

Accelerated Moisture Infusion in Composites

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4th International Conference on
**Composites Testing and Model
Identification**

20 - 22 October 2008

Dayton, Ohio, USA

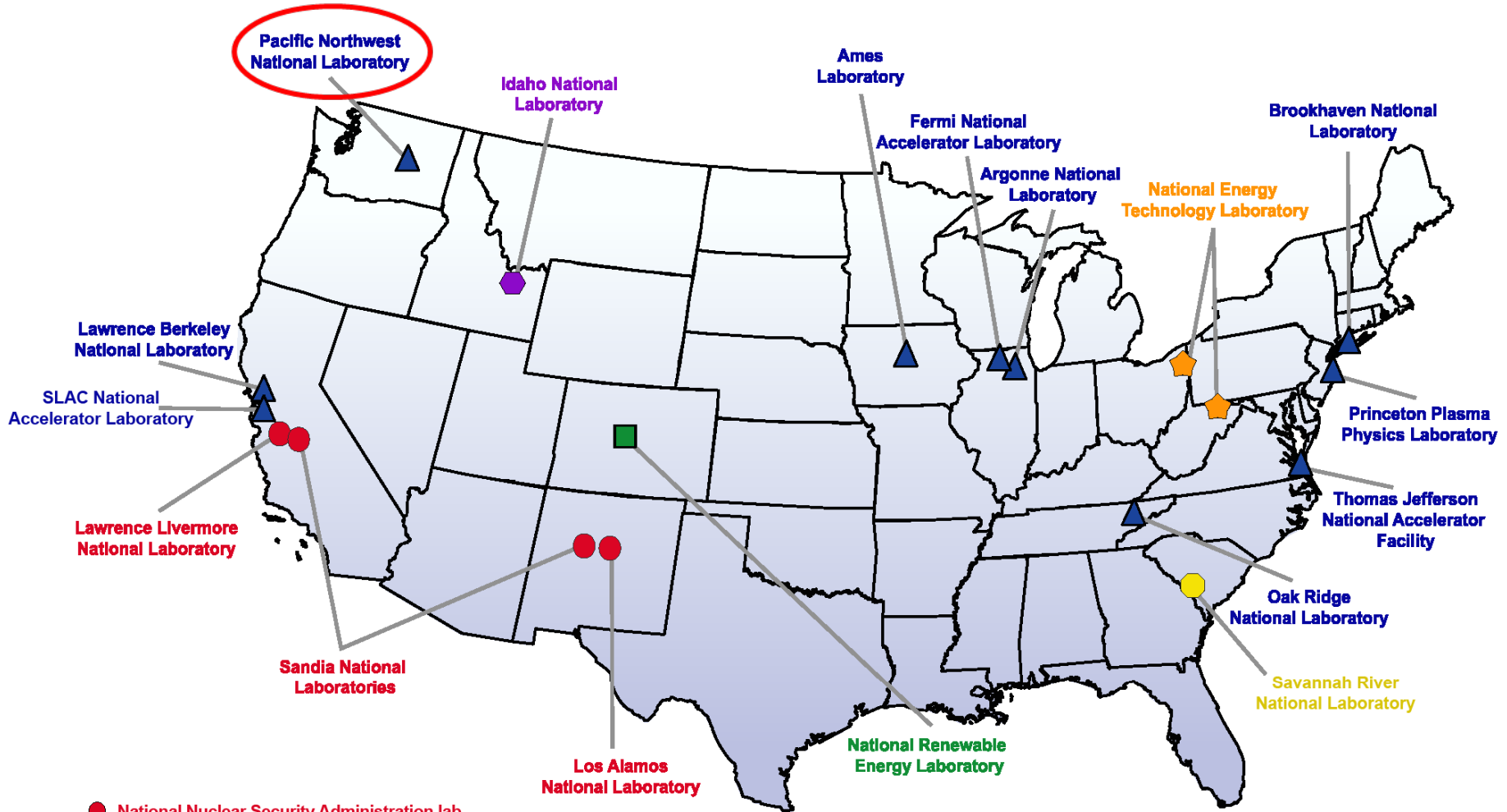


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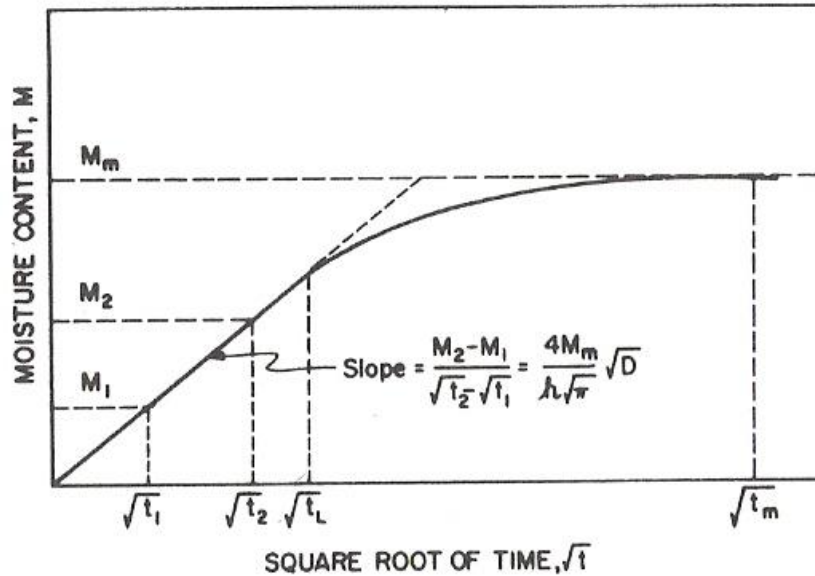


