Development of Acicular Mullite Materials for Diesel Particulate Filters Application

Aleksander Pyzik, Chan Han, Cheng Li, Mike Malanga, Frank Mao, Doug Merrick, Art Prunier, Clifford Todd, Denis Turmel, Kwanho Yang

Dow Automotive / New Products Core R&D

The Dow Chemical Company

presented at the DEER conference,
Chicago, Illinois
August 24, 2005

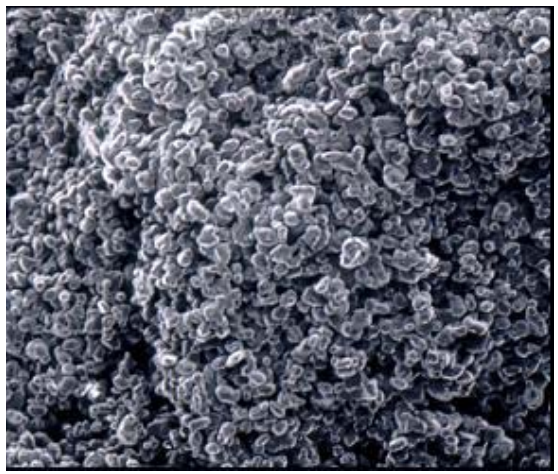
Introduction To Advanced Ceramic Material (ACM)

- ACM, i.e. Advanced Ceramic Material, is high porosity DPF formed of Mullite crystals
- ACM exhibits a unique microstructure compared to any other commercially available ceramic substrates
- This results in a range of unique properties from a physical standpoint, but also from an application standpoint

INTRODUCTION TO ACM

ACM Microstructure

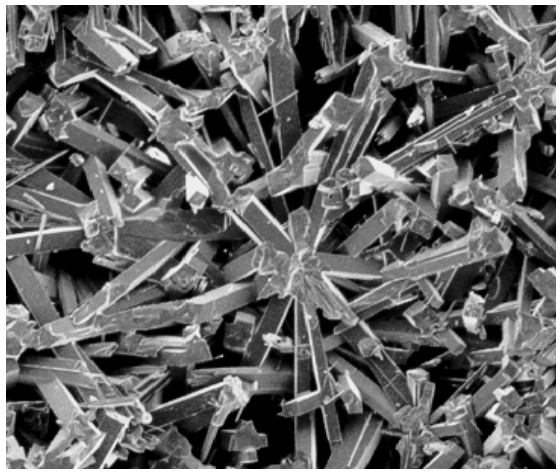
Standard ceramic microstructure: grains are fused to one another through a sintering process



Analogy with →

Washington Monument

Advanced ceramic microstructure: three-dimensional interconnected mullite crystals microstructure with open connected pores

