Non-destructive, Non-invasive Neutron Imaging of Soot, Ash and Washcoat Deposition in Particulate Filters

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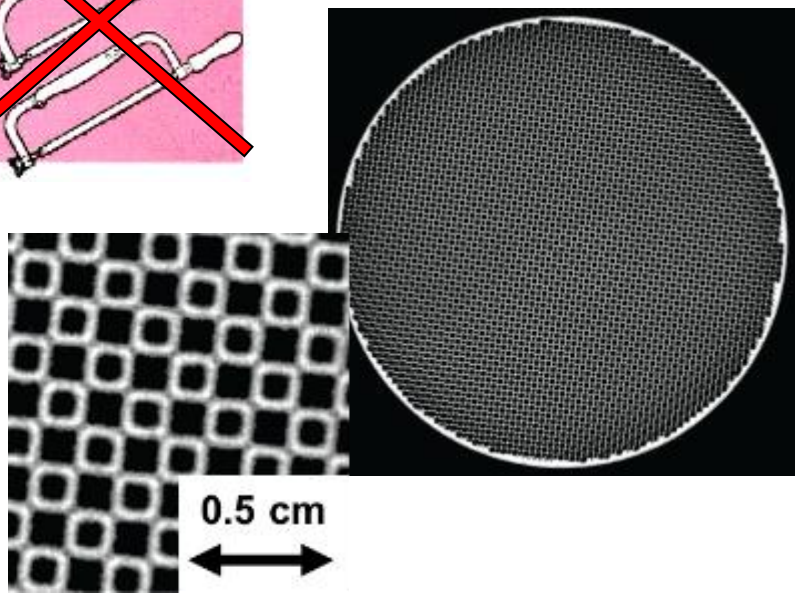
PAPER # n/a (Oral Only)

Non-destructive techniques needed for iterative approaches and to minimize disruption

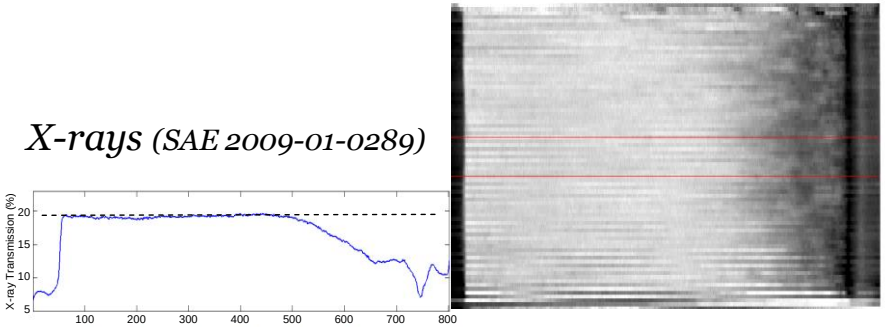
- Non-destructive techniques
  - Neutrons
    - Capable of high detail, 10-50 microns
    - strong absorbance with light elements
  - X-rays
    - Wide range of applications
      - low cost portable tomography
      - Synchrotrons
    - strong absorbance with heavy elements

- Destructive Techniques
  - Limited spatial resolution
    - Can only observe where specimen is fractured
  - TEM, SEM and EPMA
  - Iterative studies are difficult/impossible

Neutron CT scan (SAE 2009-01-2735)



X-rays (SAE 2009-01-0289)



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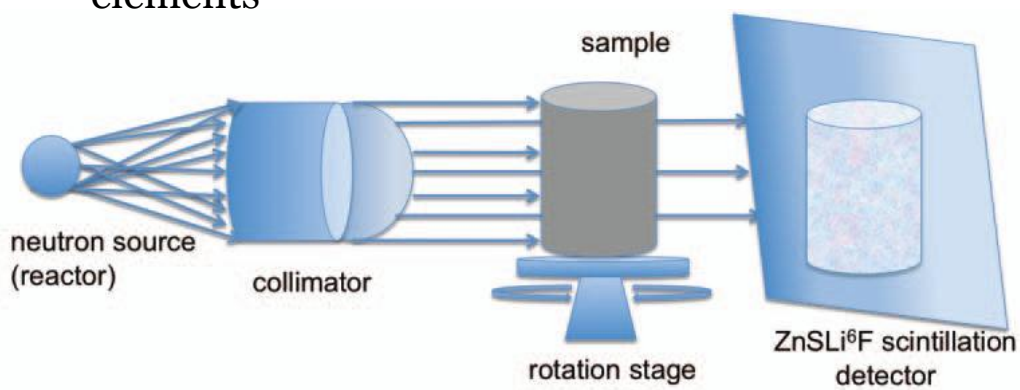
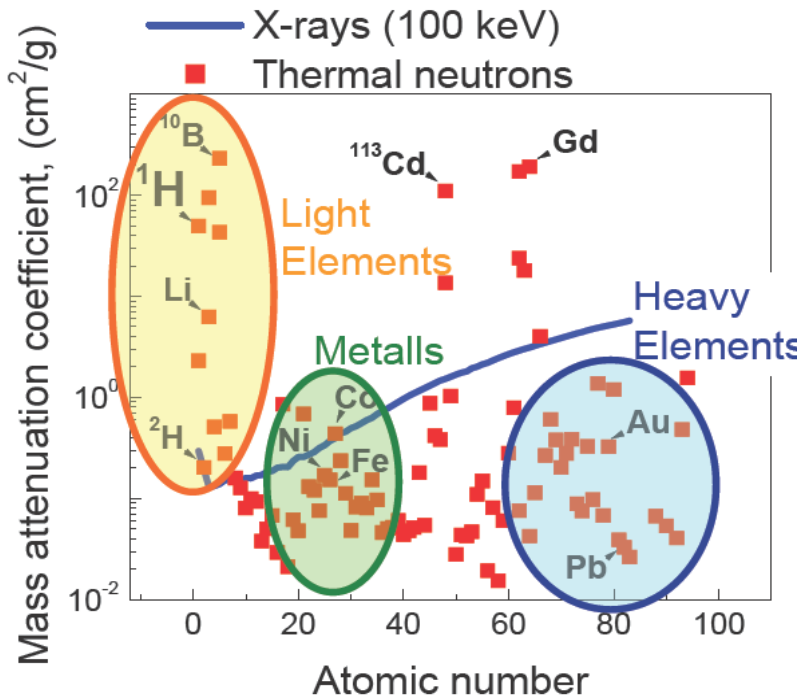
Neutrons are absorbed by a range of elements including light elements

- Neutrons absorbed by light elements such as Hydrogen and Boron
  - Can penetrate metals without absorbing
  - Sensitive to water and hydrocarbons/fuel
    - Can image carbon soot layer due to absorption of water and HC
  - Image is based on absence of neutrons