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- National Nuclear Security Administration lab
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Background



► Diffusion Coefficient

$$D = \pi \left(\frac{h}{4M_m} \right)^2 \left(\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}} \right)^2$$

► Test the material to saturation i.e. up to $t = t_m$

► $t = t_m$ = Order of several years

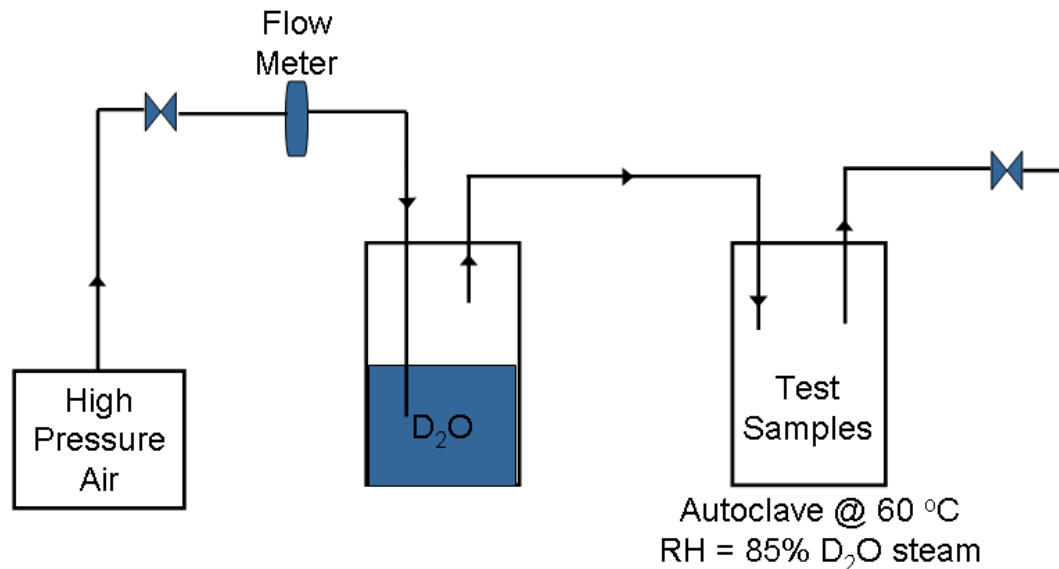
► Current Acceleration methods

- Two-step accelerated conditioning cycles
- Increase the conditioning/test temperature

► Not very effective

A Novel Accelerated Humidity Test

- ▶ High pressure is used to accelerate the moisture ingression.
- ▶ In-house built accelerated humidity chamber



