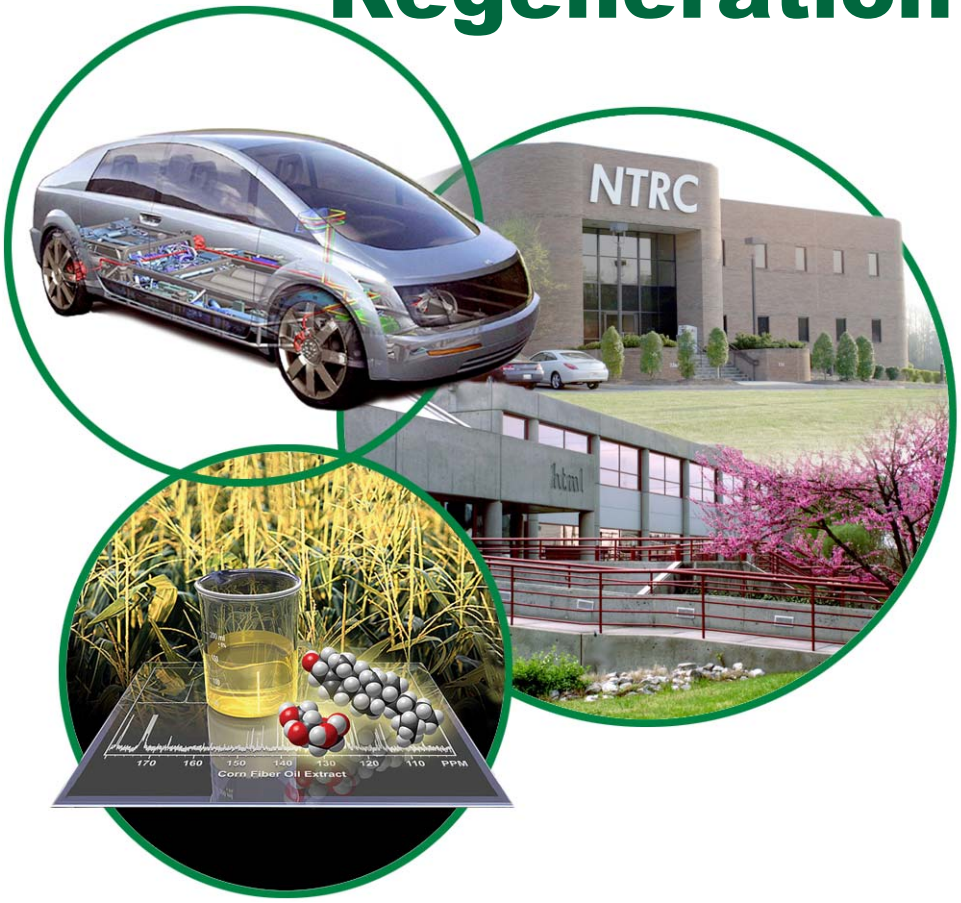
Non-Destructive X-ray Measurement of Soot, Ash, Washcoat and Regeneration Damage for DPFs



Charles E.A. Finney,
Todd J. Toops, C. Stuart Daw

Oak Ridge National Laboratory

Jan Zandhuis

3DX-RAY

Thomas Fox

X-Metrix

DEER

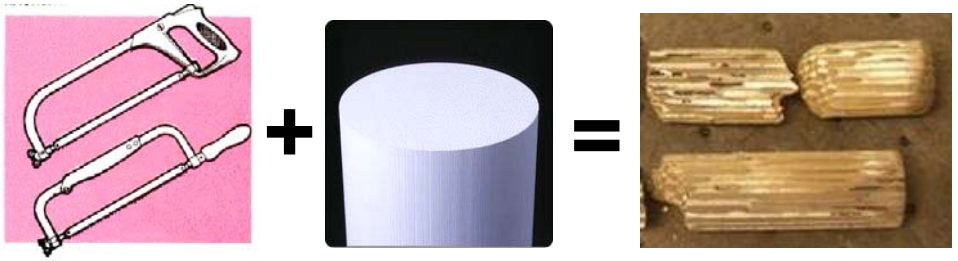
August 7, 2008

Need for non-destructive technique for ash and soot analysis

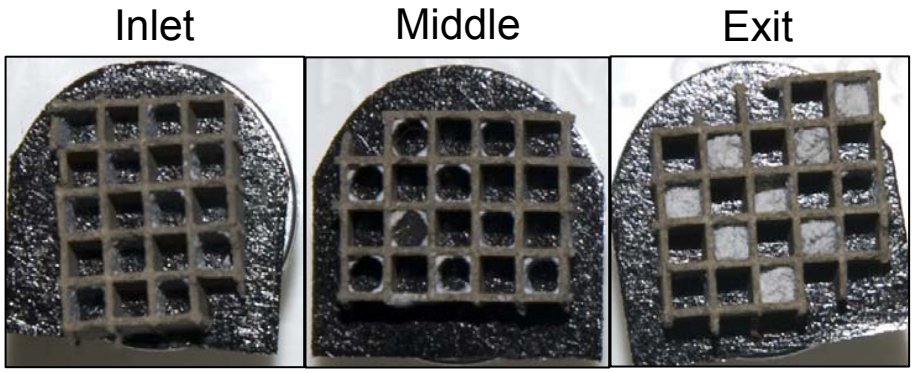
- Typical techniques to determine the soot and ash location require destruction of DPF
  - May disrupt layers
  - Sequential measurements not possible
  - Resolution along the length not possible

Typical EPMA preparation illustrates potential ash/soot layer disruption

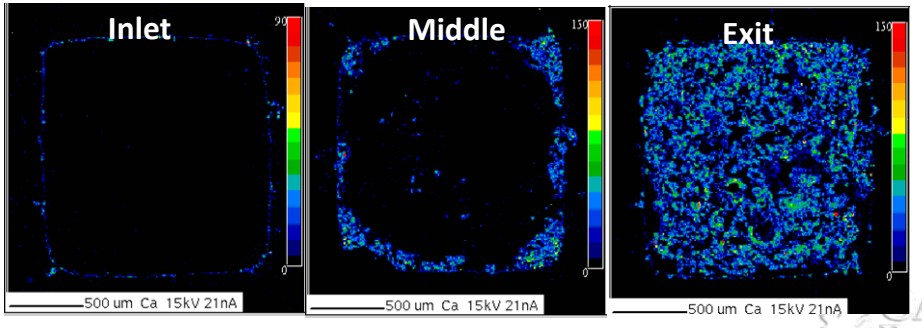
- **First step:**
  - Cut into pieces
  - Possible mechanical disruption



- **Second step:**
  - Fill channels with epoxy
  - Possible disturbance of ash from the walls; flowing