- X-ray imaging relies upon absorption of heavy elements





Neutron imaging is a complementary analytical tool

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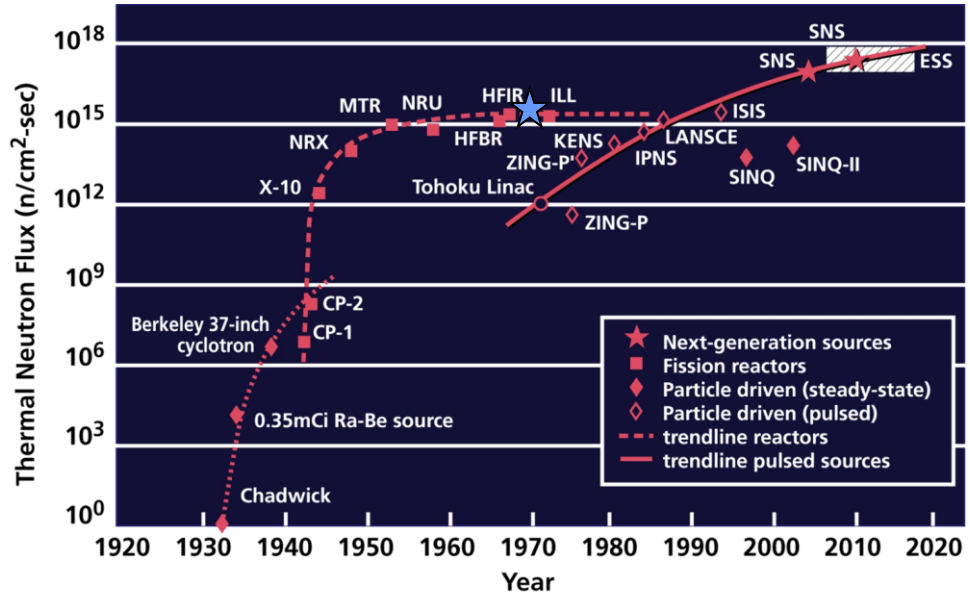
Attenuation Coefficient Reference: N. Kardjilov's presentation at IAN2006
http://neutrons.ornl.gov/workshops/ian2006/MO1/IAN2006oct_Kardjilov_02.pdf

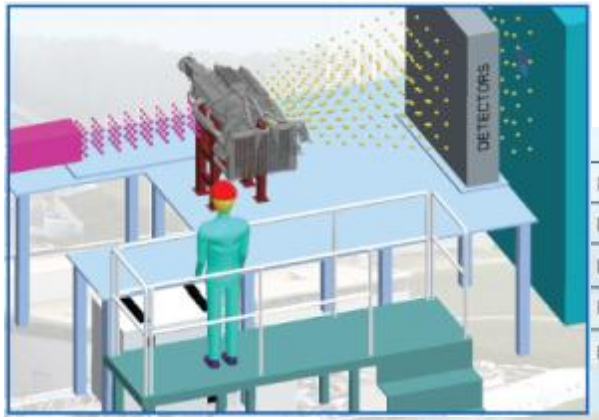
Neutron imaging capabilities at ORNL

- High Flux Isotope Reactor (HFIR)
  - Steady "white" neutron beam
  - Imaging beamline incorporated into user program
    - Accepted proposals cover neutron Neutron scientists efforts and beam time

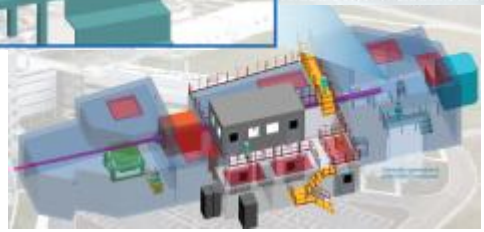
- Spallation Neutron Source (SNS)
  - Most intense pulsed neutron beams in the world
    - energy selective
  - Multi-laboratory effort funded by DOE Office of Science
  - Imaging beamline moving forward, but not reality yet



(Updated from *Neutron Scattering*, K. Skold and D. L. Price: eds., Academic Press, 1986)

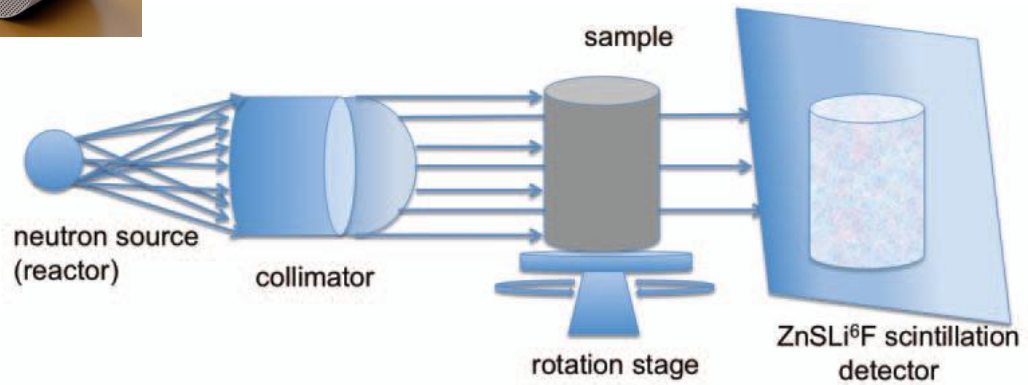


Estimated Beam Characteristics	
Maximum Field of View	90cm x 90cm
Length to Diameter Ratio (L/D)	>1000
Integrated Neutron Flux	10^8 n/cm2/sec
Neutron Beam Energy Range	0-40Å
Energy Resolution ($\Delta\lambda/\lambda$)	0.1%

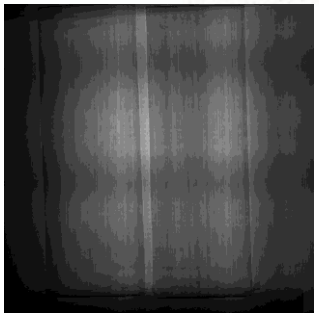


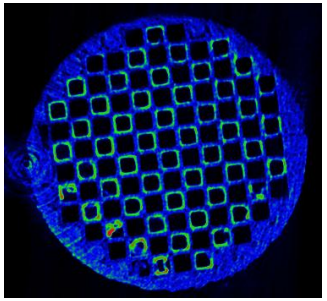
Approach enables iterative studies

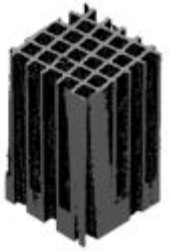
Receive devices from industrial partners or implement our own techniques



Record raw images of devices using neutron beam



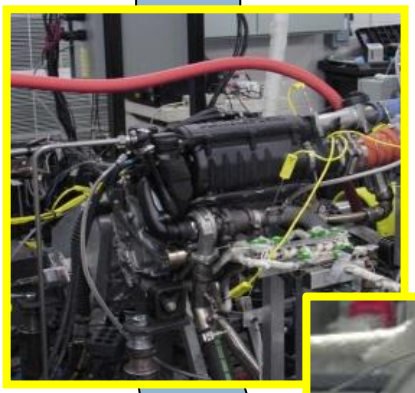




DPF Walls

Particulate

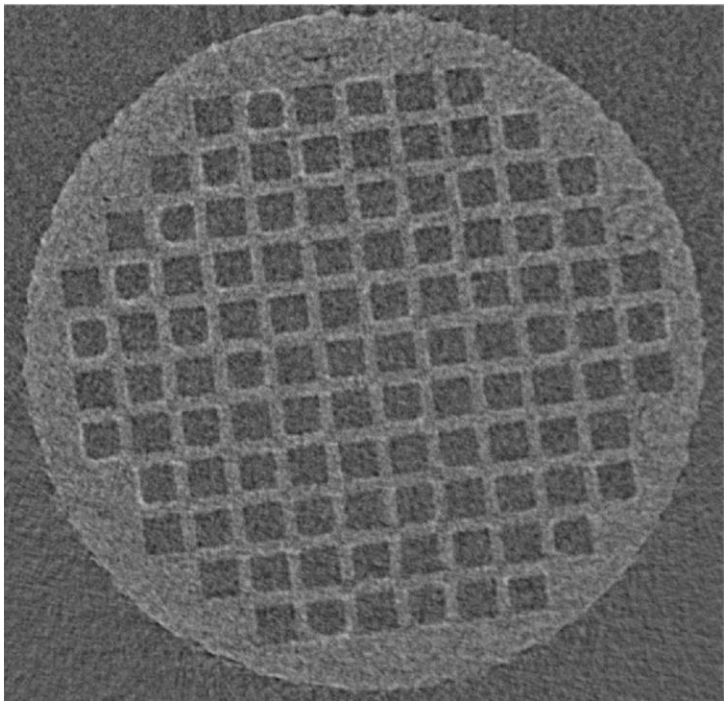
Reconstruct device using neutron computed tomographic techniques