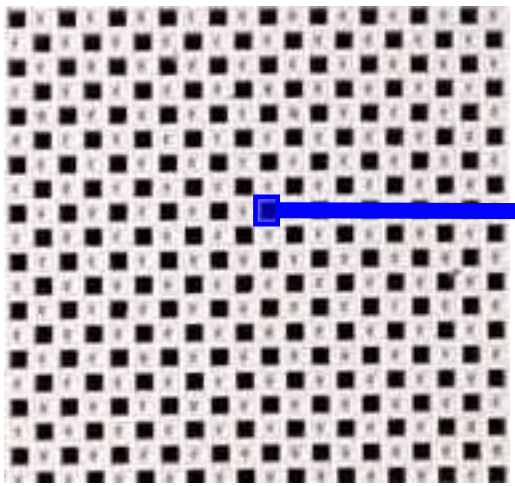


Analogy with →

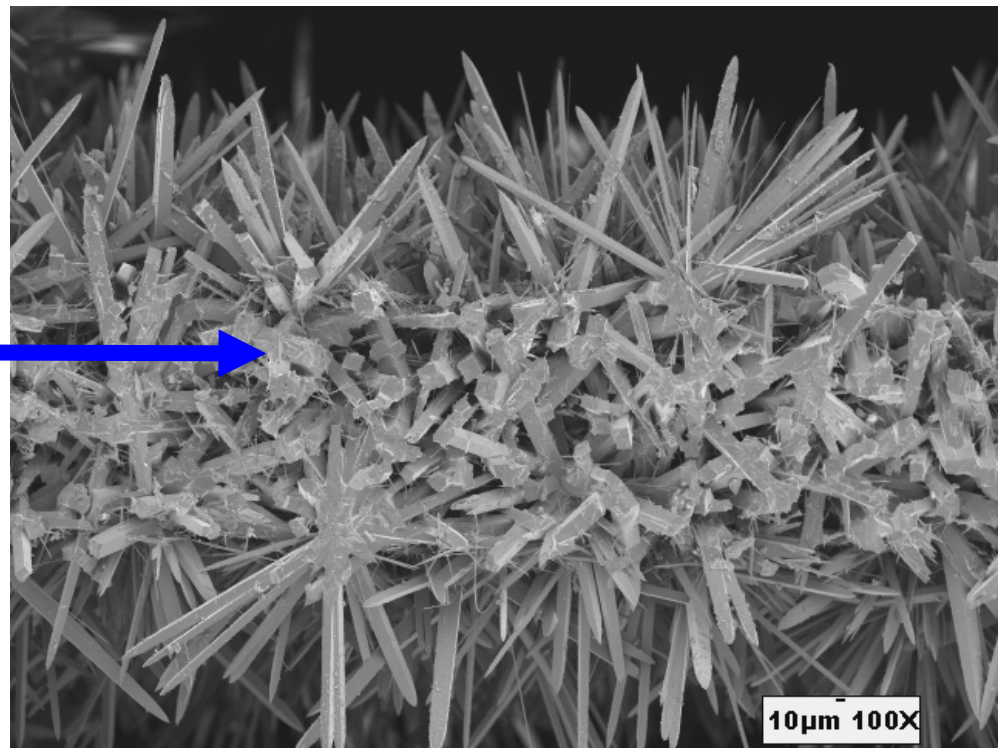
Eiffel Tower

INTRODUCTION TO ACM

ACM Microstructure

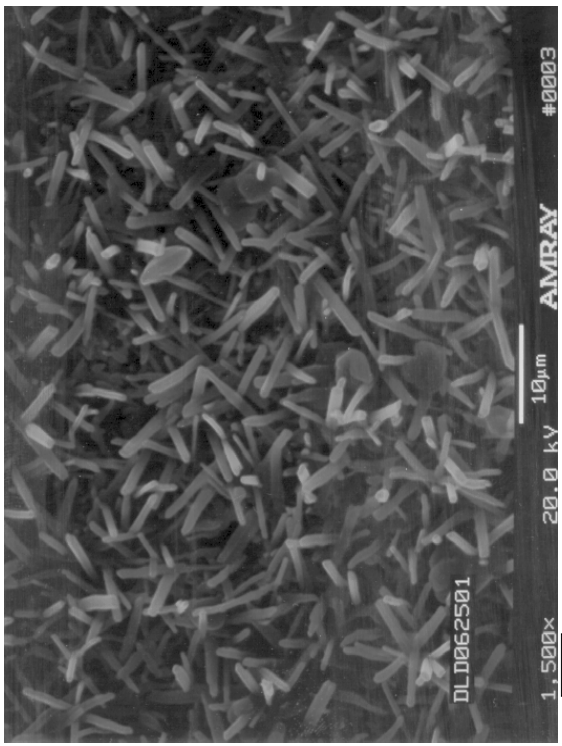


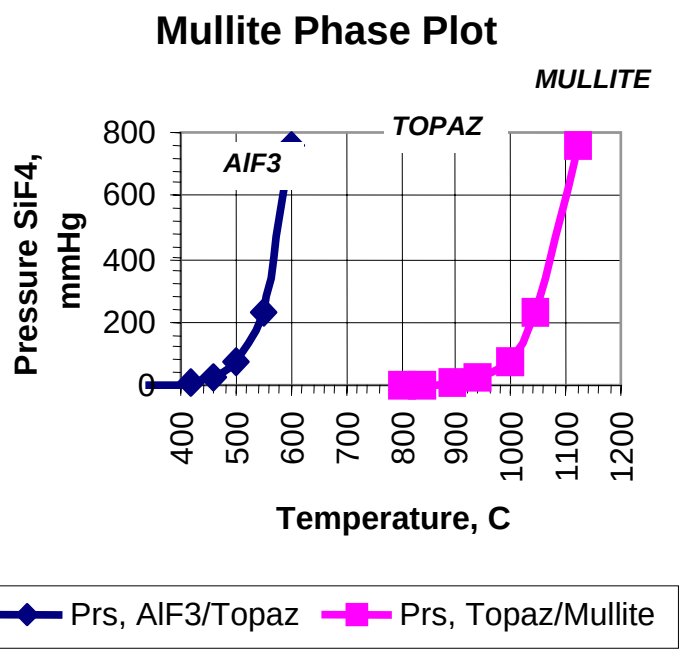
Acicular microstructure

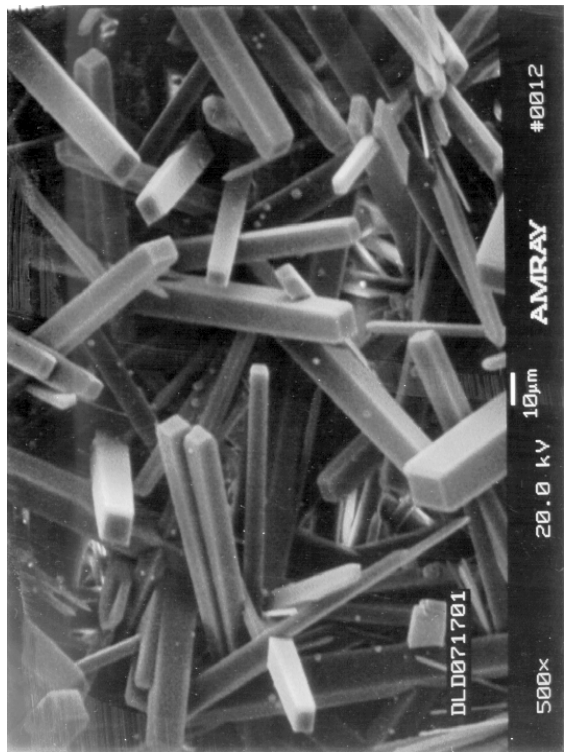


Synthesis of Advanced Ceramic Material (ACM)

