Nondestructive Measurements & Characterization Sciences Group

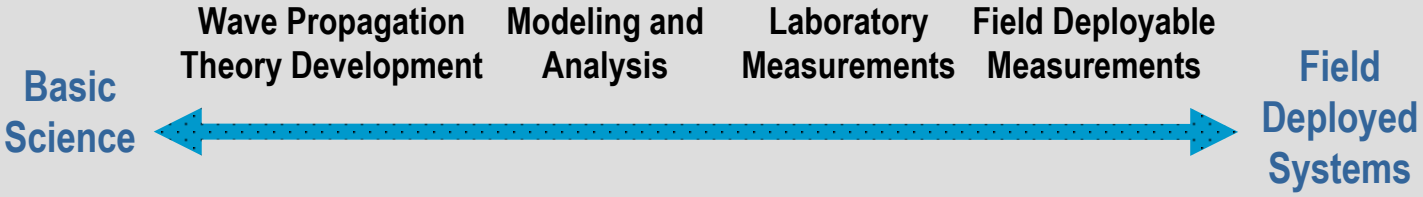
Capabilities Overview

National Security Directorate
Pacific Northwest National Laboratory

Nondestructive Measurement and Characterization Sciences Group

"We develop and apply new NDE technologies to solve unique and difficult problems".

- **Highly Capable Staff and Facilities:**
  - Over 30 highly qualified NDE experts.
  - We invest our resources in cutting-edge equipment and facilities.
- **Teaming on Multiple Levels:**
  - Ability to form multi-disciplinary teams of experts
  - Teaming across PNNL and with other national laboratories, universities, and industrial partners.
- **Broad Scientific Spectrum:**



Unique Features of NDM Group

- **Heavy Science Content (Growing Science Core):**
  - Five (5) new multi-year science focused projects in the past years (EMSP, LDRD, NETL)
  - Capabilities include an ultrasonics theorist
  - Publications have grown to over 100 per year.
- **Continuing History of "*Science to Solutions*":**
  - 4 FLC Awards & 4 R&D 100 Awards
  - 39 Patents and 90 Invention Reports since 1990
  - Strong industrial contacts – chemical, petro-chemical, food, electronics
- **Crossing the Borders of Physical, Chemical and Biological Sciences:**
  - Fundamentally a Physical Science Group
  - Linkages to Chemical Sciences – Sonochemistry, chemical composition analyses
  - Linkages to Biological Sciences – Acoustic microscopy of bio-systems, cell lysis, cell sorting and manipulation, cellular stress studies.

NDM Group Technology Focus

- **Diverse Set of NDE Technologies**
  - Ultrasound
    - Low frequency (~20 kHz - ~1 MHz)
    - Common NDE (~1Mhz- ~25Mhz)
    - High (~50 MHz - ~200 MHz)
  - EM and Eddy Current
  - Infrared
  - Visual
  - Geophysical
- **Diverse Set of Application Areas**
  - Ultrasonic Inspection
  - Ultrasonic Materials Characterization
  - Process Monitoring and Control Applications
  - EM and Eddy Current Inspection and Characterization

Application Area - Ultrasonic Inspection

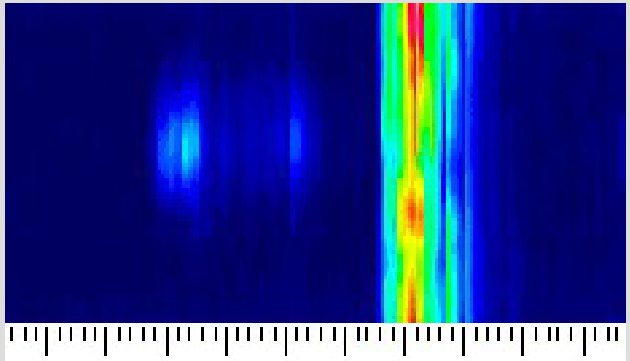
- **Focuses on identifying, locating and characterizing subsurface flaws**

- **Signal and Image Processing and Computer-Aided Data Visualization**

- **Example Projects**
  - International Nuclear Safety Program – DOE (P.I. Tom Taylor)
  - Reliability of Nondestructive Evaluation Techniques – NRC (P.I. Steve Doctor)
  - Synthetic Aperture Focusing Technique – NRC (P.I George Schuster)
  - Double Shell Tank Inspection – DOE (P.I. Alan Pardini)
  - Solid Rocket Motor Inspections (P.I. Leonard Bond)
  - Natural Gas Piping Inspection (P.I. Paul Panetta)

In-service Inspection (ISI) Program Development

- Long standing 25 year program with Nuclear Regulatory Commission (NRC) to develop ISI program requirements for commercial nuclear industry via Codes and Standards



- Reliability by Performance Demonstration
  - Parametric studies (laboratory detection and sizing)
  - Round Robin Studies (field ISI reliability)
- Solving difficult inspection problems
  - Geometric, material selection, and environmental challenges
  - Resulted in development and deployment of new imaging technologies (Synthetic Aperture Focusing Technique) and inspection guidelines

Inservice Inspection Program Development

INPUTS

- Service Conditions & History
- Geometry & Access Restrictions
- Fabrication Method & Examinations
- Material Properties
- Damage Mechanisms

ANALYSIS

Program Development Process to Manage Risk of Component Failure

- **Safety Risk**
- **Economic Risk**
- **Public Perception Risk**

OUTPUTS

- Target Flaw Sizes
- Inspection Techniques
- Inspection Locations
- Inspection Frequencies
- Performance Demonstration

Problem: Support development of a comprehensive program that reduces risks at Soviet-designed nuclear power plants.

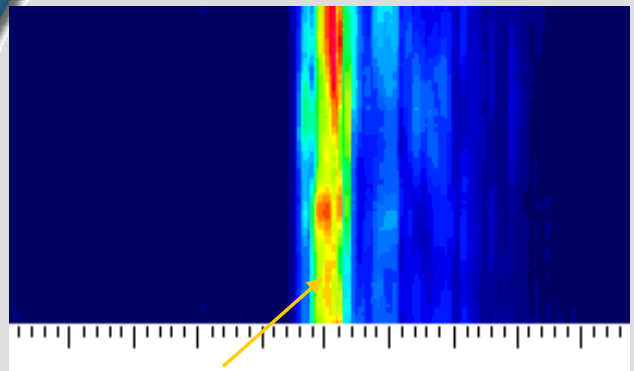
Training on Weld Inspection

Leningrad Nuclear Power Plant

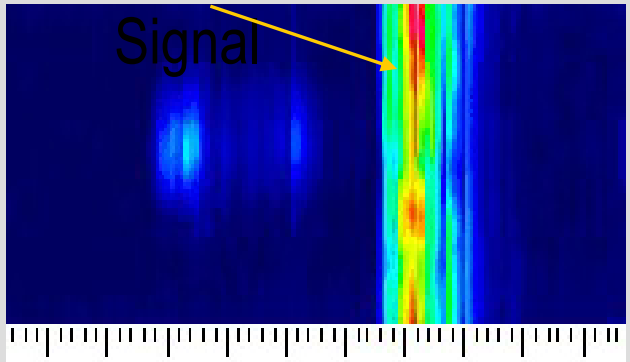
- **Provide Essential Capital Equipment to enable In-service Inspection of Critical Safety Related Components and Systems**
- **Provide Technology Transfer and Training on Proven Western Inspection Procedures**
- **Help Change Countries Certification Practice to meet international standards**
- **Set up Manufacturing Facilities to ensure sustainable infrastructure**

Client: DOE NA-23

Problem: Need an imaging technique capable of high resolution flaw characterization in hard to inspect locations.



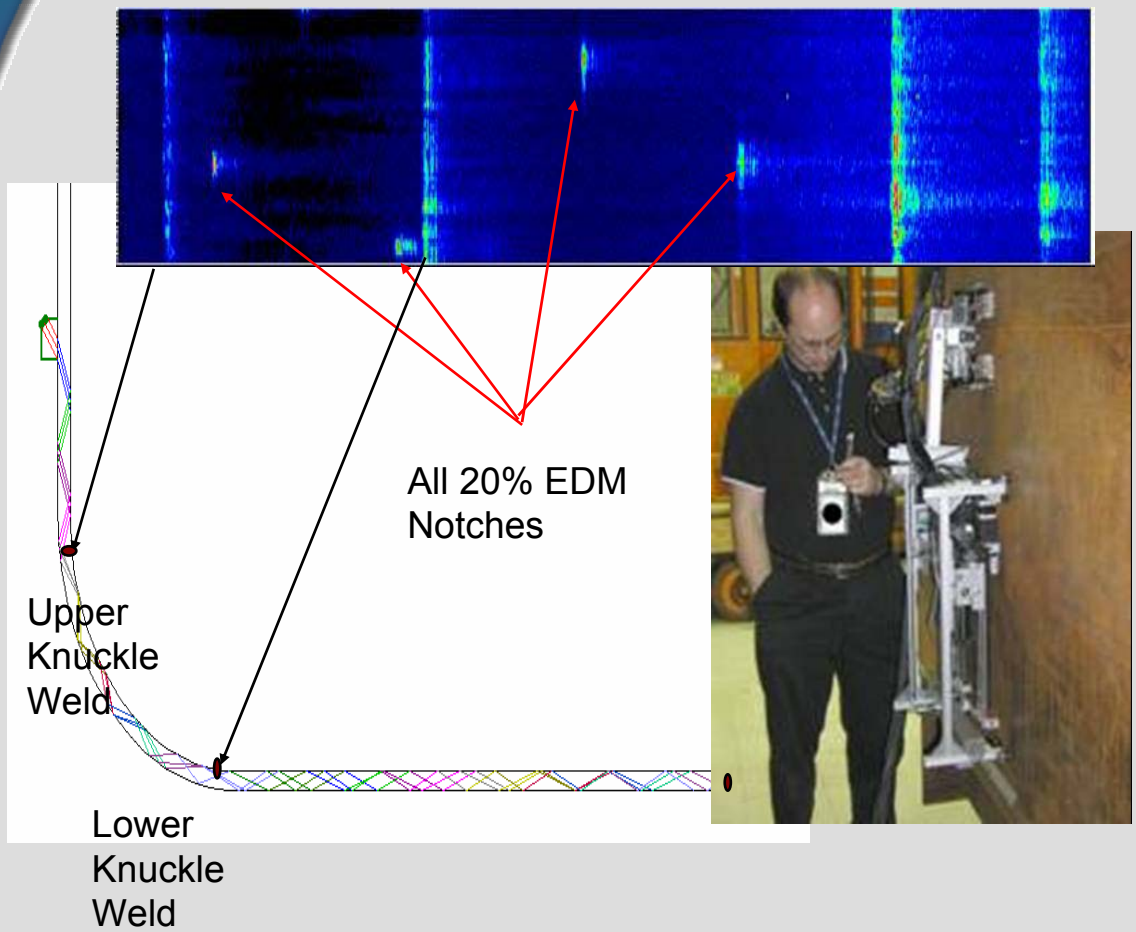
End of Plate



Upper image shows end of plate signal and lower Image shows "crack" after a 0.180" sawcut is inserted 4" from the end of Plate

- Developed Synthetic Aperture Focusing Technique (SAFT) algorithm for the NRC to characterize fabrication flaws in RPVs.
- Research indicates ability to detect and size machine made notches as small as 0.180" from 42" away.
- Non-homogeneous, coarse-grain materials lead to significant difficulties for traditional nondestructive examination techniques
- Great inspection potential for the use of low-frequency SAFT to provide high-resolution, high signal-to-noise ratio images.

Problem: Need a method to inspect hard to reach knuckle region of double shell tanks.