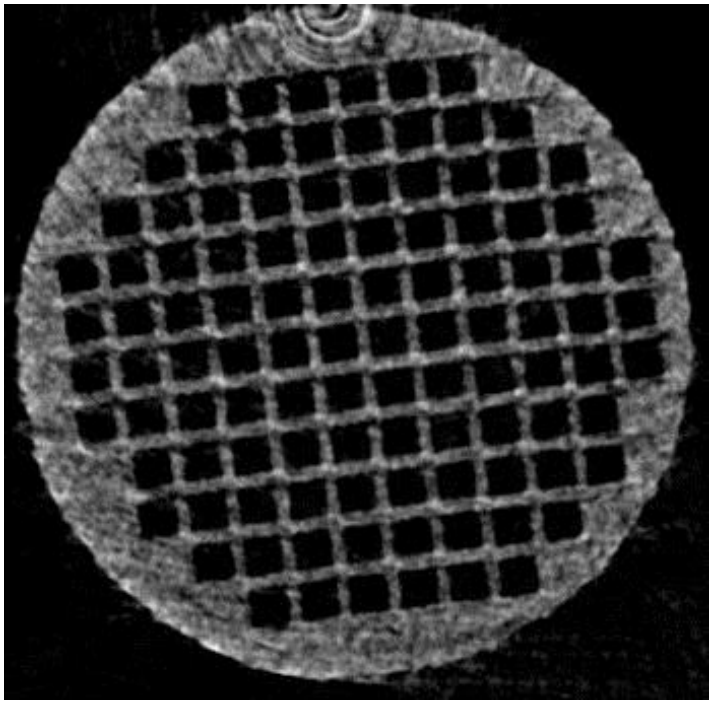


Iterate with engine/bench reactor for sequential study

Establishing high quality neutron images depends on equipment and computational technologies

- Neutrons absorbed by device are combined to build tomographic image
- To improve image quality data is processed to improve contrast
  - Techniques employed: Back-Filtered (Left) vs. Iterative (Right) Projection
  - Back Filtered Projection: Grainy aspect and noise prevent data analysis
- Filtering algorithms still under development





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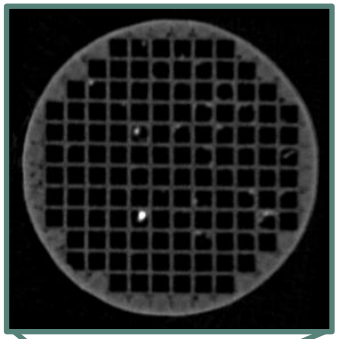
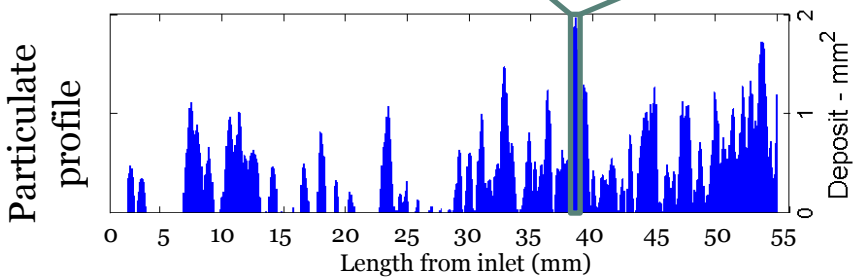
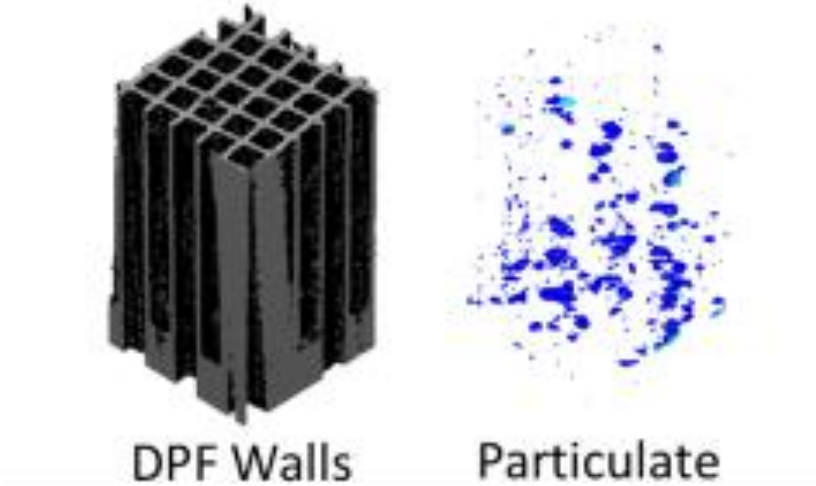
Neutron computed tomography and data analysis employed to show particulate profile in DPFs

Virtual slice at 38 mm in 1"x3" PF





- DPF with unique particulate profile
- Can be quantified to identify location of particulate as function of length or radius
- Can be visually analyzed with either:
  - video reconstruction
  - 3-D image with removable particulate



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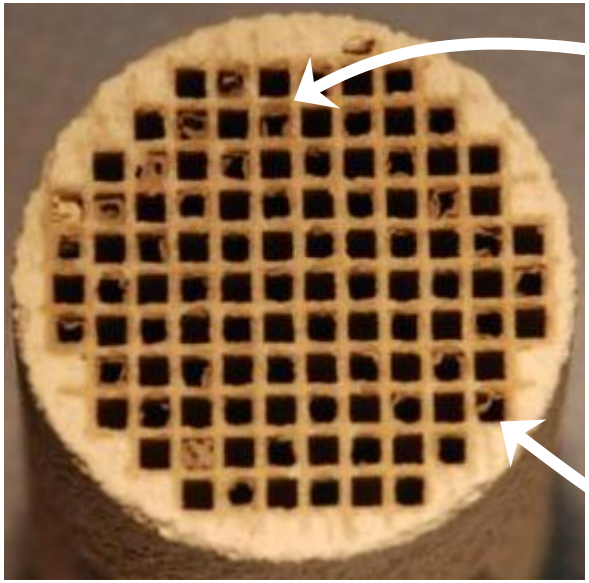
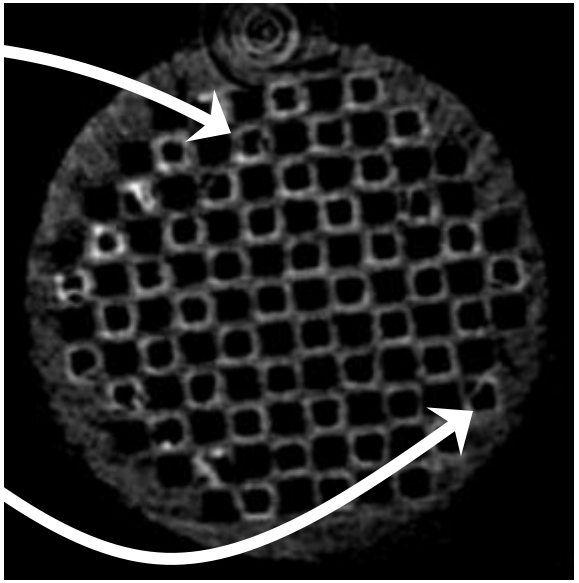
Catalyzed washcoat layer visible in DPFs on outlet channel