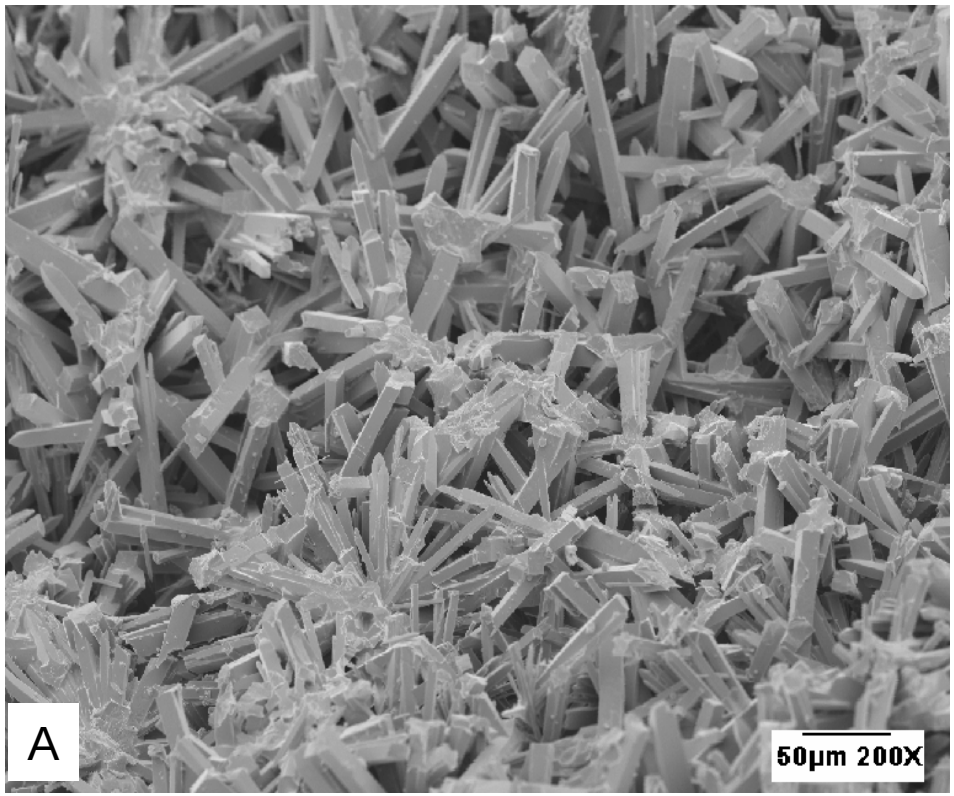
Mullite Precursor $(\sim 1.7Al_2O_3 \cdot SiO_2)$ $\xrightarrow[\sim 700C]{+ SiF_4}$ **Fluorotopaz** $(Al_2SiO_4F_2)$ $\xrightarrow[\sim 1100C]{- SiF_4}$ **Acicular mullite** $(\sim 1.7Al_2O_3 \cdot SiO_2)$

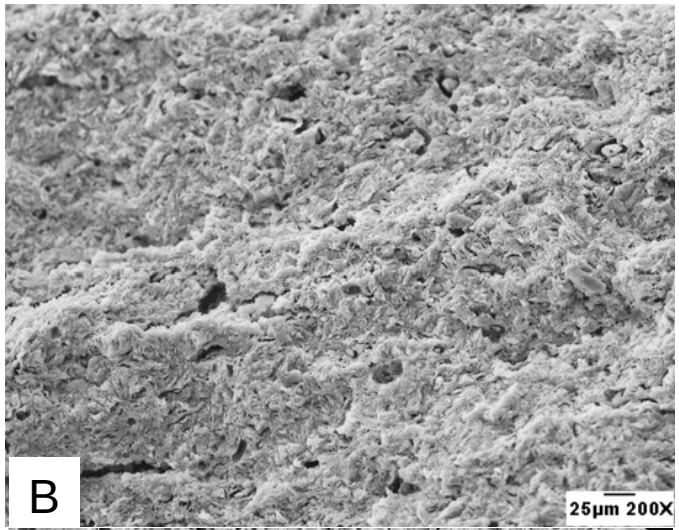


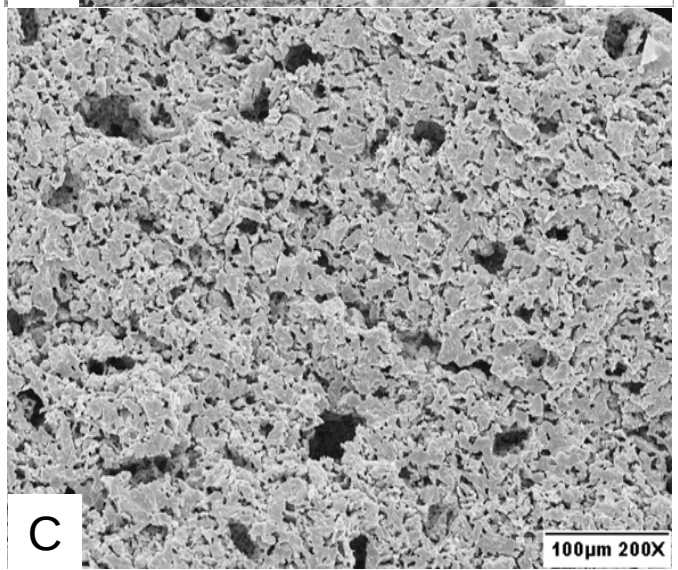




Highly Acicular Mullite Forms Under Specific Conditions Using Dow's Proprietary Process