Test Parameters

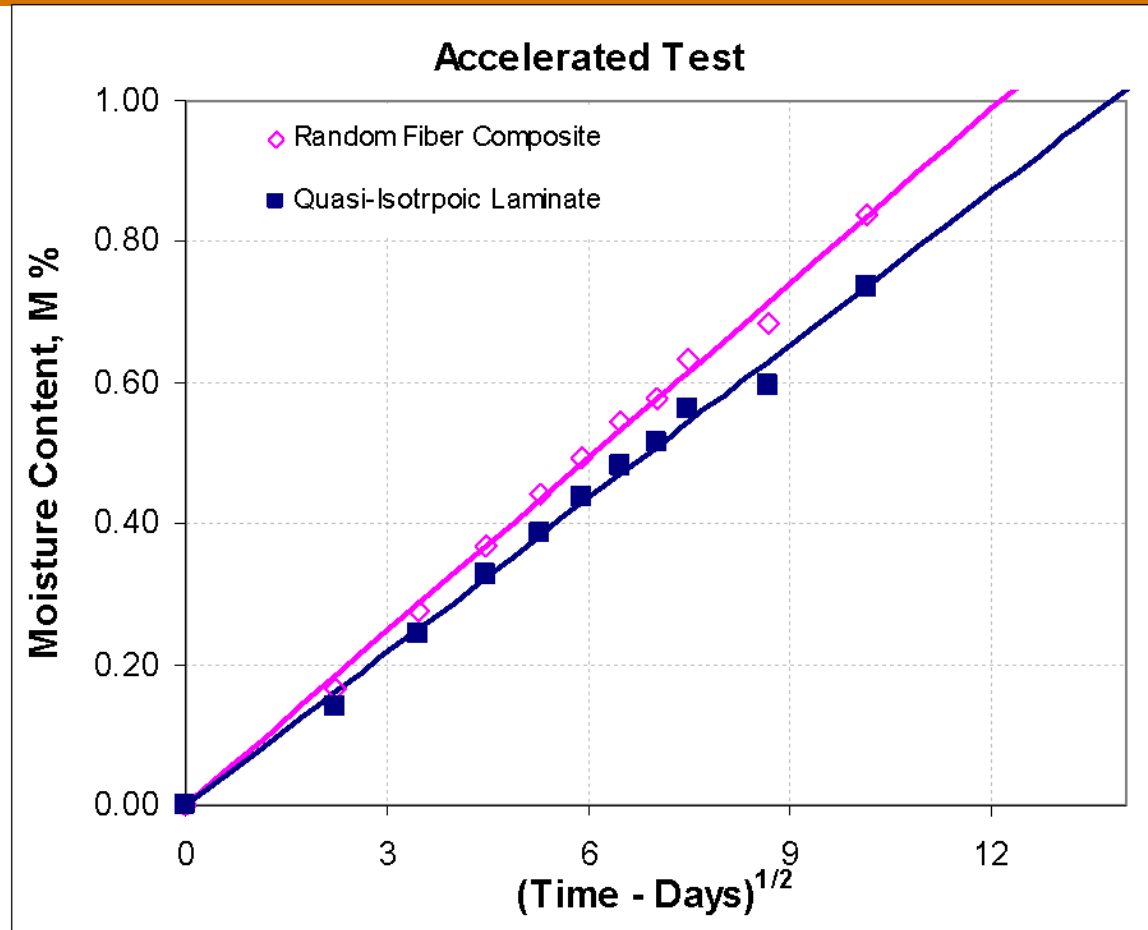
Accelerated

- ▶ In-house built accelerated humidity chamber
- ▶ 85% RH and 60° C – Similar to standard conditions
- ▶ Higher pressures of ~ 65 psi is used to accelerate the moisture ingress.
- ▶ Two Materials
 - Quasi-Isotropic Laminate
 - Random Fiber Composite
- ▶ Specimen Size
 - 50 x 50 x 4 mm

Standard

- ▶ Tenney Bench top hygro-thermal humidity chamber
- ▶ 85% RH and 60° C
- ▶ Atmospheric pressure
- ▶ Two Materials
 - Quasi-Isotropic Laminate
 - Random Fiber Composite
- ▶ Specimen Size
 - 50 x 50 x 4 mm

