# XMT Imaging and Digital Volume Correlation for Characterization of Micromechanical Performance in Wood-Plastic Composites


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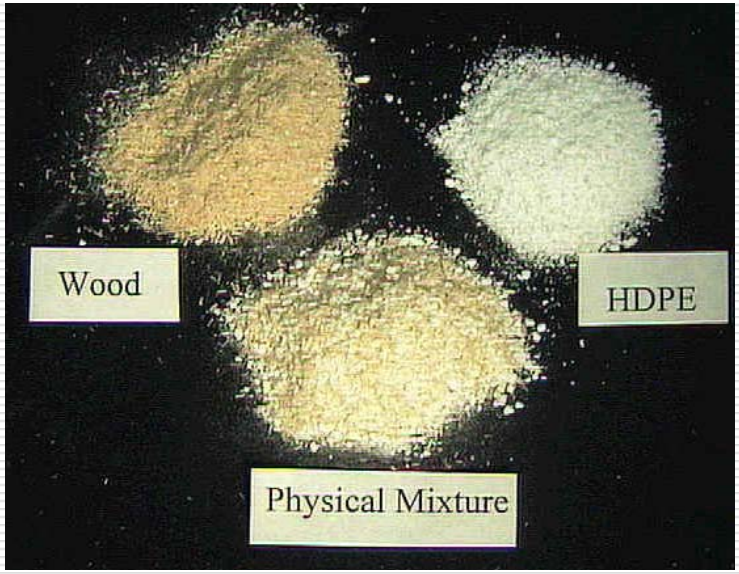
# Wood-Plastic Composites (WPCs)

Heterogeneous dispersed particulate composites comprised of 3 phases:

- **Particles:** Wood or other natural fibers
- **Matrix:** Thermoplastics
  - Polystyrene **(PS)**
  - Polyethylene **(PE, HDPE)**
  - Polypropylene **(PP)**
  - Polyvinyl chloride **(PVC)**
- **Additives…**




composites.wsu.edu/ navy/Navy1/materials.html

# Additives (up to 10%)

- Lubricants/Process aids
- Colorants
- **Coupling agents**
- Preservatives
- Antimicrobials
- Antioxidants
- Flame retardants
- Light (UV) stabilizers

# Issues…

- Durability
- Significant creep
- Thermo-expansion
- Weight/Strength

# Wood-Plastic Composites (WPCs)

- Mechanical and physical properties of WPC's may be engineered by changing phase characteristics, proportions and orientation, processing parameters, and **internal bonding**
- **Limitations:** composite design is limited by the naturally variable wood properties and the limited selection of thermoplastics that may be used with wood (melting temperature <200° C)

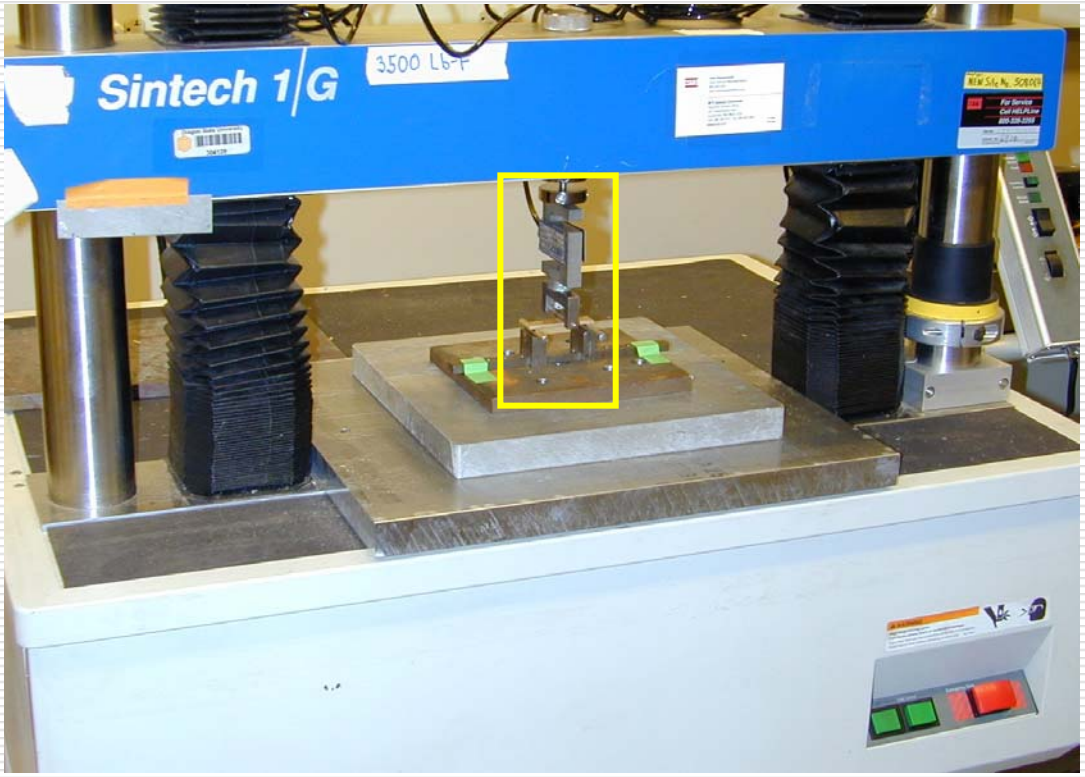
# Focus on the Internal Bond

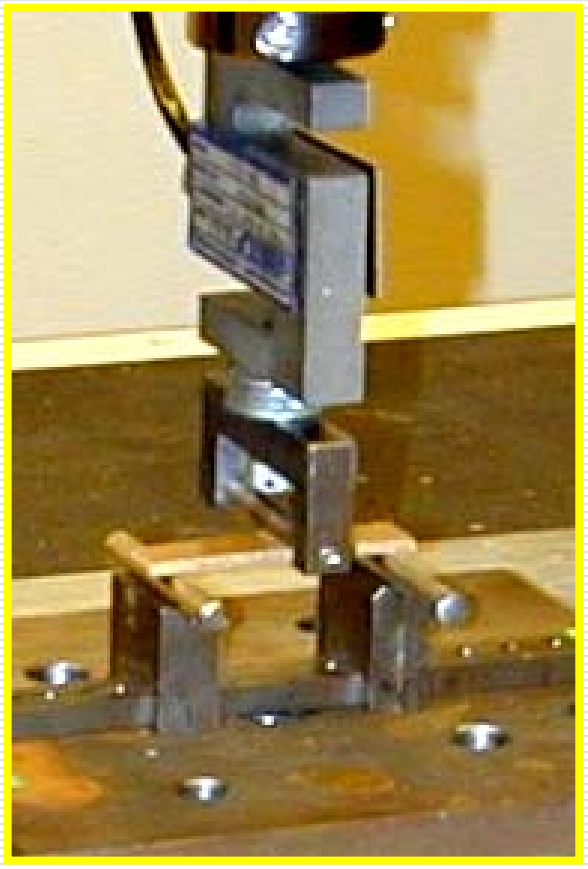
- Challenge: Bonding two dissimilar materials
  - Hydrophilic wood
  - Hydrophobic thermoplastics
- Facilitated by coupling agents

# Motivation

- Durability and mechanical performance of WPC's are decided on the μ-mechanical level
- Traditional testing methods offer indirect and limited insight to μ-mechanical performance, μ-damage accumulation and governing failure mechanisms
- Any significant progress in this field depends on better understanding of the composite performance and internal bond durability on the μ-mechanical level, and reliable modeling based on that understanding

# How much can flexural tests reveal about internal bonding?



# PROBLEM STATEMENT

- Can the external measures of the internal bond performance be correlated to micro-mechanical characteristics of the composite materials (WPC's)?
- Can WPC material degradation due to long term environmental exposure be simulated by mechanical loading?

# OBJECTIVES

To develop experimental procedures for multi-scale evaluation of micro-mechanical performance, governing failure mechanisms and micro-damage accumulation in WPC's

# OBJECTIVES

The specific objectives are to investigate:

- Statistical characterization of the local deformation and internal damage accumulation induced by degrading conditions;
- Correlations between the internal damage and the storage modulus;
- Correlations between the internal damage inflicted by mechanical loading and cyclic environmental exposure regimes: soak-dry and freeze-thaw.

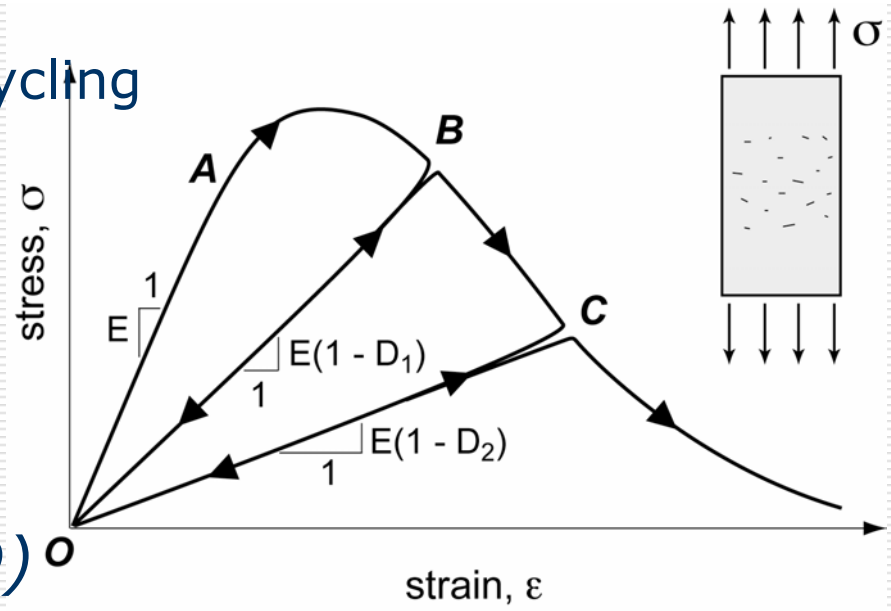
# Procedure: Multi-scale analysis

- Macro level: elastic modulus, static strength, storage modulus after degrading treatment
- Optical DIC analysis to identify strain concentrations on the specimen surfaces
- Digital Volume Correlation based on low resolution CT scans (20 μm/pxl) to identify internal strain concentrations (AOI for hi-res CT)
- High Resolution CT scans (3 μm/pxl) on the AOI's identified in the previous steps to reveal and characterize internal damage concentrations

# Damage characterization

Damage is defined as a degradation in microstructure due to an external or internal influence.

- mechanical stress
- moisture/temperature cycling
- chemical changes



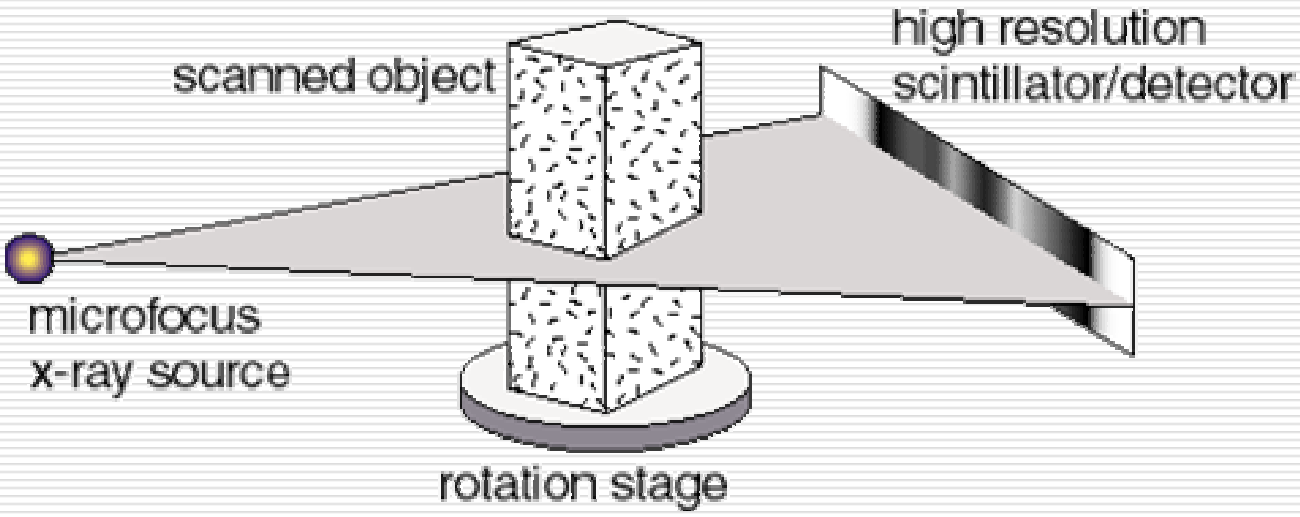

Define damage variable, $D$, such that $E = E_0(1 - D)$

# Nondestructive X-Ray Microtomography

Nondestructive measurement allows evaluation of the three-dimensional internal structure before and after the degrading treatments