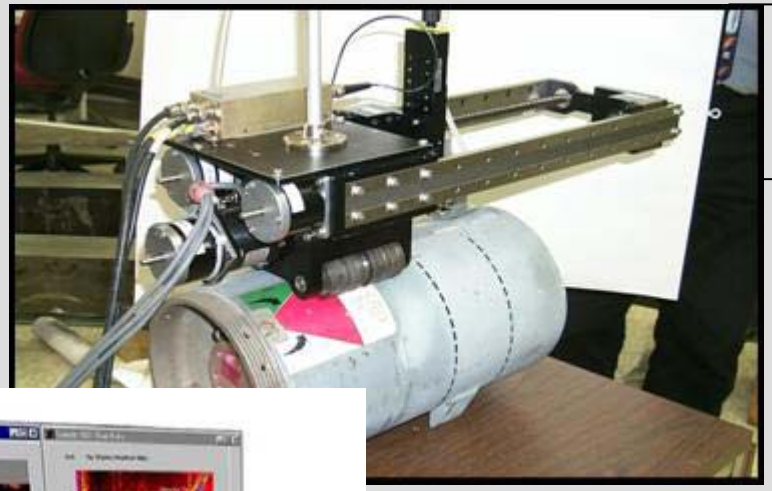


Solution:

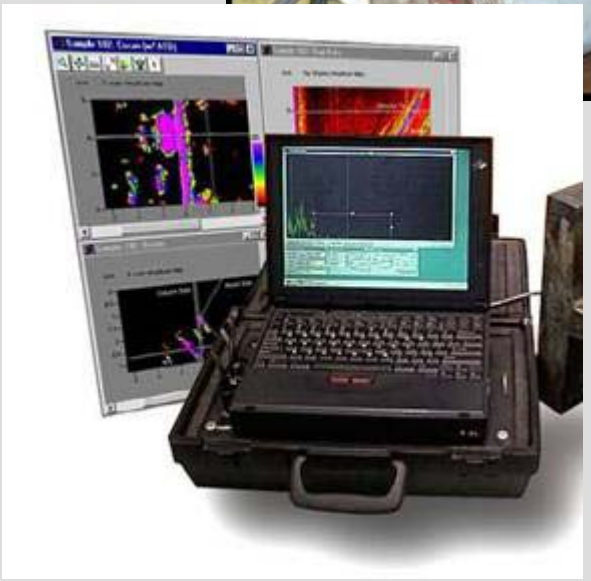
- Developed specialized UT transducers
- Deployed sensors on a magnetically couple robotic crawler.
- Modified SAFT imaging software for application to this difficult geometry
- Deployed in Hanford double shell tanks

Client: DOE-EM

Problem: Capability needed to locate un-bond flaws in rocket motors .



NDE and Surveillance of Sparrow Rocket Motors



Ultrasonic Data Acquisition and Display System

A large air unbond between case and the insulator as a result of a fabri-cation error.

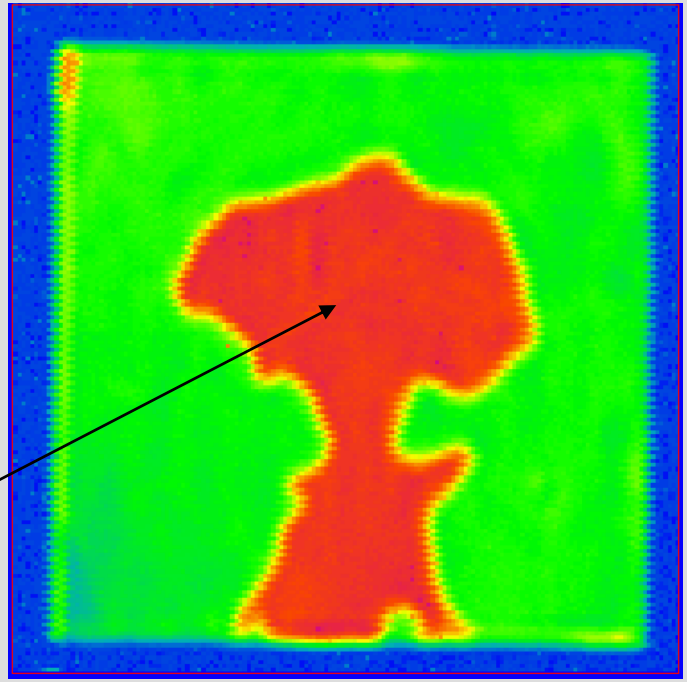
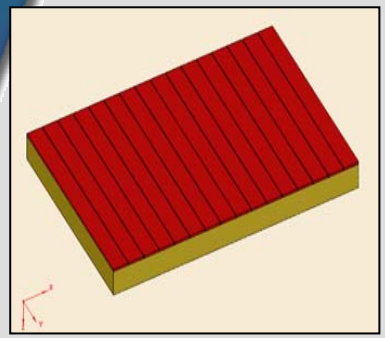
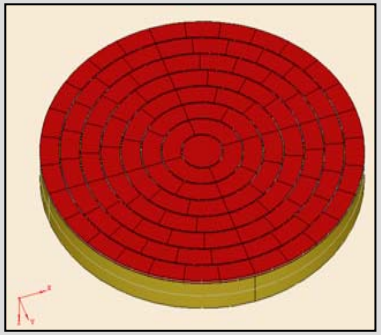


Image of the Flaw

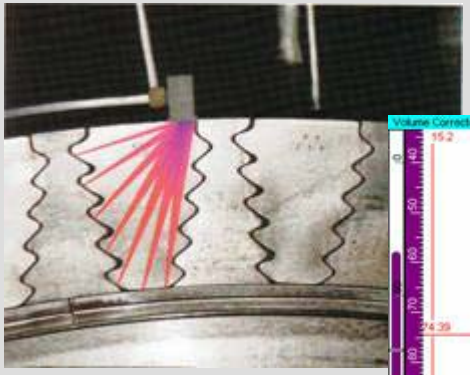
Utilization of Phased Array Technology for Multiple Project Applications



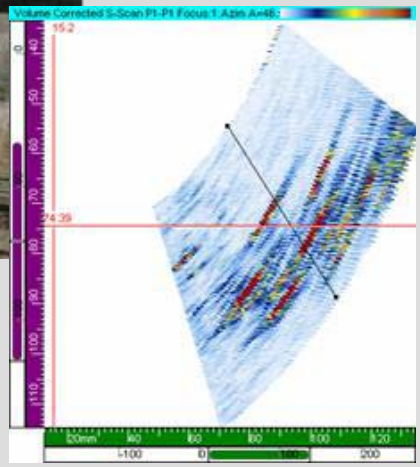
Linear Array



Sectored Array



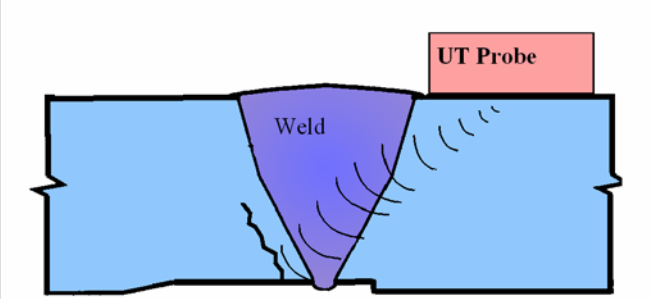
Turbine Blade Inspection

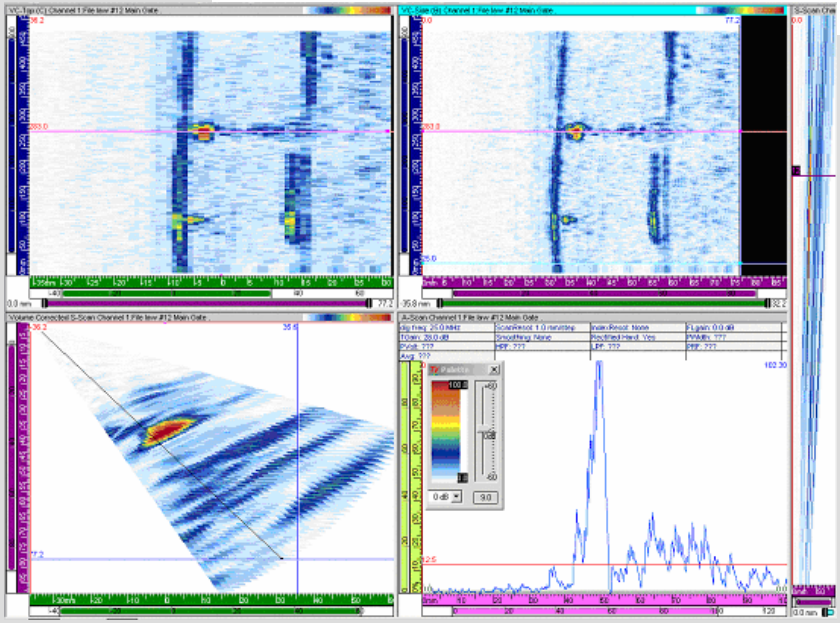


Azimuthal Sweep

- Conventional probes limitations:
  - Fixed angle, fixed focal distance, fixed focal point size
  - To vary these requires a different probe, a wedge or a lens.
- Phased-array probes advantages:
  - **Uses a single, small, electronically controlled, multi-element probe**
  - Software control of beam angle, focal distance, and scan parameters
  - Greater flexibility for complex geometry NDE and difficult for examination materials and welds
  - High-speed scans with less or no moving parts

Problem: Detect and characterize cracks on the far-side of austenitic welds in presence of geometrical reflectors.





- Access to both sides may be limited
- Welds absorb and re-direct acoustic energy
- Ultrasonic responses from weld root and counter-bore mask flaws

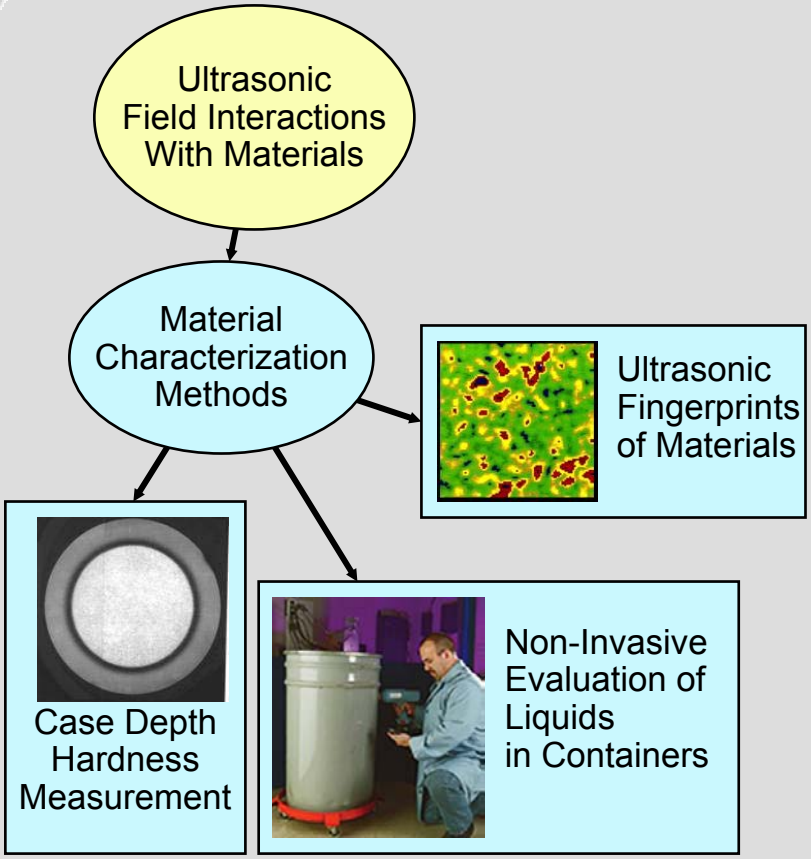
PNNL solution:

- Use phased array to sweep through multiple angles on a single line scan enabling discrimination of flaws from geometry

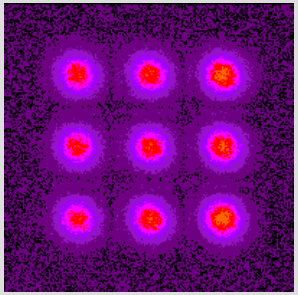
Problem: System needed to monitor the effects of use-loading on structural members & detect failure precursors

- Demonstrated detection of material stress changes using stress-driven *acoustic emission* from material.
- Demonstrated use of advanced signal analysis to discriminate material changes from system operating sounds.
- Tested on bridges, aircraft, piping.
- Capable of detecting and locating flaws as they form and predicting flaws before they form.