- Analysis at one cross-section or a complete reconstruction can provide a cross-section at any point depending on detector resolution
  - ~50 microns currently achievable at ORNL's High Flux Isotope reactor (HFIR)
- Washcoat visible on outlet channels, matches physical cross-section

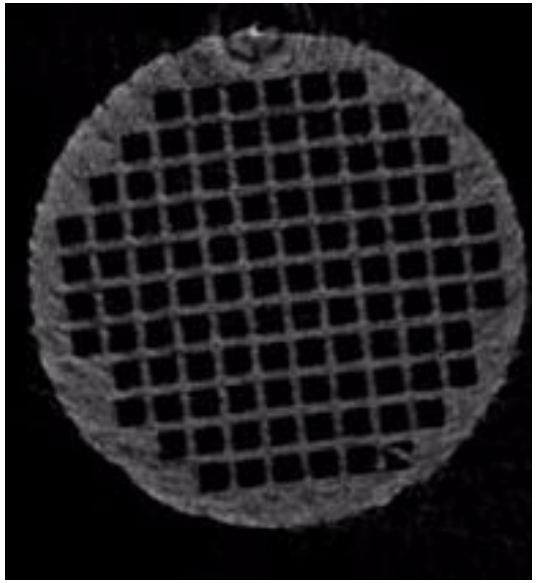
Cross-sectional view of after cutting open DPF



Neutron image "virtual" cross section (not cut)



Catalyzed filter beginning at midpoint going to inlet



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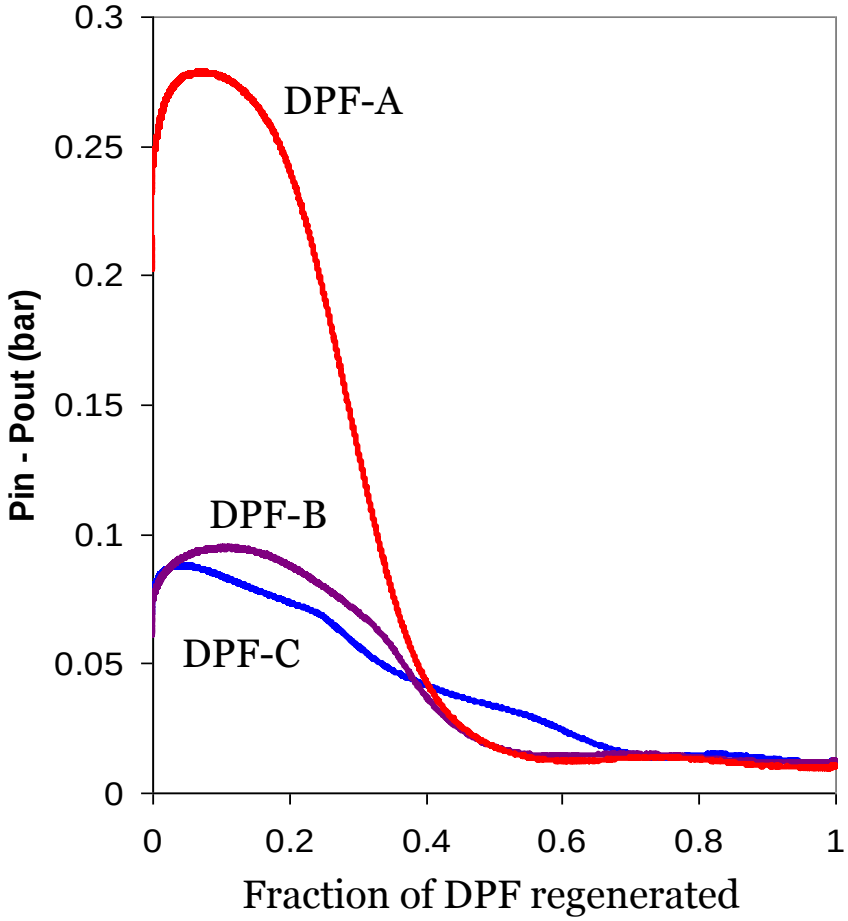
Systematic approach to investigate how particulate profiles change during regeneration

- DPF partial regeneration
  - Pressure drop goes to background levels after only 50% regenerated
  - Where is the soot being regenerated?
  - Are regenerations complete?
- DPFs loaded in collaboration with Navistar
  - Loaded to a total of 3, 5, or 7 g/L
    - used engine exhaust slipstream
- Regenerate to 0%, 20%, 50%, 75% and 100% for neutron imaging
  - **<u>Completed 0% and 20% to date</u>**



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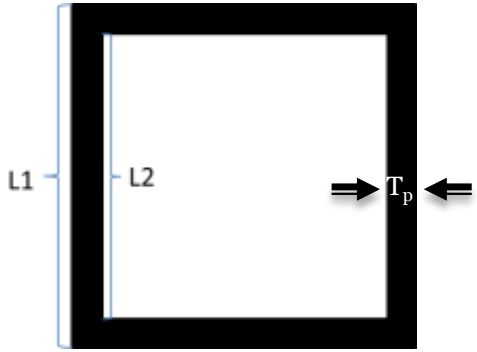
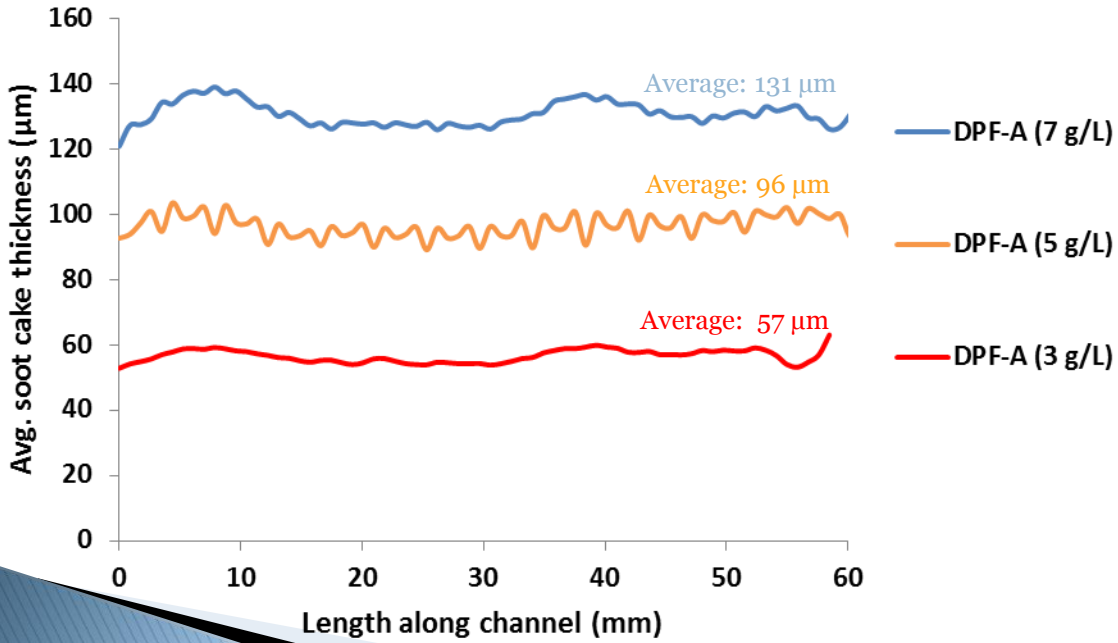
Initial soot loading profiles quantified with image analysis; illustrate soot cake growth