- **Third step:**
  - Electron microprobe
  - Low probability of disruption

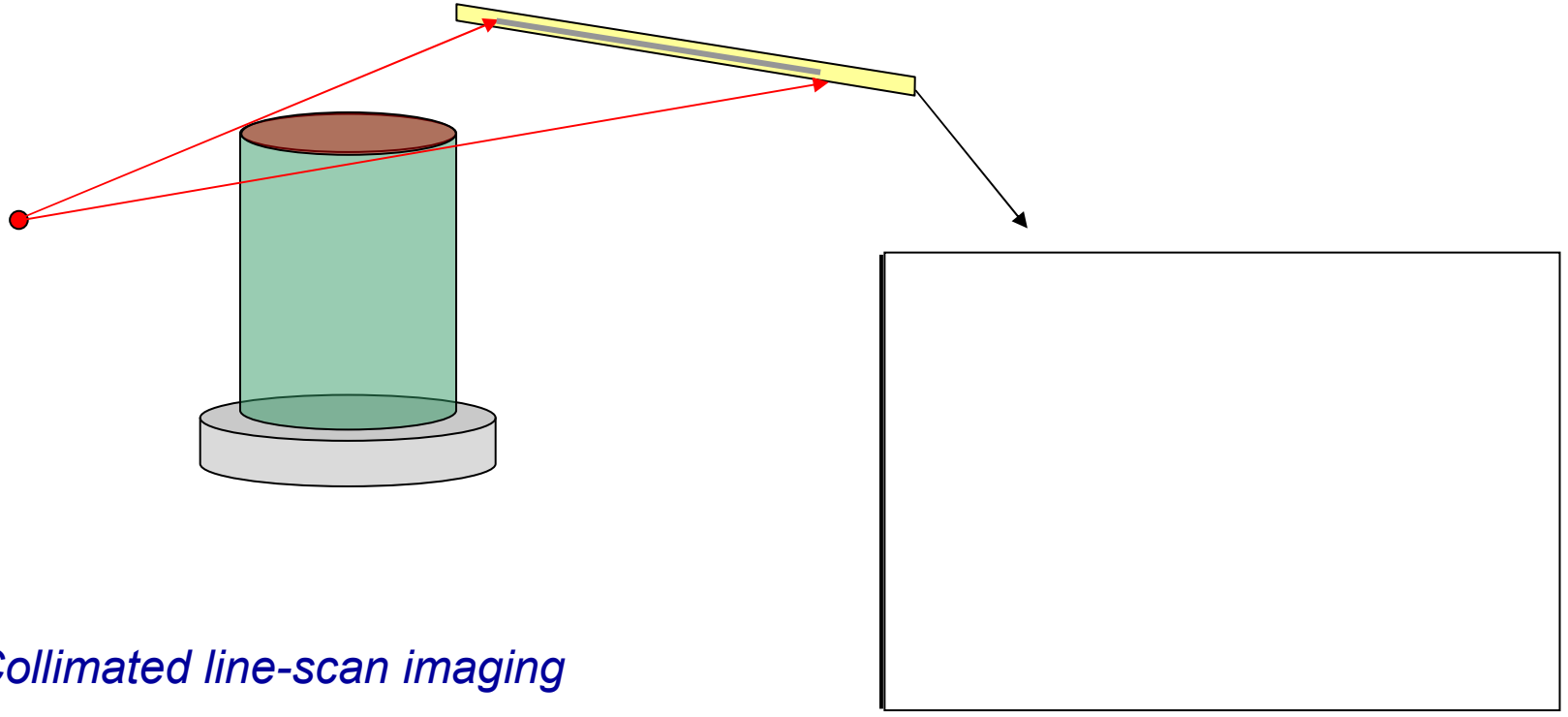


Opportunity to use X-ray instrument at ORNL for short-term DPF analysis

- Loaned from 3D X-ray (Ltd.) for 6 weeks
  - MDXi 400
    - Linear low noise detector
    - 12 bit, 4096 gray scale levels
    - Control over sample height
    - Control over sample angle
    - Batch file capability
  - Evolved from airport security screening devices
  - Scan can be recorded in 15s

- Can it be used to detect ash and soot distributions in a DPF?