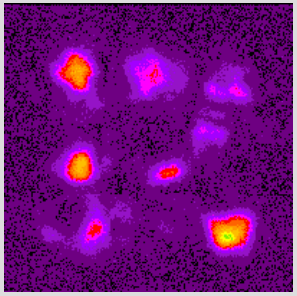
Application Area – Ultrasonic Material Characterization



Powdered Nickel



Ti-17 Billet



- **UT Inspection is concerned with flaw detection and sizing.**
- **Materials characterization is concerned with microstructural properties or physical properties.**

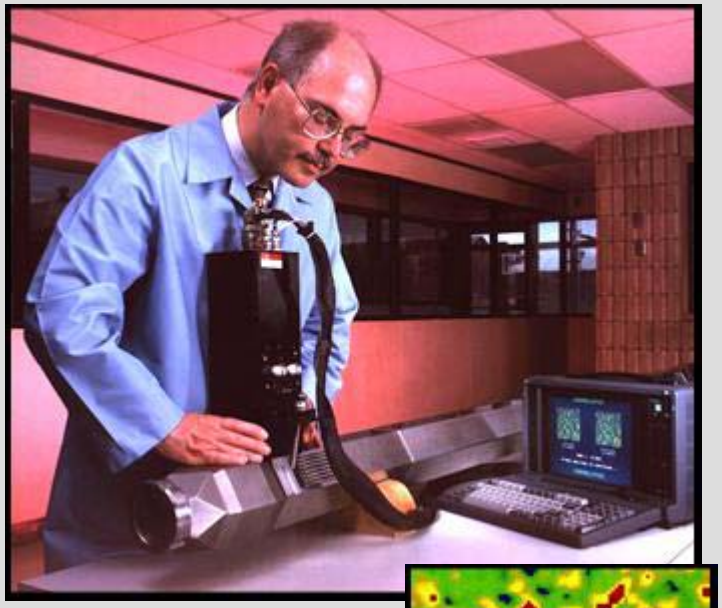
Problem: Develop method & equipment to perform non-destructive case depth hardness measurement on steels.

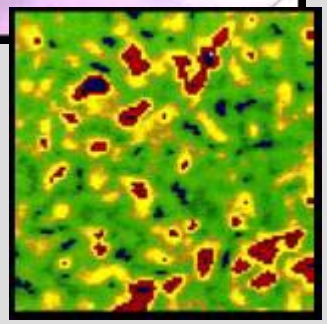
PNNL Solution:

- Developed ultrasonic test whose signal correlated with case depth hardness from destructive tests
- Developed instrument to perform in-plant sample measurements
- Won R&D 100 and FLC Awards
- Technology commercialized

Client: General Motors

Problem: Provide definitive method to identify & authenticate individual high-value metal & composite samples





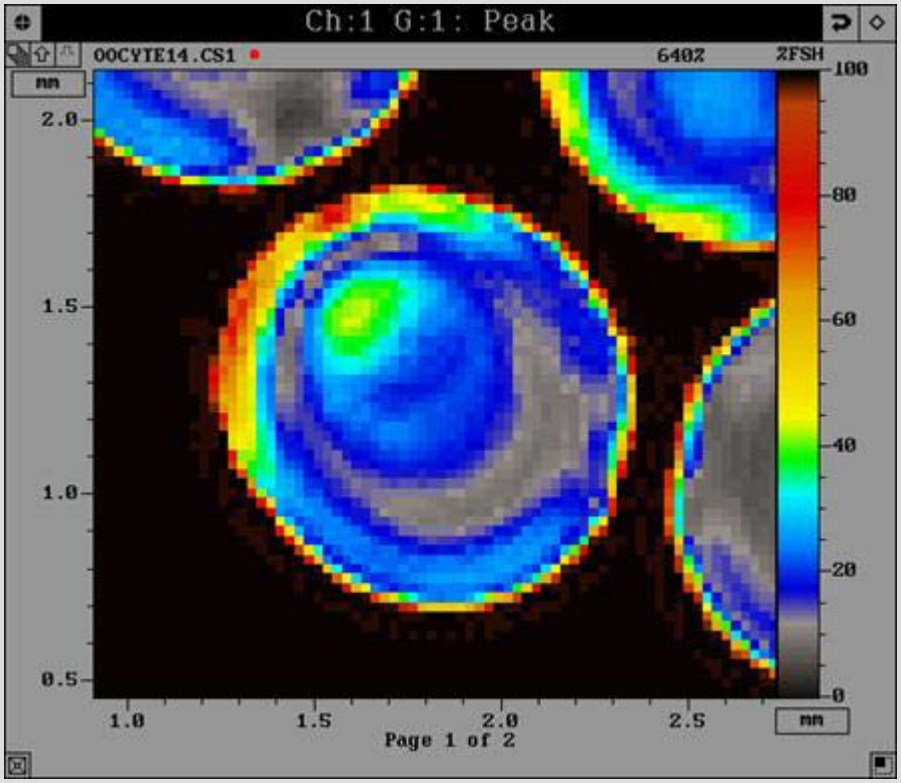
Typical Ultrasonic Fingerprint

PNNL Solution:

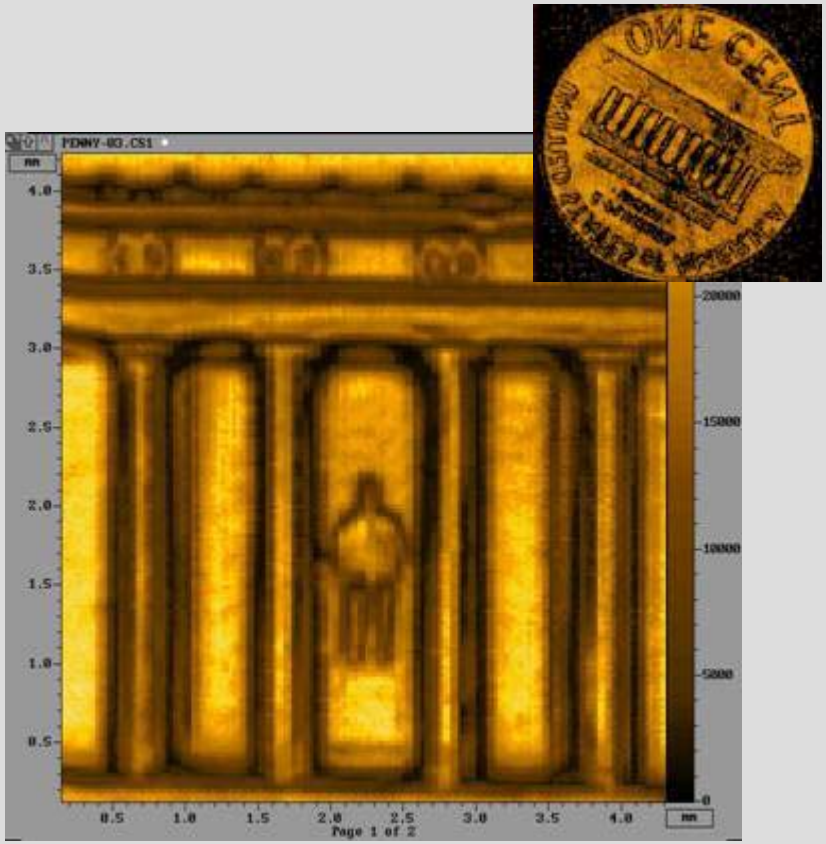
- Found ultrasonic (UT) “fingerprints” allow reliable sample identification.
- Developed small UT scanner to obtain in-field sample “fingerprints”.
- Developed method & software for automatic “fingerprint” verification.
- Delivered portable “fingerprinting” instruments tailored to client needs.

Client: DOE & IAEA

Acoustic Microscopy Images



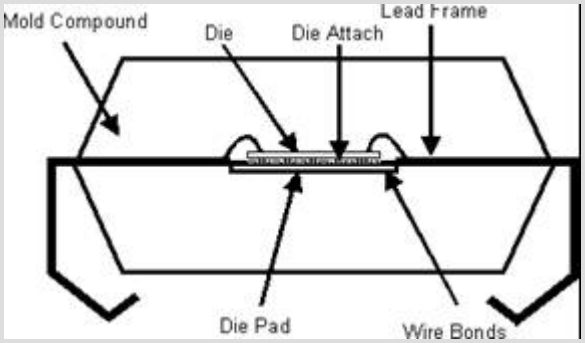
Xenopus Oocyte
(A Single Living Cell)



Lincoln in the Lincoln Memorial

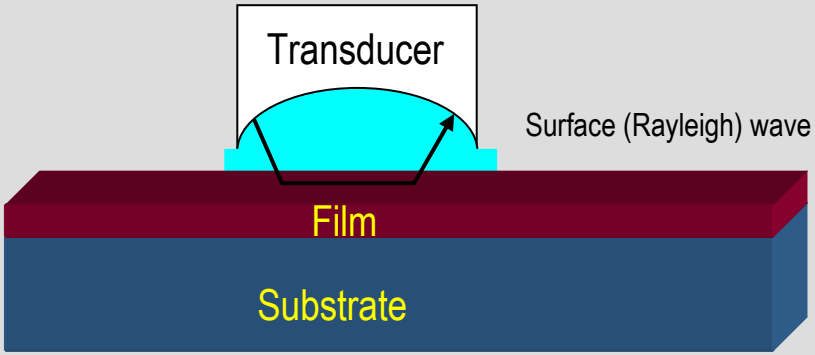
Acoustic Microscopy For IC and Thin Film Characterization

Delamination in Electronic Packages



- Flaw detection in microelectronic circuits
  - Delaminations, Cracks, Voids, Flip chips
- Thin film characterization
  - Elastic properties
  - Thickness

Thin Film Characterization



Problem: Need a reliable, non-invasive method to identify artillery shells containing chemical weapon liquids.

PNNL Solution:

- Reliable ultrasonic signature determined for chemical weapons (CW) material identification
- Backpack instrument developed with automated CW identification capability (3 mo. elapsed time)
- Second generation hand-held instrument developed & deployed

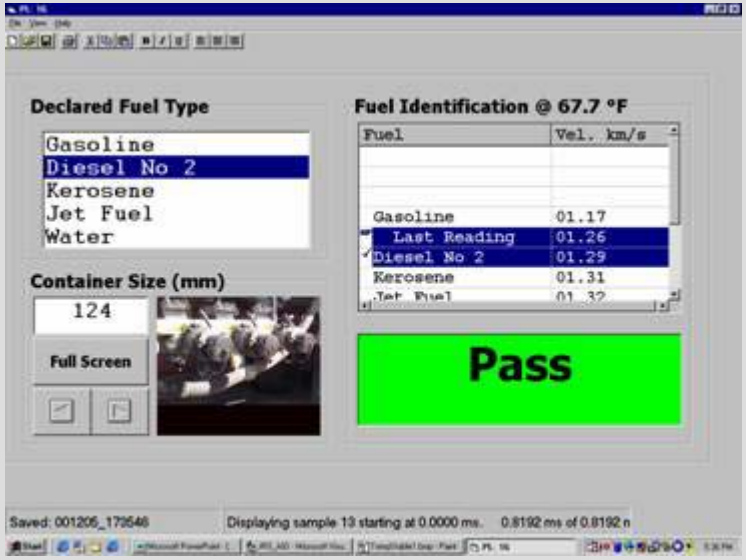
Client: DOE NN-20

Problem: Need a reliable, non-invasive method to characterize contents of a liquid filled container.

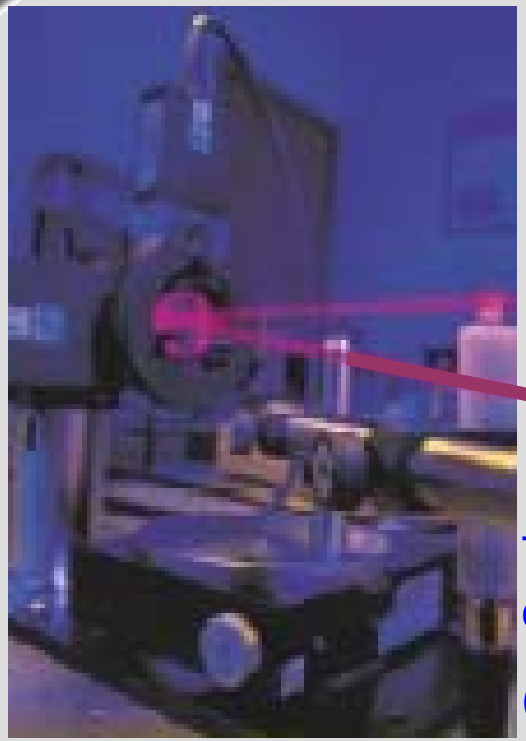
PNNL Solution:

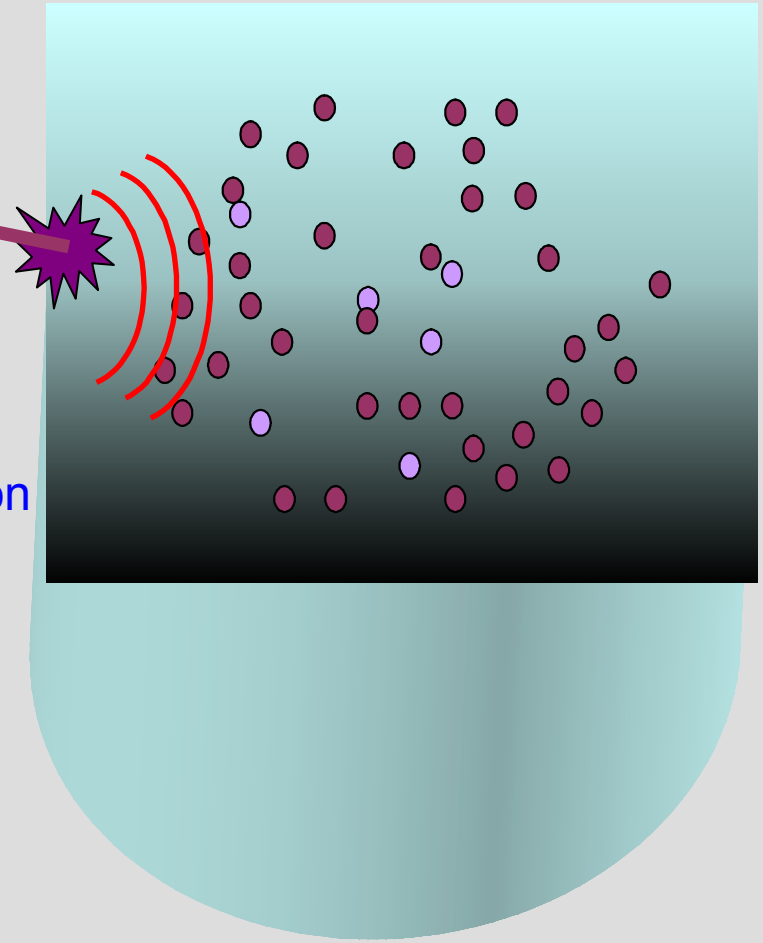
- Developed Hand-held acoustic inspection device
- Can discriminate between various types and grades of liquids and determine fill level
- Can identify hidden compartments or undeclared solids.