Oregon State University

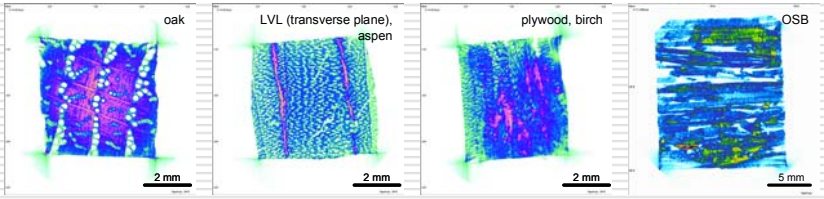
# X-ray Microtomography: Measurement principles



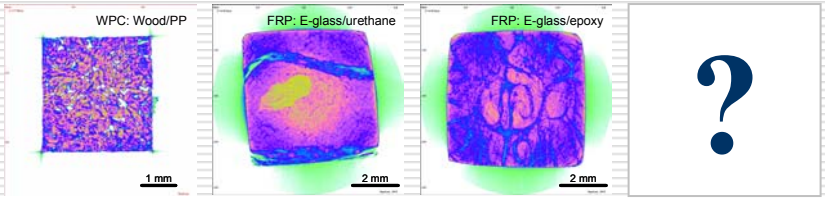

- 3D maps of x-ray absorption reconstructed from projection images
- High resolution through high performance x-ray source and detector

# Nondestructive 3-D X-ray microtomography

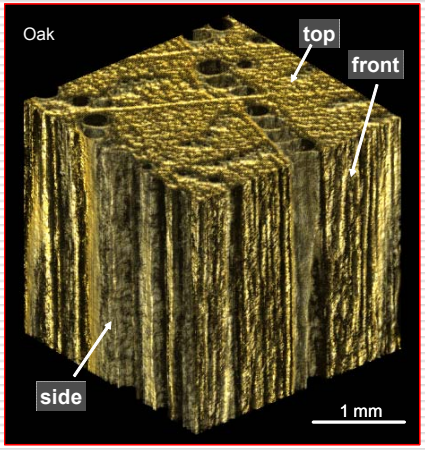
## Is it good for more than just cool pictures?

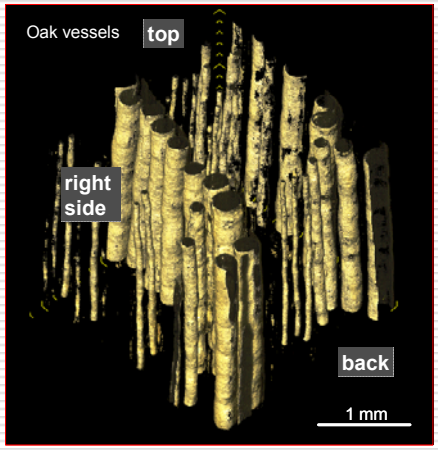



oak LVL plywood OSB




Wood/PP GF/Urethane GF/Epoxy Who's next?



# Nondestructive 3-D X-ray microtomography

Is it good for more than just cool pictures?

- Digital tools are available to identify visible features and different material phases (solid phases, voids etc.), quantify connected pore structure, and visualize complex microstructure
- Internal strains can be evaluated (Digital Volume Correlation)

# Digital Image Correlation
# Digital Volume Correlation

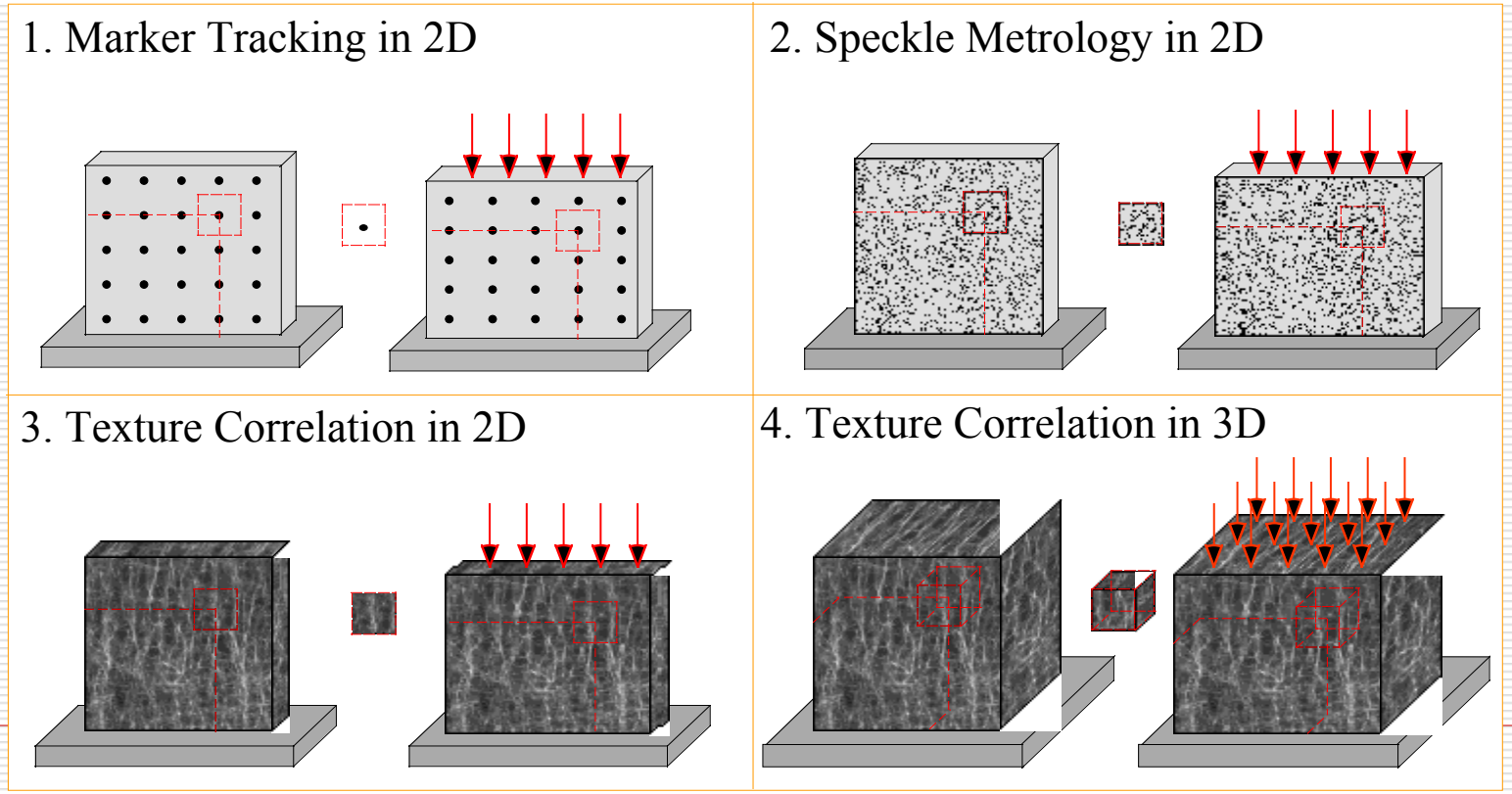
OSU
Oregon State
UNIVERSITY

# Types of Image Correlation

- In-plane deformations from single-camera surface images
- Out-of-plane deformations from multiple-camera surface images
- Volumetric deformations from tomographic data sets (Digital Volume Correlation)
  - A 3D extension of DIC
  - Applicable to materials with inherent texture
  - Porous materials, composites, large-scale microarchitecture

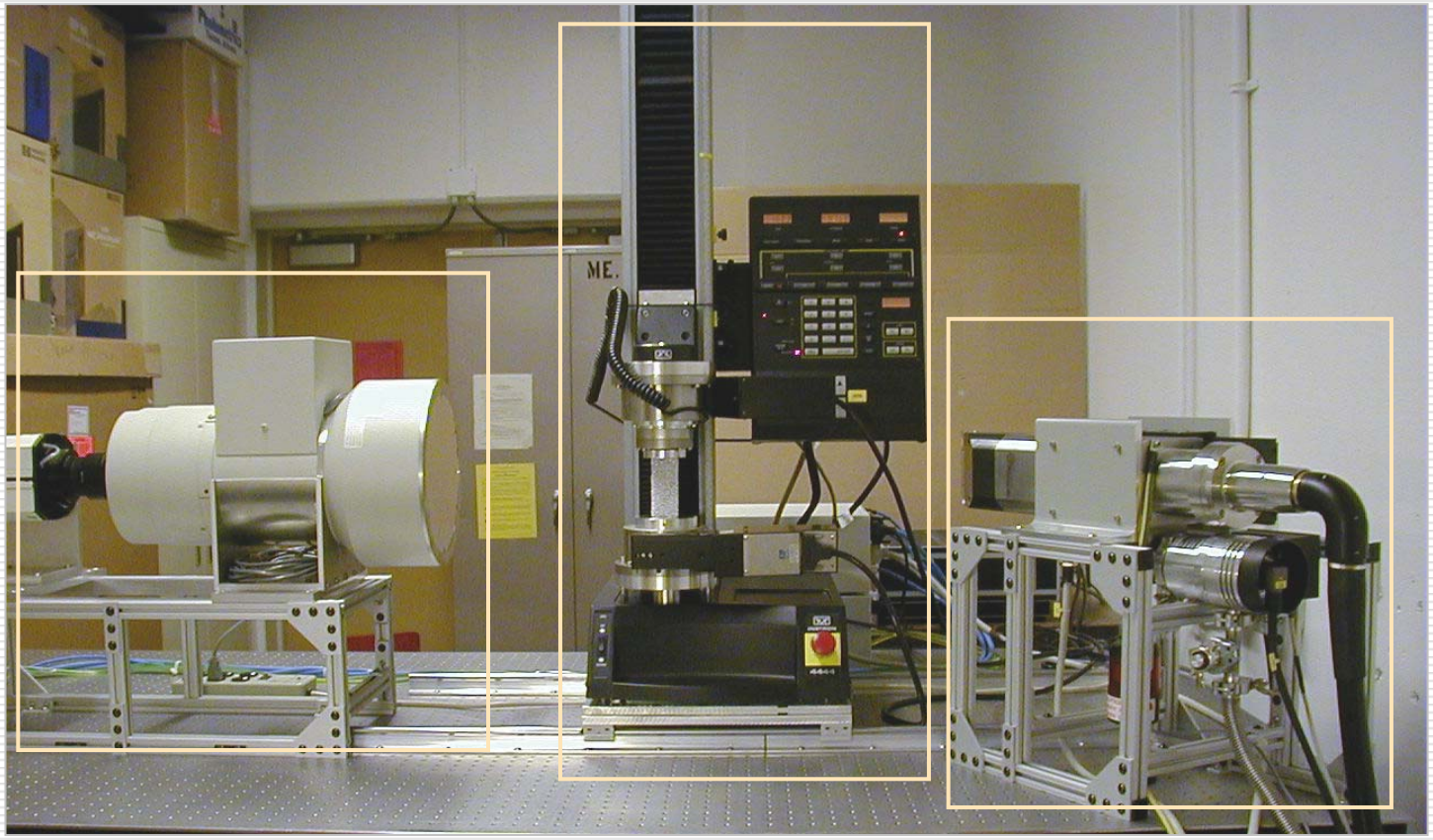
# Volumetric Strain Measurement - Concept

Strain is quantified using correlation methods that compare loaded and unloaded data volumes.