Accelerated Gravimetric Results



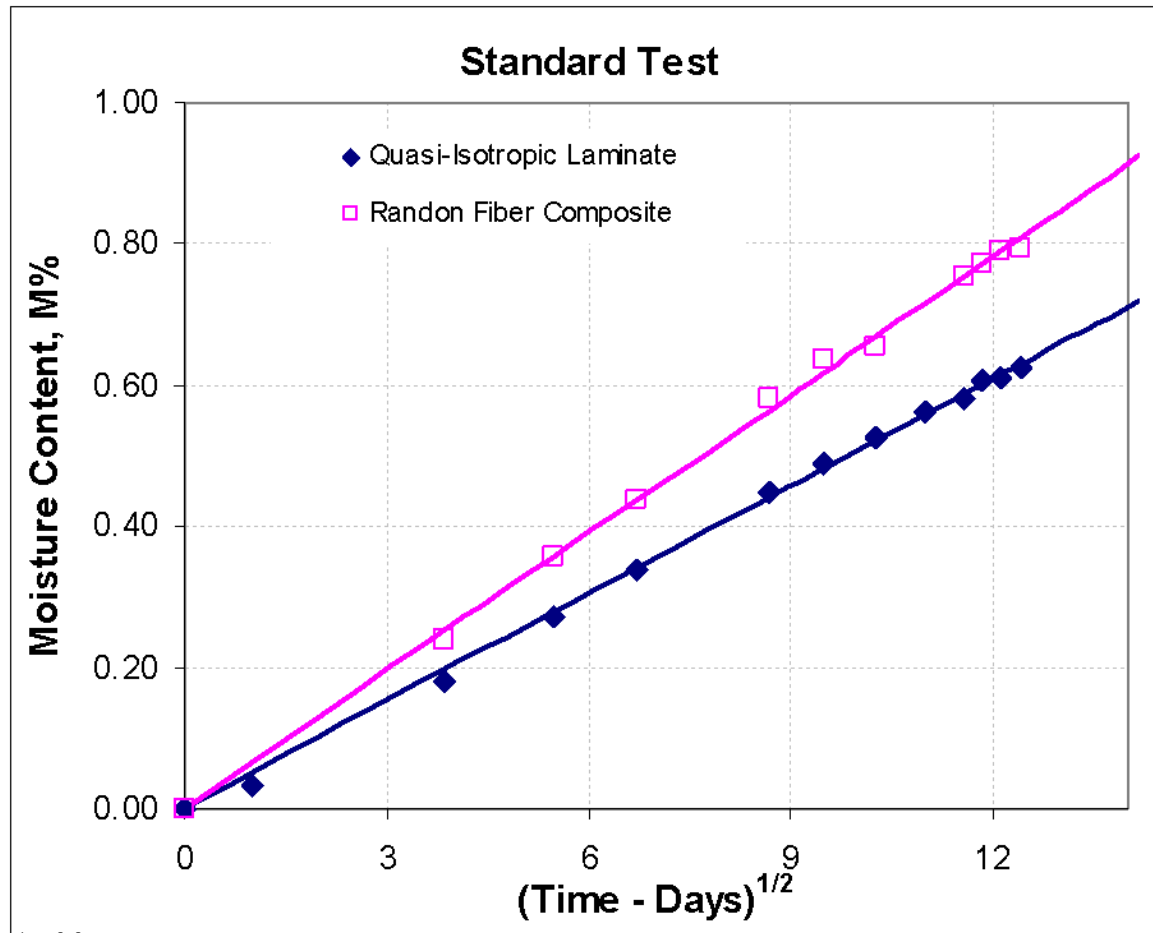
► Estimated diffusivities

■ $D_{\text{Random Fiber}} = 1.4097\text{E-}07 \text{ mm}^2/\text{s}$

■ $D_{\text{Laminate}} = 8.6415\text{E-}08 \text{ mm}^2/\text{s}$



Standard Gravimetric Results

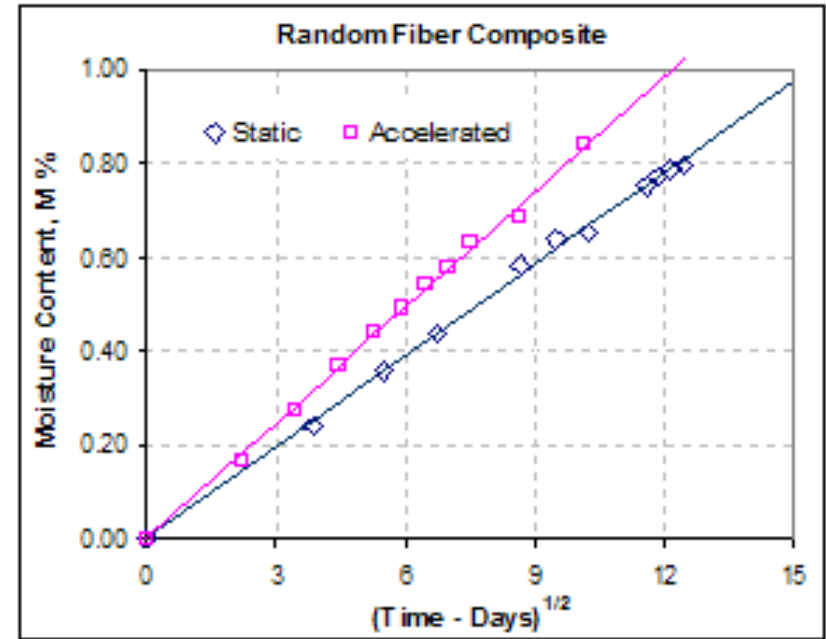
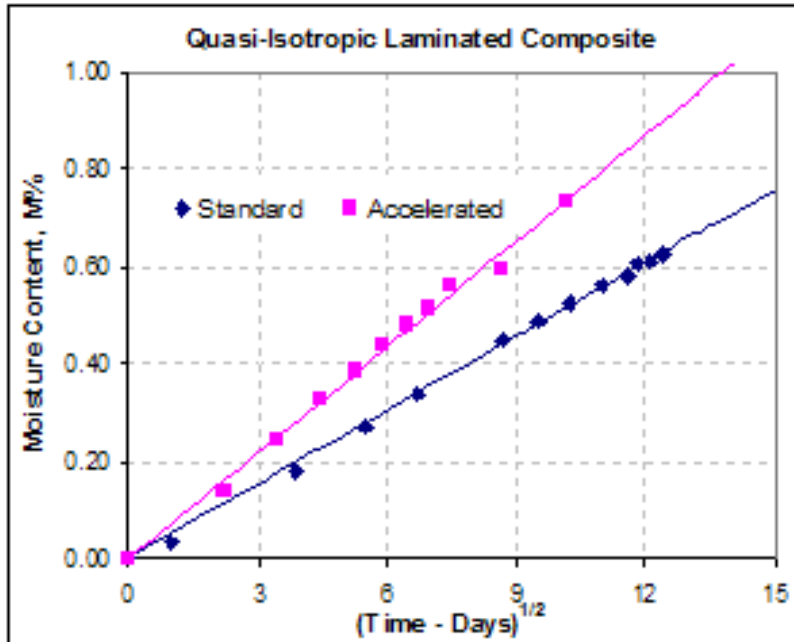


► Estimated diffusivities

■ $D_{\text{Random Fiber}} = 8.0009\text{E-}08 \text{ mm}^2/\text{s}$

■ $D_{\text{Laminate}} = 4.8687\text{E-}08 \text{ mm}^2/\text{s}$

Accelerated vs. Standard - Comparison



Diffusivity mm ² /s	Standard	Accelerated
Quasi-Isotropic Laminate	4.87E-08	8.64E-08
Random Fiber Composite	8.00E-08	1.41E-07

- ▶ Diffusivities increased by a factor of ~ 80%
- ▶ Successfully demonstrated that the moisture diffusion can be accelerated using pressure.