Client: IRS, USCS



Laser Ultrasonics



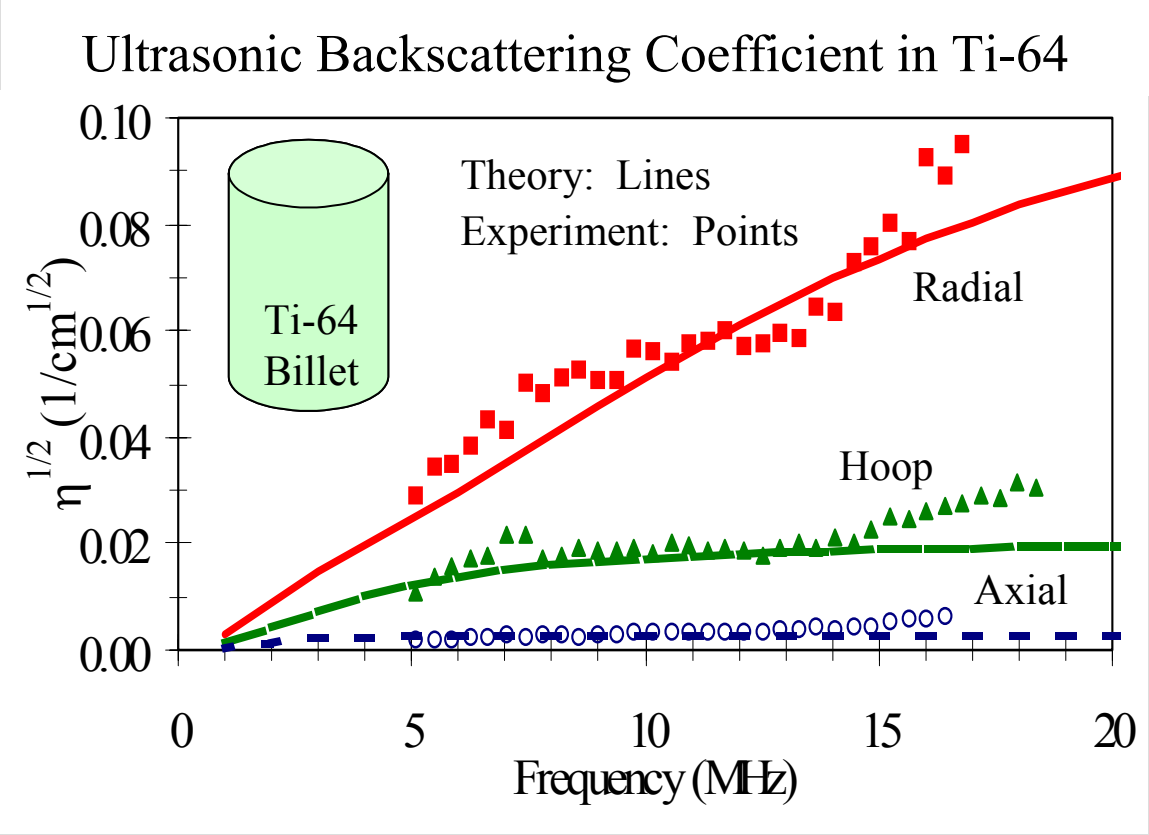


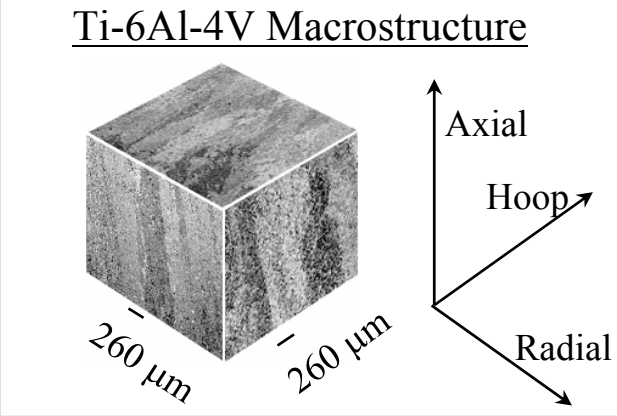
Thermal expansion

(no ablation needed)

- Stand off – many meters to 10's of meters
- Same measurements available as conventional ultrasonics
- Ablation nice but not necessary
- Commercially available

Problem: Fundamental Theory - Need to provide microstructural parameters/properties nondestructively, such as grain size and shape (morphology)



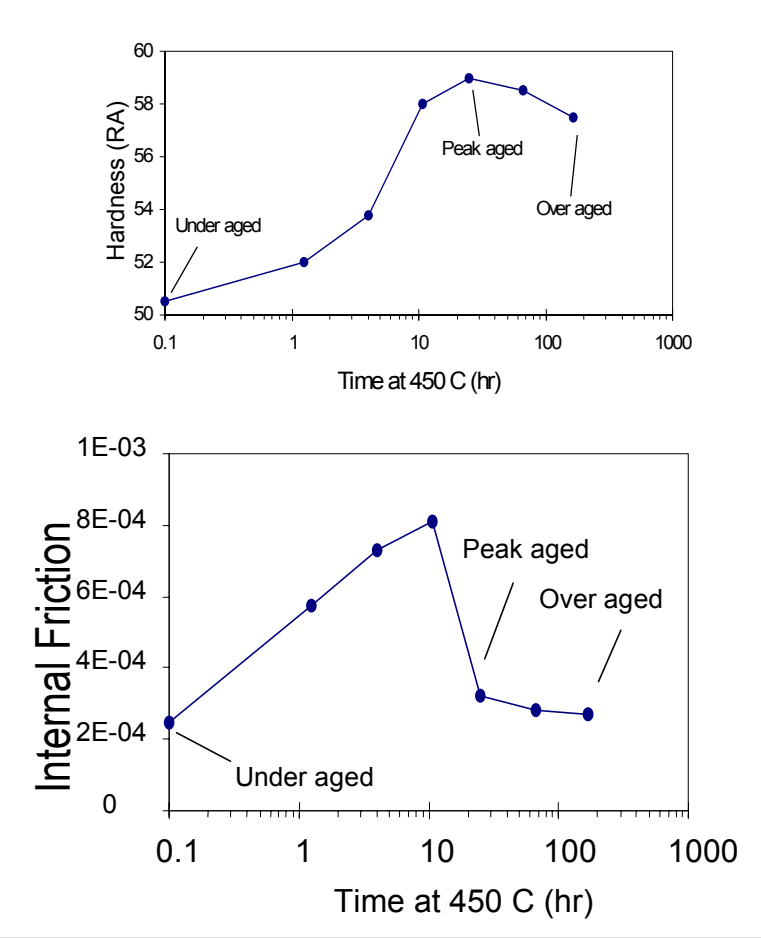


- Anisotropic micro/macrostructure controls scattering.
- Theory accurately predicts backscattering in this duplex titanium alloy.
- Theory currently being extended to describe scattering in iron and other alloys.

Client: LDRD

Problem: In-service monitoring of degradation of nuclear reactor components.

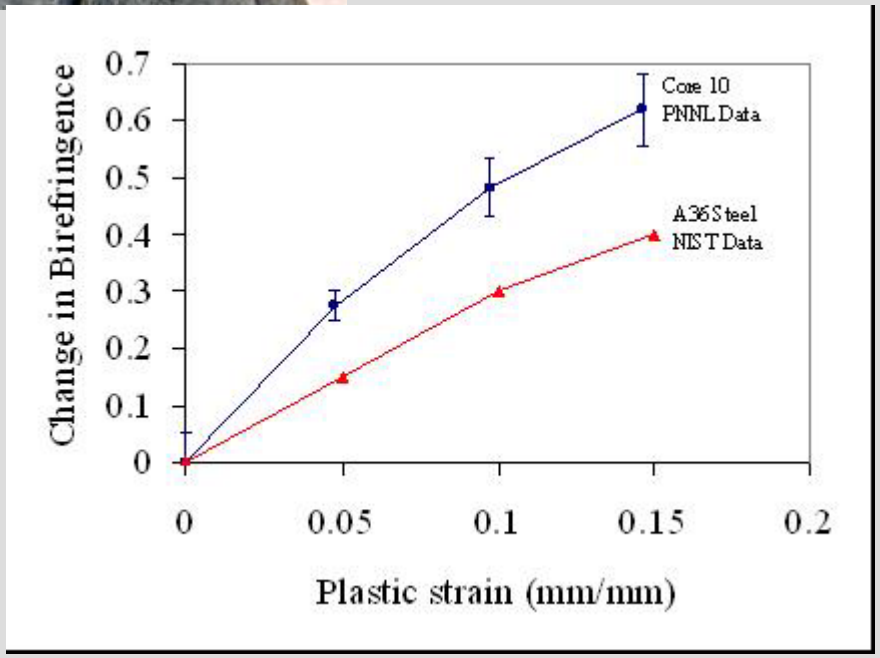
Precipitation induced hardening



- Developing ultrasonic and EM methodologies to characterize embrittlement due to void swelling and precipitate evolution

Client: LDRD

Problem: Need a method to quantify damage to piping infrastructure (natural gas pipelines).



- Current methods can find damage but are inaccurate in determining severity
- Ultrasonic velocity is sensitive to stress/strain (damage)
- Developing ultrasonic methodologies to characterize damage to pipelines and other structures
- Methods are applicable to many types of structures

Client: DOE-NETL

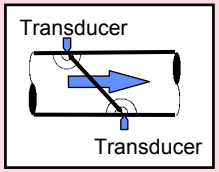
Application Area – Process Monitoring and Control Applications

Ultrasonic methods initially developed for government applications are finding applications in food processing and industrial process control

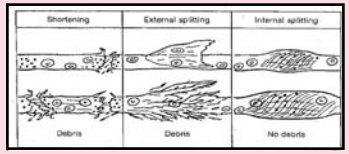
Process Monitoring of Gas and Liquid Stream

In-Line Density & Viscosity Measurement

Particle Size & Solids Concentration Probe



In-Situ Gas Flow Measurement



Wood Pulp Characterization

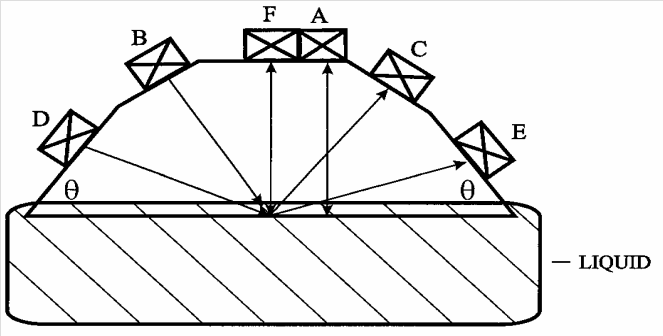
- Effectiveness of mixing, settling, or uniformity in vessels
- Detection of process changes, precipitate formation, colloid formation, gelation, agglomeration, crystallization
- Determination of density, viscosity, concentration, and particle size in-vessel or during pipeline transport of a slurry
- Detection of stratification within a flowing pipeline or the onset of pipeline plugging

Problem: Need a method to determine both particle size and solids concentration in slurries.

