

Figure S1: FTIR spectra of PVMSZ, PVMSZ_T and **PVMSZTi2.5** samples.



Figure S2: liquid-state ¹³C NMR spectrum of TDMAT.



Figure S3: experimental ¹³C CP MAS NMR recorded for the PVMSZTi2.5 sample from -20 to 160 ppm.



Figure S4: TG curves recorded during decomposition of PVMSZ in flowing ammonia, nitrogen and argon.



Figure S5: experimental ²⁹Si CP MAS NMR for the **P-1400NH**, **P-1400N**, **P-1400A** and **P-1800A** samples at 7 T.



Figure S6: identification of the α and β -Si₃N₄ phases in the XRD pattern of **P-1500N**.



Figure S7: HRTEM micrographs of **P-1500NH** (a), **P-1500N** (b) and **P-1500A** (c) samples with FFT images obtained from HRTEM images of **P-1500N** (b) and **P-1500A** (c) samples as insets.



Figure S8: EDS mapping of the **P-1600A** sample.



25µm

25µm

Figure S9: EDS mapping of the **P-1800A** sample.