Nuclear Reaction Analysis - Composites

▶ Nuclear Reaction Analysis – Basic Principle

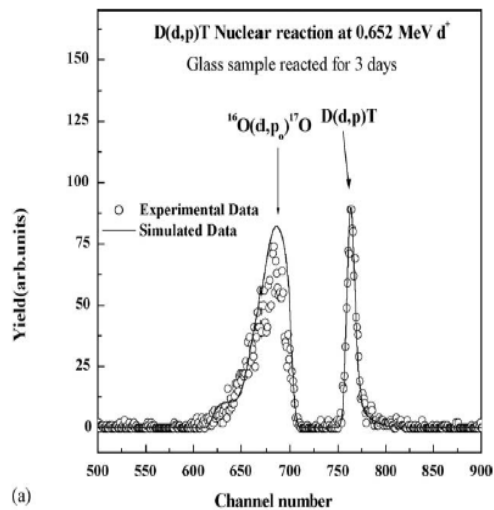
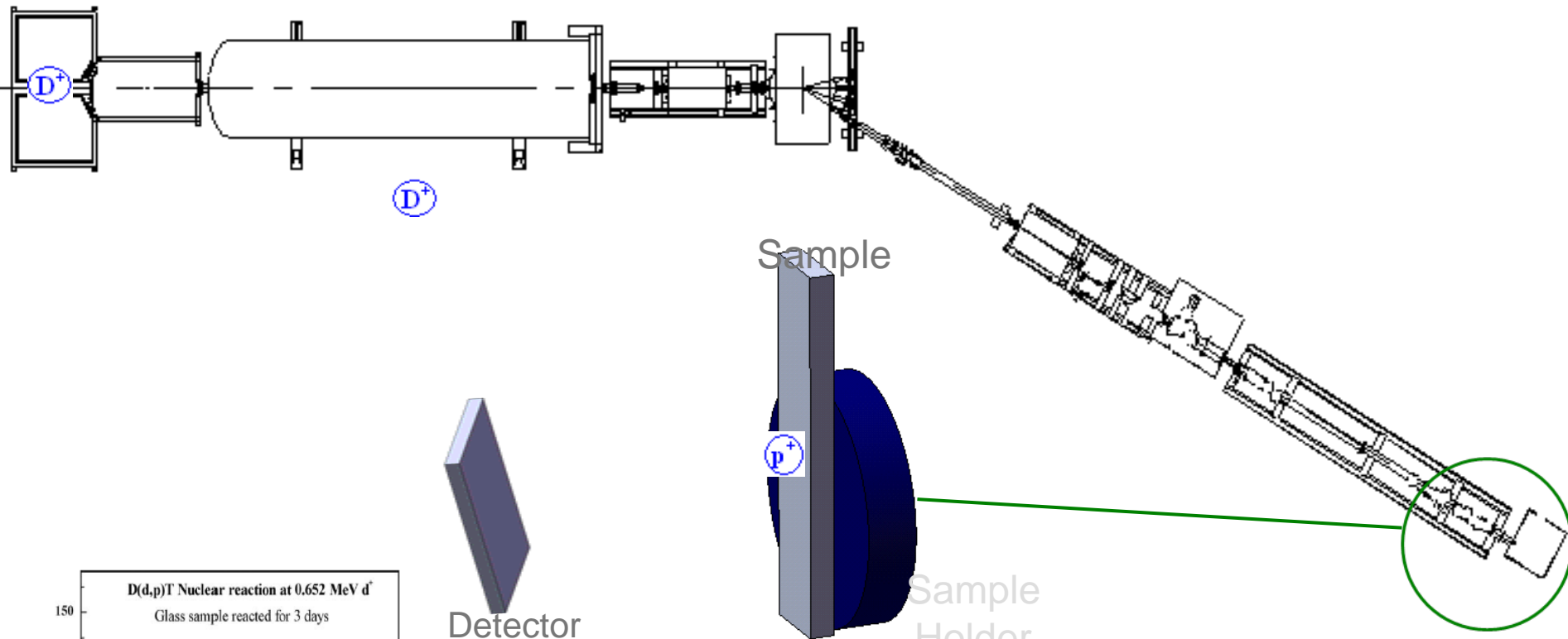
- Charged particles (an ion beam) with energy – produced in an accelerator and bombards the sample.
- Nuclear reactions with low-Z nuclei in the sample are induced by this ion beam.
- Products of these reactions (typically p, d, t, ^3He , α particles, and γ rays) are detected using a spectrum of particle yield versus energy.