- Particle size and concentration are determined based on ***ultrasonic signal attenuation*** over a frequency range (0.3 to 12 MHz)
- Ultrasonic signal attenuation falls into three regimes
- The frequencies of the transitions between these regimes provides particle size and size distribution width
- The magnitude of the signal provides slurry concentration

Client: DOE EM

Problem: Need a method to determine fluid density, stratification and viscosity in pipelines.



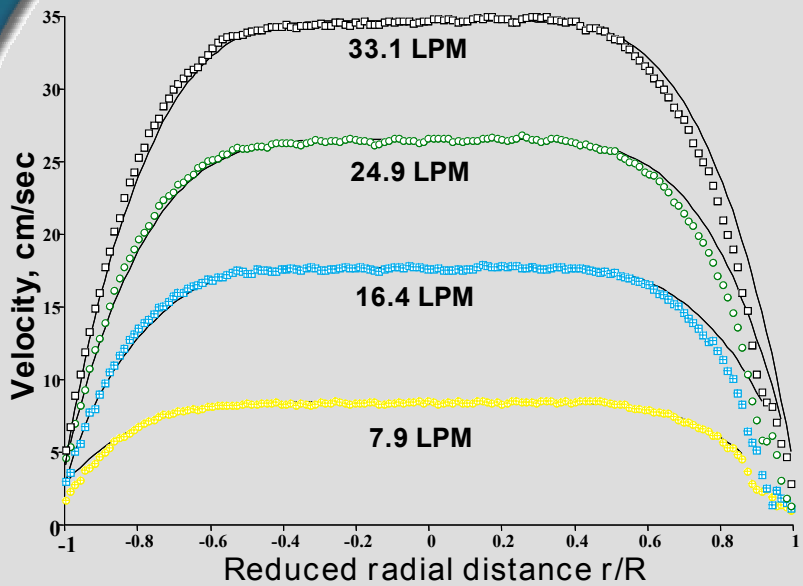
Ultrasonic pulses are reflected at the wedge/fluid interface.

U.S. Patent 5,708,191 and 5,886,250

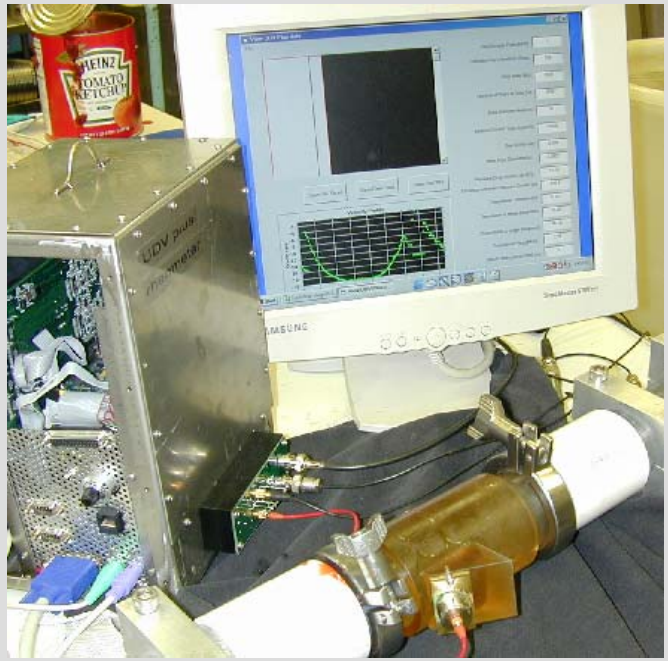
- Ultrasonic reflection coefficients are measured experimentally for longitudinal and transverse wave modes
- Reflection coefficient obtained with the base of sensor wedge immersed in water
- Reflection coefficient measurements for longitudinal and shear waves provide a measure of fluid density and viscosity in near vicinity of probe face

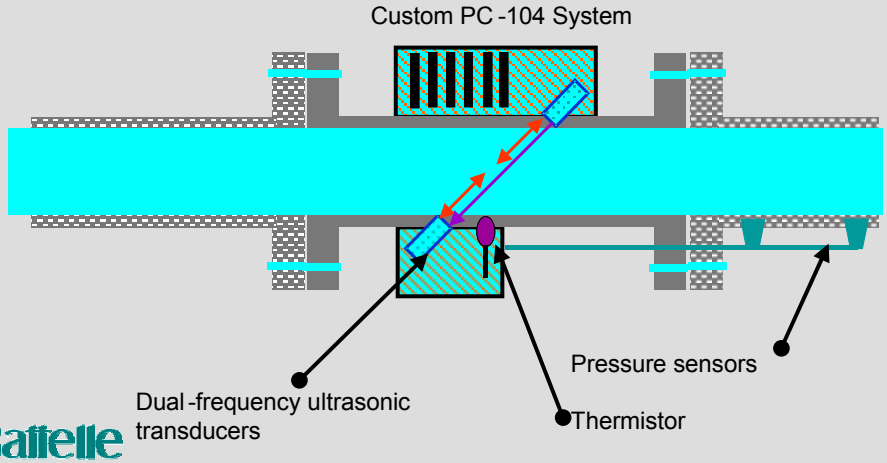
Client: DOE EM

Problem: Need a method to determine flow rate and viscosity in non-Newtonian fluids.



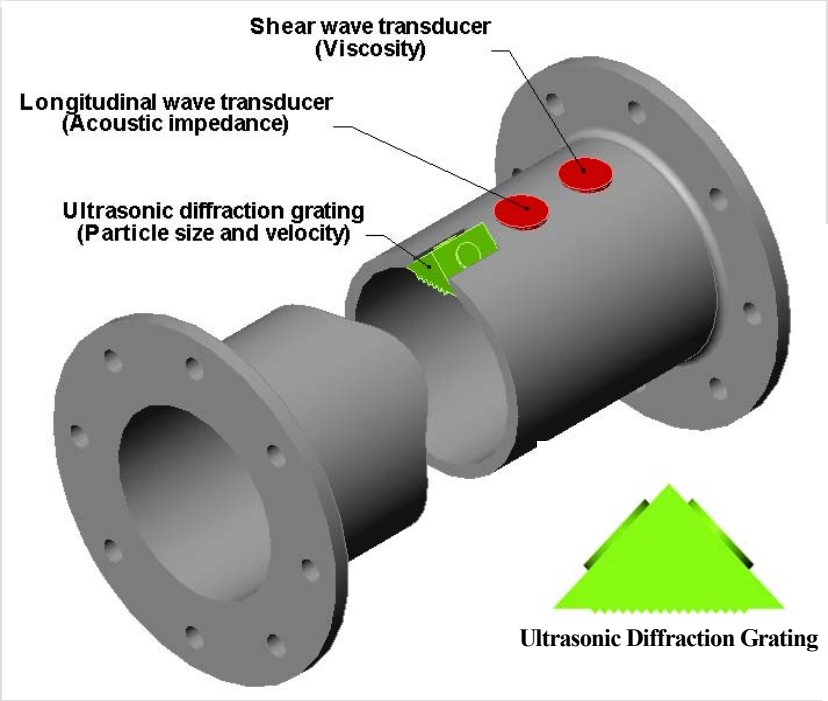
Velocity Profile Generated by Velocimeter



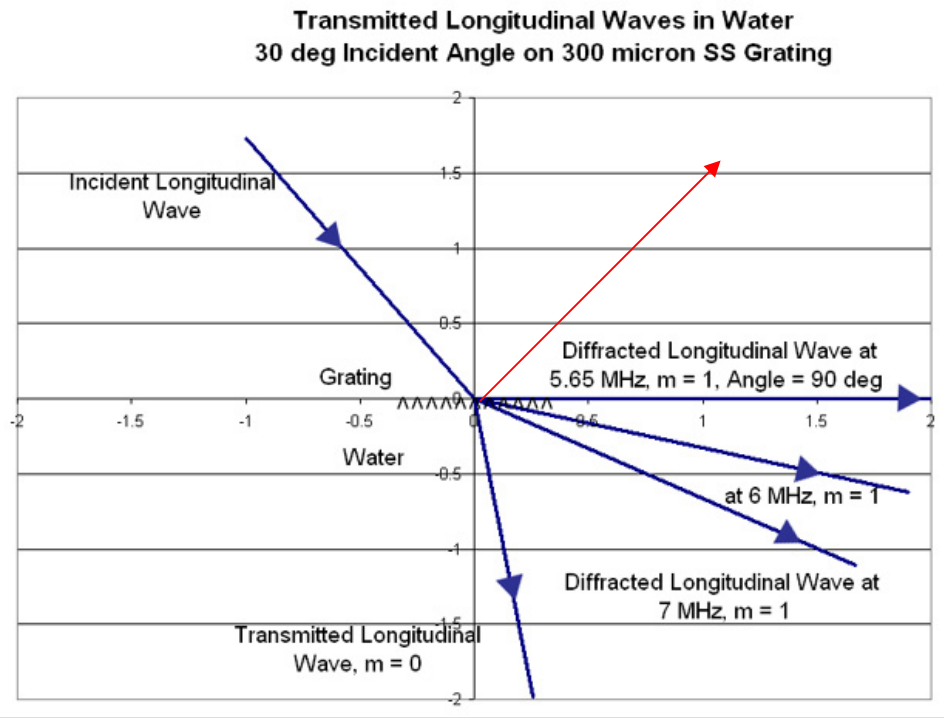


- Measures frequency shift of backscattered wave to determine velocity profile
- Computes viscosity from velocity profile and pressure drop

Problem: Need a method to determine particle size, fluid density, and viscosity in pipelines.

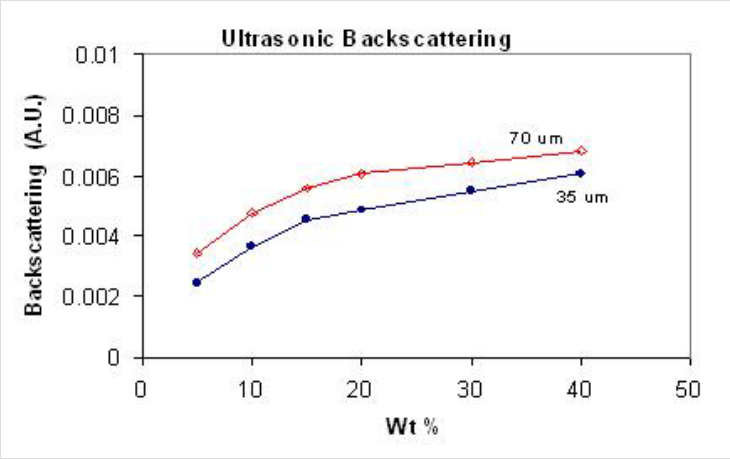


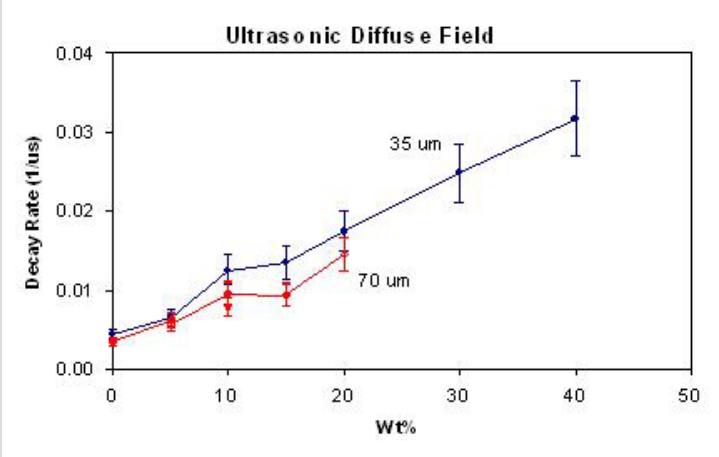
- Utilizes optical diffraction theory
- Adjust frequency to obtain particle size and velocity
- With velocity and impedance obtain density



Client: DOE EMSP

Problem: Need a method to accurately determine particle size in high concentration liquids.





