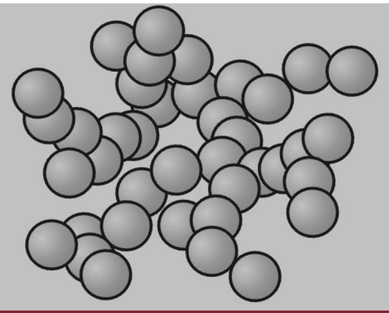

Design of Prototype Monolithic Aerogel Catalytic Converter

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INTRODUCTION

nal combustion engines utilize catalytic converters to combat the production of toxic compounds found in car exhaust, such as nitrogen oxides, carbon monoxide, and hydrocarbons, by converting them to more environmentally friendly gasses, such as carbon dioxide, water vapor, and nitrogen gas. Aerogels' high porosity and high surface area, as well as the ability to maintain textural stability at high temperatures, make them good candidates for heterogeneous catalysis applications. Past research in the Union College Aerogel Lab has shown that the catalytic performance of granular or powdered aerogels can be augmented by adding metal salts in the synthesis process. Unfortunately, free-standing granular aerogels would be an unaccommodating substitute for traditional catalytic converters as they are difficult to contain. While the low-difficulty, low-cost pathway into the commercialization of aerogel catalytic converters is through the introduction of aerogels into the conventional wash-coating process used to coat the catalytic converter substrate with a catalyst material, monolithic models may prove to be more feasible to produce and more effective at improving the catalysis of automotive emissions. Aerogels have high surface areas and are very porous, however, the rate of diffusion of gases through a monolithic aerogel is not suitable for use in a catalytic converter. The purpose of this research is to develop an aerogel catalytic converter prototype, and by doing so accomplish three things: 1) create multiple monolithic aerogel substrate models; 2) quantitatively compare the substrate models against each other to prescribe the most effective model; 3) and lastly, test the catalytic performance of the model when doped with catalytic metals.

RSCE METHOD

When applying the engineering design process, it is important to exhaustively brainstorm protentional areas for improvement. Figure 1 displays the parameters that affect the performance of catalytic converters. With subject matter knowledge of both aerogels and catalytic converters, substrate material was isolated as a key driver for catalytic converters performance.

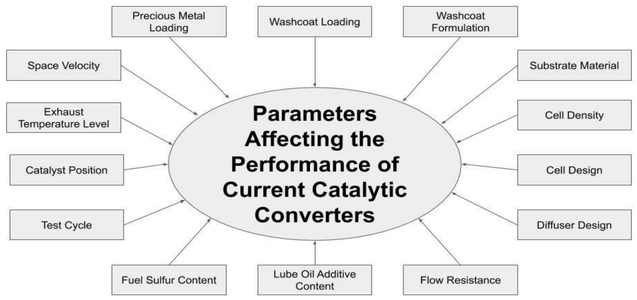


Figure 1: Parameters Affecting the Performance of Catalytic Converters

RSCE METHOD

The most common supercritical extraction method for making aerogels uses an autoclave (pressure vessel) and carbon dioxide (low temperature) supercritical extraction [1,2]. However, in the Aerogel Lab at Union we use a Rapid Supercritical Extraction (RSCE) technique [3-5]. The equipment we use includes a 30-ton hydraulic hot press, an alloy steel mold, a high temperature gasket, and a stainless-steel foil (Figure 1). The advantage of this method is that the entire process can be accomplished in one step and in a few hours [4, 5].

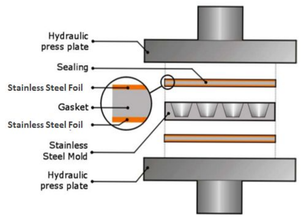


Figure 2: Schematic of the Mold and Press Configuration for Aerogel Processing.