# Integrated Testing and Imaging

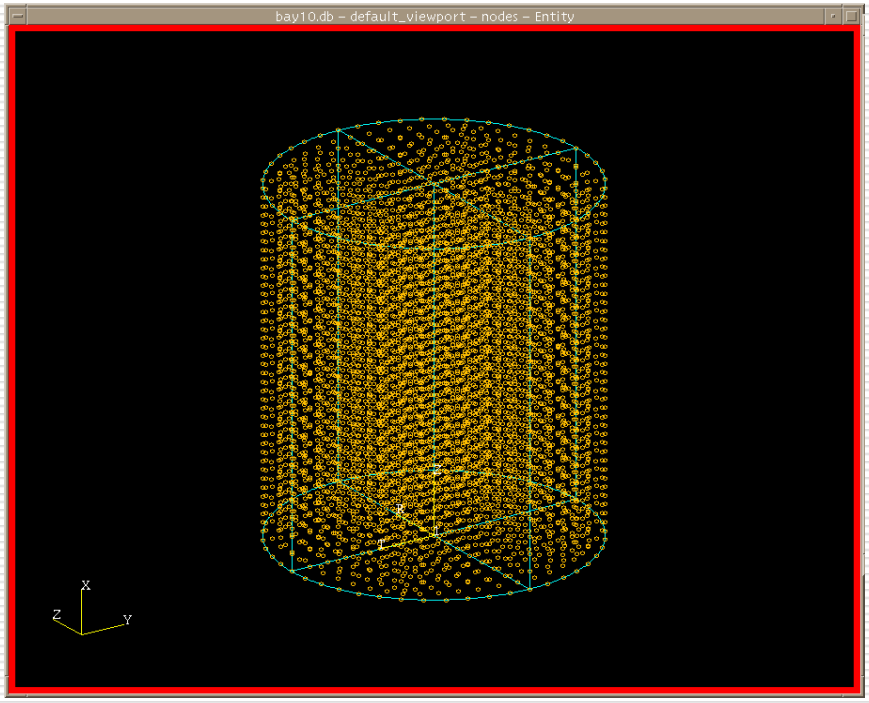
**Detector** **Sample Stage** **X-ray Source**



# Hardware Details

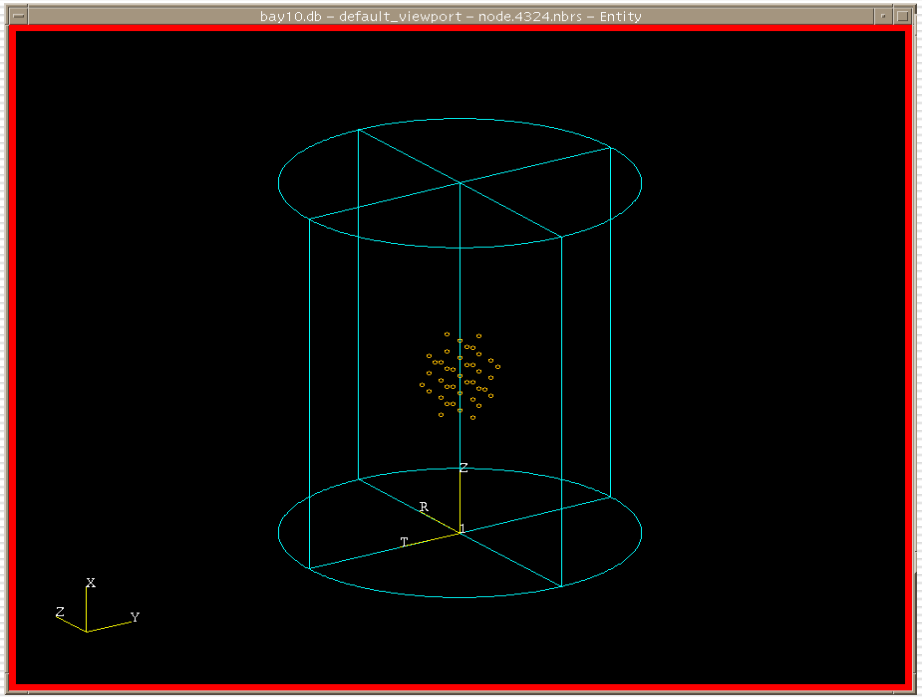
- **The x-ray source:**
  - FeinFocus 160 kVp
  - 10 micron focal spot
- **The detector:**
  - Thompson TH9438HX image intensifier
  - Retiga 1024x1280 10-bit CCD, lens coupled
- **The sample stage:**
  - Newport RV120 high load rotational stages, opposed
  - Instron 4444 load frame, 2000N capacity

# Strain Measurement - Details




**Displacement Vectors Measured at Many Points Throughout a Sample**

**Strain Tensors Calculated at Each Point From Groups of Displacement Vectors**



# Software Details

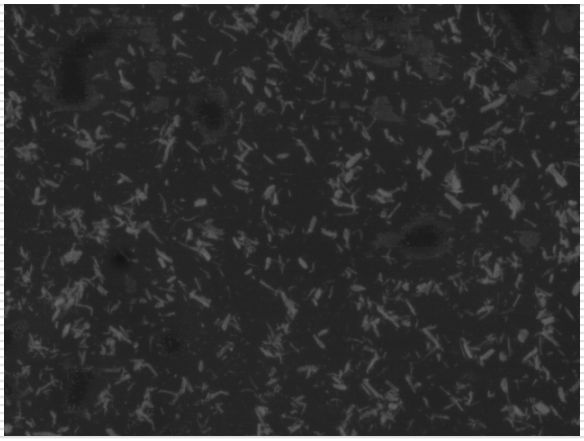
- **Image collection:**
  - Field of view between .5 and 10 cm
  - 1000 projections (360 degrees)
- **Reconstruction:**
  - Feldkamp-style filtered back-projection
  - ~ 1billion voxels, res. range 5 - 100 μm
- **Data volumes:**
  - Pojection images ~ 2GB, reconstruction volumes ~1 GB
  - Collection ~ 20 min (PC/GPIB based)
  - Reconstruction (2 vols) ~14 hrs (2 proc Sun)

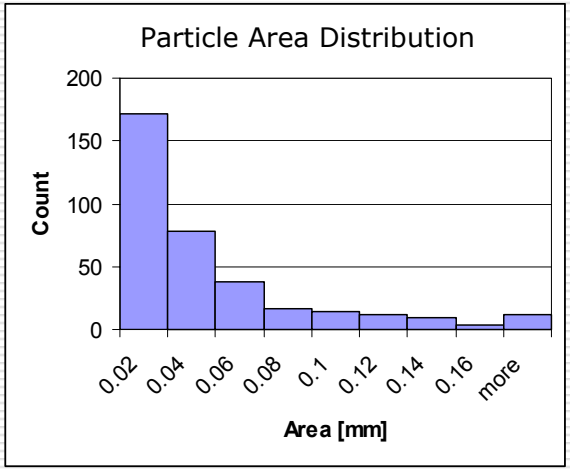
# MATERIAL

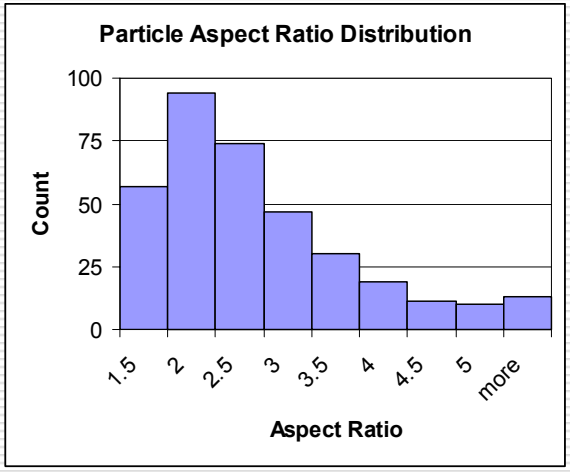
# Component Materials

- Wood flour
  - 40 mesh pine from American Wood Fibers
  - Oven dried prior to use for 24 hours at 103°
- Plastic
  - High Density Polyethylene:  BP Solvay B53 35H FLK, melt flow = 0.49 g/10 min
- X-ray attenuation **contrast enhanced** by doping the matrix with chemically inert gold nano-spheres

# PARTICLE CHARACTERIZATION





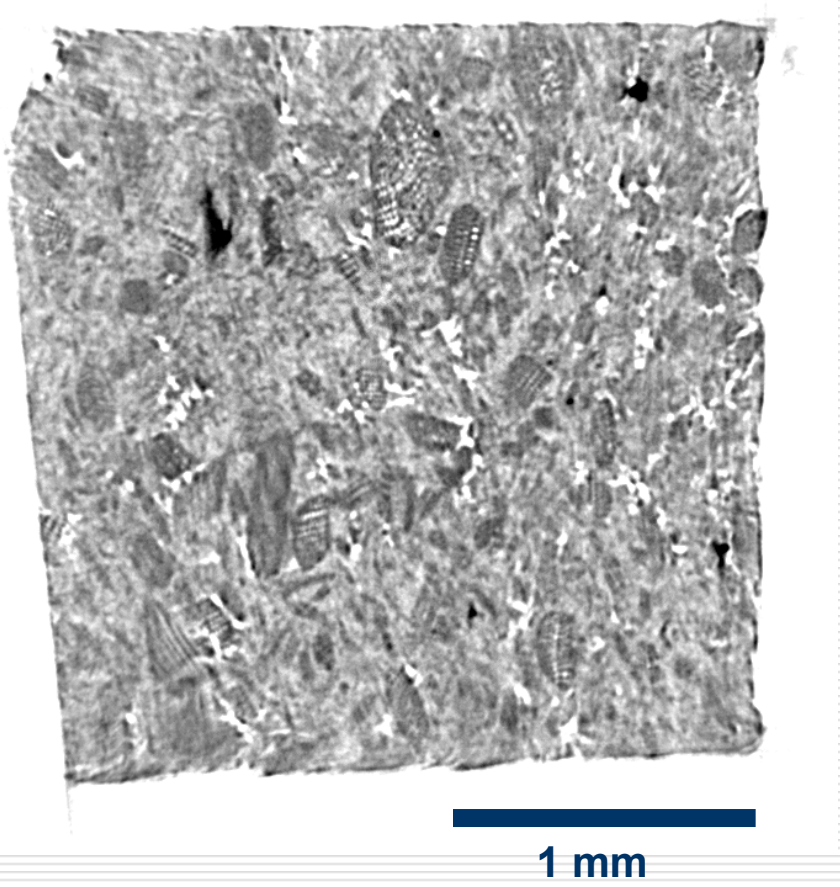



# Phase Characteristics

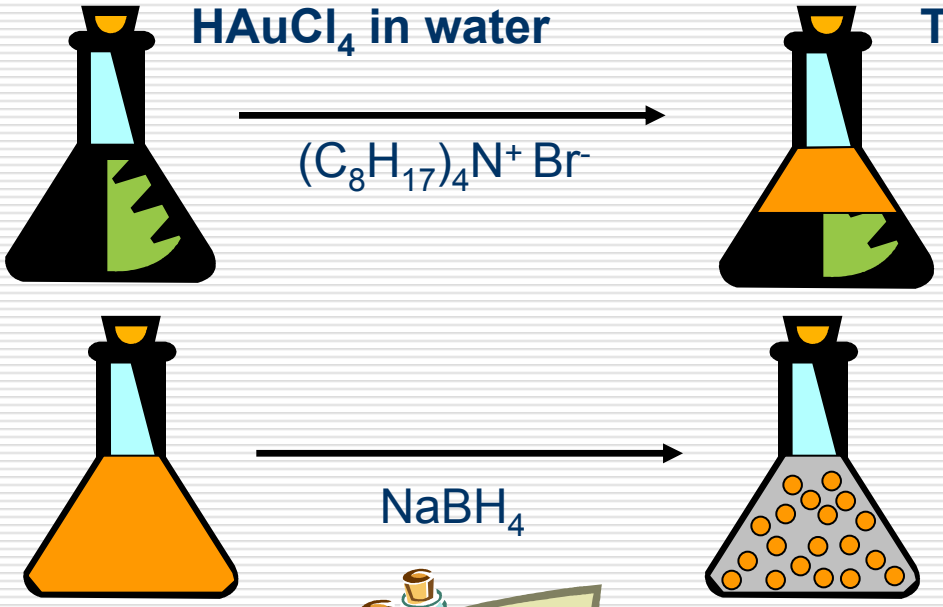
**Density profiles**

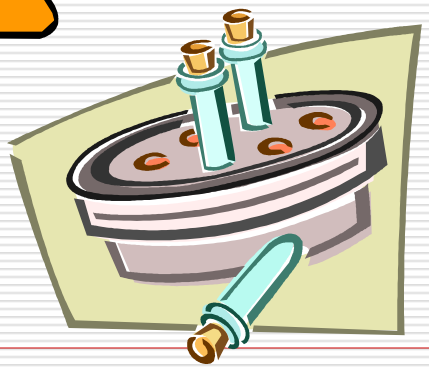
| | |
|---|---|
| Wood cell | 1.5 g/cc |
| HDPE | 0.95 g/cc |
| PVC | 1.3 to 1.58 g/cc |
| PP | 0.9 g/cc |

Wood particles and th polymer matrix material have similar densities and x ray absorption levels (CHO).