- Current methods work well at low concentrations:
- Developing ultrasonic methodologies to work at high concentrations
  - Particle size distribution
  - Agglomeration
  - Gelation
- Applications:
  - Pharmaceuticals
  - Paints
  - Petroleum Products
  - Bioreactors

Client: DOE-EMSP

Problem: Need sensors/instruments suitable for an on-line, automated, field deployed oil analysis system

Bench-top, Integrated Prototype System

Lube Oil Condition
- *Viscosity*
- *Oxidation*
- *Total Acid/ Base*
- *Additives*

Lube Oil Contamination
- *Fuel*
- *Coolant*
- *Dirt*
- *Water*

Lube Oil Wear / Debris
- *Ferrous, Non-Ferrous Metals*
- *Wear Metals / Elemental Analysis*
- *Particle Characteristics*

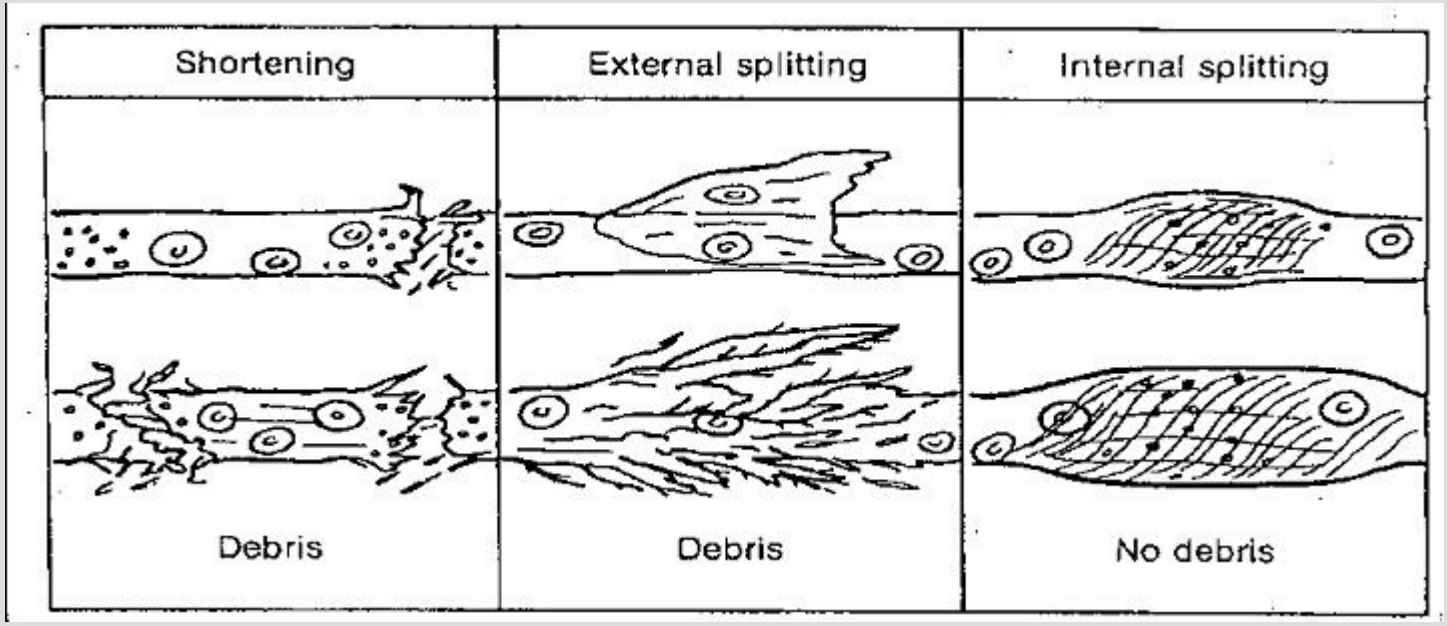
Commercial sensors for viscosity, in-line debris, & particle monitoring

- Fully portable systems can provide near-real time analytical data related to lubricating oil condition and machine wear materials.
- Uses optical signatures for key indicators of oil degradation (e.g., oxidation, nitration, sulfation, and water contamination)
- PNNL developed XRF sensor with oil flow interface that measures 22 elements.

Client: DOE - ITP

Problem: Need a method to monitor fiber changes during refining of wood pulp.

- Frequency-dependent attenuation measurements may characterize wood pulp refinement



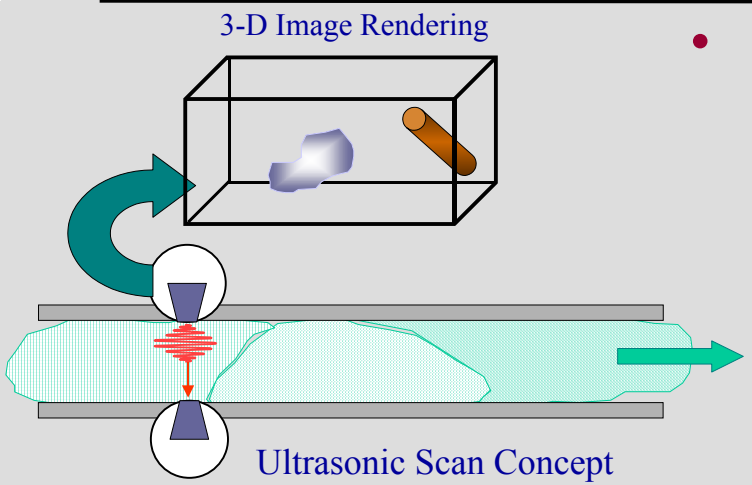
Wood pulp morphology in process stream

Problem: Develop an on-line, at-consistency measure of cationic demand *and* fiber charge on paper pulp.

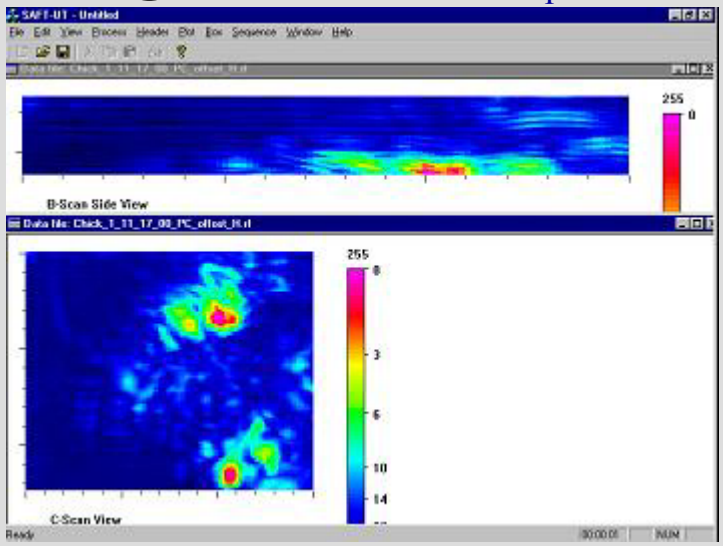
- Developed an Electrokinetic Sonic Amplitude system
- Benefits include:
  - Deployed at line for near-real-time measurements
  - Optimized chemical additives
  - Reduced drying energy
  - Increased process control
  - Increased retention & drainage

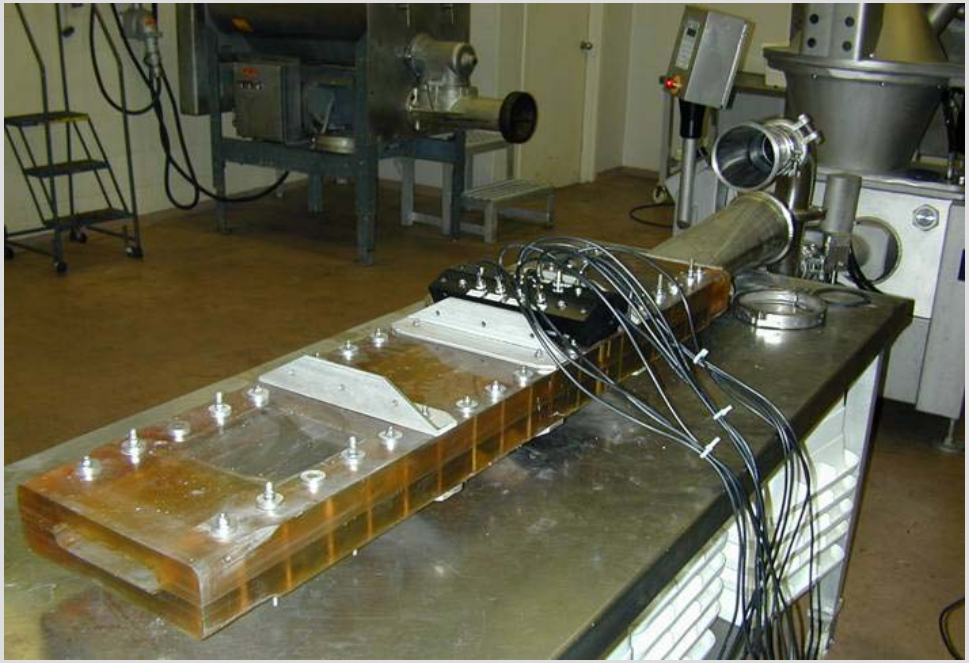
Client: DOE-OIT

Problem: Need a method to detect foreign material in meat in a high speed processing line.



- Developed automated ultrasonic inspection system for detection of bone in chicken breast processing
  - Ultrasonics detect embedded bones
  - Candling system detects surface bone





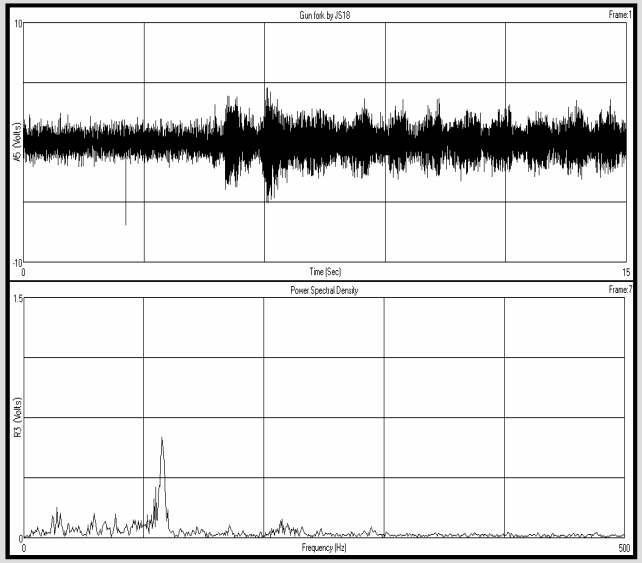
Problem: Need a method to detect 3rd party contact to pipelines to assess damage or prevent tampering.

- Listen for sound traveling in pipe
  - Pipe acts as a waveguide
- Due to sensitivity of accelerometers can also hear sounds around pipeline

Accelerometer

Amplifier

PC with D/A board and filter card



Problem: Need a method to detect 3rd party contact to pipelines to assess damage or prevent tampering.

- Detect events occurring to and within pipe
  - Construction, theft, vandals, compressor shutdown, etc.
- Detect events occurring along pipeline
  - Encroachment, traffic, over flights, etc.