Table 1. Silica Aerogel Recipe

TMOS (g)	MTMS (g)	MeOH (g)	DI Water (g)	NH_4OH (µL)
9.78	0	24.47	4.04	302

Table 2. Hot-Press Conditions

Step #	Temperature (℉)	Temp. Rate (℉ /min)	Force (kips)	Force Rate (kips/min)	Dwell Time (min)
1	90	200	50	600	0
2	550	4	50	600	30
3	550	200	1	1	15
4	90	4	1	1	15
5	END				

SUBSTRATE MODELS AND PRESSURE TESTING

nolithic aerogel substrate for use in a catalytic converter it was necessary to ensure that the substrate could withstand the flow conditions necessary for catalytic testing. Thus, three substrate models were tested in a system specifically designed to study the pressure across the substrate at varying flow rates, the Union Mechanical Airflow Testbed (UMAT). The ultimate goal of creating new monolithic substrates rather than using a standard Silica monolithic aerogel was to increase the maximum allowable flow rate through the sample while decreasing the subsequent back pressure across the model at the achieved flow rates. The two new substrate models invented were 1) the Carbon Burn Out model, where activated carbon was added to the aerogel precursor solution and removed with heat treatment after hot press fabrication, and 2) the laser cut model where standard silica aerogels were laser cut after removal from the hot press.

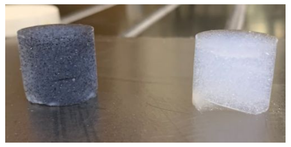


Figure 3: Carbon Burn Out Samples Before (Left) and After Heat Treatment (Right)

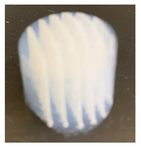


Figure 4: Laser Cut Sample

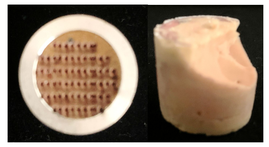
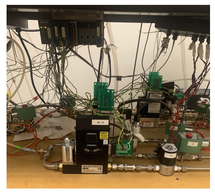


Figure 5: HTCA Sample in Threaded Sample Holder and Laser Cut (Left) and Unaltered (Right)

UMAT DESIGN



Figures 6-8: UMAT (Top Left) UMAT Flow Controllers and Flow Meters (Top Right) Test Chamber with Gasket, HTCA Sample in the Ring Holder, Spacers, and Screens (Bottom)

The UMAT was designed to receive an input flow rate of up to 15 SLPM, provide airflow at and display the actual flow rate, and output the pressure across the test chamber in psi. The airflow was controlled with a flow controllers and measured with flow meters, and pressure was recorded with both a pressure transducer and a DAQ.

The test chamber was designed such that it fit seamlessly into the UMAT with little head loss. The sample was produced within tubing that was threaded to provide more stability to the sample. Gasket was used to fix the substrate model vertically within the test chamber such that flow could not exist between the sample ring or spacer and the inner wall of the test chamber. Screens were used to pervert broken samples from entering the rest of the UMAT.

Table 3: Results of UMAT Tests

Substrate Model	Activated Carbon	Laser Cut
Back Pressure at Max Flow Conditions (psi)	1.108 ± 0.578	1.289 ± 0.134

BET SURFACE AREA TESTING RESULTS

Table 4. BET Surface Area with Uncertainty Accounting for Mass and Surface Area

Sample		
Activated Carbon	470.5	47.4
Silica	455.0	28.3
Laser Cut	396.1	19.0

In order to understand the effect of burning activated carbon and laser cutting on aerogel properties, the ASAP 2020 Gas Adsorption instrument was used to analyze the porosity and surface area of various samples. To prepare samples for analysis, a portion of the aerogel was scraped off the monolith and then crushed using a mortar and pestle. Approximately 0.2 g of sample was put into a sample tube and massed. The sample was then degassed and remassed. The degas process heated the sample to 90 C for two hours and then 200 C for 6 hrs.

REFERENCES

[1] R. M. Heck and R. J. Farrauto, *Catalytic air pollution control: commercial technology*. Hoboken, NJ: Wiley, 2009. [2] S. Sabatini, "A New Semi-Empirical Temperature Model for the Three Way Catalytic Converter", IFAC-PapersOnLine, Volume 48, Issue 15, Pages 434-440, 2015, ISSN 2405-8963. [3] Donlan, E., Anderson, A.M., 2017,"Fabrication and Analysis of Metal-Nanoparticle Containing Silica Aerogels"

