## Supporting Information

## Robust Silicone Aerogel via Copolymerization of Difunctional Organoalkoxysilane and Polymethylmethoxysiloxane for High-Temperature Thermal Insulation

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Figure S1. (a) FT-IR spectra of precursors PMMS, DMDMS, VMDMS, PMDMS. (b)

Solid-state <sup>29</sup>Si NMR spectra of typical aerogels.



Figure S2. Superior hydrophobicity of the aerogel shown by floating on water for

more than a month.



**Figure S3.** Schematic diagram of the crosslinked network and gel skeleton structure formed by using DMDMS, VMDMS, and PMDMS as crosslinking structures, respectively.



Figure S4. SEM images of typical aerogels: (a) PM-0.2, (b) PM-0.3, (c) PM-0.4, (d)

PM-0.3-3.8, (e) PM-0.3-2.2, and (f) PM-0.3-1.6.



Figure S5. (a)  $N_2$  adsorption/desorption isotherms of typical aerogels. (b) pore size distributions of typical aerogels.



**Figure S6.** (a,b) Comparison of the Si–O–Si mass percentage of typical aerogels and their residual weights in air or nitrogen atmosphere at 800 °C. (c,d) TGA curves of typical aerogels in air and nitrogen atmosphere.

Sample	Initial decomposition Temperature in	Initial decomposition	Residual weight	Residual weight
	air / °C	N <sub>2</sub> / °C	m an 7 70	III 1 <b>v</b> 27 70
DM-0.3	342	332	83.5	27.8
VM-0.3	272	360	82.1	72.3
PM-0.3	350	371	68.7	66.9
PM-0.2	341	346	74.2	69.7
PM-0.4	352	378	65.0	69.3

Table S1. TGA results of typical aerogels in air and nitrogen atmosphere



**Figure S7.** FT-IR spectra of reaction gas of sample DM-0.3 under nitrogen atmosphere at different temperatures. At an initial decomposition temperature of 332 °C, virtually no reaction gas is detectable. However, at the peak weight loss rate corresponding to 506 °C and the conclusion of weight loss at 642 °C, a substantial release of Si–O–Si oligomers was detected.



**Figure S8.** Comparison of the thermal stability of our aerogels and the other reported aerogels prepared using different reinforcement strategies: Spu-reinforced silica aerogel,<sup>1</sup> cellulose-reinforced silica aerogel,<sup>2</sup> PR-reinforced silica aerogel,<sup>3</sup> PVPSQ aerogel,<sup>4</sup> PBA-crosslinked silica aerogel.<sup>5</sup>



**Figure S9.** Schematic diagram of alcohol blowtorch heating device: A 150×150×20 mm aerogel plate is placed on the iron frame table, the alcohol blowtorch is placed right under the iron frame table, and the flame nozzle of alcohol blowtorch is 40 mm

away from the aerogel plate. A thermocouple is attached to the back of the aerogel plate, and the temperature of the back of the aerogel plate is displayed in real time by the electrical instrumentation.



Figure S10. Live view of alcohol blowtorch heating aerogel plate.

## Reference

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