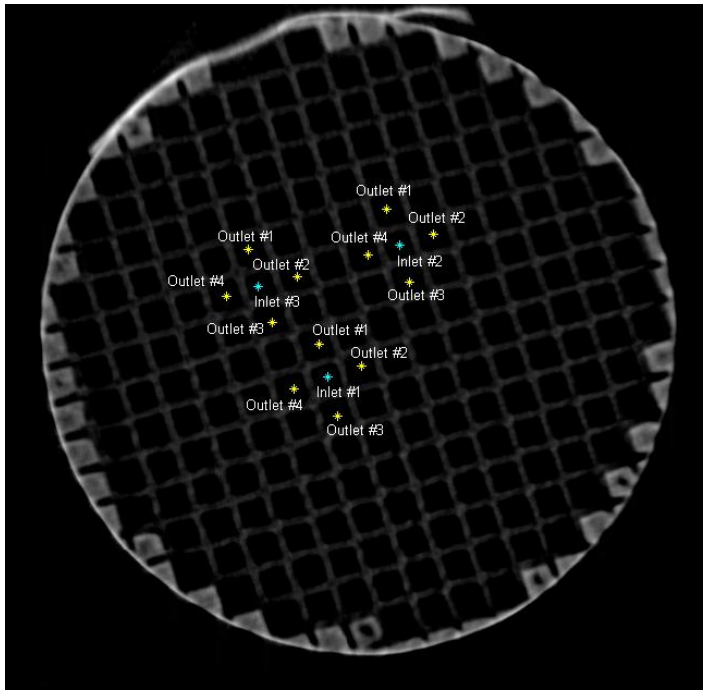
- Particulate was difficult to distinguish from wall
  - However, inlet channels definitely have smaller pore openings than outlet channels
- Employ inlet versus outlet calculation routine
  - Does not take into account cake densities
- Sequential loading clearly identified in filters
  - Relatively even distribution during loading



A_{outlet} = *L1 x L1 (open channel area)*
A_{inlet} = *L2 x L2 (filled channel area)*

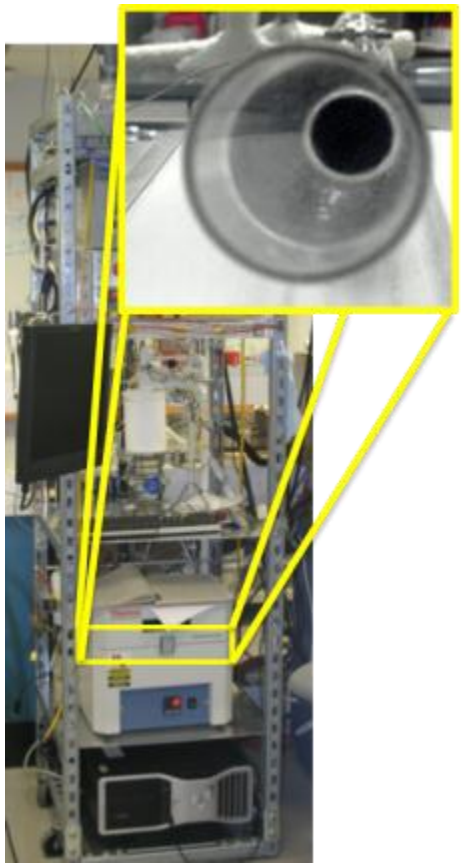
Particulate layer thickness: $(T_P) = (L1-L2)/2$

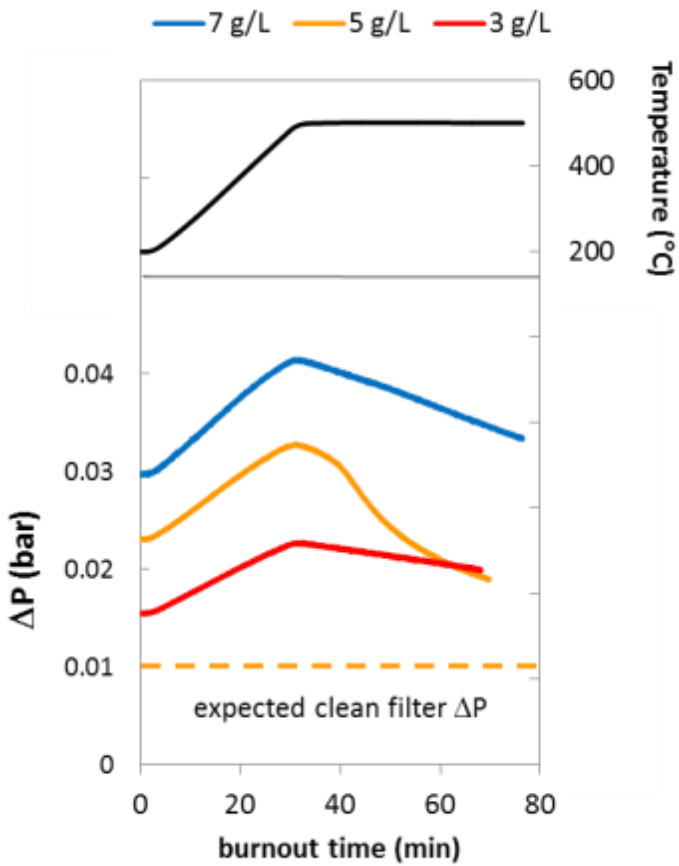




PF Regeneration sequence

- Regeneration in feedback controlled bench reactor
  - Flow and temperature coordinated with FTIR and integration of CO and CO_2 products