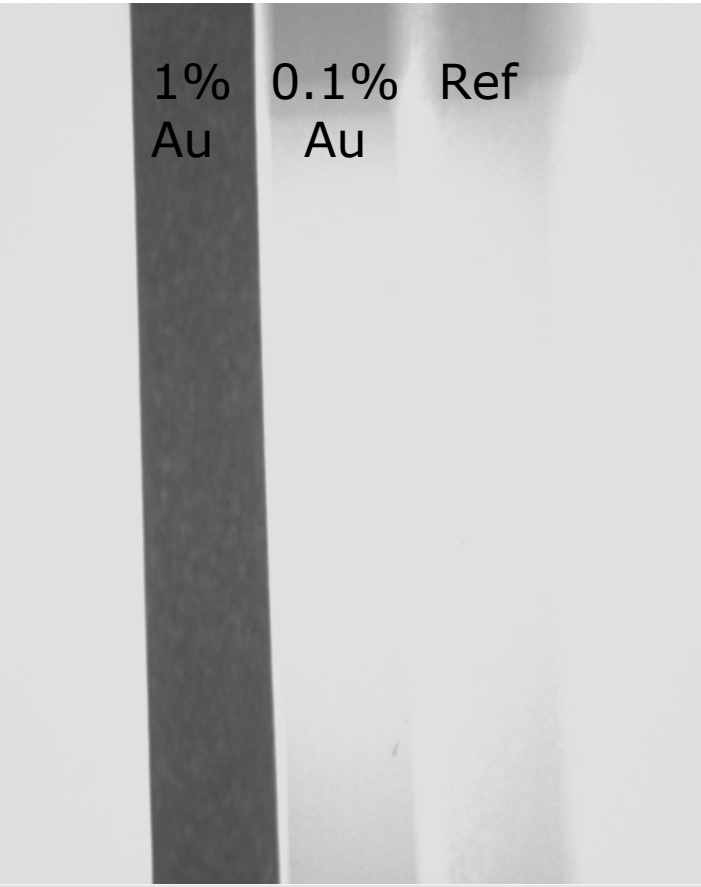
# GOLD NANOSPHERES

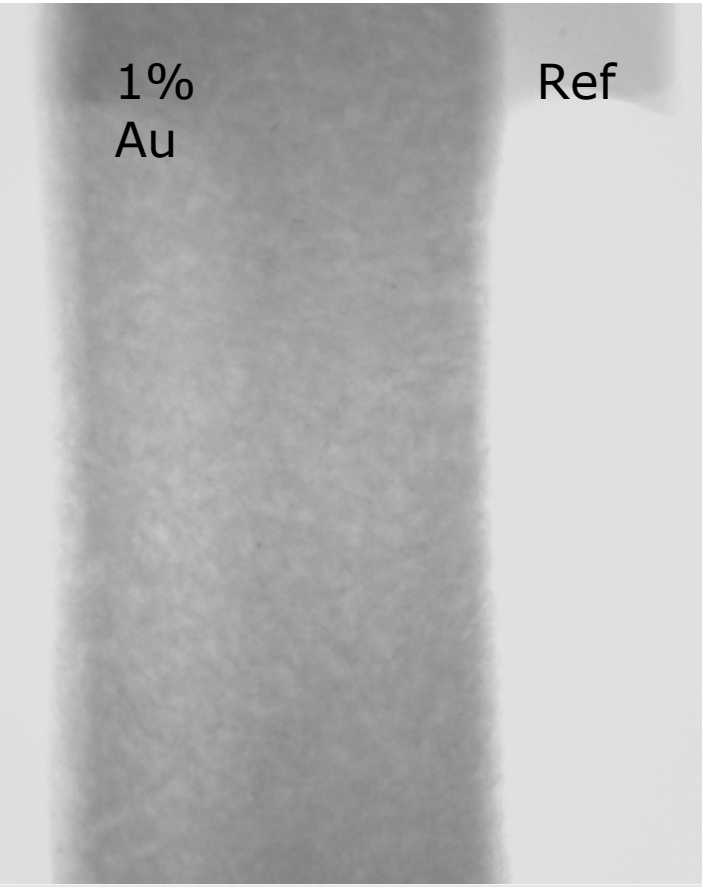


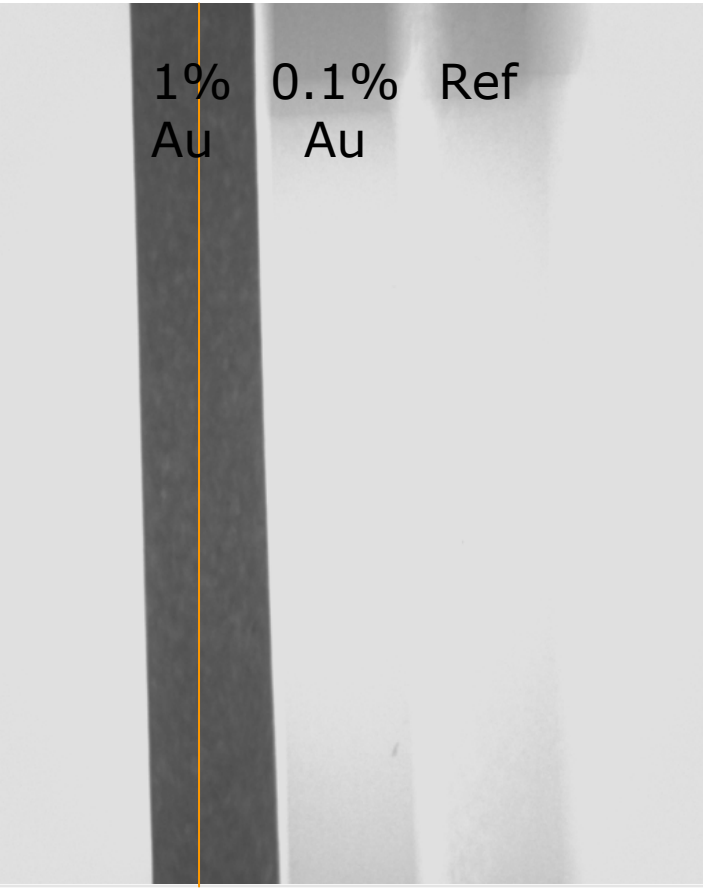
Centrifuge to remove gold nano-spheres
Add to HDPE in Brabender Plasticorder mixer

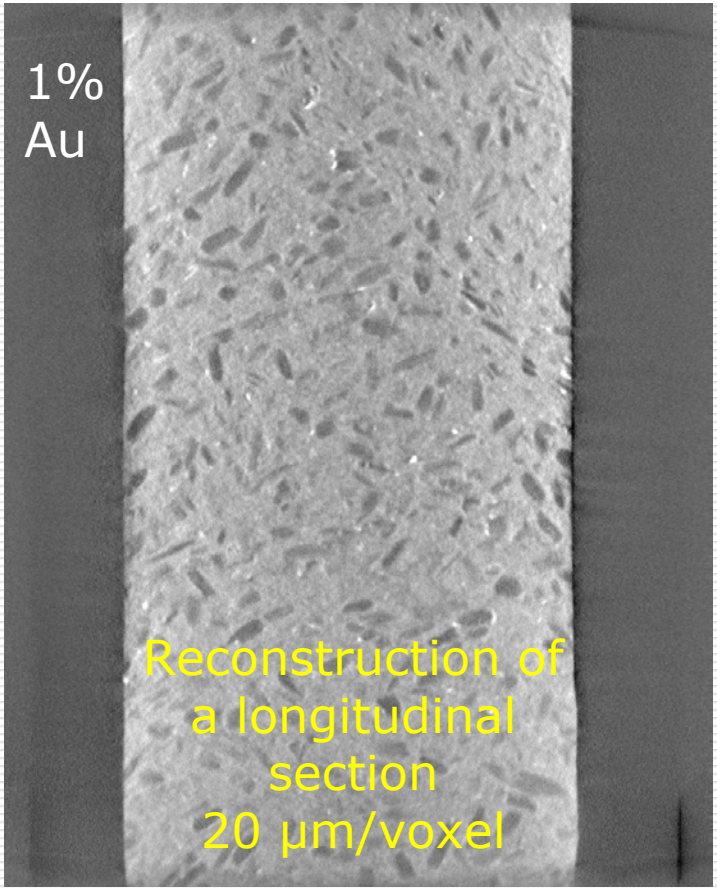
# Enhanced CT contrast



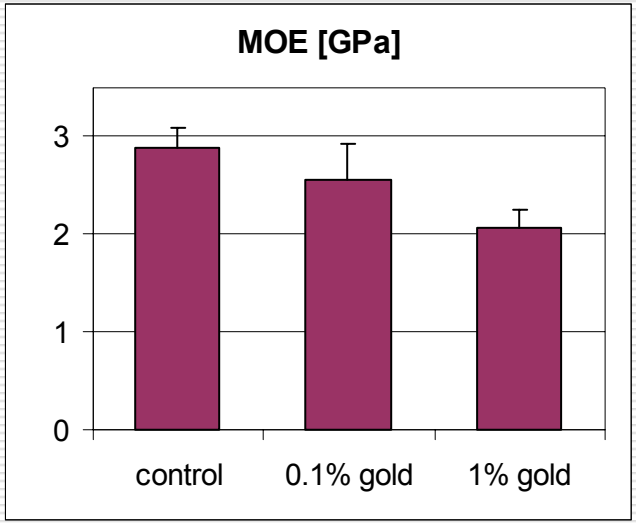



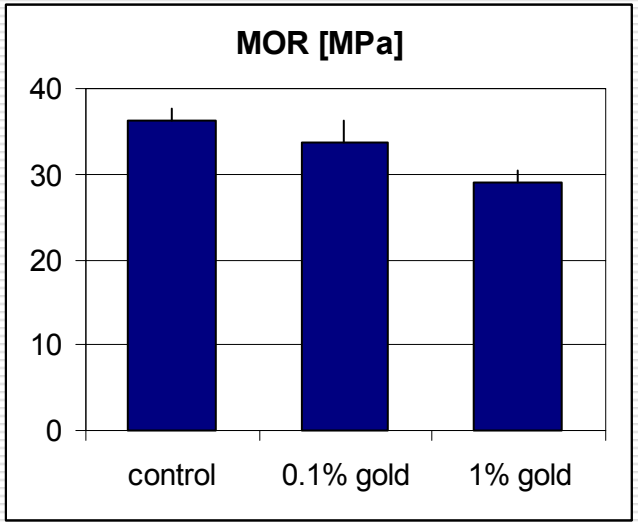
# Enhanced CT contrast

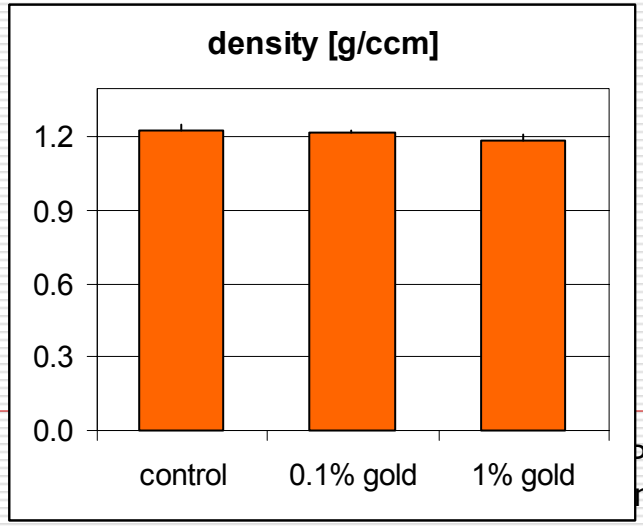


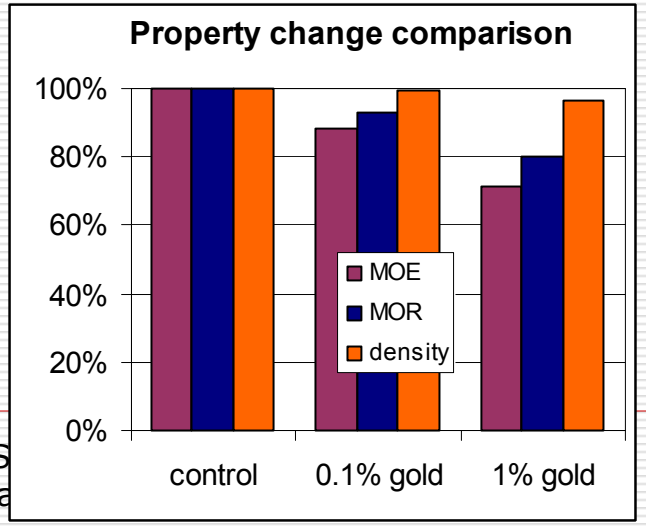



# Enhanced CT contrast: with a price tag…



# Blending

## Brabender Intelli-Torque Plasticorder

- Melt HDPE at 170° C
- Add gold n-spheres
- Add wood flour
- Mix 10 min
- Remove and store for compression molding