Riser Impact

Compressor Shutdown

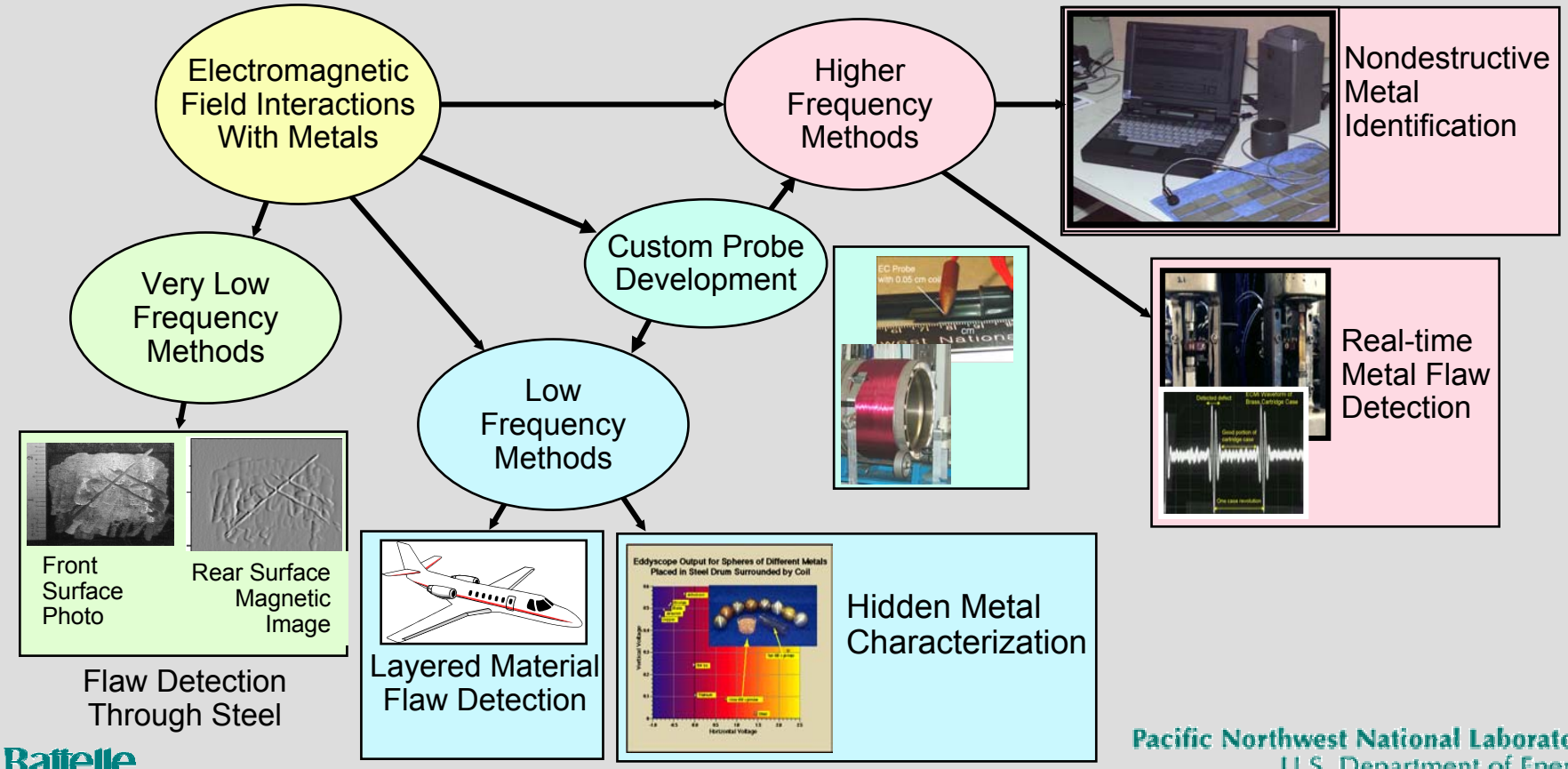
Debris Passing Through Pipeline

Thunder

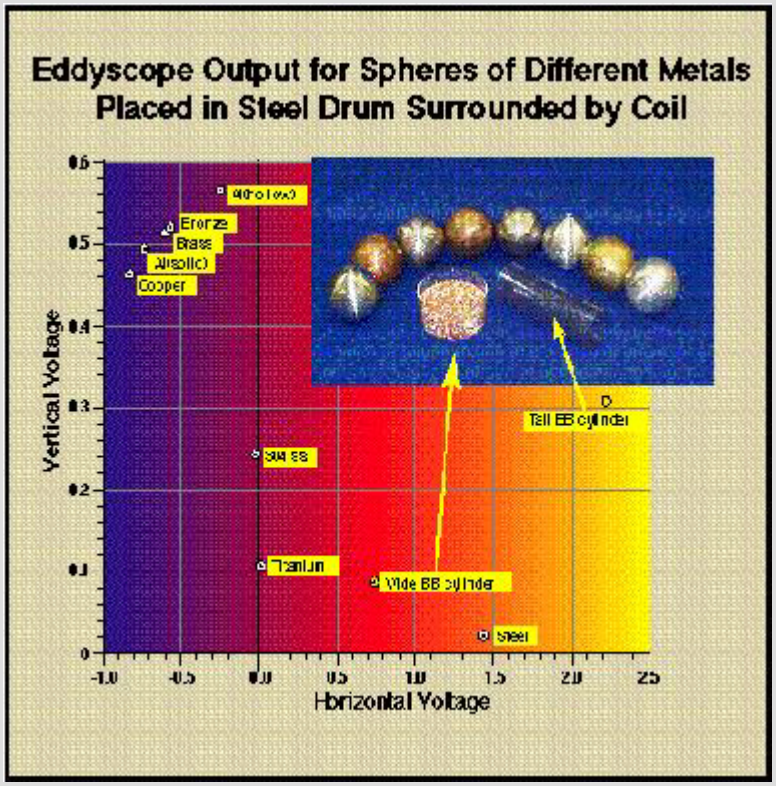
Car and Dog

Application Area – Electromagnetics & Eddy Current Material Inspection and Characterization

- Attractive features associated with EM measurements include - fast (~1millisec), non-contact for many applications, high-precision (1 part in 10,000 or better), simple and rugged enough for field use.



Problem: Develop non-invasive method to obtain unique signatures of metal objects within closed containers.

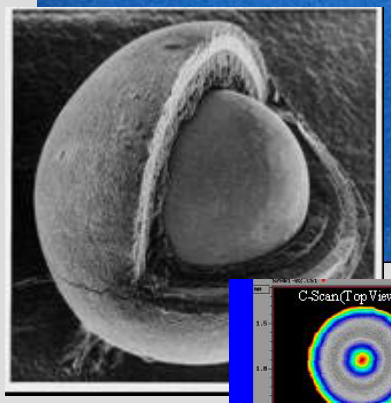


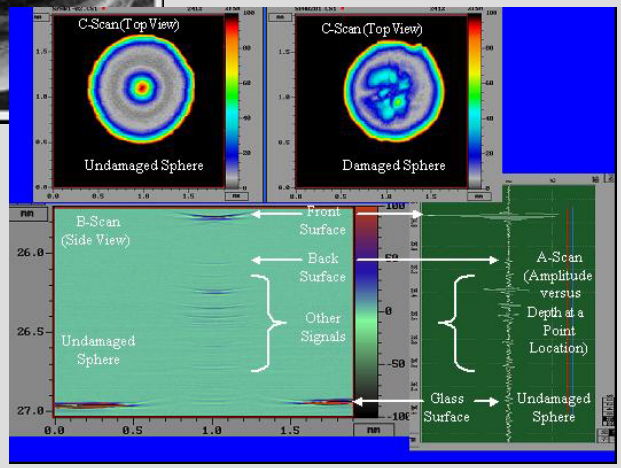
PNNL Solution:

- Developed electromagnetic (EM) sensing system to examine metal objects inside containers
- Low-frequency EM fields penetrate container & interact with contents
- Sensor impedance measurements distinguish between various possible enclosed metal object(s)
- Characterizes both type of metal and mass of object.

Client: DOE NA-22

Problem: Capability needed to rapidly inspect millimeter sized particles to detect coating flaws.

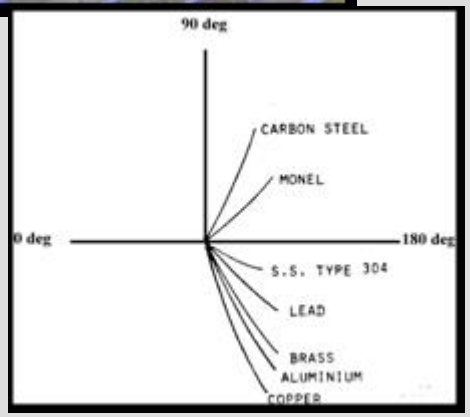




- Detect flaws in large batches of pellets to increase throughput
- When a flaw is known detected, use alternative technique to localize it to a given pellet.
- Evaluating several NDE techniques including ultrasonic, eddy current, optical, and acoustic microscopy

Client: DOE-NE

Problem: Determine electromagnetic signatures of metals to support rapid, non-destructive metal sample identification.



Distinct Impedance Signatures of Metals

PNNL Solution:

- Conducted lab tests to determine properties of a variety of metals
- Established complex impedance "signatures" for metals important for import/export control
- Developed data analysis algorithm to allow rapid metal identification
- Developed practical instrument

Client: DOE & US Customs

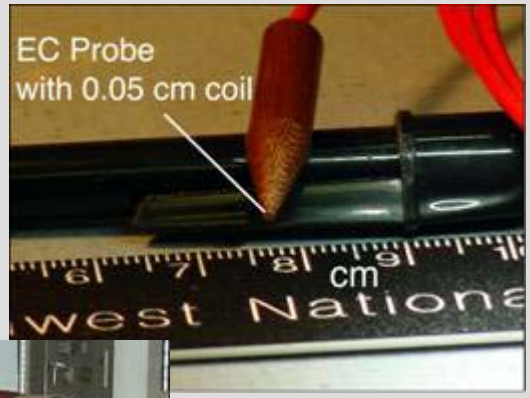
Problem: Develop a high speed eddy current inspection method to evaluate thin coatings on ID and OD of tubing.

PNNL Solution:

- Developed customized probes and an automated high speed scanning system (aluminide & nickel).
- Characterize probe performance & revise design as needed
- Developed an advanced method to inspect both ID and OD simultaneously

Client: DP

Problem: Specialized electromagnetic probes are needed to meet various measurement & inspection requirements.



Large Coil Being Wound on 55 Gallon Drum Mandrel

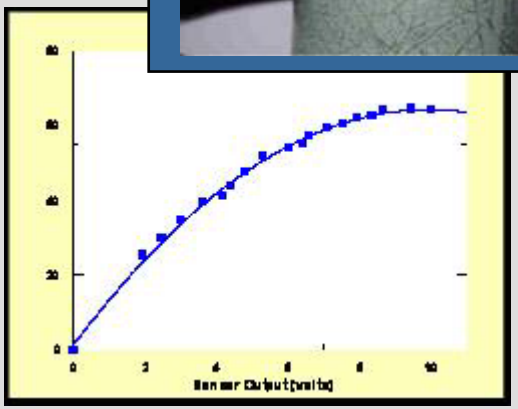
PNNL Solution:

- Determine application specific measurement requirements
- Design & fabricate unique probes tailored to meet needs
- Characterize probe performance & revise design as needed
- Fabricate final probes for client application

Client: DOE, DOD, Industry

Problem: Develop non-contact method to determine moisture percentage in radioactive-chemical waste materials.

Lab Test of Sensor in Simulated Waste



Physical Property Sensors

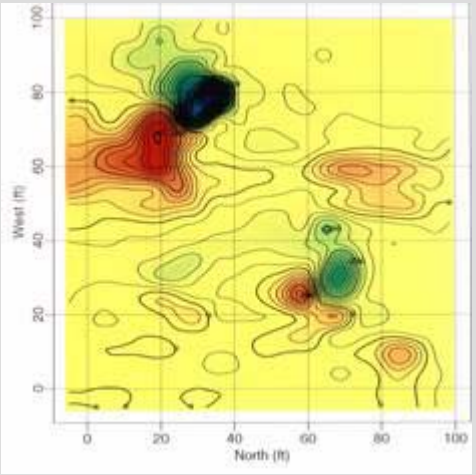
PNNL Solution:

- Completed laboratory tests showing eddy current signal correlation to moisture level
- Established techniques for quantitative data interpretation
- Successfully demonstrated method in waste tank to meet application requirements

Client: DOE EM-30 & 50

Problem: Locate buried objects (e.g. UXO) and underground structures at large sites with hazards or surface obstructions

Cypher UAV



Magnetic contours of detected targets

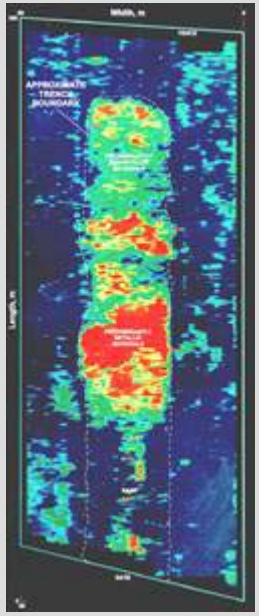
PNNL Solution:

- Adapt Sikorsky unmanned aerial vehicle (UAV) as a platform for geophysical sensors.
- Integrate multi-sensor instrument suite (GPR, magnetometers, EMI, GPS, others) with aircraft systems.
- Develop data acquisition system with real-time data display.

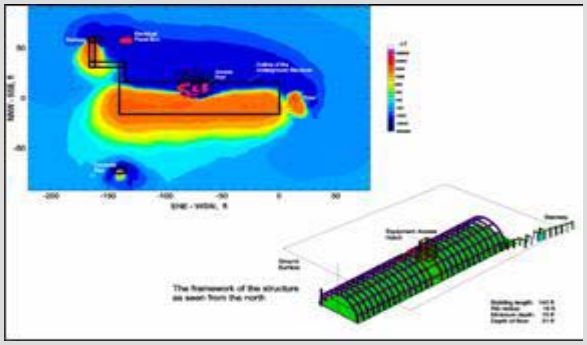
Client: DOE and DOD

Problem: Detect, map, and characterize buried objects such as UXO, waste, utilities, geologic and cultural features.