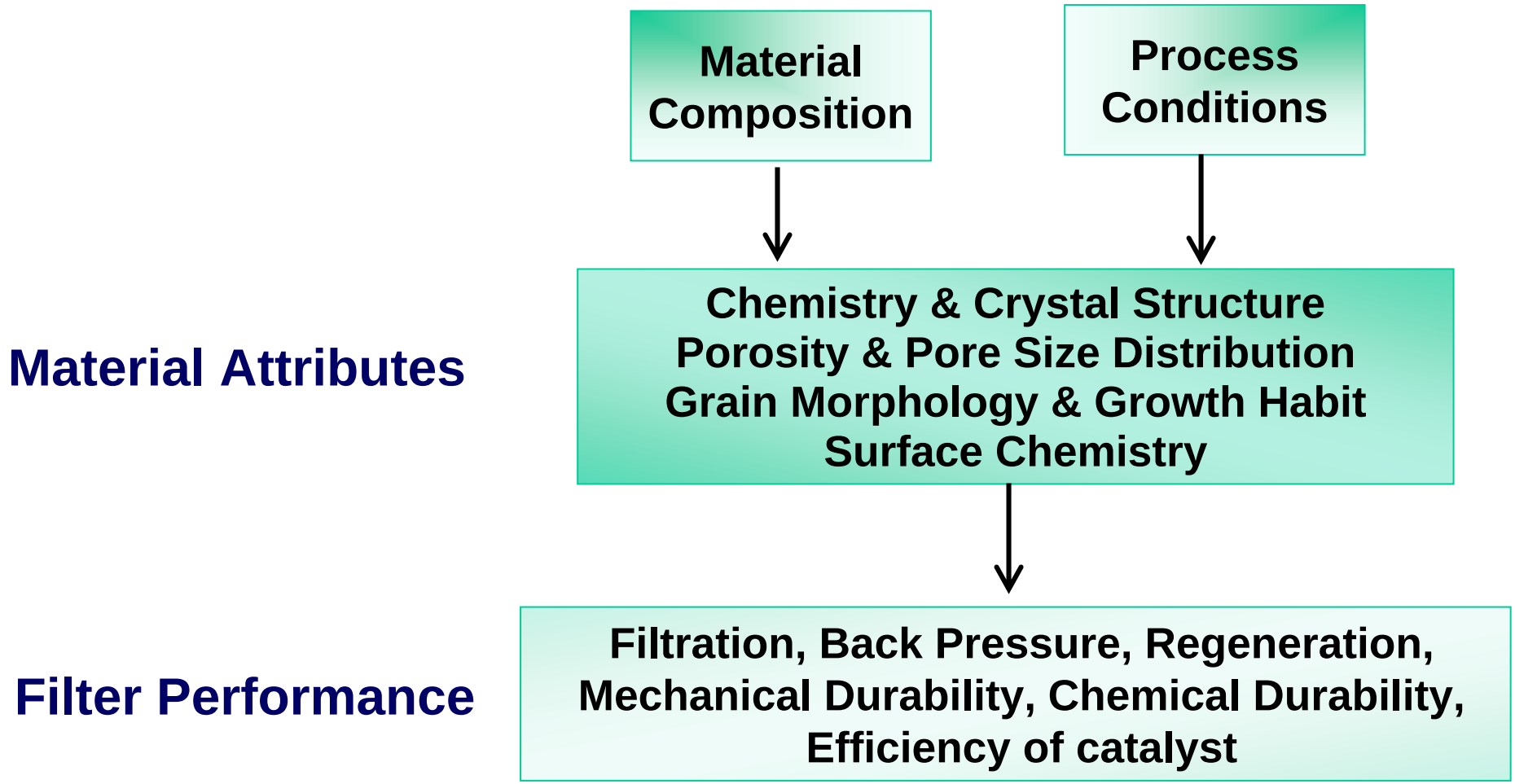


A. Typical Microstructure (Dow process)

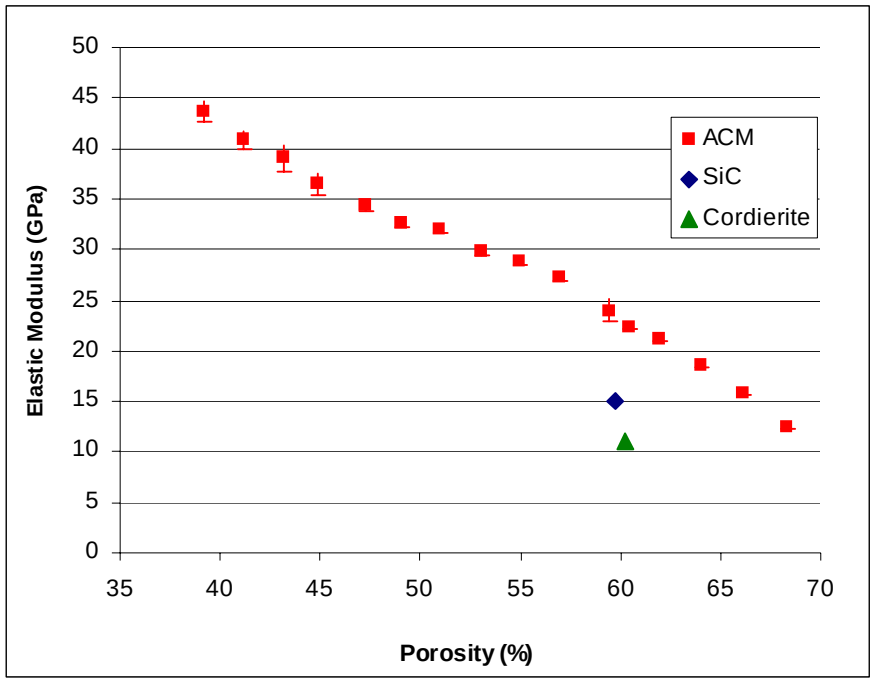
B. 1200°C Calcination in air

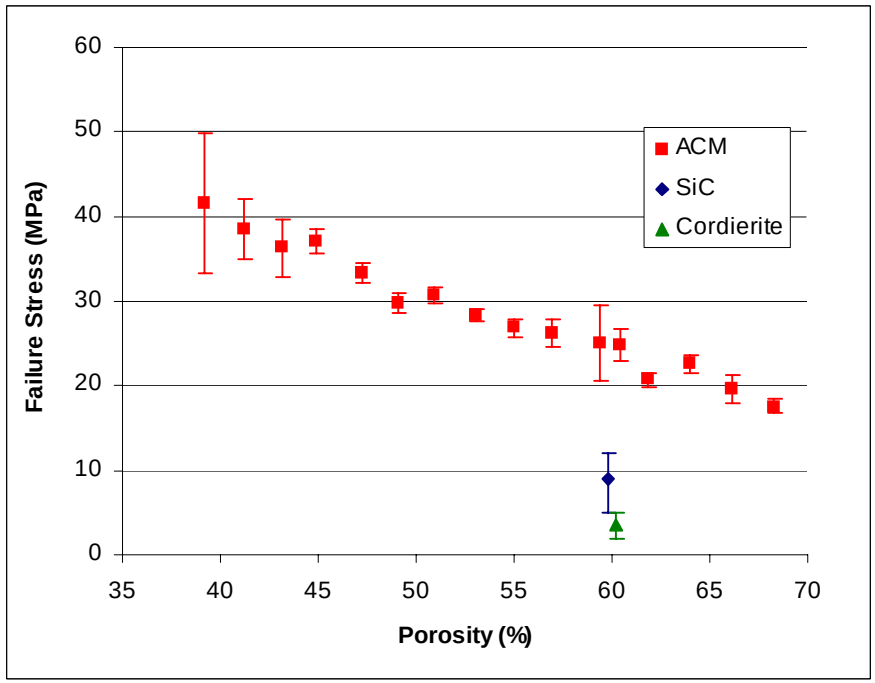
C. 1500°C Calcination in air