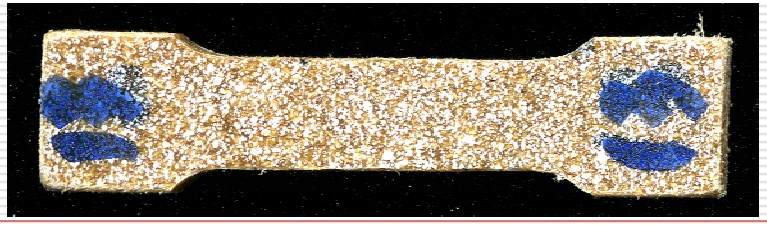
# Pressure molding

## Carver Press

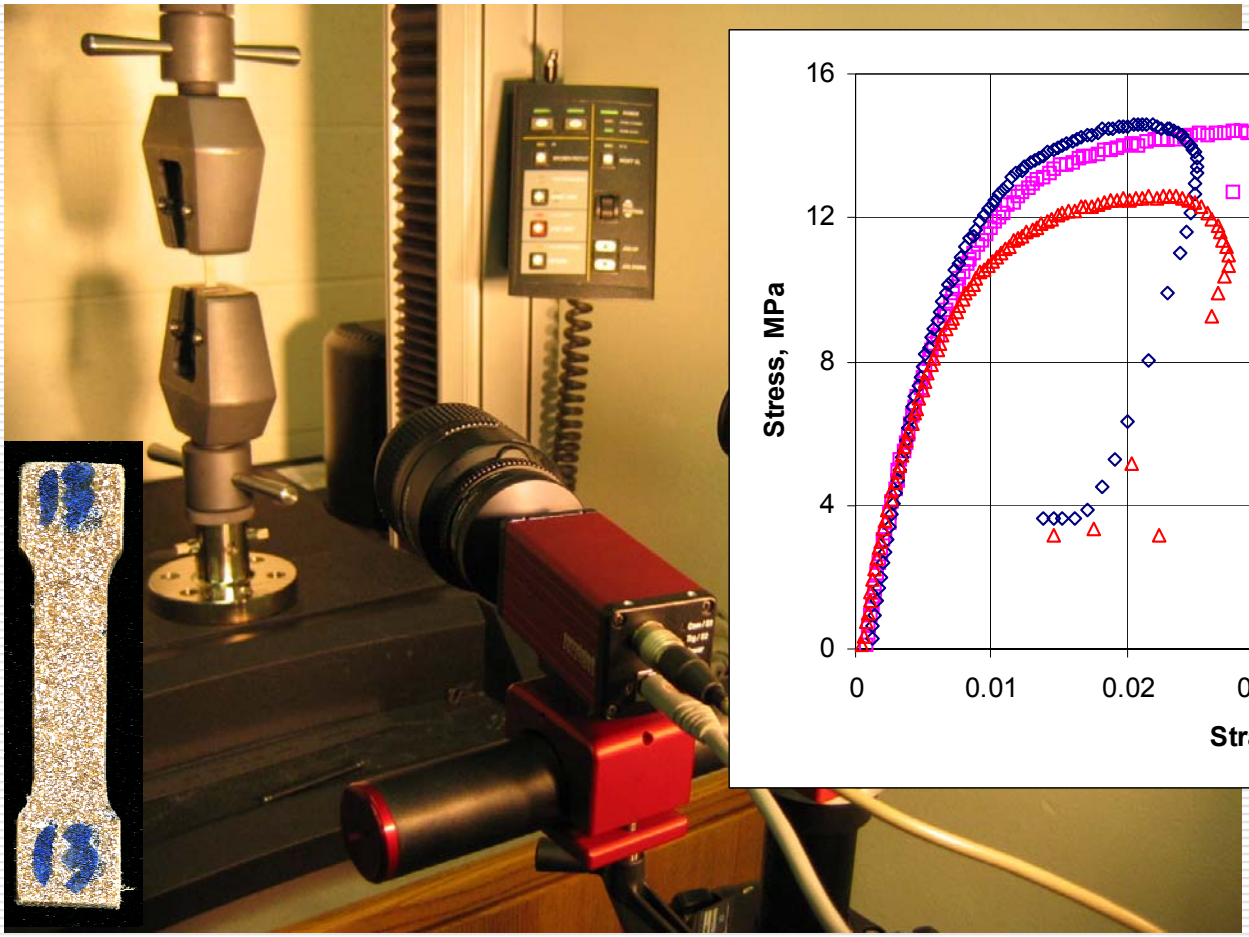
- Mold: 101.6 x 101.6 x 2 mm
- Temperature: 185$^{o}$ C
- Preheat time: 10 min
- Press time: 10 min
- Press pressure: 344.8 kPa
- Cooling pressure: 344.8 kPa

# Specimens

- 10 specimens for each wood-PE composite board
- Specimen size
  - L: 54.5 ±2.0 mm
  - W: 12.5 ±2.0 mm
  - (w: 9.2 ±0.1 mm)
  - t: 2.6 ±0.5 mm

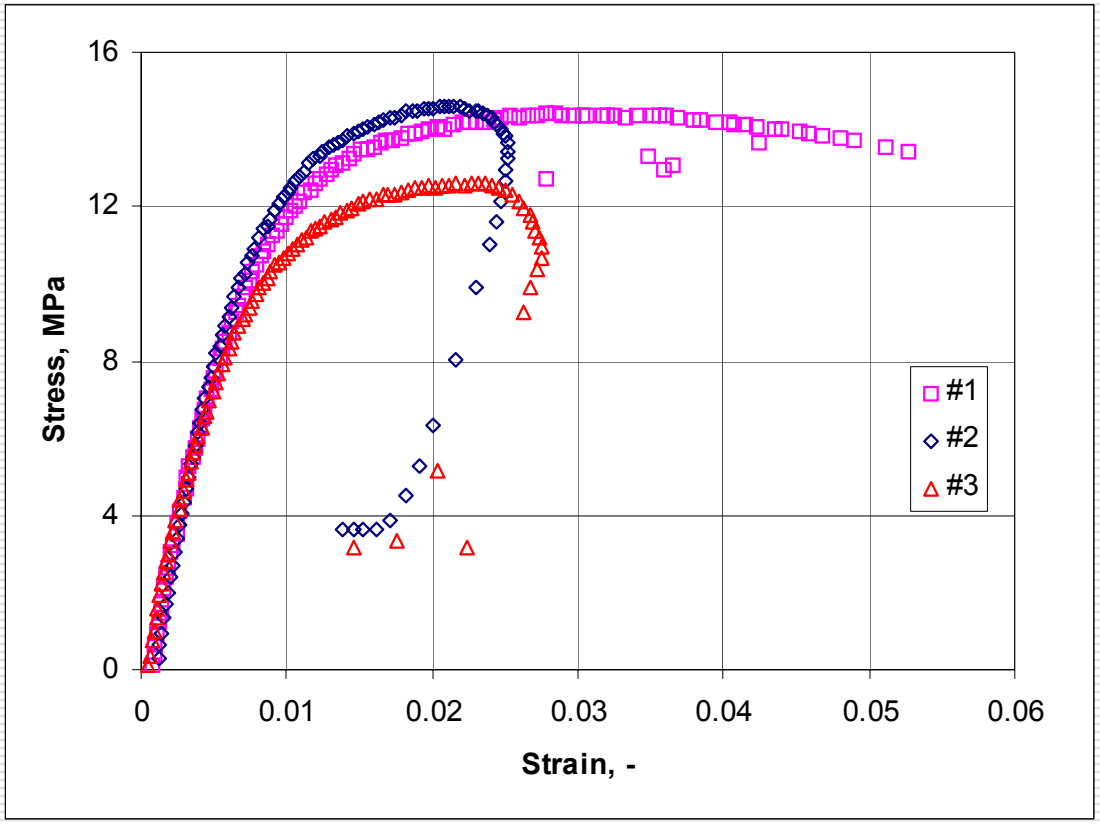


# Static tensile tests



# Static tensile tests

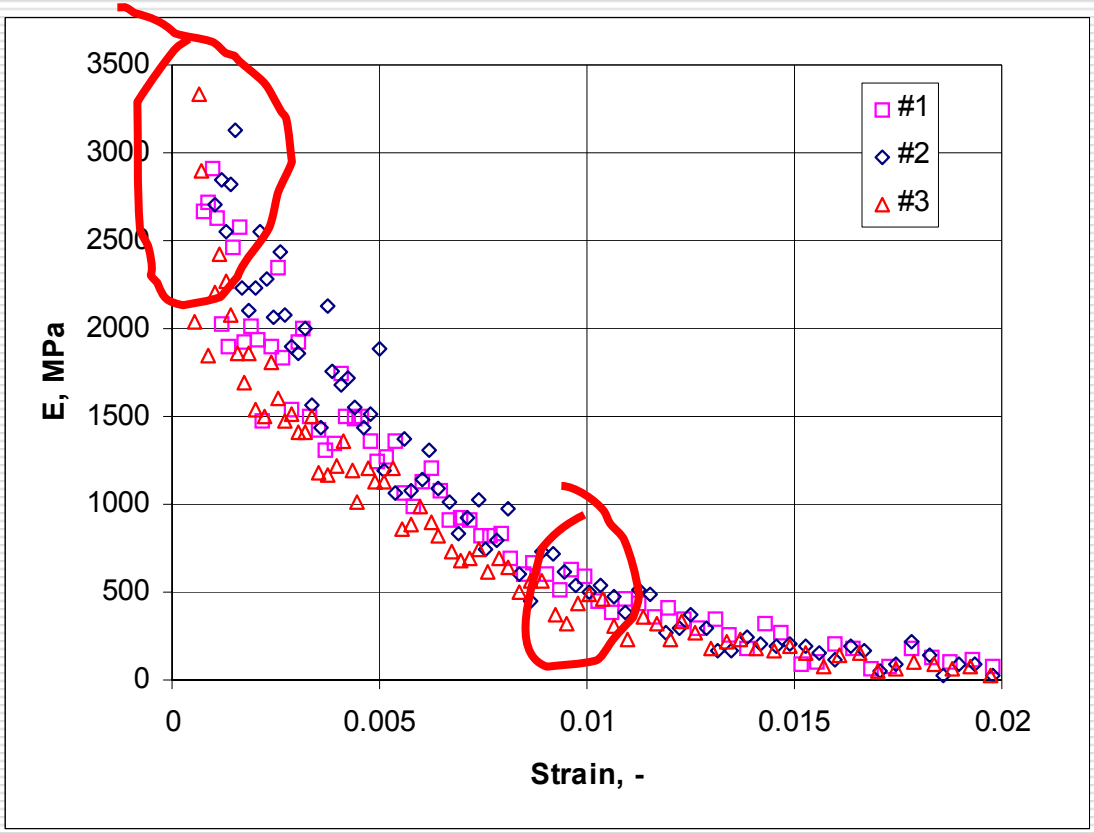
| | E* | UTS |
|---|---|---|
| # | *MPa* | *MPa* |
| 1 | 1755 | 14.80 |
| 2 | 1934 | 14.68 |
| 3 | 1527 | 12.71 |




***linear fit to a section of non-linear data**

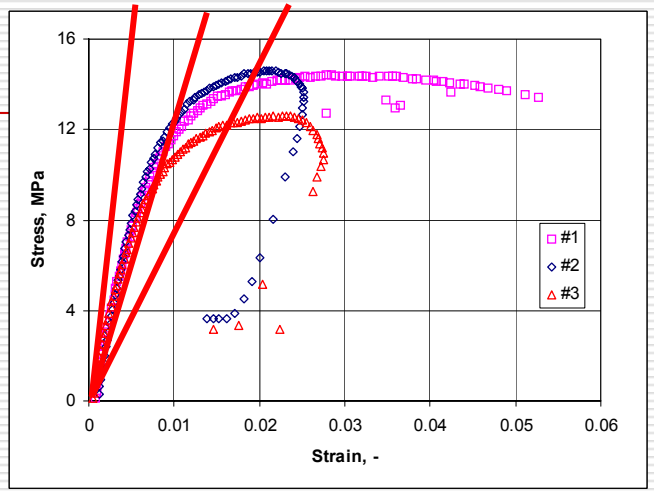
# Static tensile tests: nonlinearity

| | $E_{tan}$* | $E_{1\%}$* |
|---|---|---|
| # | *MPa* | *MPa* |
| 1 | 3131 | 487 |
| 2 | 3846 | 481 |
| 3 | 2776 | 387 |