Basics of X-ray imaging



Collimated line-scan imaging

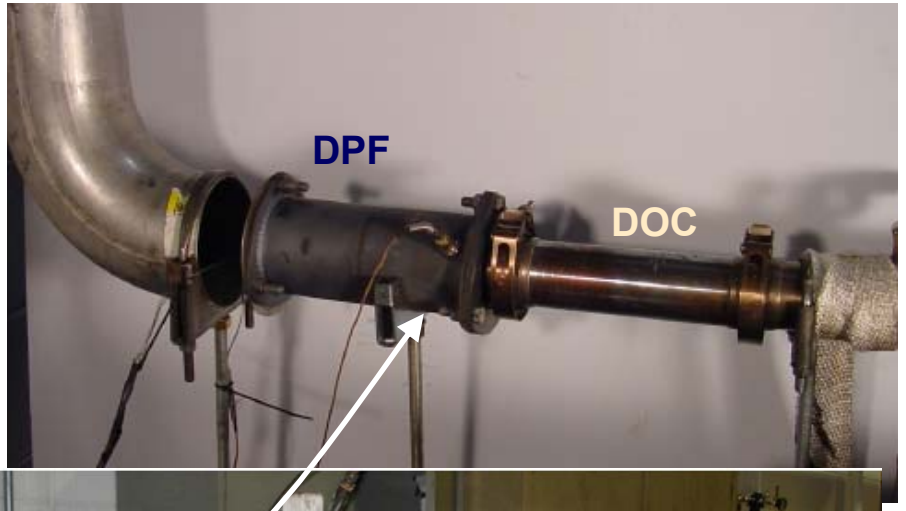
Goals of analysis

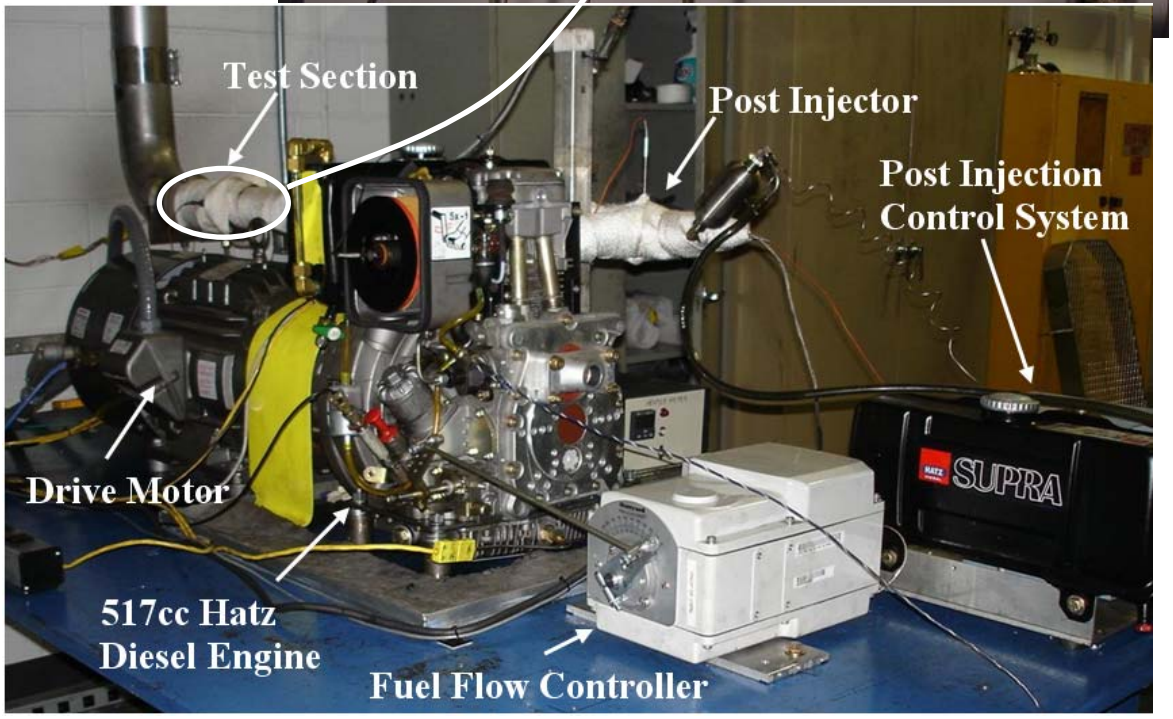
- Measure and corroborate ash distributions from rapid ash loading projects
- Measure soot distribution
  - In-can soot distribution from engine
  - Quantification and calibration with simulated soot loading
- Measure thermal damage in DPFs
  - Purposefully damaged

Ash Loading and Distribution

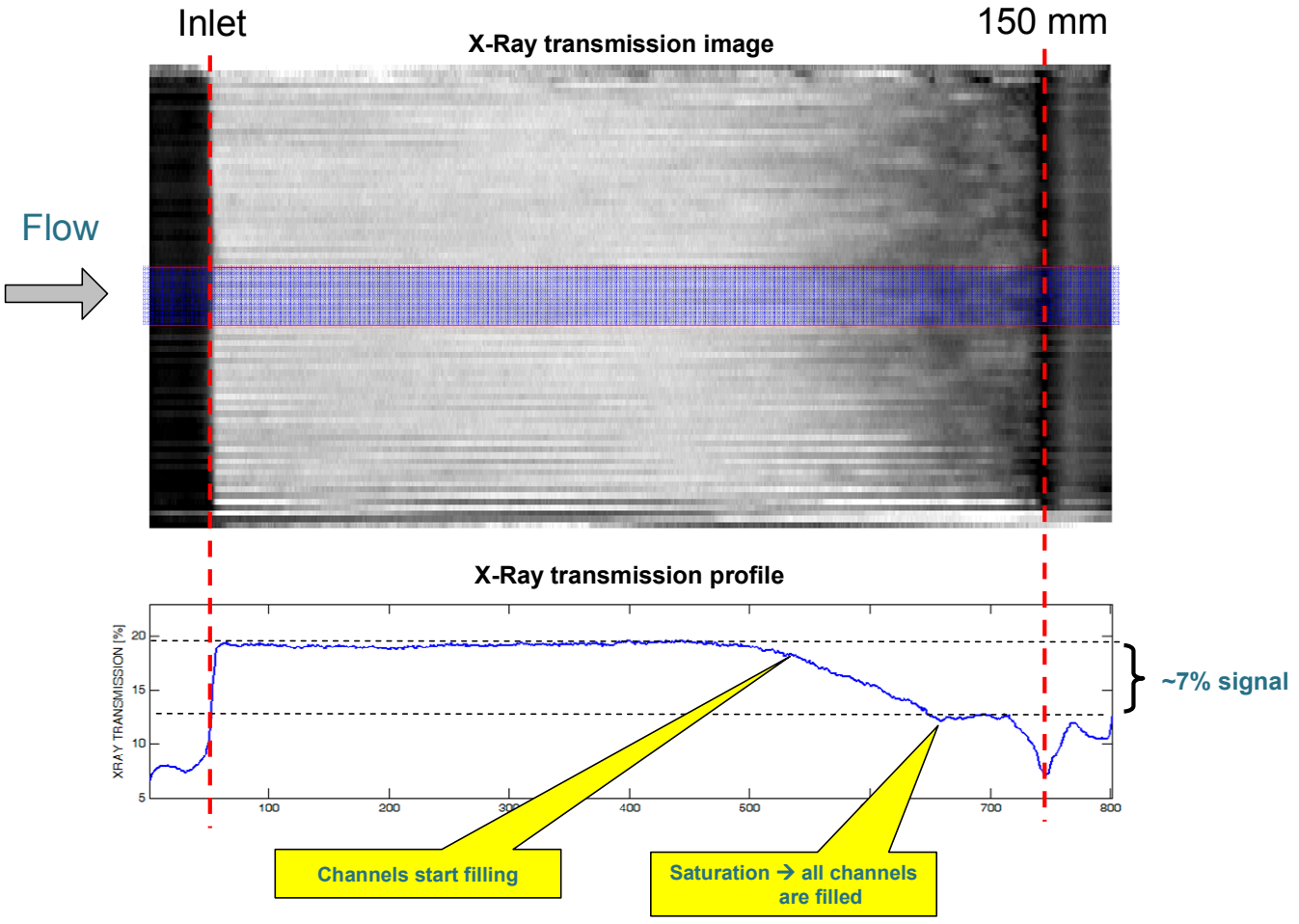
Experimental setup for accelerated ash loading at ORNL (ash part 1)

- 517cc Hatz Engine is operated at 1500 RPM continuously
- 5% lube oil mixed with ULSD fuel
- 700cc DPF
- Active regeneration