▶ Nuclear Reaction Analysis – Applied to Composites

- Need to use D_2O instead of H_2O – other wise unable to distinguish the moisture content (H_2O) from the hydrogen and oxygen within the specimen.
- Uses either **D (d, p) ^1T** or **D (^3He ,p) ^4He** nuclear reaction
- Proton yield is directly proportional to the deuterium concentration within the reaction volume



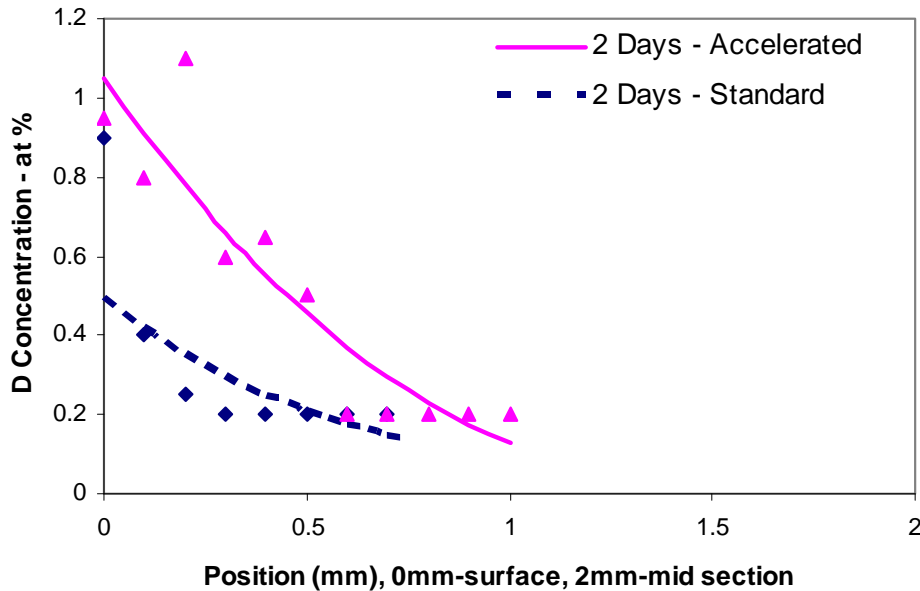
Animated View of the Accelerator System



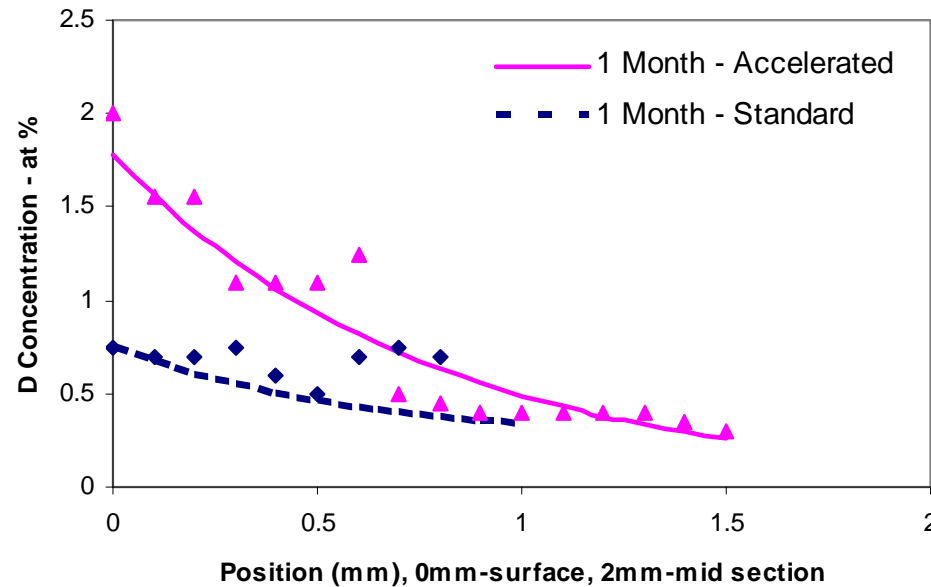
(a)