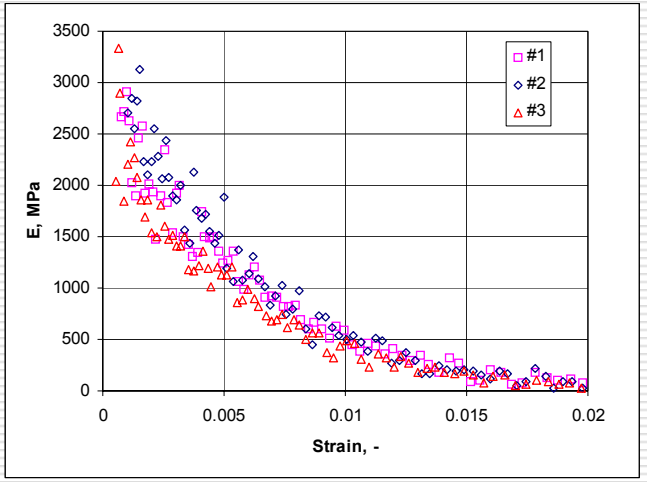



***from exponential fit to E(eyy) data**

# Static tensile tests: nonlinearity



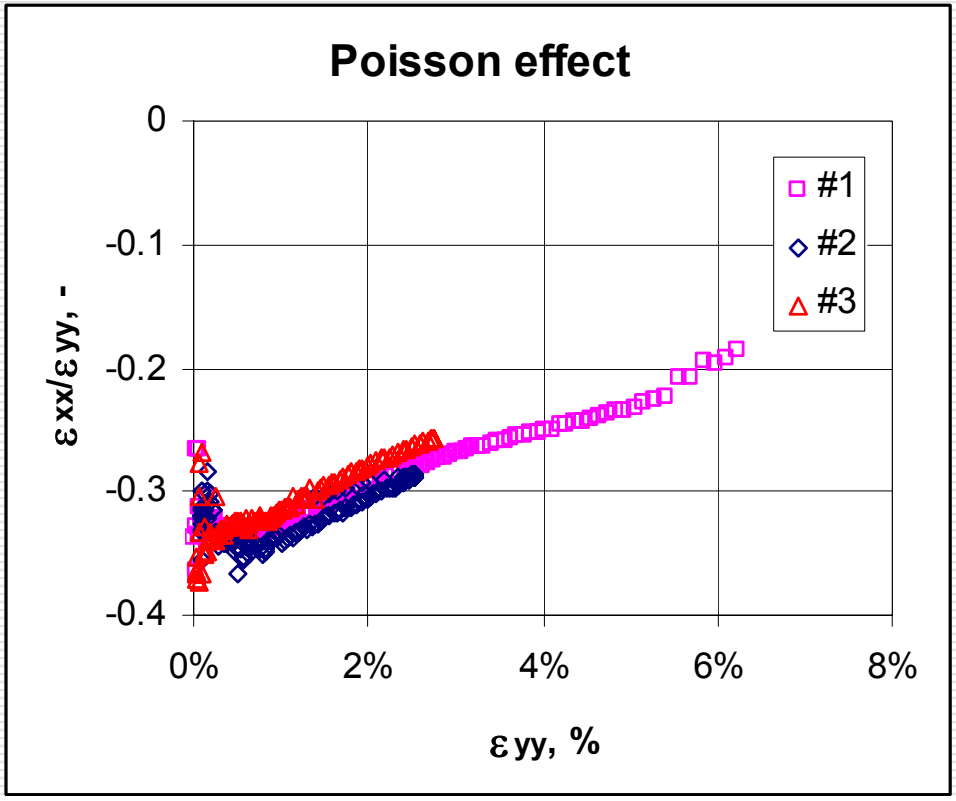

| | $E_{tan}$* | $E_{sec1\%}$ | $E_{sec2\%}$ | $E_{1\%}$* |
|---|---|---|---|---|
| # | *MPa* | *MPa* | *MPa* | *MPa* |
| 1 | 3131 | 1221 | 721 | 487 |
| 2 | 3846 | 1242 | 731 | 481 |
| 3 | 2776 | 1082 | 629 | 387 |




***from exponential fit to E(eyy) data**

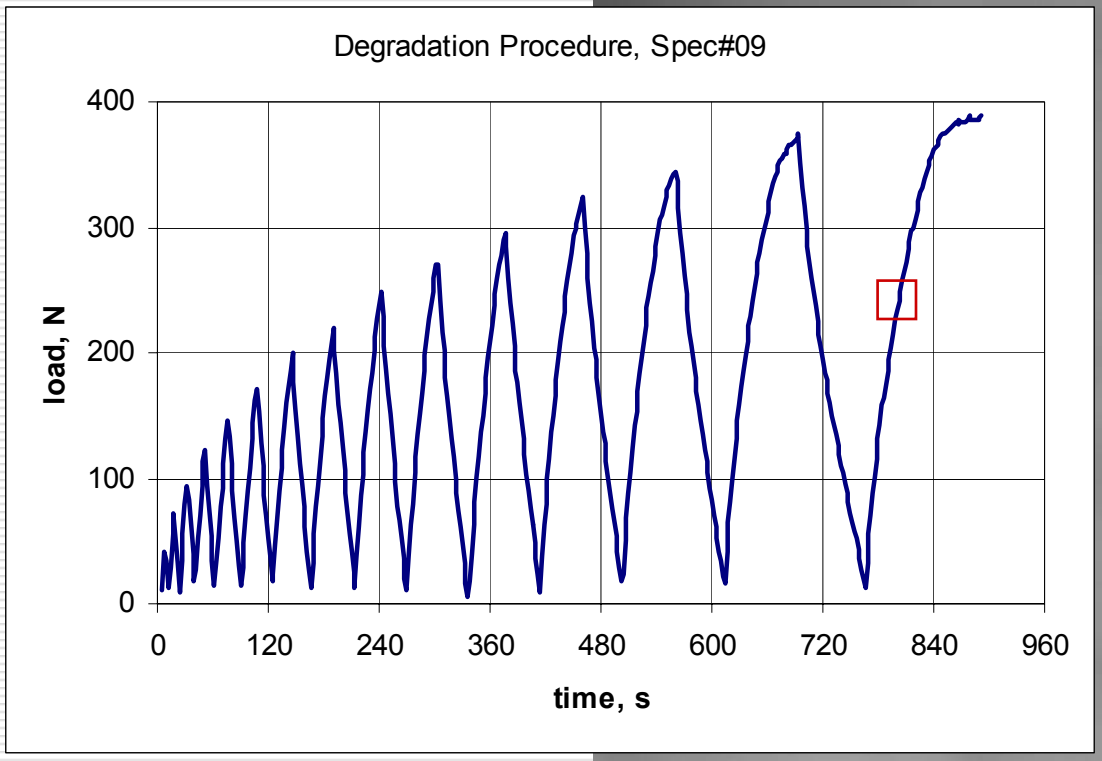
# Static tensile tests: Poisson effect

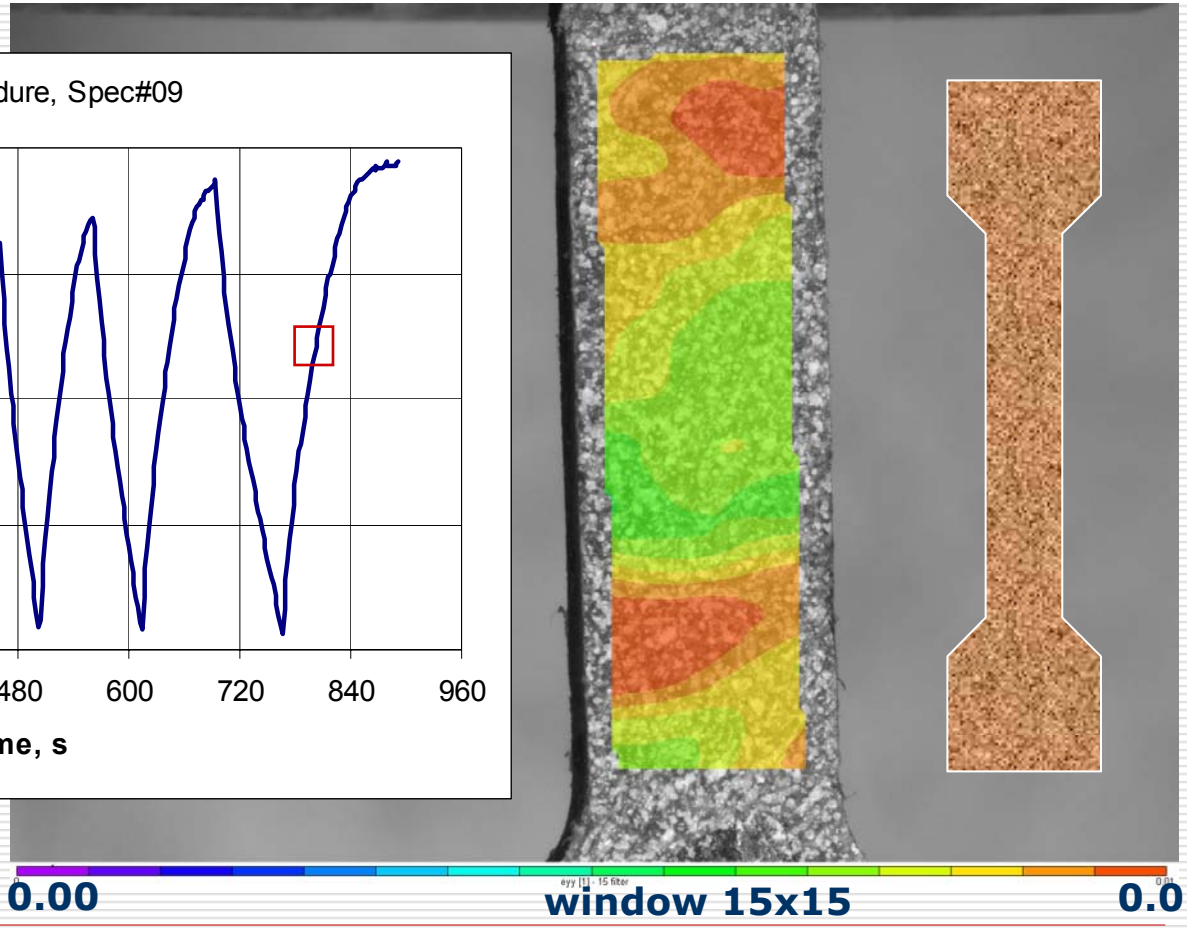
| | $\nu_{0\%}$* | $\nu_{2\%}$* |
|---|---|---|
| # | - | - |
| 1 | -0.3516 | -0.2959 |
| 2 | -0.3597 | -0.3033 |
| 3 | -0.3412 | -0.2805 |