Ash deposits observed in rear section



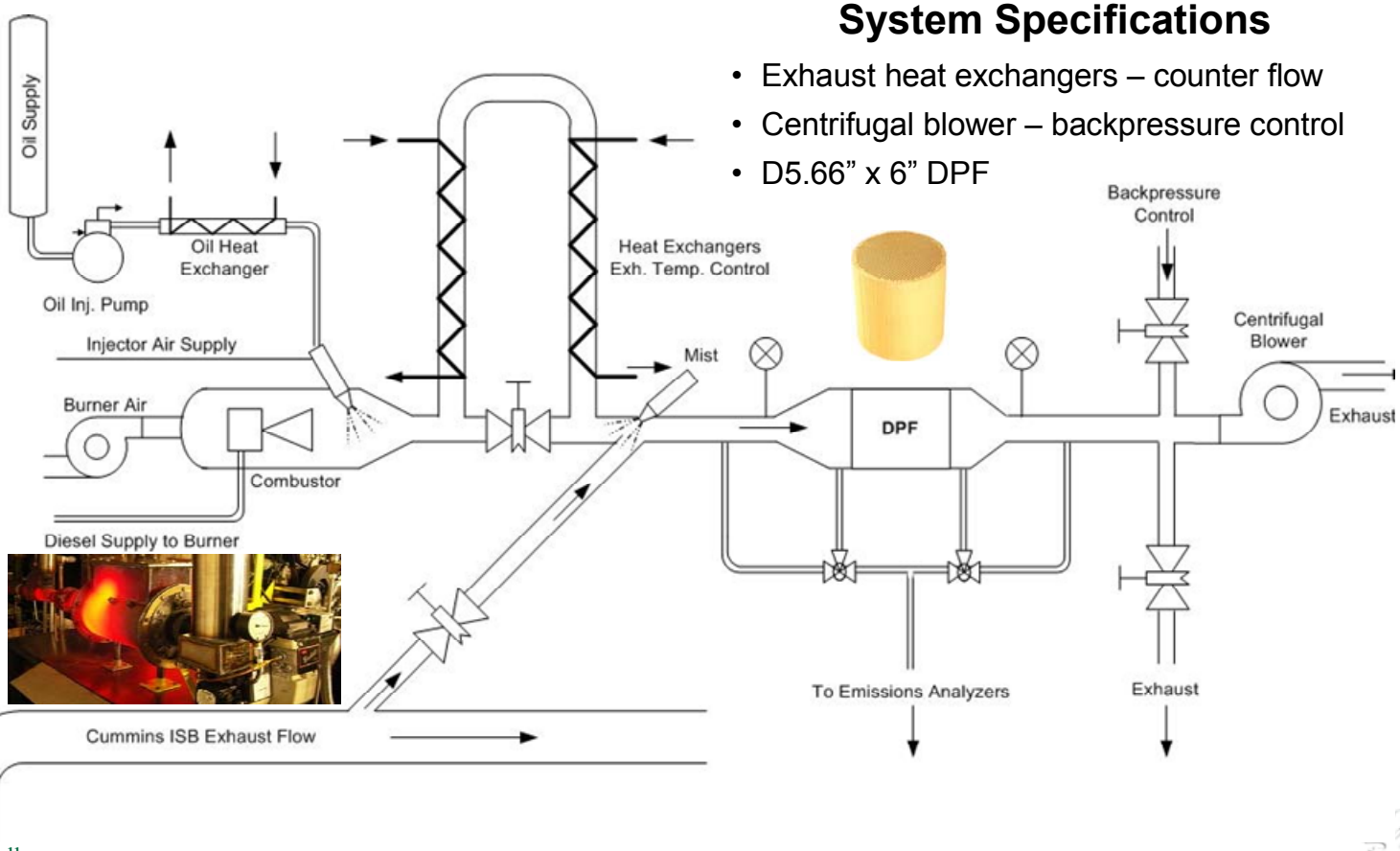
- **Shows good correlation to EPMA results**

Accelerated ash loading at MIT (ash part 2)

- **Sloan Automotive Laboratory accelerated ash loading system (Sappok *et al.* – DEER 2008)**
- **DPF received in two halves**
  - **One with ash loaded, one unused**

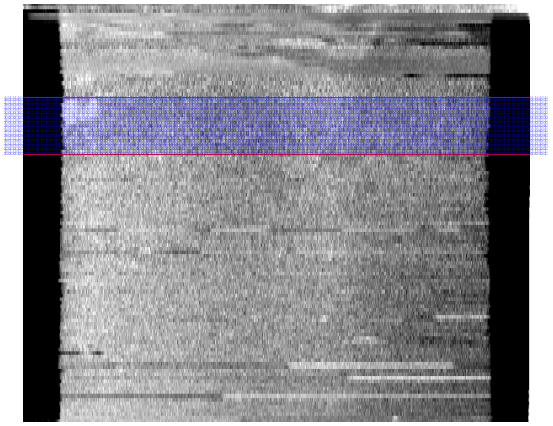
System Specifications

- Exhaust heat exchangers – counter flow
- Centrifugal blower – backpressure control
- D5.66” x 6” DPF