GPR Survey



GPR map of waste burial site



Magnetic Modeling

PNNL Solution:

- State-of-the-art instrumentation (ground-penetrating radar, EM induction, magnetometers, GPS, vehicle-mounted sensors, digital data processing).
- Many field surveys performed in the US, Europe, and the Middle East.

Client: DOE, DoD

Problem: Enhance operator safety & measurement efficiency for subsurface characterization of hazardous sites

Remotely Controlled Vehicle

Control Station

PNNL Solution:

- Telerobotic Geophysical Survey System
- Remotely controlled, nonmetallic survey vehicle with control station, RF communications, real-time data display, and vehicle-mounted video.
- Integrated sensor suite (ground-penetrating radar, EM induction, magnetometers, and GPS).

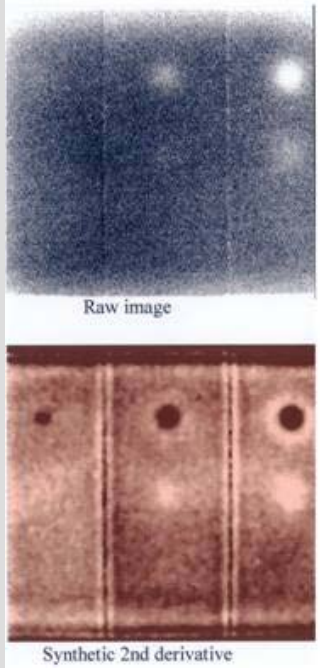
Client: DOE, DoD

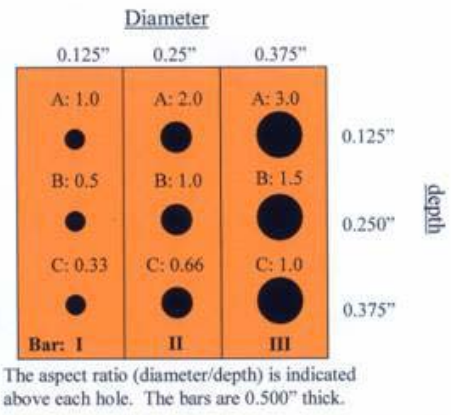
Other Applications and Capabilities

- **Infrared Imaging**
  - Ariel (P.I. Steve Doctor)
- **Optical Inspection**
  - Internal Stress in Glass (P.I. Chet Shepard)
- **High Power Ultrasonics**
  - Cell Lysis (P.I. Jerry Posakony)
- **Sono Chemistry**
  - Mexican Petroleum
- **Acoustic Microscopy**
  - Cellular studies
  - IC Applications

Problem: Need a method to detect flaws using infrared technology.

- Flashlamp or other heating source rapidly deposits heat onto sample surface
- Evolution of sample surface temperature is recorded in time with a digital infrared movie camera (40 frame/sec)
- Subsurface features (flaws) show up as time-dependent anomalies in the surface temperature
- Flaw size and depth can be determined from the time-resolved record of images

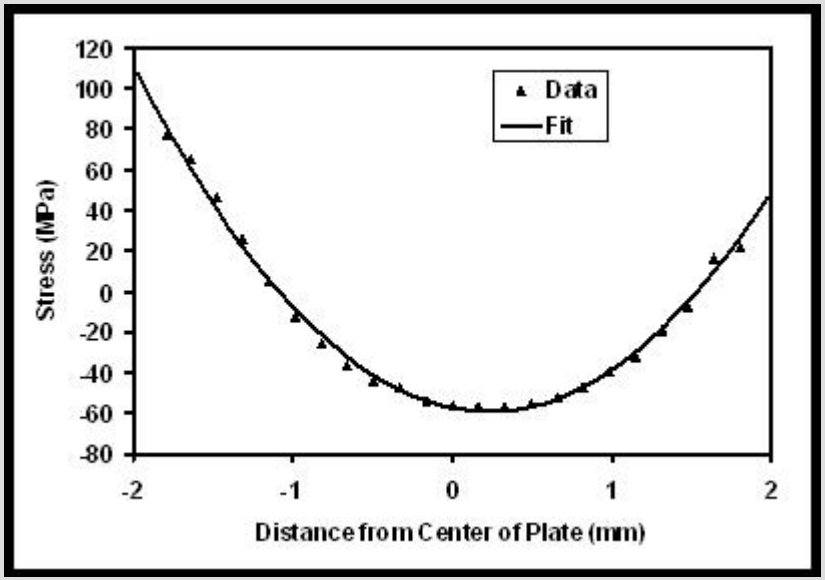




The aspect ratio (diameter/depth) is indicated above each hole. The bars are 0.500" thick.

- Holes with aspect ratio < 1 are not detectable.
- Holes with aspect ratio < 2 are difficult to detect using conventional analysis.
- Holes with aspect ratio > 1 (except IIIC) are detectable with synthetic processing.
- Edge effects may effect detection of IIIC.

Problem: Internal stress profiles in glass sheet govern glass strength but no method existed for measuring these stresses.



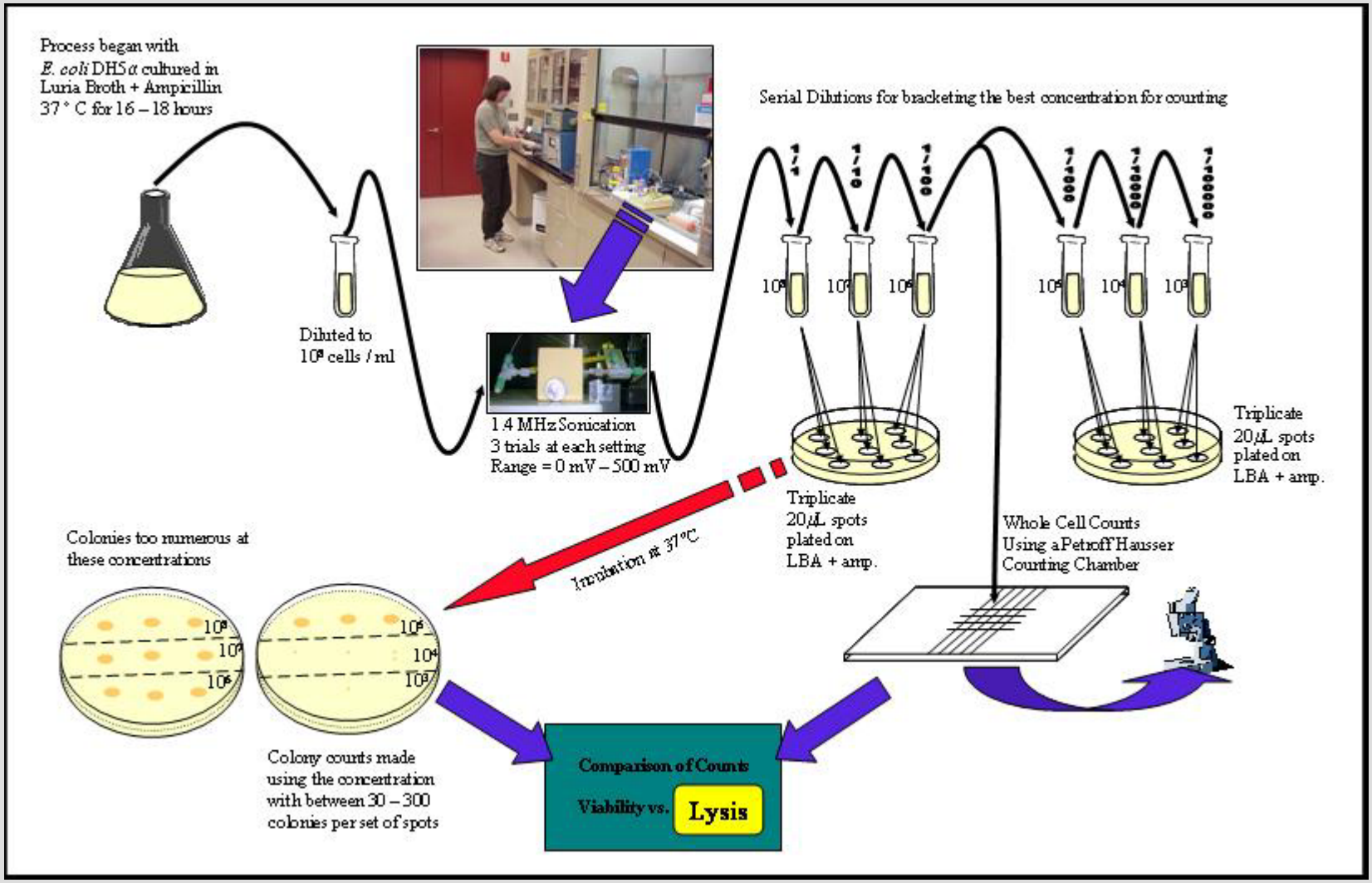
Experimental Stress Distribution in Tempered Glass Sample

PNNL Solution:

- Developed unique optical method for measuring stress profiles
- Established a laboratory scale measurement system
- Performed experiments which validated measurement principle
- Efforts are continuing to develop an instrument for routine use

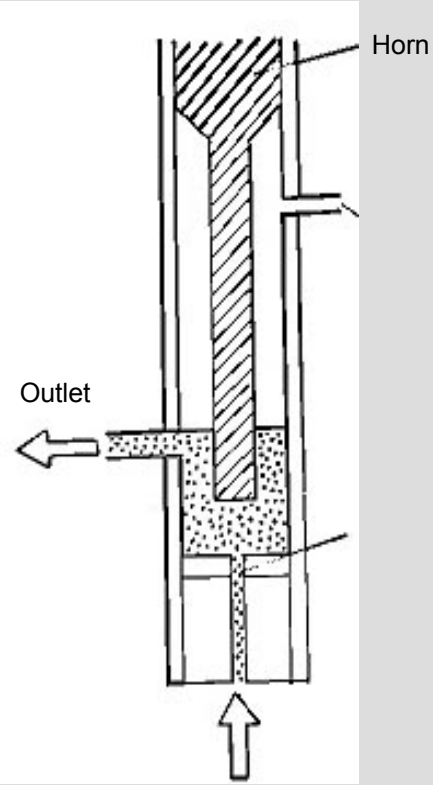
Client: DOE - OIT

High Power Ultrasound – Cell Lysis



Advanced Ultrasonic Processing Methods For Petroleum Refinement Applications

Example of a Flow Through Reactor Cell



Sonochemistry:

- use acoustic energy (high-power ultrasound) to influence chemical reactions in fluids.
- generates greater quantitative yields through higher reaction rates.
- Utilizes novel transducer designs to optimize volume ensonified and power delivered.
- Utilizes nanocatalysts to enhance reaction rates.

Applications:

⇒ Crude Oil Refinement (asphaltene, sulfur, and heavy metals reduction)

⇒ Refinement of Lighter Fractions (Gasoline, Diesel, etc.)

⇒ Breakdown of Volatile Organic Compounds (i.e. CCl_4)

NDE Measurement and Characterization Sciences Group
Making A Difference

The Sphere of Influence of the NDE Measurement and Characterization Sciences Includes:

- **Influencing International programs on NDE Reliability by Providing Technical Guidance to International Activities such as PISC studies and European/American Workshops on NDE Reliability**
- **Influencing International programs on In-Service by Providing Technical Guidance to IAEA programs**
- **Influencing National Codes and Standards by Participating on ASME committees, ASNT Ad Hoc Committees**
- **Influencing National and International Law Enforcement through Technology**
- **Influencing the "State of NDE Characterization Science" through Technical papers**

Group
Awards & Recognition

- R&D 100 Awards -

- 2003 - Product Acoustic Signature System
- 2000 - Multi-Blade Knife Failure Detector
- 1995 - Real Time Ultrasonic Imaging System
- 1994 - Ultrasonic Microstructural Analyzer

- FLC Awards -

- 2003 - Acoustic Inspection Device
- 2001 - Multi-Blade Knife Failure Detector
- 1995 - Ultrasonic Microstructural Analyzer
- 1986 - Synthetic Aperture Focusing Technique

- US PATENTS -

- 39 Patents and 90 invention reports awarded or pending between 1990 and 2004 that relied on S&E technical capability
- Primary areas of concentration
  - –Electro-optic & electromagnetic methods & devices
  - –Nondestructive evaluation methods & devices
  - –Material Characterization Devices
  - –Process monitoring and control technologies