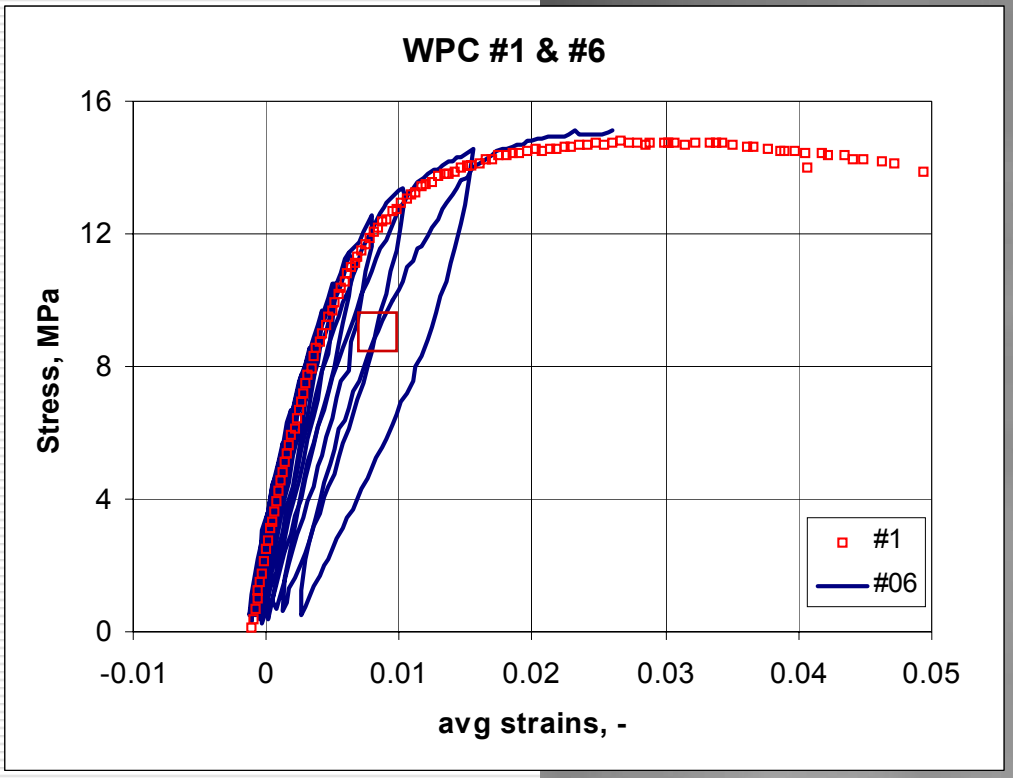

***linear fit to a section of experimental data (0.5% - 2.5%)**

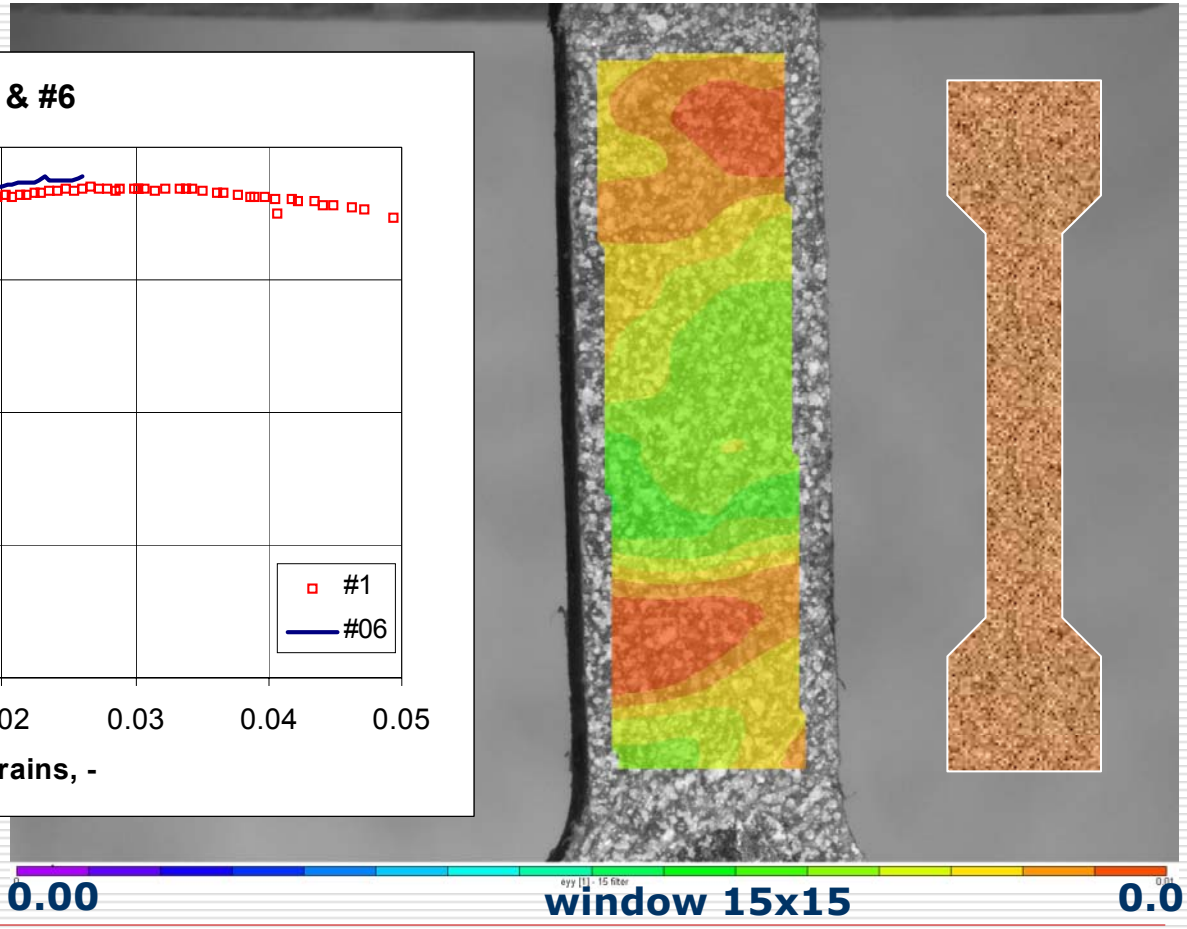
# Degrading procedure

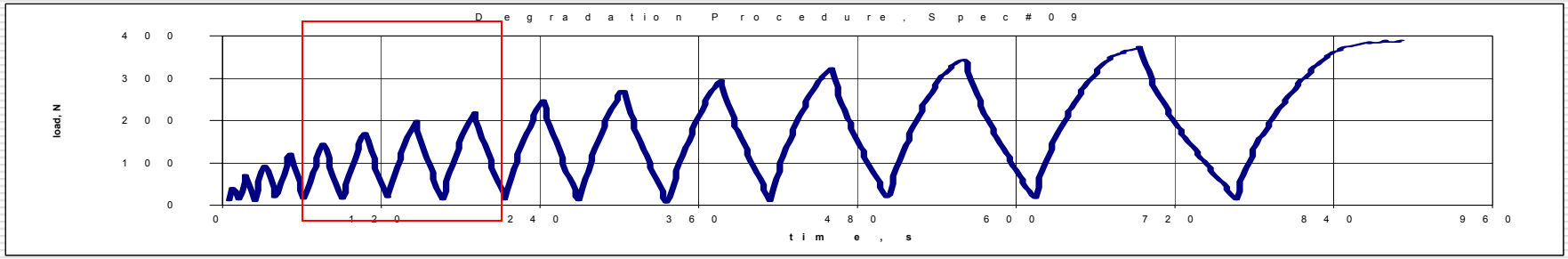


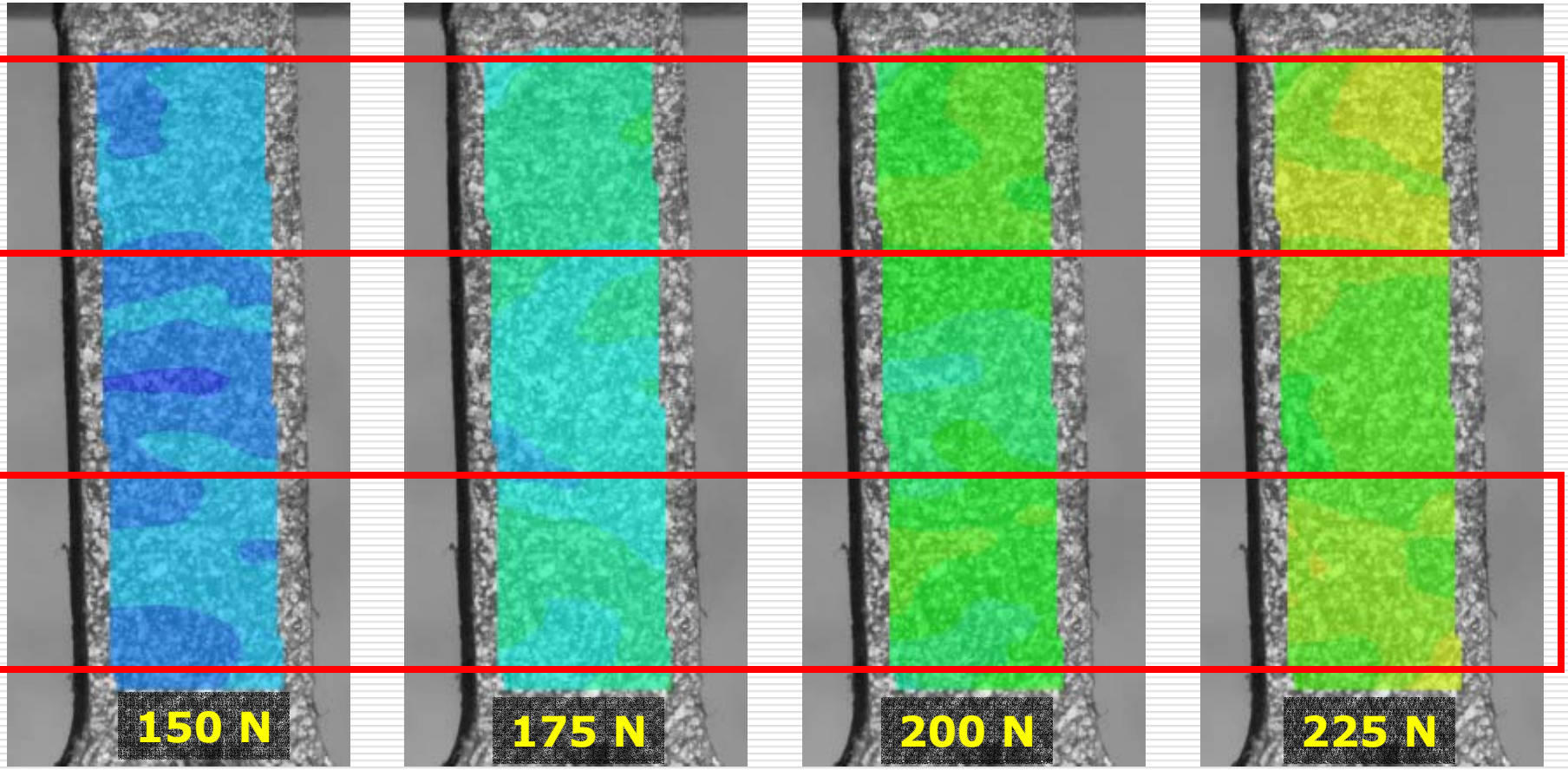



# Degrading procedure

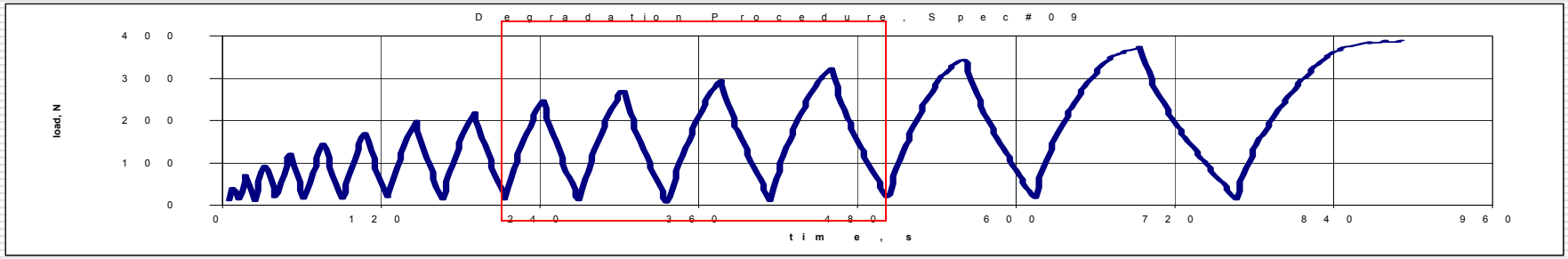


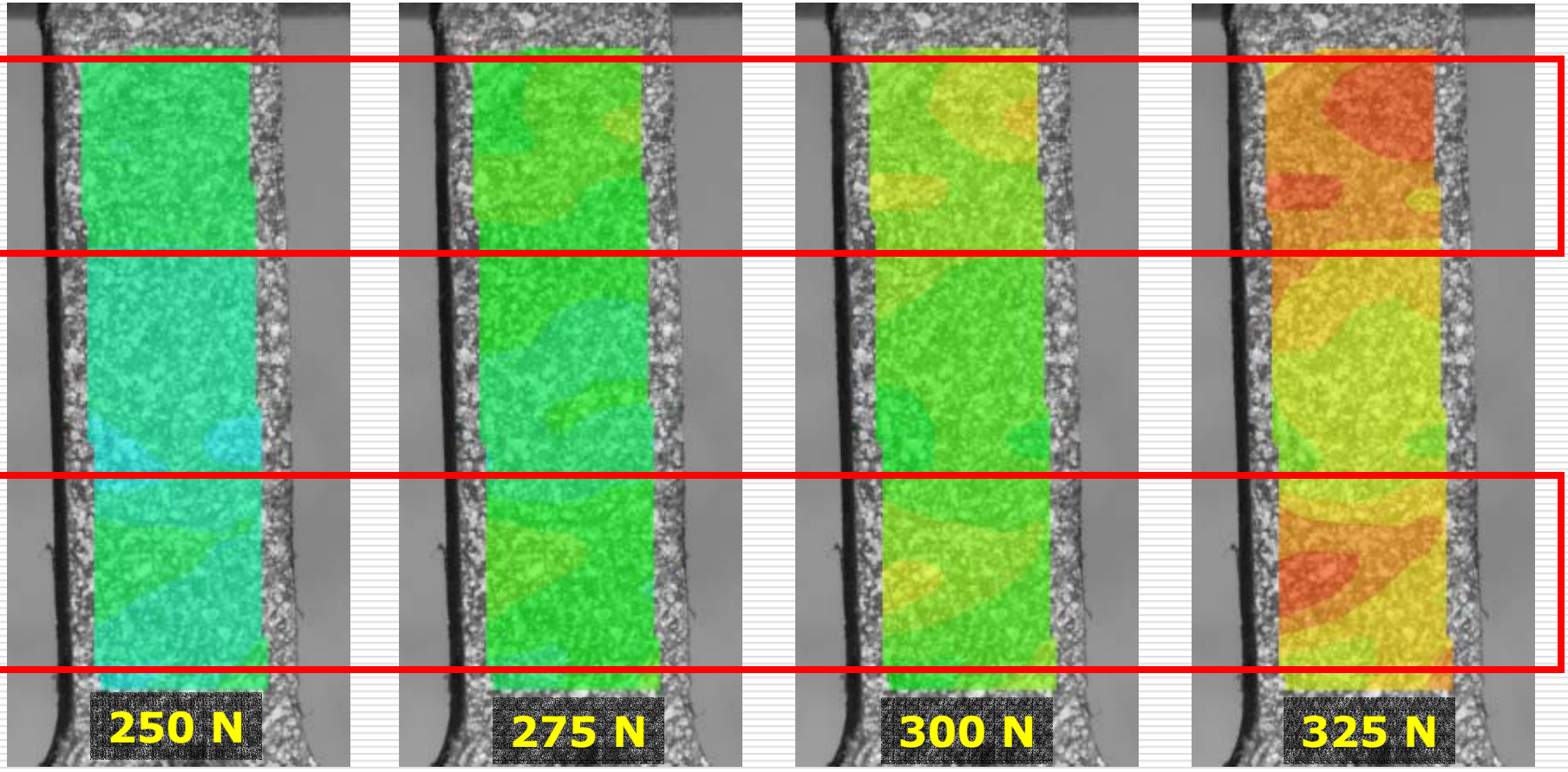



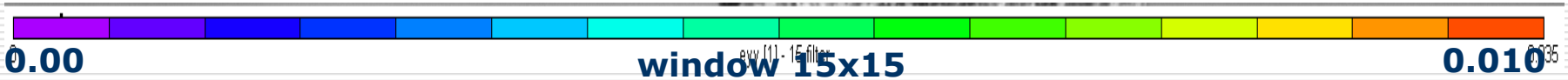

Degradation Procedure, Spec#09
load, N
400
300
200
100
0
0
120
240
360
480
600
720
840
960
time, s