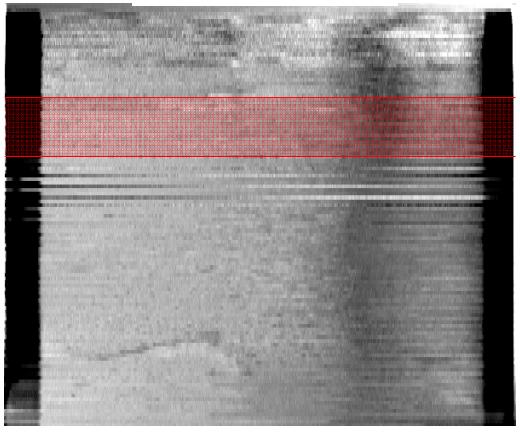


Ash in segmented DPF

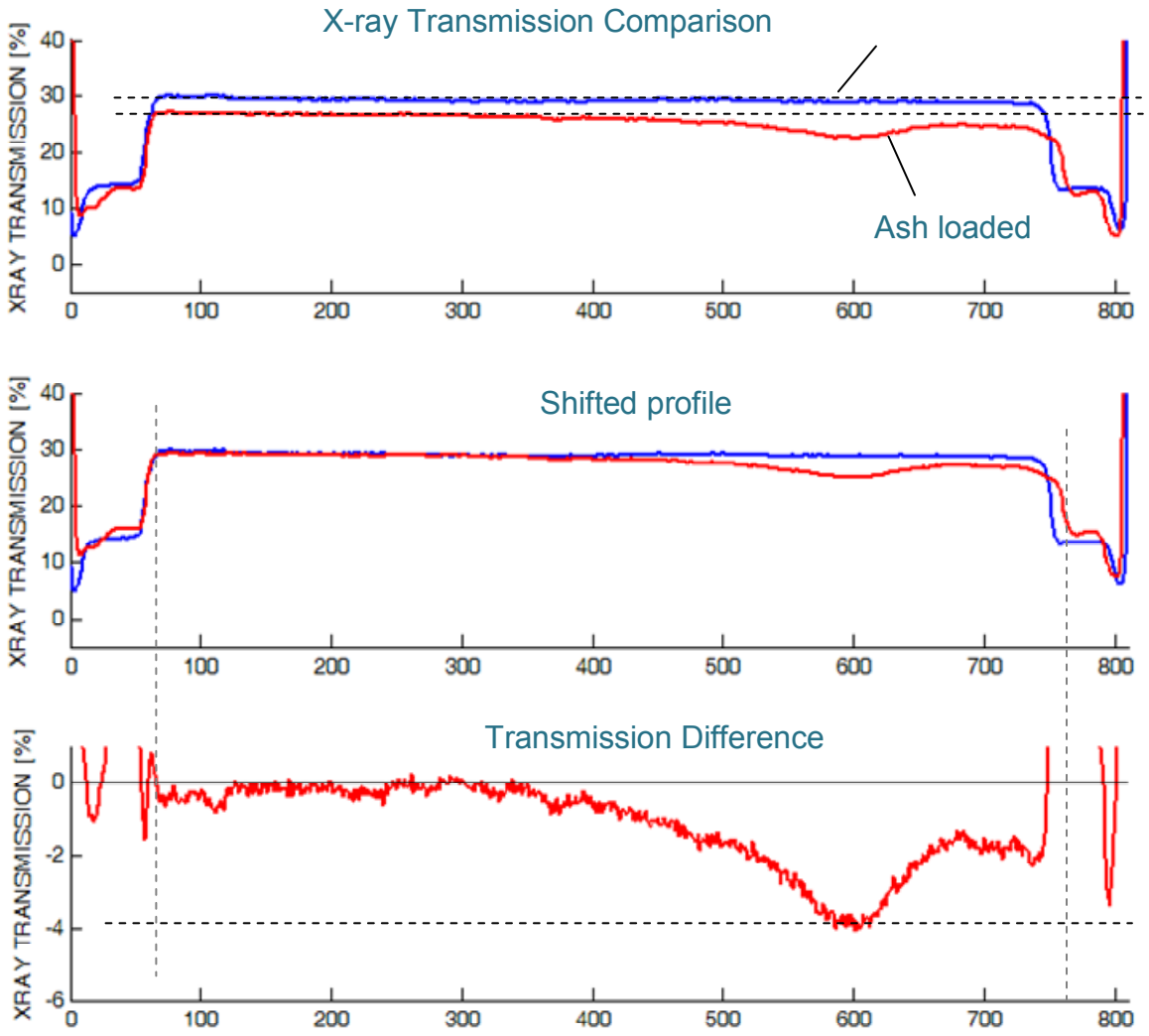
Clean



Ash loaded



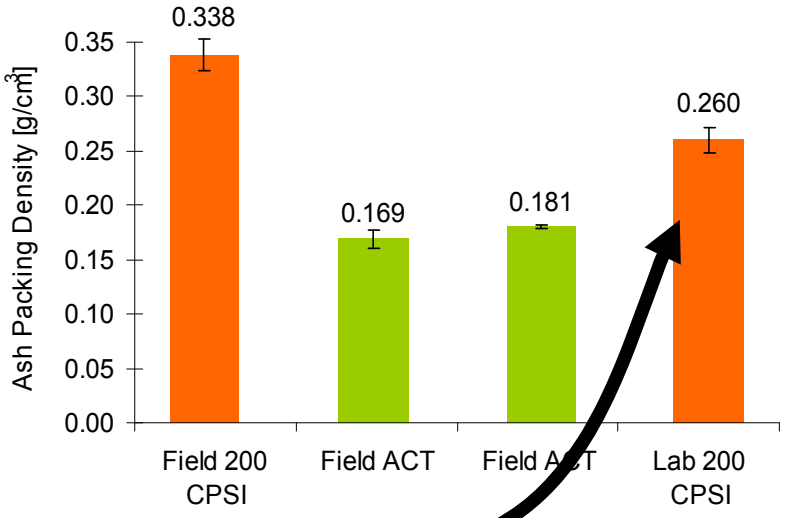
FLOW

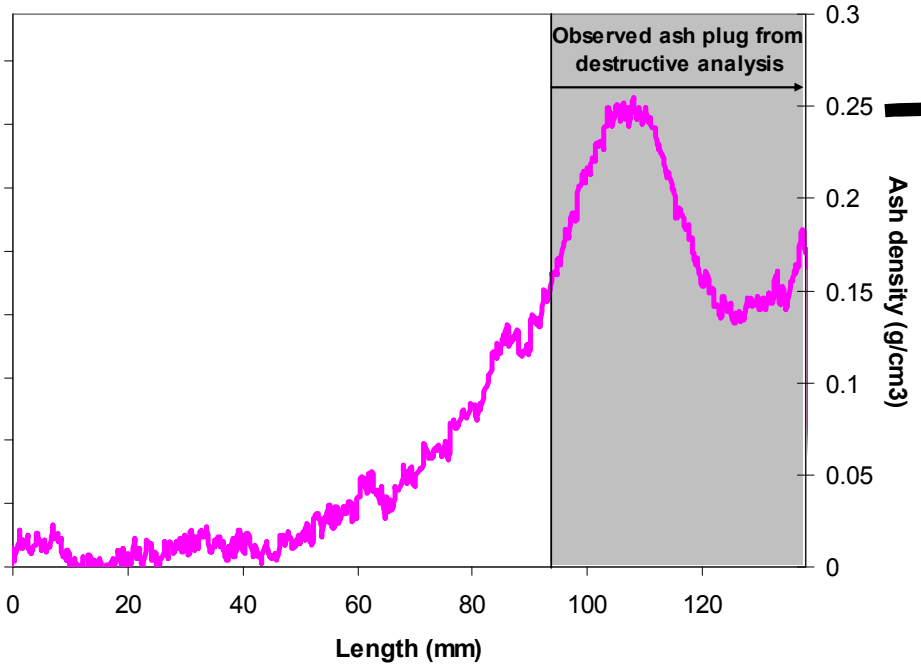


- **X-ray confirms ash plugs ~4 cm from end**

Quantification of ash loading determine ash density and specific loading profile

- Area under curve correlates to ash mass
- Local packing densities can be calculated

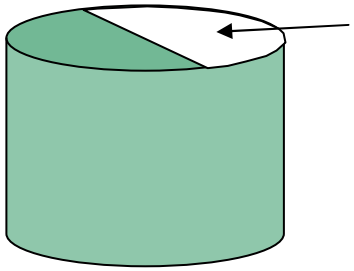




- X-ray corroborates destructive analysis
  - density measurement
  - approximate plug length
- Unusual packing distribution not detected during destructive analysis
  - Difficult to do through lab measurements