Filter Performance Is Enhanced By The Unique Material Attributes Of ACM



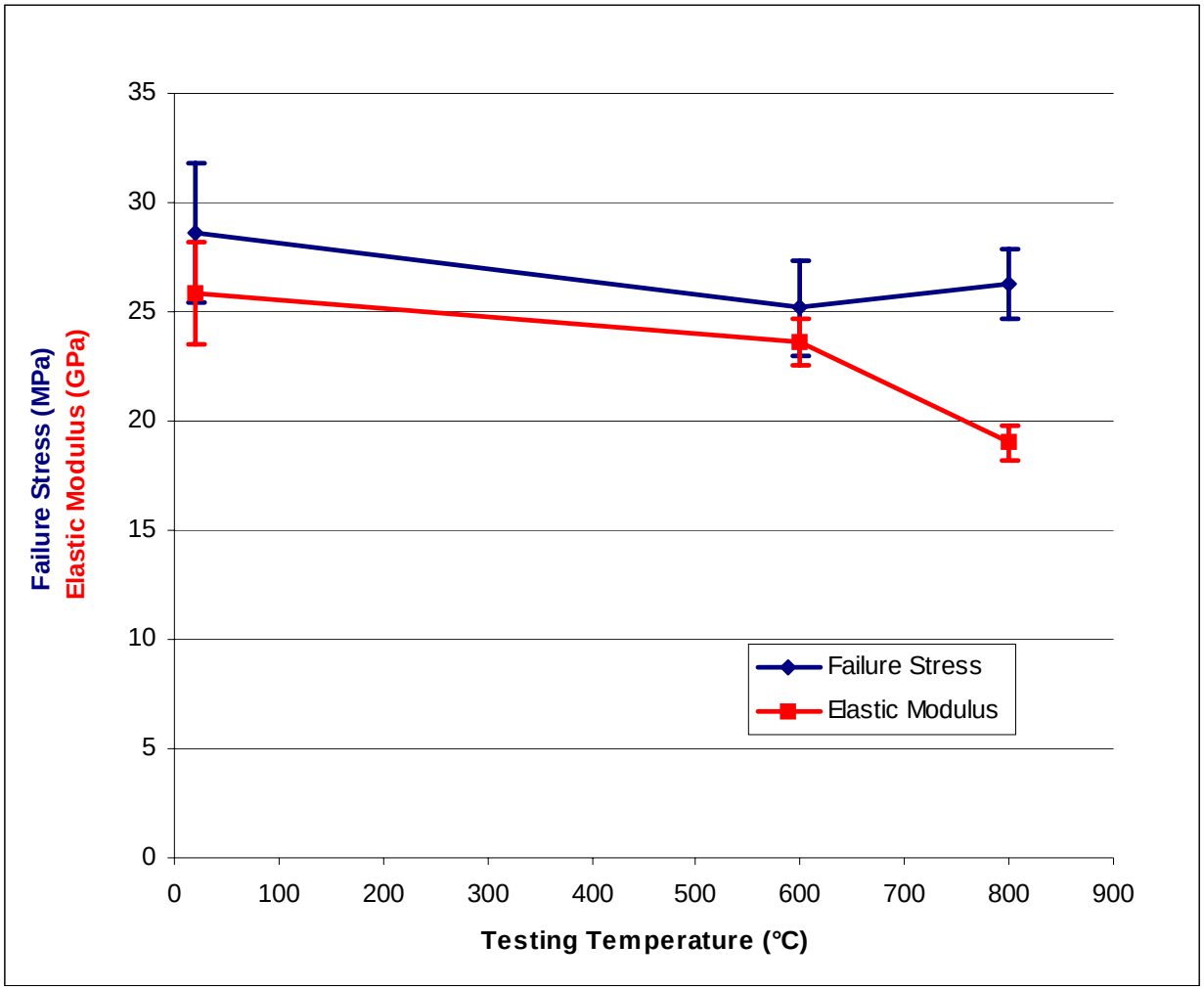
Acicular Structure Allows Combination Of High Porosity And Excellent Mechanical Integrity