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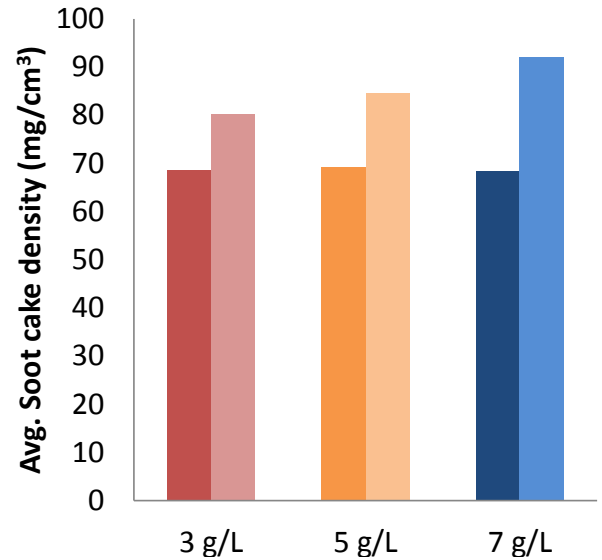
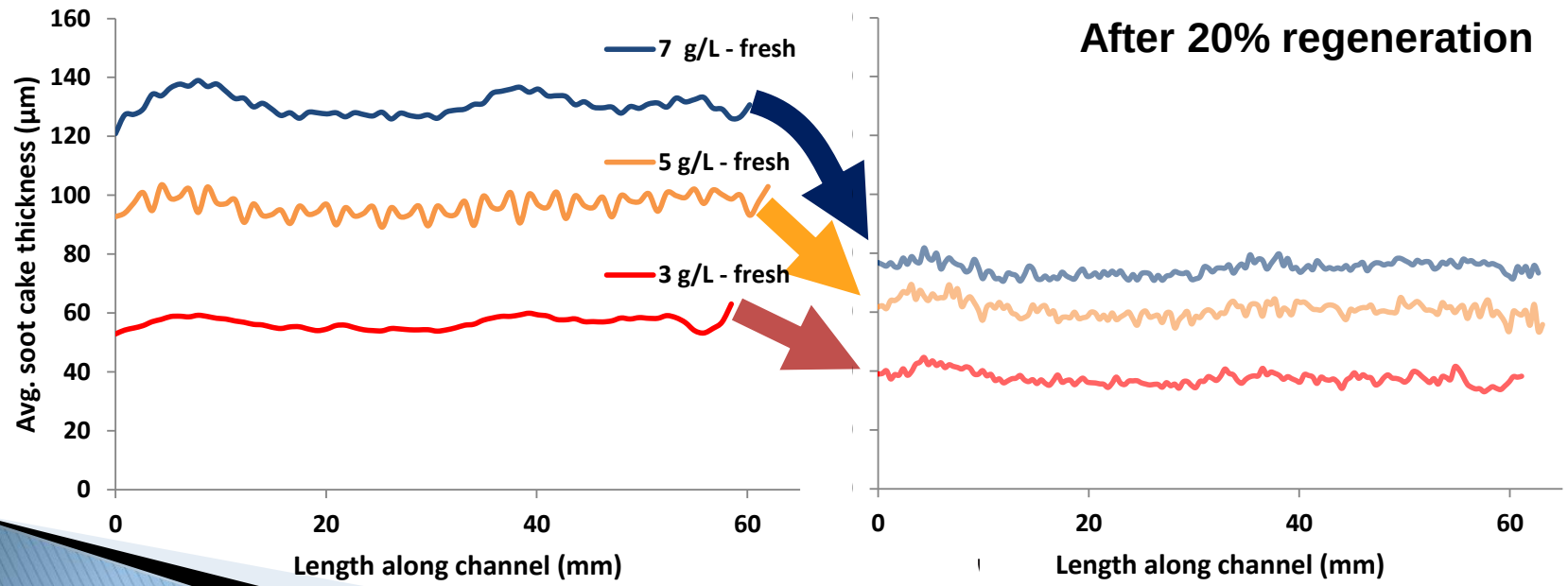
20% regeneration increases average packing density and maintains uniform distribution

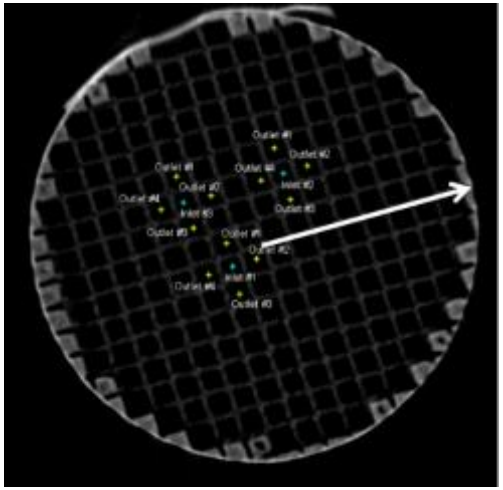
- After 20% regeneration soot cake density increases 15-25%
  - Increases more for higher loading
- Distribution profiles not significantly affected