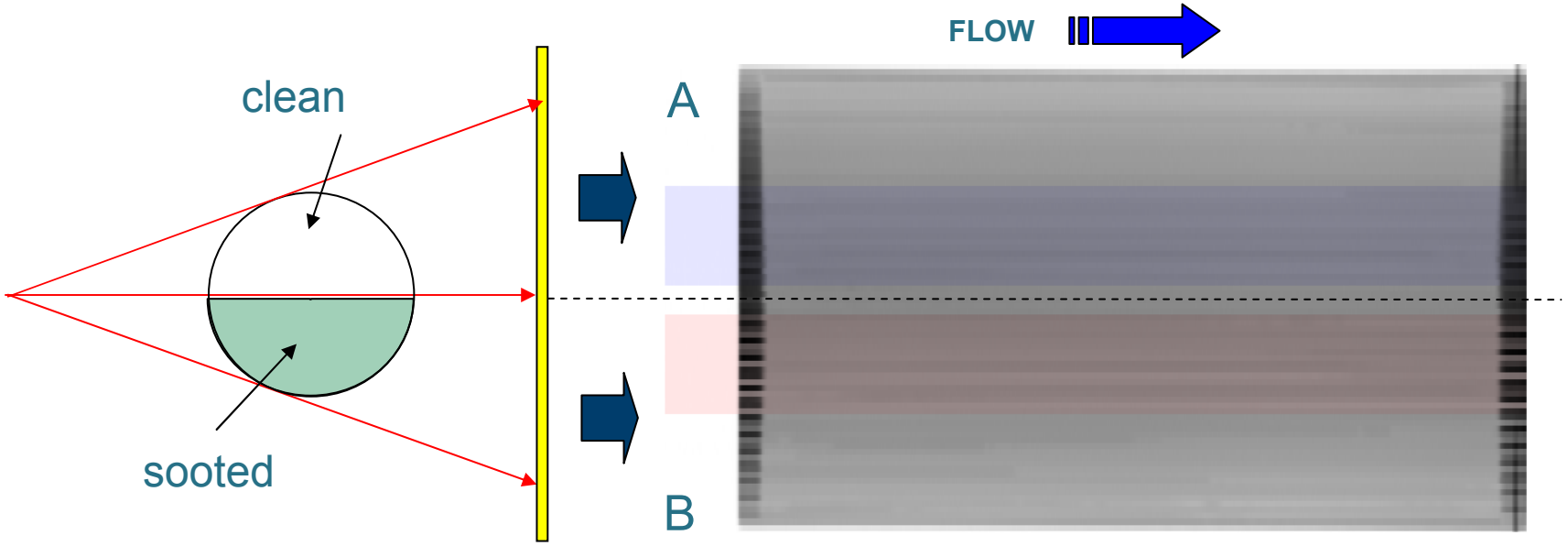
Soot Loading Studies

Controlled soot loading for calibration and quantification (part 1: laboratory soot loading)

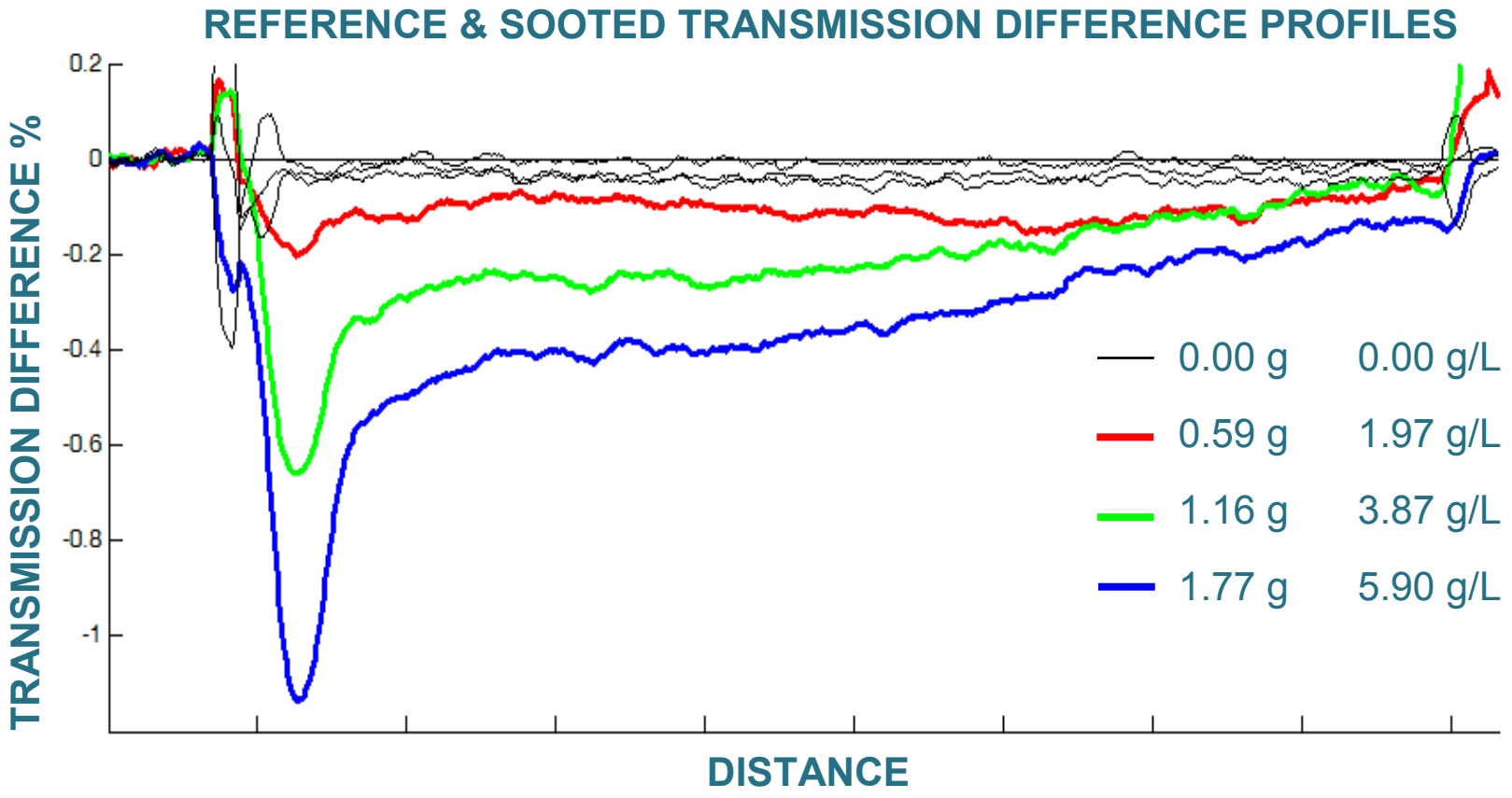


- 75mm (0.6L) uncatalyzed Cordierite
- Artificially vacuum loaded
- One half sooted (0.3L), other half clean (masked off)

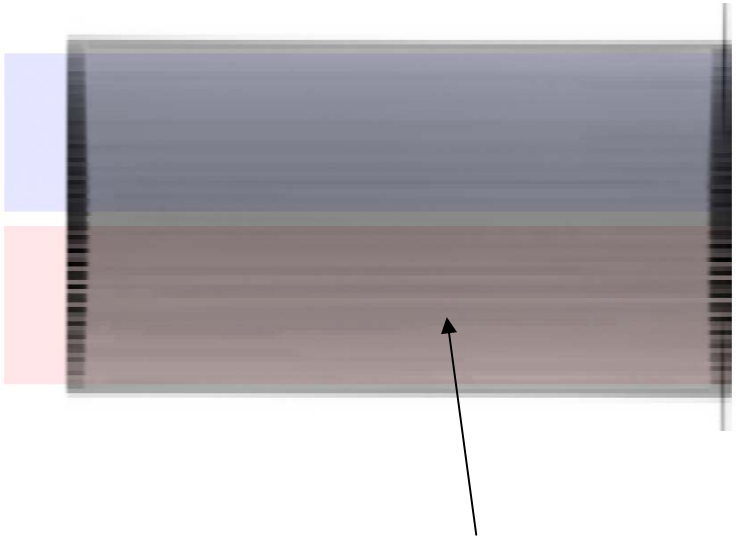
	Added Weight	Loading
0	reference	0.00 g/L
1	0.59g	1.97g/L
2	1.16g	3.87g/L
3	1.77g	5.90g/L