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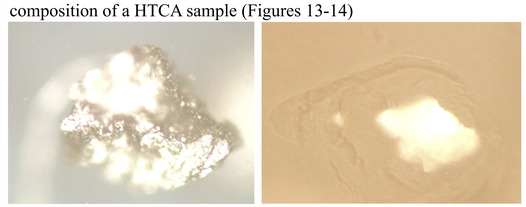
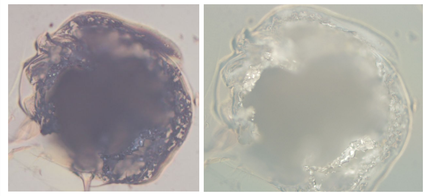
MICROSCOPY

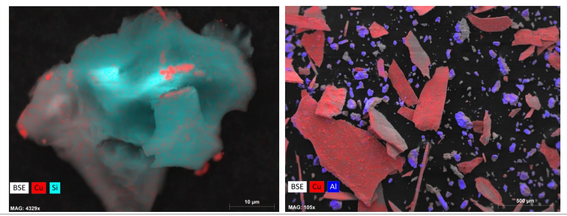
Microscopy was utilized to qualitatively analyze the new monolithic aerogel substrate models invented in this research. Optical microscopy was performed on the carbon burn out sample before and after heat treatment (Figures 9-10), and on laser cut samples (Figures 11-12). Scanning Electron Microscopy (SEM) was performed by Chris Avenessian to yield a better understanding of the composition of a HTCA sample (Figures 13-14)



Figures 9-10: Optical Imaging at 200x Magnification of Carbon Burn out Samples Before (left) and After Heat Treatment (Right)

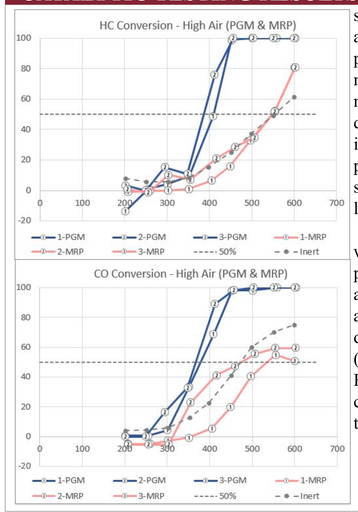


Figures 11-12: Optical Imaging at 200x Magnification of Laser Cut Pores



Figures 13-14: SEM imaging of HTCA Samples

CATALYTIC TESTING RESULTS



To dope the samples with catalytic metals a new substrate model had to be invented. Heat treated copper alumina (HTCA) aerogel granules were added to the precursor solution at a proportion of 1% by mass. The new solution was mixed on a stir plate until the viscosity noticeably increased. Once gelation was observed qualitatively, the methanol was super critically extracted in the hydraulic hot press with the same hot press procedure used in all the previous samples. Finally, the samples were laser cut following the laser cut two sides lined up procedure.

For reference, the monolithic HTCA silica aerogels were compared to PGM aerogels composed of 50% platinum and granular silica samples known not to have any catalytic activity. In both tests, the HTCA silica aerogels performed very closely with the inert samples, displaying a lack of catalytic activity where the platinum (PGM) samples did. The lack of catalytic activity in the HTCA samples could be due to a low percentage of catalytic metals or the high space velocity used in the tests

Figure 8: Catalytic Performance of Granular PGM Aerogels, Monolithic HTCA Silica Aerogels, and Granular Inert Silica samples.

CONCLUSION

The purpose of this study was to improve the performance of catalytic converters. To integrate monolithic aerogel substrates into catalytic converters, the stability of the aerogel substrates had to be studied. It was determined that while the two best substrate models were the carbon burn out and laser cut models, with top pressures of 1.108 ± 0.578 and 1.289 ± 0.134 respectively. The laser cut two sides lined up had more consistent top pressures and was more suitable for the inherent commercial application, thus it was selected as the model to be catalytically doped.

Although the sample was determined to be essentially inert through catalytic testing, may opportunities for further studies arose. Primarily the same sample could be tested with less harsh flow conditions, such as a lower space velocity. Additionally, the amount of HTCA aerogel doped into the silica sample was 1% by weight. Increasing the ratio of HTCA would theoretically increase the catalytic activity but may also lower the stability of the substrate. Lastly I am interested in testing the laser cut and carbon burn out models with different amounts of alteration, at higher volumetric flows.