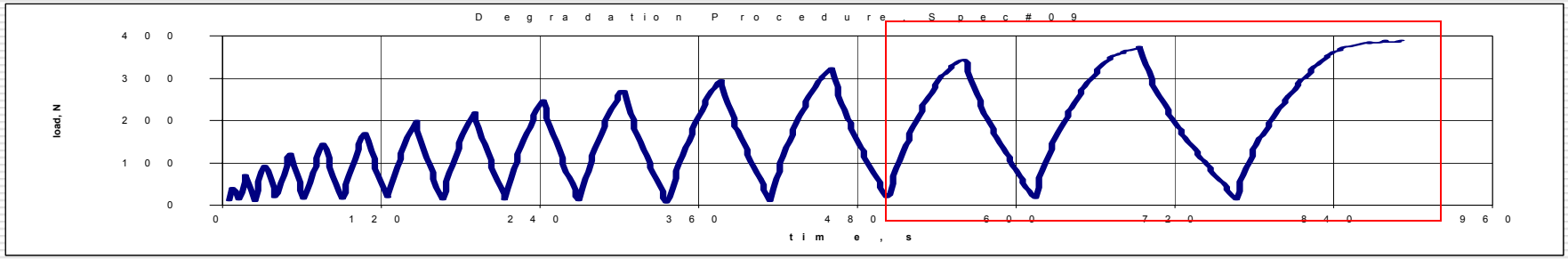

150 N
175 N
200 N
225 N

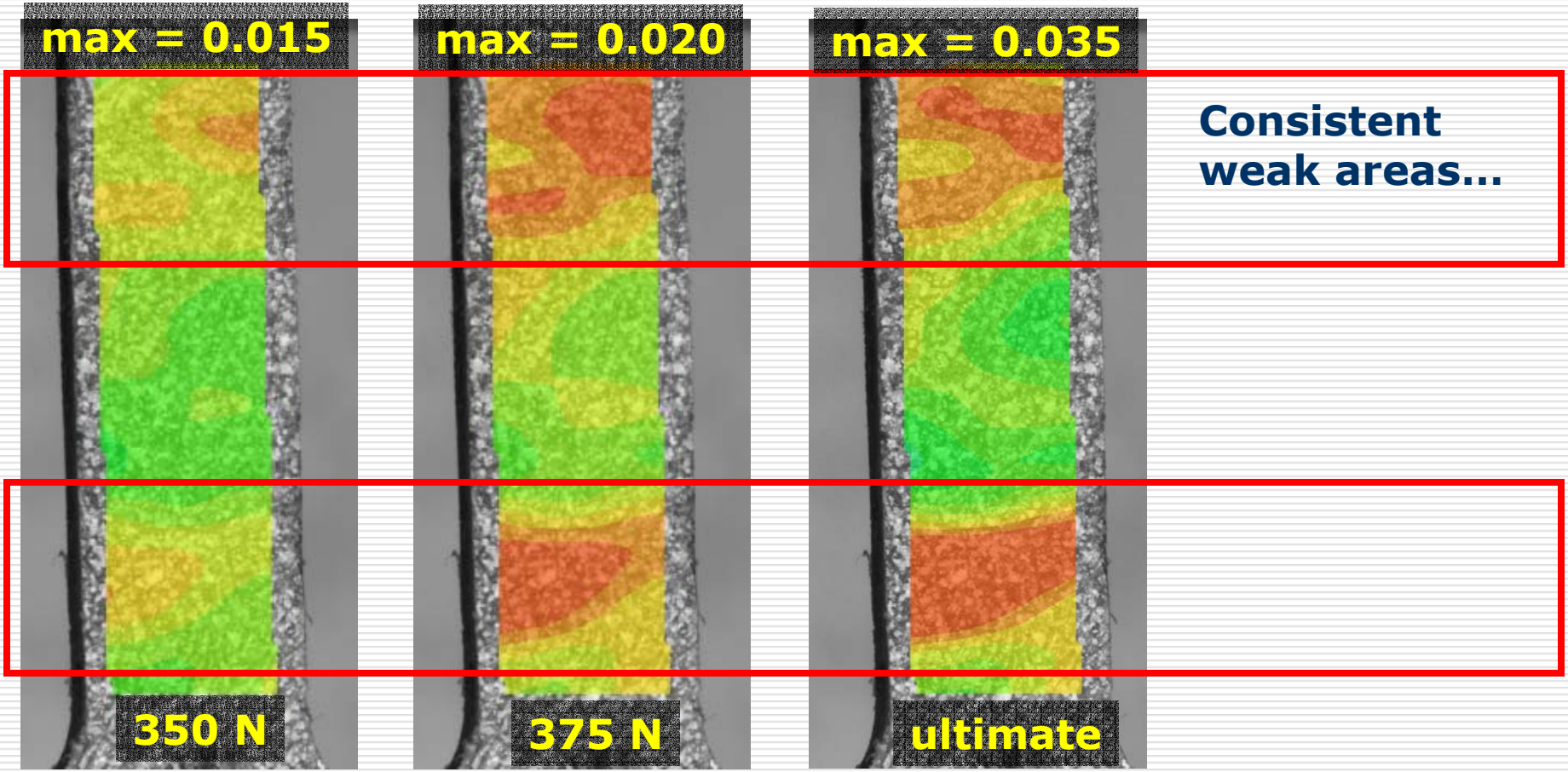

0.00
window 15x15
0.005


Degradation Procedure, Spec #09
load, N
400
300
200
100
0
0
120
240
360
480
600
720
840
960
time, s

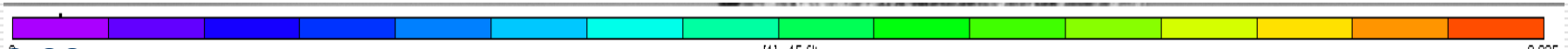

250 N
275 N
300 N
325 N


0.00
window 15x15
0.010

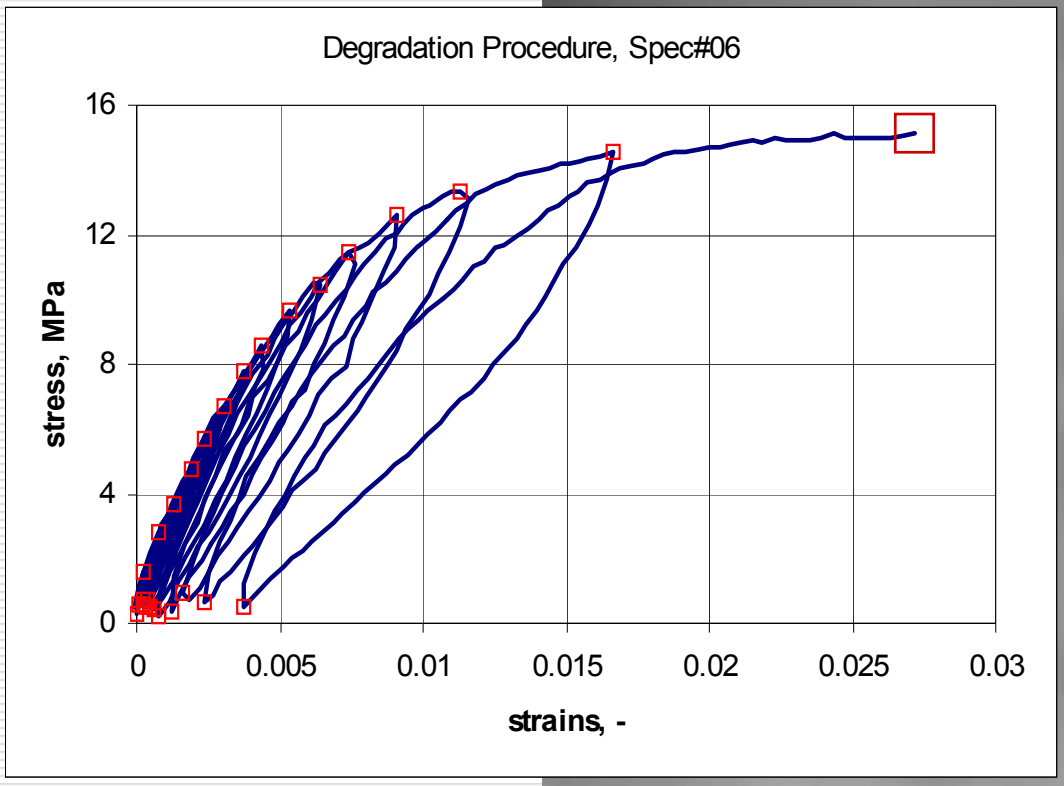


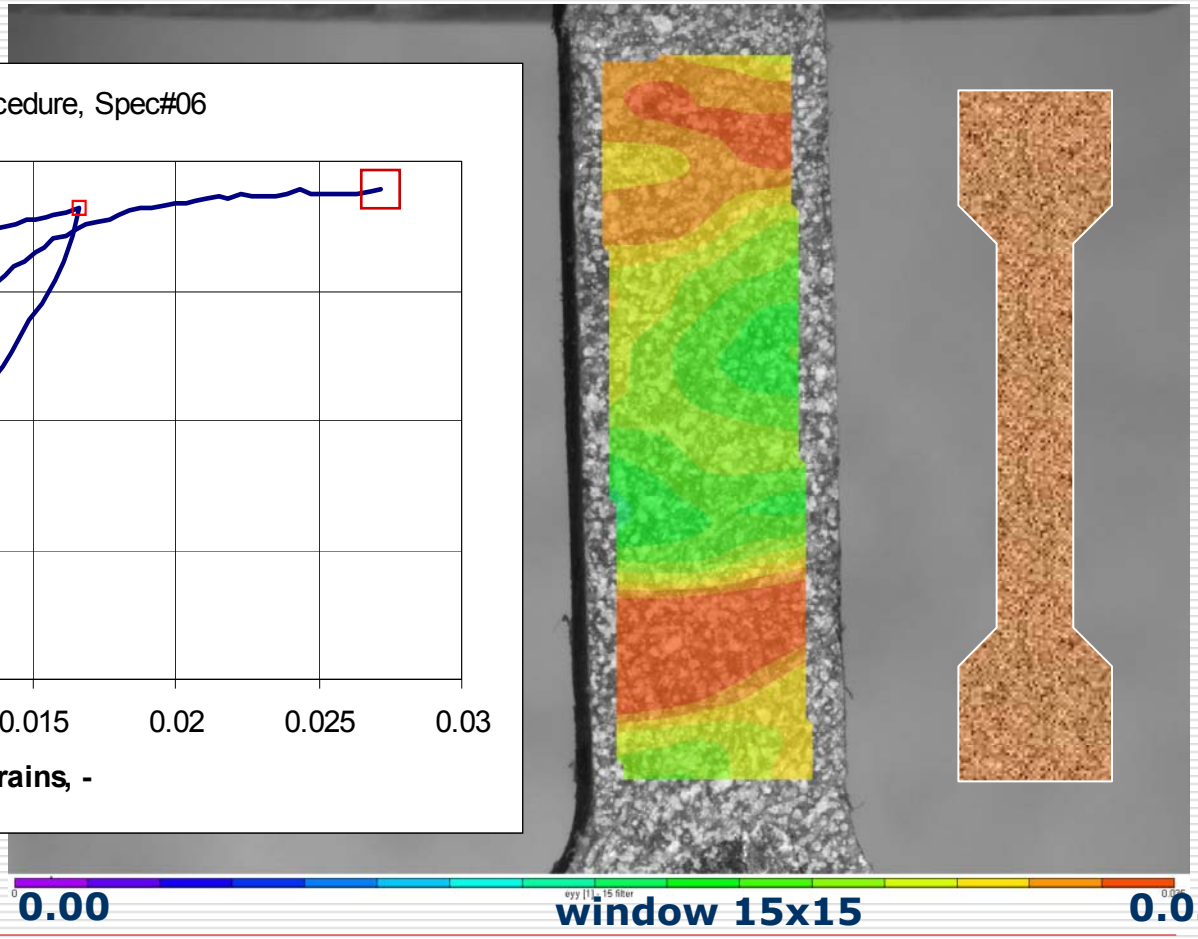




**Consistent weak areas…**



# Degrading procedure



# Work in progress...

# Acknowledgement

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# Questions?

# Preliminary Conclusions

- Digital images carry wealth of quantitative information…
- Find an alternative contrast enhancing treatment