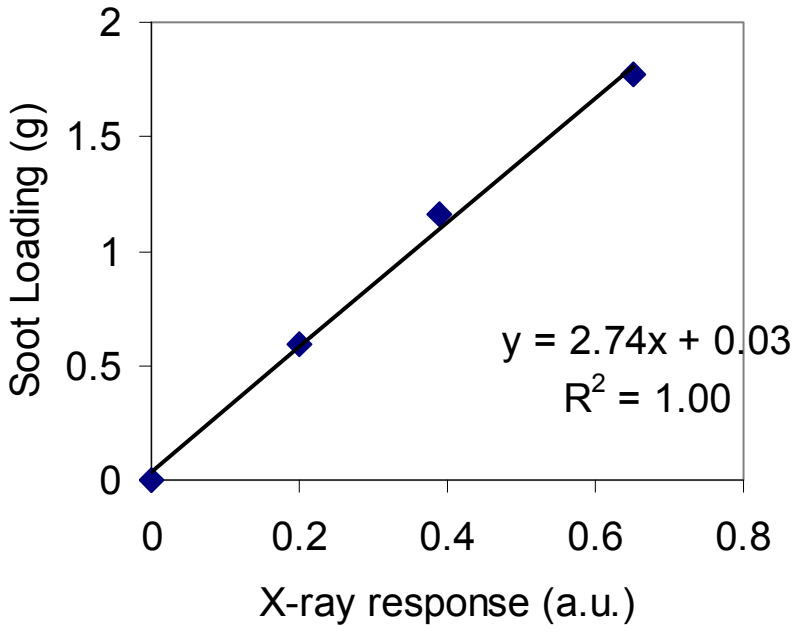
Lab-loaded DPF shows gradual decrease in X-ray transmission with increasing soot



X-ray response shows good correlation to soot mass in lab-loaded DPF



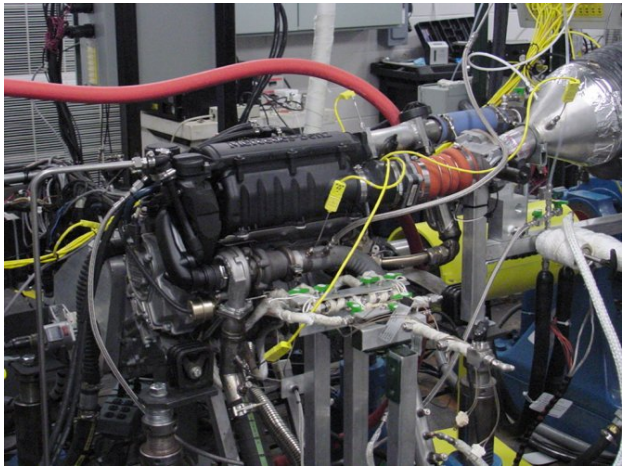
X-ray measurement of soot mass by accumulating difference signal across sooted section

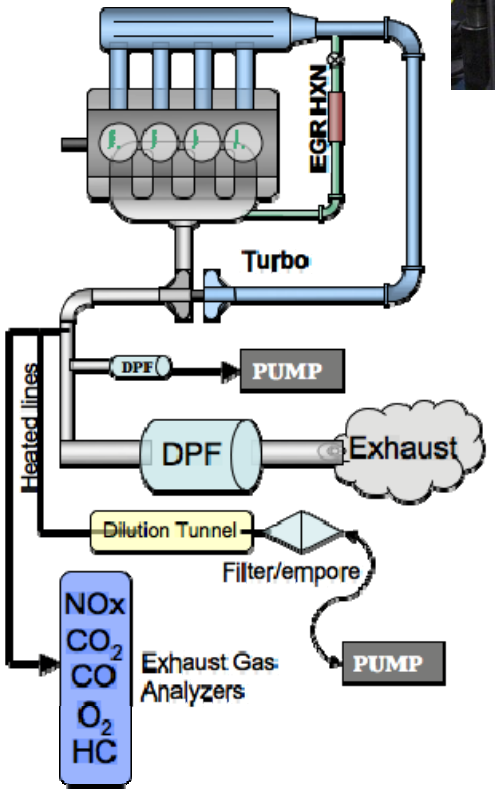


- **Linear regression shows good correlation to soot mass**
- **Suggests detection limits of ~0.1 g**

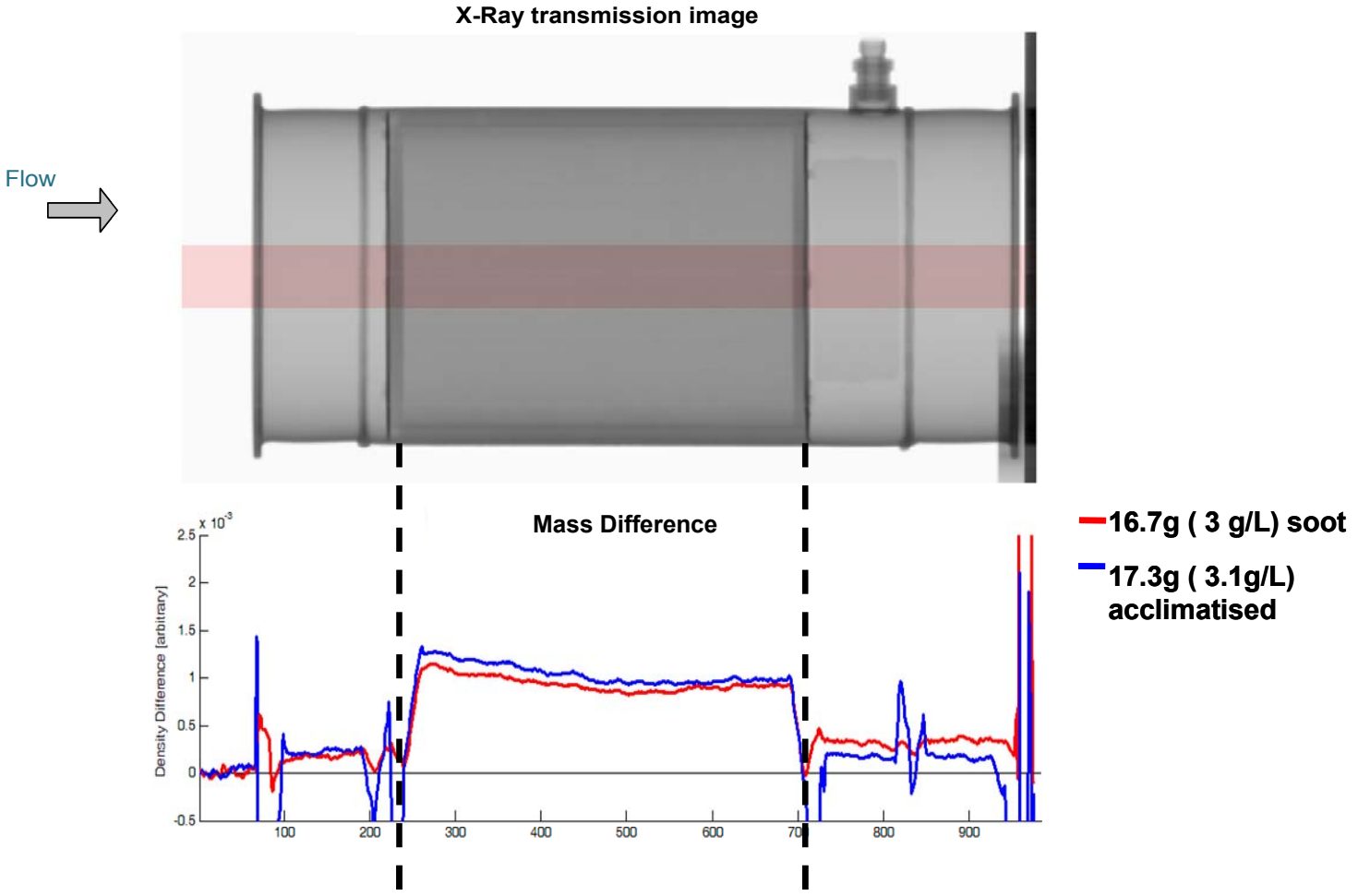
Soot loading conditions (part 2 in-can)