NRA Results – Random Fiber Composite

Random Fiber Composite - 2 Days



Random Fiber Composite - 1 Month



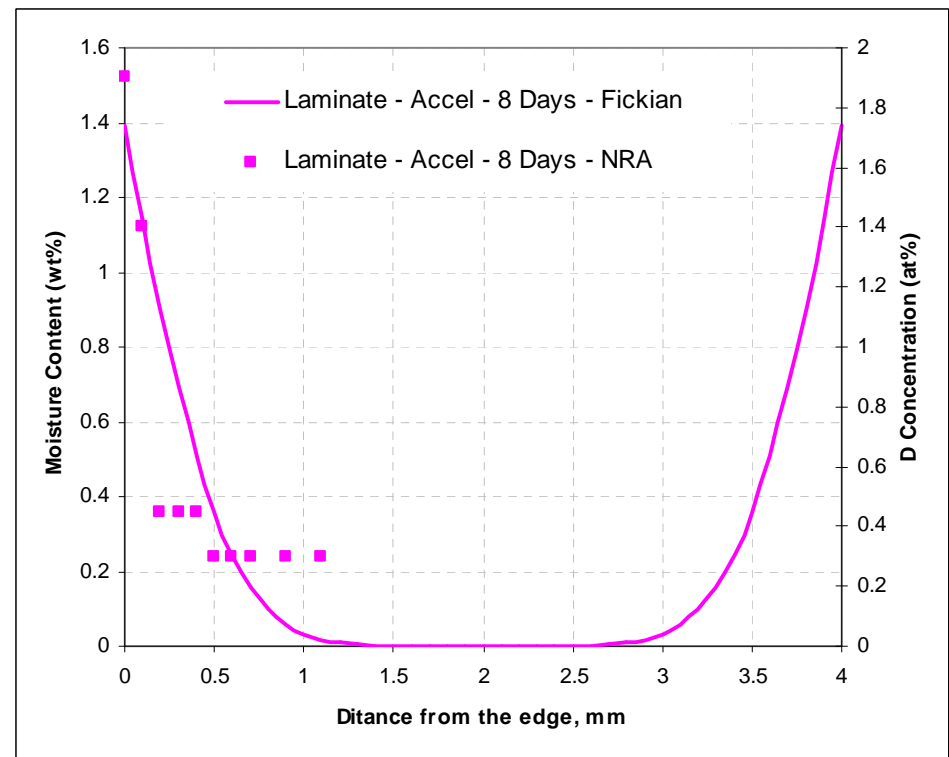
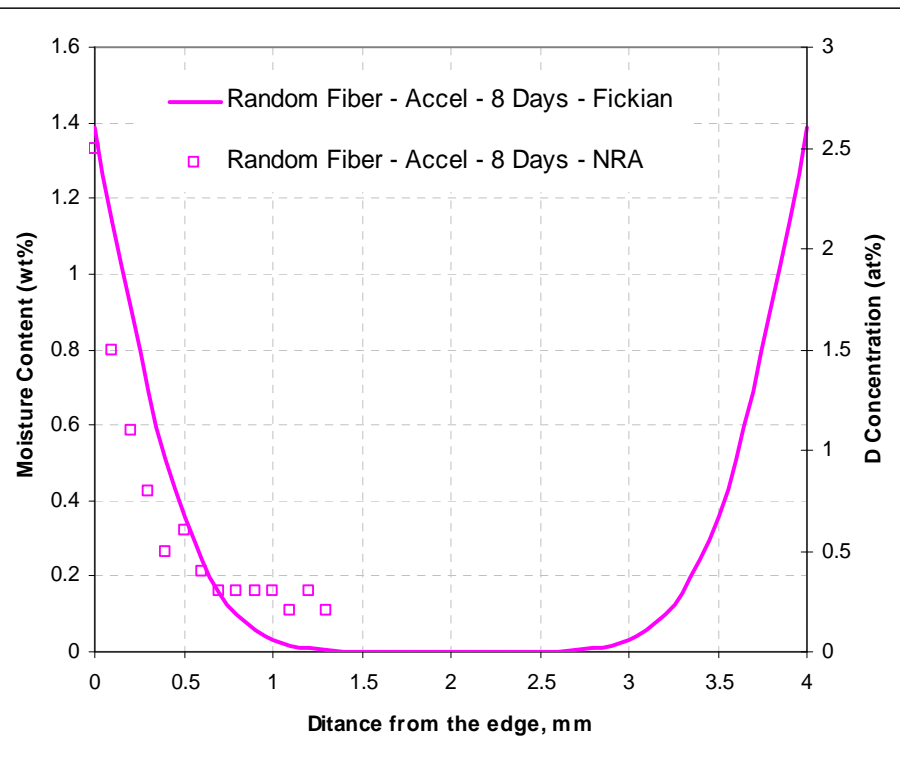
- ▶ Moisture absorption is increasing as a function of time – compares very well with gravimetric analysis