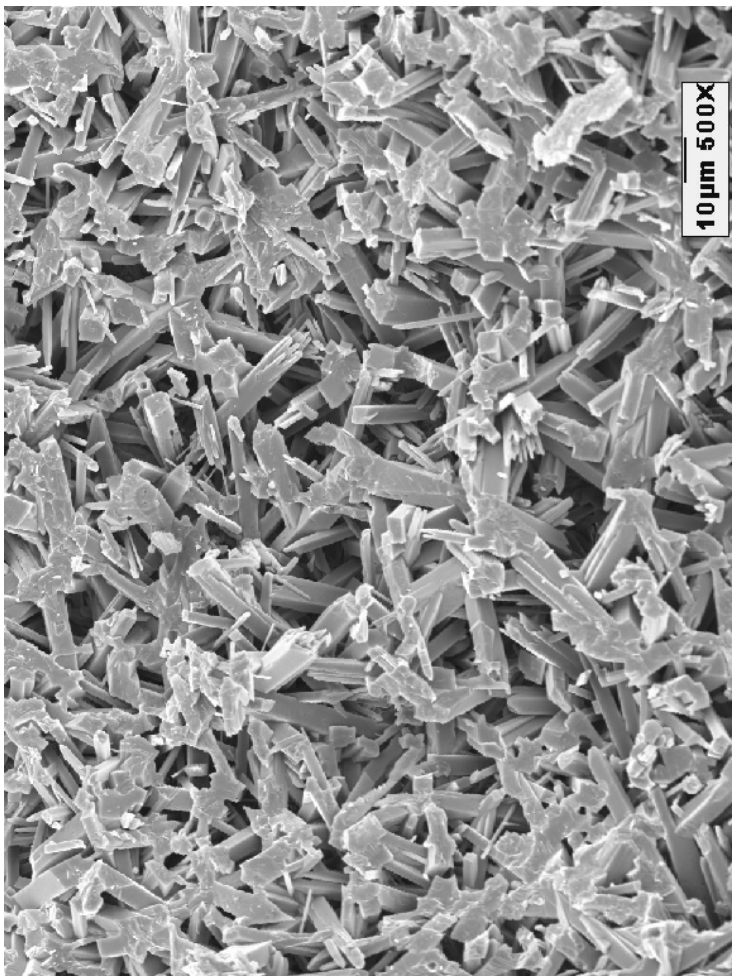


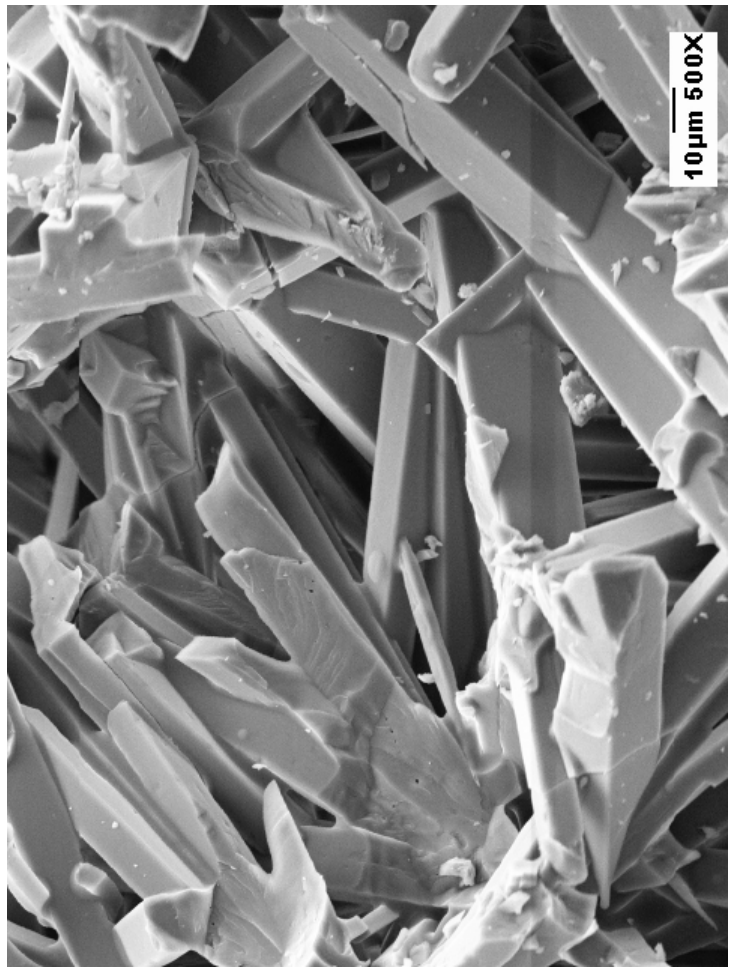
Data source for cordierite and SiC: - SAE 2003, Article 2003-01-0380
- Aachener Kolloquium Fahrzeug und Motorentechnik 2002, pp. 819-840

