Substantial Weight Gain by Oxidation of a Porous SiCO Gel under Inert Atmospheres

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Introduction

- Porous SiCO have a wide range of applications due to their low density, high specific surface area and microporous structure
- Impurities can impact the structure and properties of the material
- Argon and helium are inert gases
- Nitrogen is considered non-reactive for SiCO materials below 1000°C
- No inert gas is truly pure. Just 10 ppm of impurities can have a large impact

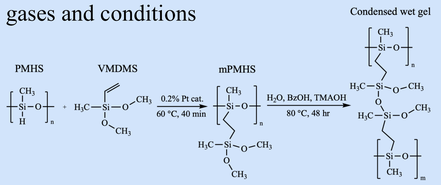
Motivation & Hypothesis

Motivation: Understanding the polymer to ceramic conversion is important to optimize processing routes

Hypothesis: Trace impurities from O_2, H_2O, and CO_2 cause oxidation of the sample resulting in substantial apparent mass gain

Method

- Porous SiCO gel prepared though hydrosilylation and condensation with subsequent ambient drying
- Thermogravimetric analysis (TGA) under different gases and conditions



Scheme 1. Two-step synthesis procedure for the preparation of the PMHS-based gel.

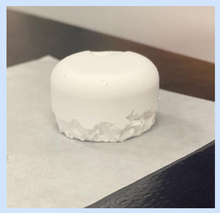


Figure 1. PMHS-based gel

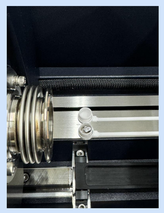


Figure 2. Image of TGA

Results

- TGA under argon, nitrogen, helium, and air
- Large mass gain under inert-conditions between 750 and 900°C
- Results confirmed at University of Trento, Italy
- Use of in-line oxygen trap reduces mass gain
- Covering sample with lid (semi-closed system) removes the apparent mass gain
- FTIR indicates oxidation in open sample
- Oxidation confirmed with chemical analysis

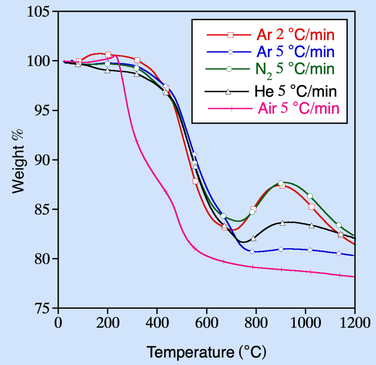


Figure 3. TGA under argon, nitrogen, helium, and air

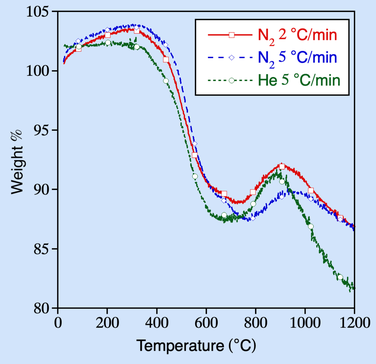


Figure 4. TGA under nitrogen and helium at U Trento

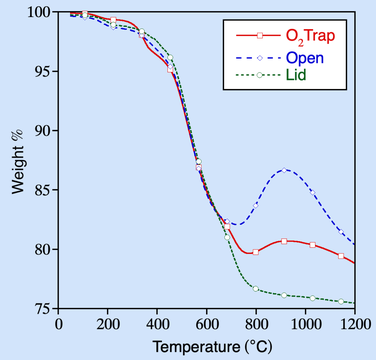


Figure 5. TGA with an open crucible, oxygen trap and lid

Figure 7. Tube furnace for bulk heating

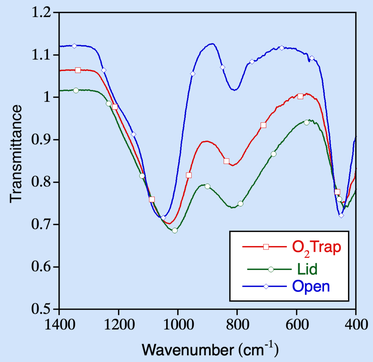


Figure 6. FTIR of TGA residue with an open crucible, oxygen trap and lid

Table 1. Chemical analysis for samples heated to 700 and 800°C

Temperature	C wt-%	O wt-%	H wt-%	Si wt-%	Composition
initial	32.9	21.9	6.8	38.4	$Si_1C_{2.0}O_{1.0}H_{5.0}$
700°C	24.83	29.1	4.0	41.07	$Si_1C_{1.4}O_{1.2}H_{2.6}$
800°C/4h	21.60	34.2	2.0	42.2	$Si_1C_{1.2}O_{1.4}H_{1.3}$

Future Work

- Continue collaboration with MPI to get TGA-MS (evolved gas analysis)
- Study oxidation reaction mechanism and kinetics

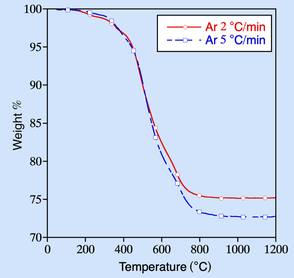


Figure 8. TGA under argon inside glovebox at the MPI

Summary

- SiCO material was oxidized due to ppm level impurities during pyrolysis in TGA
- Mass gain observed under nitrogen, argon, and helium
- Results confirmed on separate instruments at Max Planck Institute and University of Trento
- Addition of oxygen trap within gas stream reduced apparent mass gain
- Use of lid reduced mass gain further
- Chemical analysis confirmed oxidation

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Acknowledgements

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