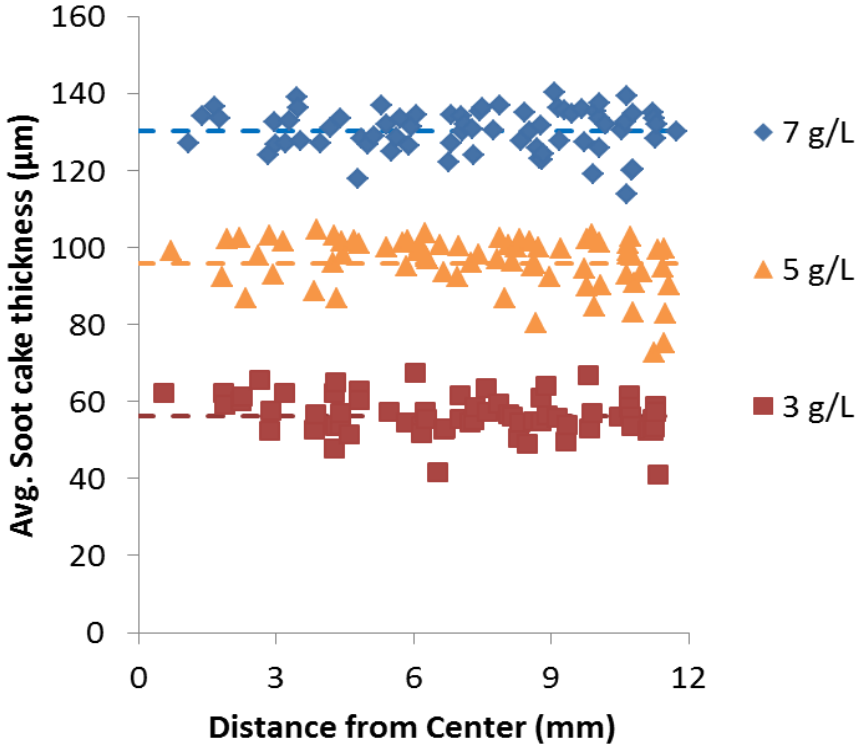


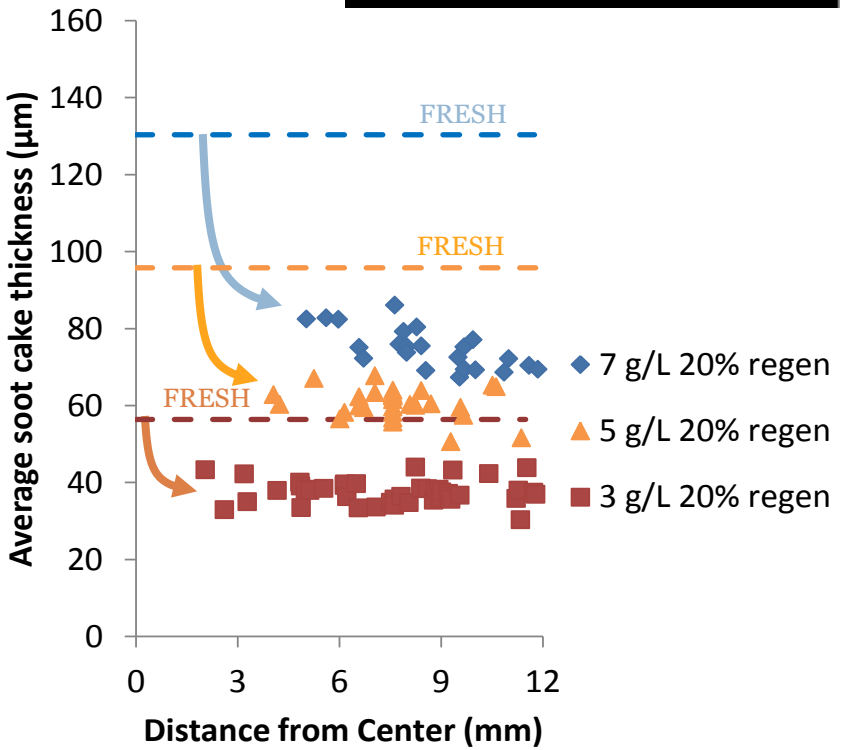


Particulate filter loading also analyzed as function of radius

- Each data point below represents one inlet channel
- For fresh PFs studied, the radial variation was not significant
  - Increased variability was observed near wall
- After 20% regeneration, small slope observed with 7 g/L
  - Other profiles maintain flat distribution



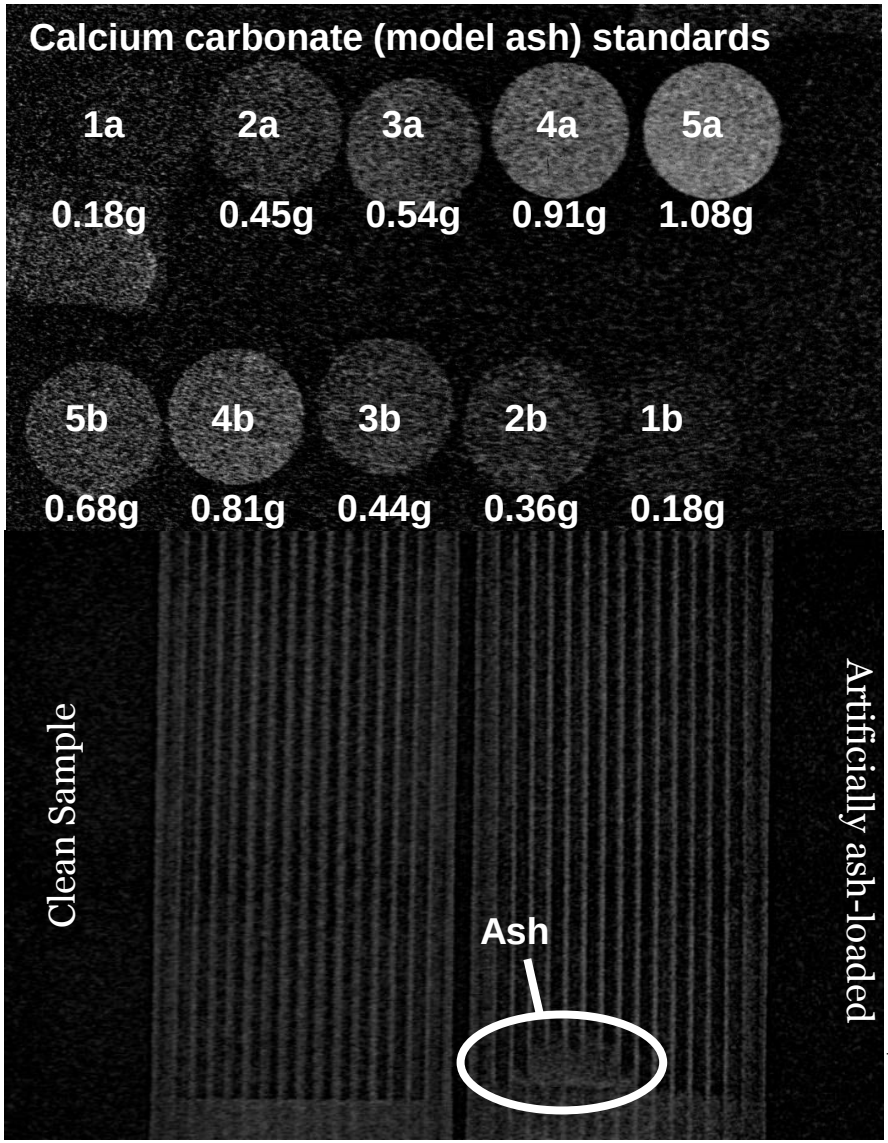




Distinguishing Ca-based model ash is difficult but similar approach as in soot loaded samples can be applied

- Model ash (calcium carbonate) standards
  - pressed into disks
  - two densities studied
    - a: 507 g/cm^3
    - b: 370 g/cm^3
  - Model ash approach by NGK*
  - Illustrates calibration potential

- Ash artificially loaded in portion of DPF
  - difficult to distinguish from sample
- Ca adequately simulates exhaust ash, BUT has poor Neutron absorbtivity
  - Visualization relies on water adsorption
- Similar approach can be applied as in the soot study
- Best approach is to have clean PF for at start of iterative study



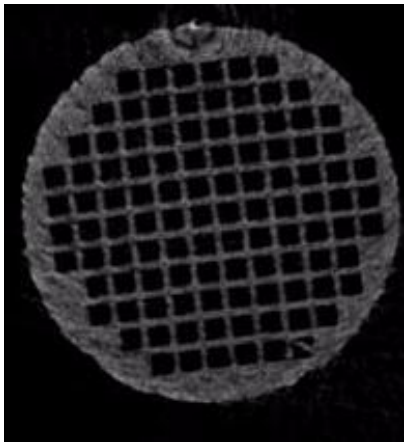
* - S. Fuji and T. Asako, SAE 2010-01-2171

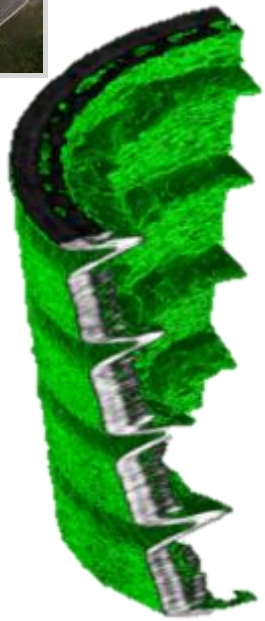
Summary

- Coming…

Summary

- Neutron Imaging enables nondestructive view of entire device
  - Can be used to guide control strategies

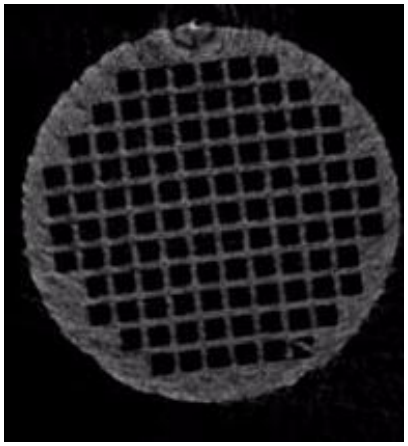


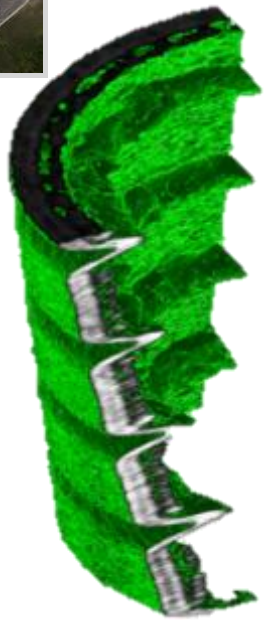


- Neutron imaging efforts underway at ORNL
  - Prototype being developed for use in VENUS at SNS
  - Currently in use at HFIR and being used in conjunction with DPFs, EGR coolers and fuel injectors