Flexure Strength And Elastic Modulus Is Maintained At Elevated Temperatures

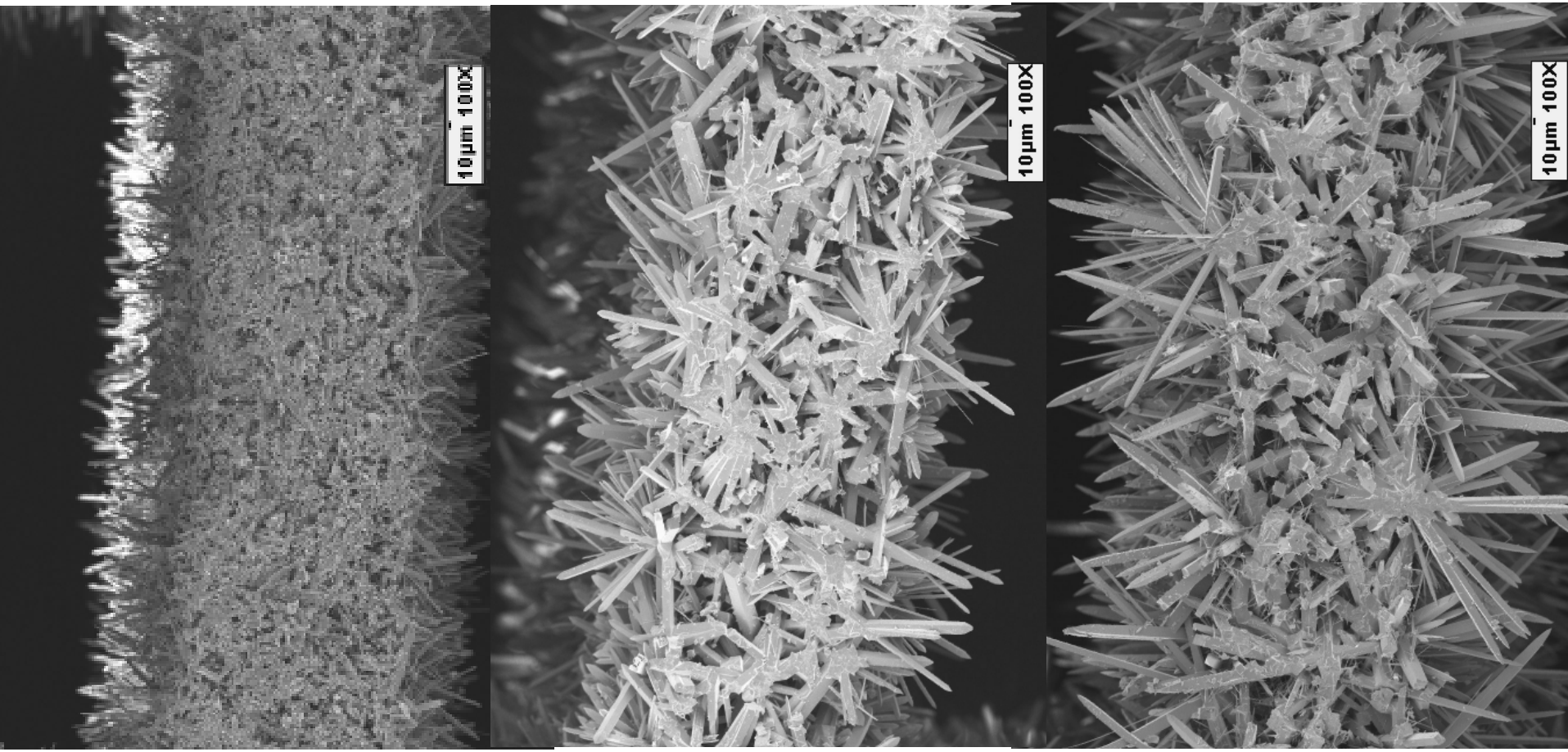


Controlled Nucleation And Crystal Growth Produces Different Grain Size And Consequently Different Pore Size





The Ability To Control Channel Surface Texture Allows Maximization Of Soot – Catalyst Interactions



Dow ACM Has Superior Chemical Resistance To Acids

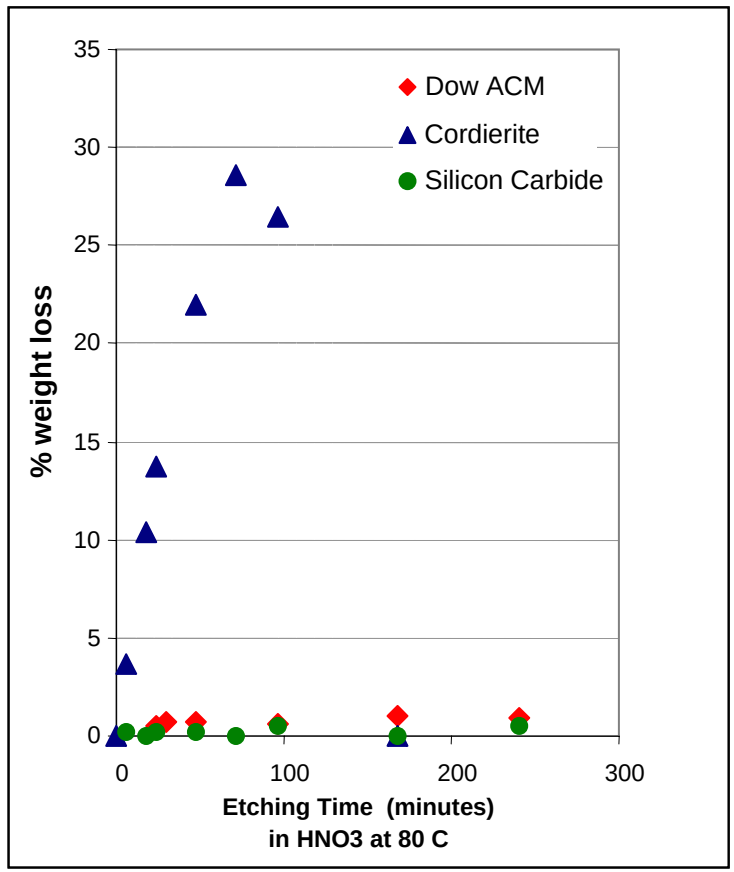
Effect Of 10% Nitric Acid On Various Honeycomb Materials (80°C)

