Electrospun SiOC Fibre Mats from Polyvinylpyrrolidone/preceramic

Siloxanes Hybrid Systems

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Supplementary Information

1. XPS

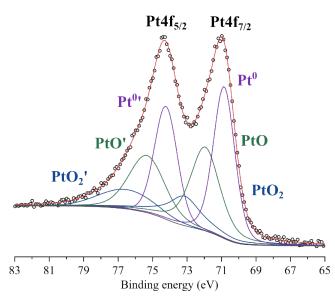


Figure S1 High resolution XPS Pt4f scan of Pt-TTCS/PVP pyrolyzed mat.

Pt4f high-res XPS is recorded from the same fibre mat that the survey scan (Fig. S2, left) and other (O1s, C1s and Si2p) high-res are performed. As shown in Fig. S1, the deconvoluted peaks are shown and marked with corresponding oxidation status at Pt⁰ (70.9 eV), PtO (72.0 eV) and PtO₂ (73.2 eV).

To investigate the compositional changes by exposing the fibre mats to acetylene torch flame for 20 s, XPS survey scan is recorded from all four pyrolyzed samples (Fig. S2). The detailed composition is given in the manuscript (Tab. 1 and Tab. 2). From the spectra, 4-TTCS/PVP, Pt-TTCS/PVP and 3-TTCS/PVP samples show a high consistency before and after heattreatment. However, a significant drop of C1s is observed in DTDS/PVP sample. This result is

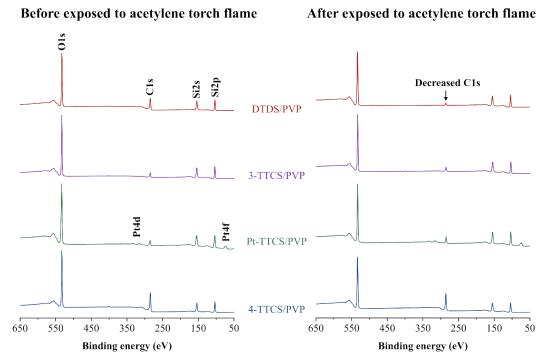


Figure S2 XPS survey scans of pyrolyzed ceramic mats, spectra of as-prepared mats are shown left and fibre mats exposed to acetylene torch flame (20 s) are shown right.

well matching with

2. NMR

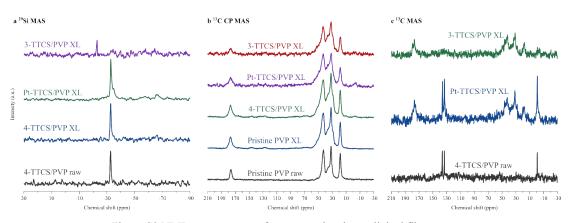


Figure S3 NMR spectroscopy of as prepared and crosslinked fibre mats.

NMR spectra of as spun fibre mats (in raw and crosslinked forms) are obtained and plotted in Fig. S3. Compared to powdered samples, ²⁹Si signals and Si-CH_x signals are much weaker and hard to make comparison between different preceramic polymers. PVP shows less or no

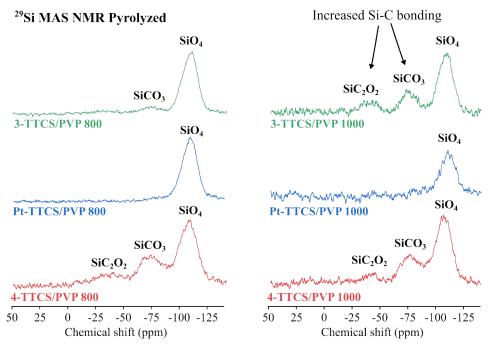


Figure S4 ²⁹Si MAS NMR spectra of samples pyrolyzed at 800 °C and 1000 °C.

crosslinking behavior when comparing the pristine PVP raw and XL samples in ¹³C CP MAS spectra.

NMR spectra of samples pyrolyzed at 1000 °C. As the increase of pyrolysis temperature, increase of SiC_2O_2 and $SiCO_3$ structures were observed only in 3-TTCS sample. However, almost unchanged microstructures were shown for 4-TTCS and Pt-TTCS samples.

3. Nano-indentation

Nanoindentation is performed on 4-TTCS/PVP fibres to investigate the transverse modulus of the ceramic fibres. The ceramic fibre mat is embedded in hard epoxy resin environment and

polished until the fibre mat is revealed on the surface. As a comparison, indentation is performed equally to the epoxy surface. The results are respectively 5.68 GPa and 1.84 GPa for

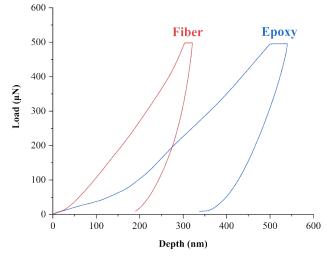


Figure S5 Nanoindentation result of 4-TTCS/PVP pyrolyzed mat embedded in epoxy. The plot shows two individual indentation on a single ceramic fibre and the hard epoxy resin surrounding the fibre.

the fibre and epoxy.

4. EDX

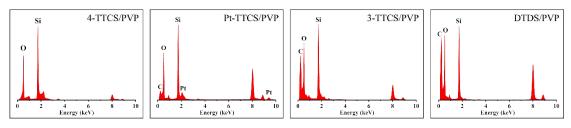


Figure S6 EDX spectra of fibre samples pyrolyzed at 800 °C

EDX analysis is performed to determine elemental composition of fibre surface. The EDX results are obtained using SEM setup, therefore, the atomic percentage of lighter elements, such as C in this case, are not accurate compared to other heavy elements. EDX of Pt-TTCS/PVP sample confirms the existence of residual Pt after pyrolysis.

5. BET Surface Area Analysis

BET surface area analyses are performed on as-spun ceramic fibre mats. N_2 is used as analysis adsorptive. The adsorption curves are plotted in Fig. S5. As a result, the BET surface area and

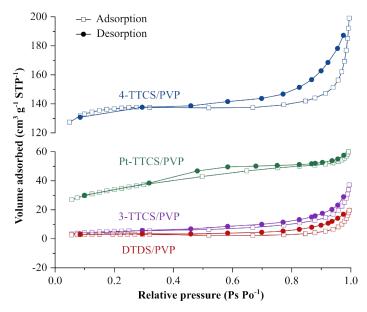


Figure S7 BET Surface Area Analyses of pyrolyzed fibre mats.

the adsorption pore sizes are calculated and provided in the Tab. S1. As seen from the results, 4-TTCS/PVP sample show the highest BET surface area.

Sample	BET surface area $(m^2 g^{-1})$	Adsorption pore size (nm)
4-TTCS/PVP	416.7	2.3
Pt-TTCS/PVP	115.6	2.8
3-TTCS/PVP	18.3	6.2
DTDS/PVP	9.2	5.0

Table S1 BET analysis results.