Summary

- Neutron Imaging enables nondestructive view of entire device
  - Can be used to guide control strategies





- Neutron imaging efforts underway at ORNL
  - Prototype being developed for use in VENUS at SNS
  - Currently in use at HFIR and being used in conjunction with DPFs, EGR coolers and fuel injectors
  - Still learning what is possible

- Neutron Imaging is

Summary

- Improved visualization tools to enable separation of neutron active particulate from filter walls
  - Illustrated dense particulate pattern could be independently visualized from PF wall

- Identified particulate depth as a function of length, radius, and particulate loading
  - Particulate filters filled to 3, 5, and 7 g/L
  - Imaged with neutrons to identify particulate profile
  - Investigating during partial regenerations

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Summary

- Relevance:
  - Non-destructive, non-invasive analysis to improve understanding of lean-burn vehicle systems targeting fuel economy improvements and durability
- Approach:
  - Neutron Imaging as a unique tool applied to Automotive Research areas to visualize, map and quantify H-rich deposit (soot/ash) in engine parts as well as looking at fuel dynamics inside spray (not achievable with x-rays)
  - DPFs, EGR coolers, Fuel injectors
- Collaborations:
  - BES, Industrial (NGK and Navistar), and Academic (U. Alabama and U. Tennessee)
- Technical Accomplishments:
  - Improved visualization tools to enable separation of particulate from filter walls

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Technical back-up slides

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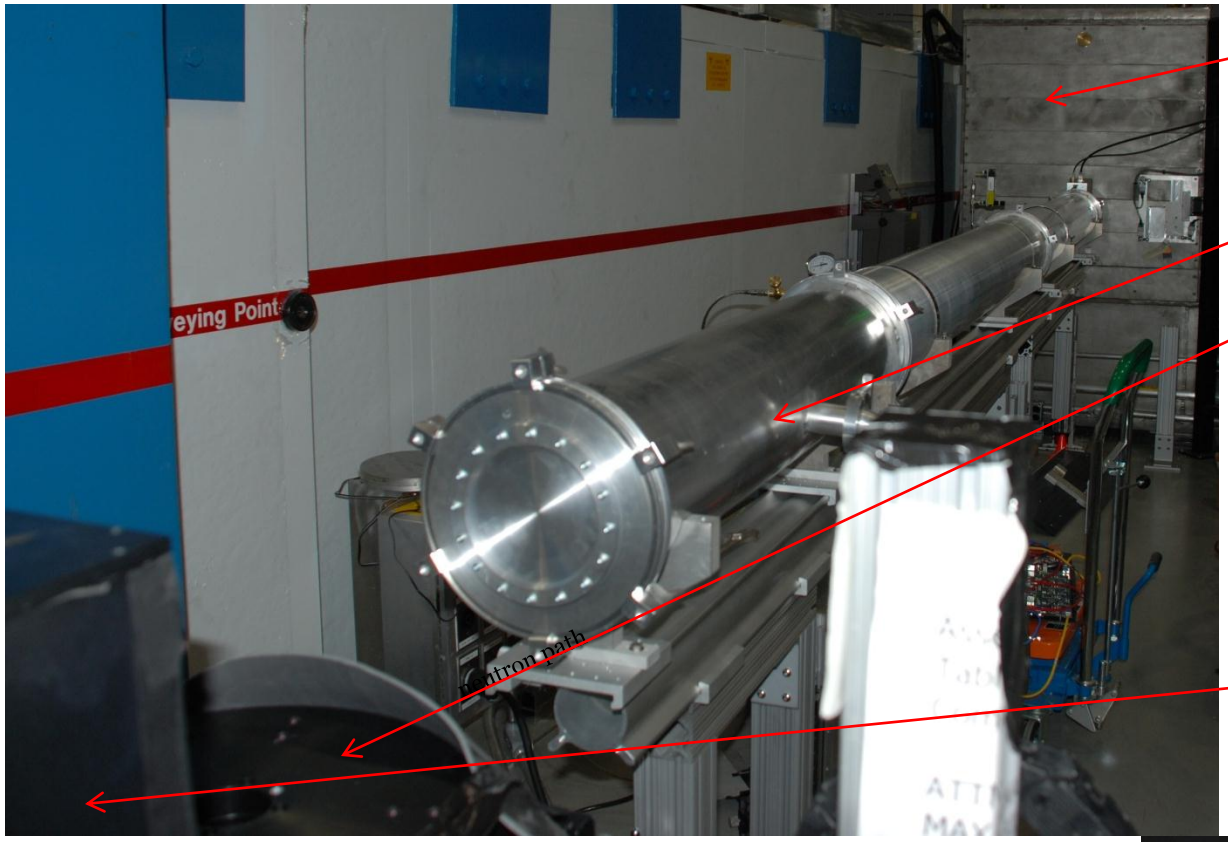


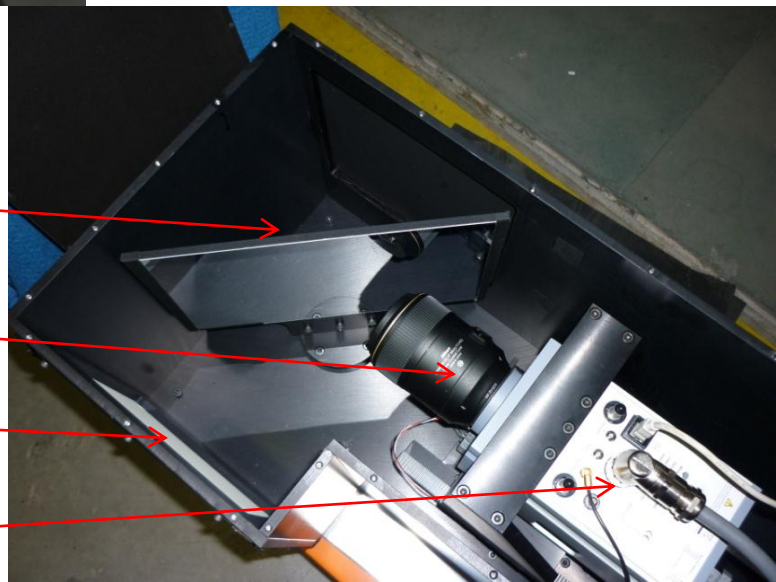
Chopper Box

He-filled Al flight tubes

Sample stage
(translation and rotation for neutron Computed Tomography)

Detector housing
(CCD, lens, mirror and scintillator)

HFIR CG1D beamline
Achievable Resolution:
-50 microns
- $\Delta\lambda/\lambda \sim 10\%$ (in TOF mode)



Mirror

Lens

LiF/ZnS scintillator
(25 to 200 microns thick)

CCD

Presentation Title

- Landscape Format
- Minimum of information on each slide
  - Highlight Main Points
  - Use Photos & Drawings
    - Contrasting Colors
- No Commercialism