Honeycomb material	Etching time		
	24 hours	100 hours	170 hours
Acicular Mullite	0.85 wt%	1.22 wt%	1.32 wt%
Cordierite	13.7 wt%	26.5 wt%	---
Silicon Carbide	0.17 wt%	0.48 wt%	0.51 wt%

Behavior Of ACM In Different Acidic Environments (96 hours)

Type of etchent	Etching temperature	
	25° C	80°C
10% HNO_3	0.6 wt%	1.07 wt%
10% H_2SO_4	0.54 wt%	1.2 wt%

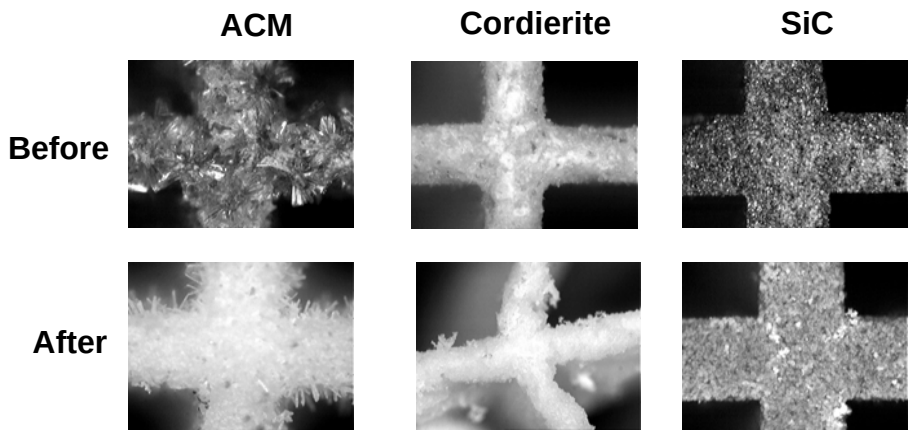
Weight Loss As A Function Of Time



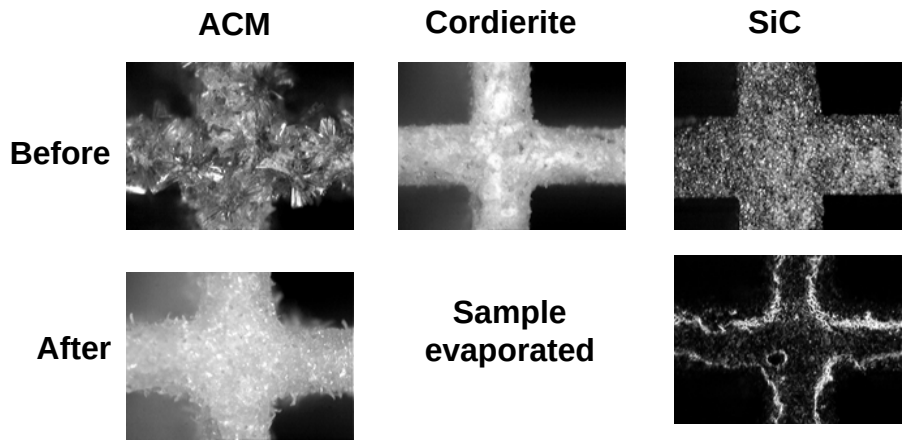
Dow ACM Has Excellent Resistance To Ash

Exposure to ash components at 1300°C for 5 hours in air.

Metal oxides	Acicular Mullite	Cordierite	SiC
Ca(OH) 2	Pass	Failed	failed
CeO2	Pass	Pass	Pass
MgO	Pass	Pass	Pass
ZnO	Pass	deformed	Pass
NaCl	Pass	melt	deformed
Na2SO4	Pass	Melt	Melt



ZnO deposited on the filter surface



NaCl deposited on the filter surface

Typical Mechanical Properties of DOW ACM

Property	Nominal value	Unit
Mullite density*, **r**	**3.17**	g/cm^3
Wall porosity (Hg)	**60**	%
DPF bulk density, $\mathbf{r}_b$	**0.52**	g/cm^3
DPF mass	**1300**	g
Melt temperature	**>1500**	°C
Young's modulus, E	**30**	GPa
Flexural strength, **s**	**30**	MPa
Poisson's ratio*, **n**	**0.20**	
Thermal expansion coefficient, **a**	**2.7**	µm/(m.K)
Mullite specific heat capacity*, C_p	**0.77**	J/(g.K)
Wall thermal conductivity, **l**	**1.3**	W/(m.K)

Note: All data are typical values measured at room temperature from 5.66" x 6.00" honeycomb monolith, unless otherwise specified (*)

Conclusions

- ***ACM DPFs fabricated by the Dow Chemical Company proprietary process offer improved filter performance due to the unique microstructure and designing capability.***
  - ACM DPF can be produced with controllable grain size, pore size distribution, total porosity and channel surface texture.
  - ACM DPF are characterized by the high temperature stability, excellent chemical inertness, and very good mechanical integrity even at porosities well above 60%.
  - **These material attributes result in the improved filtration characteristics, lower back pressure and more efficient catalyst-soot interactions.**