NRA Results – Comparison to Fickian

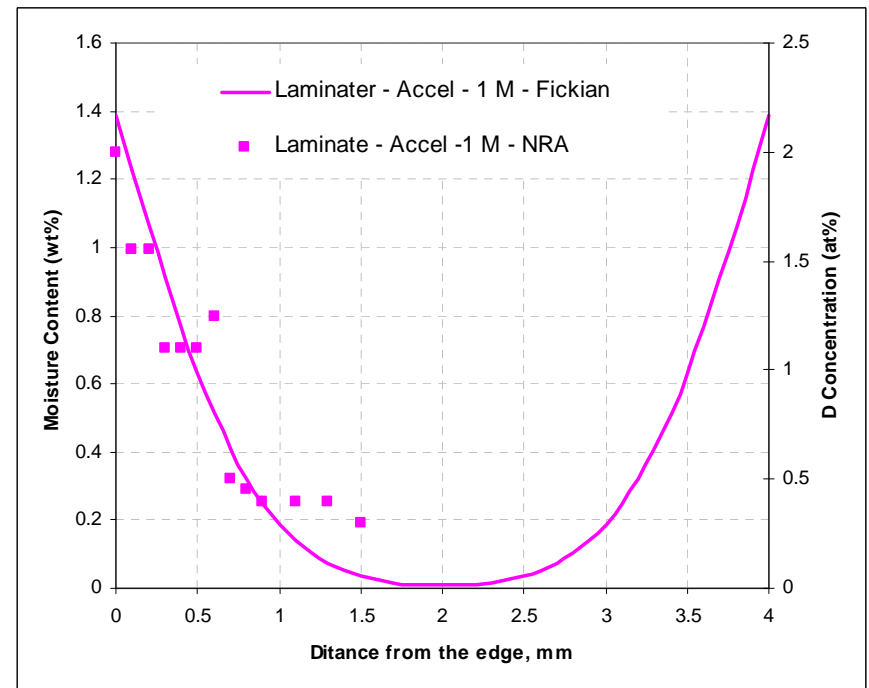
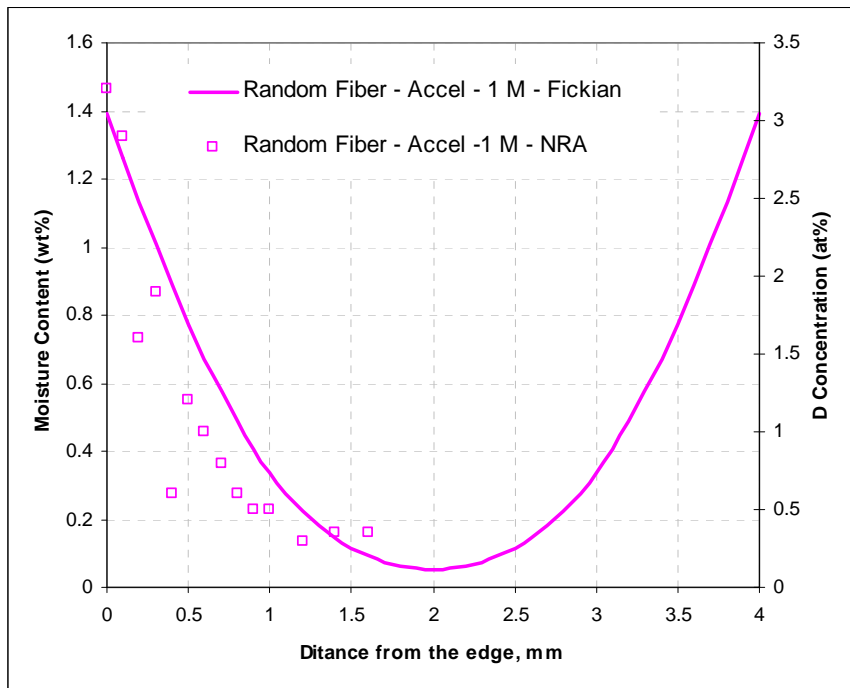
▶ Fickian solution

$$C(x, t) := C_0 \left[1 - \frac{4}{\pi} \cdot \sum_{j=0}^{\infty} \left[\frac{1}{2 \cdot j + 1} \cdot \sin \left[(2 \cdot j + 1) \cdot \frac{\pi \cdot x}{h} \right] \cdot \exp \left[-\frac{(2 \cdot j + 1)^2 \cdot \pi^2 \cdot D \cdot t}{h^2} \right] \right] \right]$$

▶ Compare accelerated NRA curves with Fickian curves



NRA Results – Comparison to Fickian



- ▶ The trend of NRA measurements shows similar trends when compared to Fickian absorption curves
- ▶ The scatter observed within the NRA measurements is inherent to the composite material system (resin rich vs. fiber rich area)

Conclusions

- ▶ Successfully demonstrated that moisture ingress can be accelerated using pressure
 - Diffusivities increased by a factor of ~80%
- ▶ Successfully demonstrated that NRA can be used to measure deuterium (moisture) concentration

Time to Saturation, Years	Static	Accelerated
Quasi-Isotropic Laminate	7.0	3.9
Random Fiber Composite	4.2	2.4

- The above values are based on the diffusivities calculated from the 160 days for static and 103 days for accelerated tests
- ▶ Accelerated testing at higher pressures might further decrease the time to saturation

Acknowledgements

- ▶ Thank You ALL
- ▶ I acknowledge Dr. James Holbery of PNNL and Dr. Patrick Stickler of Boeing Company for their support and review.
- ▶ Pacific Northwest National Laboratory (PNNL) is a multi-program national laboratory operated by Battelle Memorial Institute for the **United States Department of Energy** under DE-AC06-76RLO 1830.
- ▶ Questions? Comments!!