- DPF was filled using exhaust from Mercedes 1.7 L engine for 5 hours
  - 2300 rpm, 4.2 bar condition with 2007 ULSD
- Cordierite DPF
  - 14 cm x 15 cm
  - 5.6 liters
- 16 grams of soot loaded (2.9 g/L)





Soot measurement in can



- X-ray device can detect soot distribution at loadings of 3 g/L
  - Adsorption of 0.1 g/L H_2O is detectable
- Marginally higher soot accumulated in front section

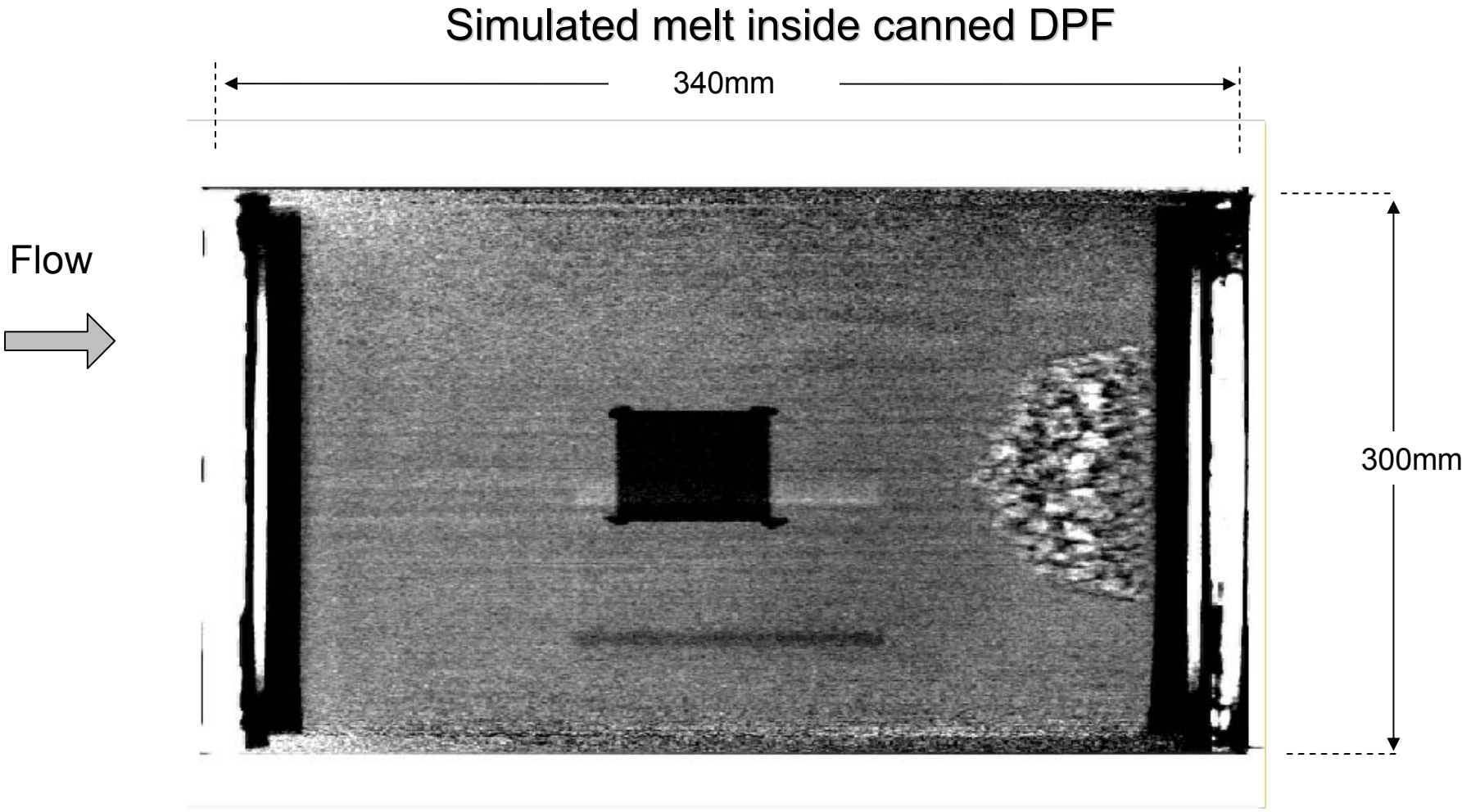
Soot and ash response summary

	Transmission	Signal
Ash	**30-40%**	**4-7%**
Soot	**30-40%**	**~1%**
Soot in can	**~5%**	**~0.1%**

- **X-rays much more sensitive to ash than soot**
- **Ease of measurement may limit absolute resolution**
  - **However, small changes still detectable in can**

Substrate Damage

Thermal damage inside can



Conclusions

- Ash deposits are readily detectable and can be quantified
- Corroborates other analysis techniques with increased detail and without sample destruction
- DPF soot distribution can be measured inside and outside the can
- Soot mass distributions can be measured

- Possible approach that we weren't able to perform:
  - Sequential ash and soot loading to determine the distribution of soot as a function of ash accumulation
  - Direct calibration of soot/ash density and transmission attenuation

Supplemental slides

Contact:

Contact details:

Jan Zandhuis
3DX-Ray Limited
Pera Innovation Park
Melton Mowbray
Leicestershire,LE13 0PB
UK
Tel: +44 (0) 1664 503 600
Email: info@ish.co.uk
URL: www.3dx-ray.com

Dr Thomas J. Fox
President
X-Metrix Inc
(US Distributor for 3DX-Ray Ltd)
2513 Early Court
Virginia Beach, VA 23454
Tel: 757-450-5978
Email: tfox@x-metrix.com
URL: www.x-metrix.com