 % Mg₂Si; **** 1% silicon, 99 % Mg₂Si



Fig. ESI 1: XRD pattern of Mg₂Si samples with different reaction holding time (a) 650 °C - 2h; (b) 650 °C - 5h

The original silica monolith exhibits a type IV isotherm with H1-type hysteresis loop supporting the mesoporous character of the sample with a monomodal pore size distribution (Fig. ESI 2). Upon removal of MgO after HCl treatment, mixed isotherms of type II and IV with a H1 hysteresis loop (Fig. ESI 2) in the mesoporous region are obtained, indicating the presence of meso- and macroporous silicon. Washing with HF leads to a type II isotherm with a H1 hysteresis loop (Fig. ESI 2). For monolithic porous Mg₂Si, the N₂ isotherm shows a low amount of adsorbed nitrogen with no distinct hysteresis anymore. Therefore, pore size distributions will not give any reliable results.



Fig. ESI 2- N₂ sorption isotherms of precursor, intermediate and final products