IIMEC 2012

SUMMER SCHOOL IN ADVANCED COMPOSITE MATERIALS

International Institute for Multifunctional Materials for Energy Conversion

Composite Materials:
Structural health monitoring using acoustic methods

By Alkis Paipetis
University of Ioannina

Technological Education Institute of Serres, Greece. July 2 – 6, 2012

Structural Health Monitoring (SHM) Definition [1]:

"the acquisition, validation and analysis of technical data to facilitate life-cycle management decisions."

SHM role:

- ✓ the realization of a reliable system for Detection and interpretation of adverse "changes" in a structure due to damage or normal operation.

SHM major challenge:

- ✓ Design and benchmark the appropriate NDE techniques
- ✓ Identify the monitored "changes"

Problems:

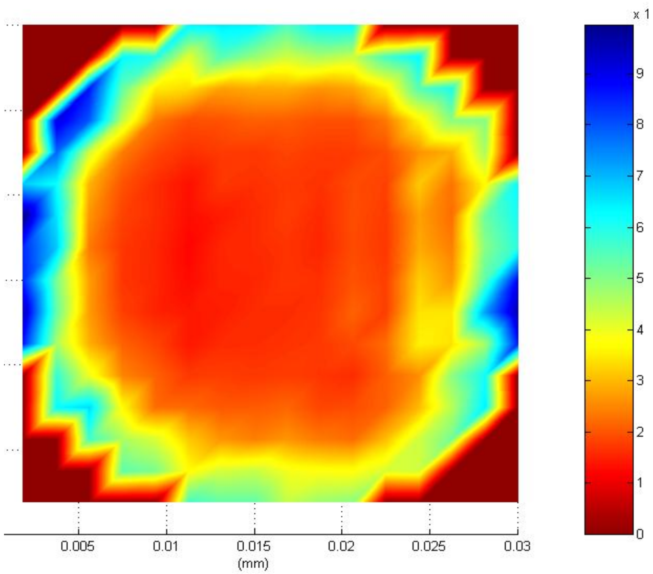
- ✓ Interpret the acquired data
- ✓ Detection limitations (resolution)
- ✓ Location algorithms
- ✓ Integrate monitoring system with minimal structural aggravation

[1] Hall S.R., *Workshop on Structural Health Monitoring*, 265-275, Technomic, Lancaster PA, 1999.

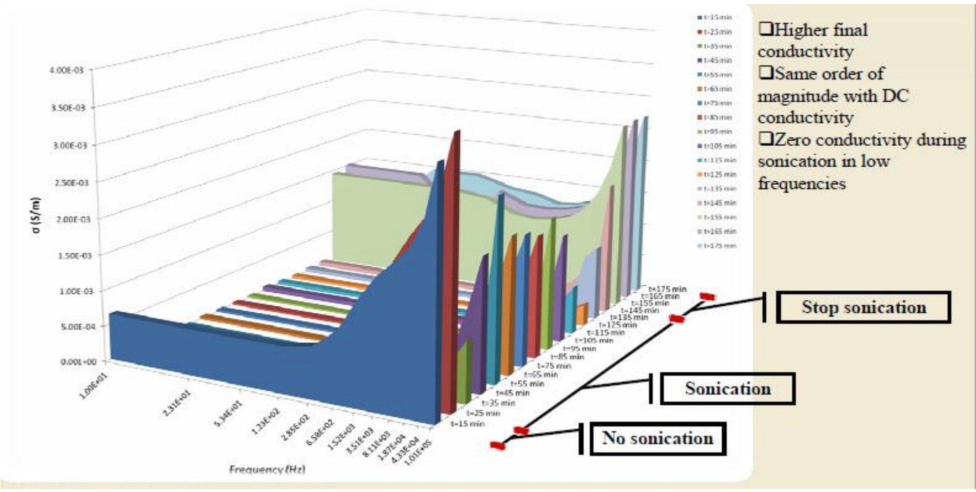
Non-Destructive Evaluation (NDE)

- the characterization of material properties and/or defects without detrimental effects on the structure examined.
- NDE can be performed using
  - Ultrasound
  - Acoustic emission
  - thermography
  - x-rays
  - microwaves
  - magnetic flux, etc.

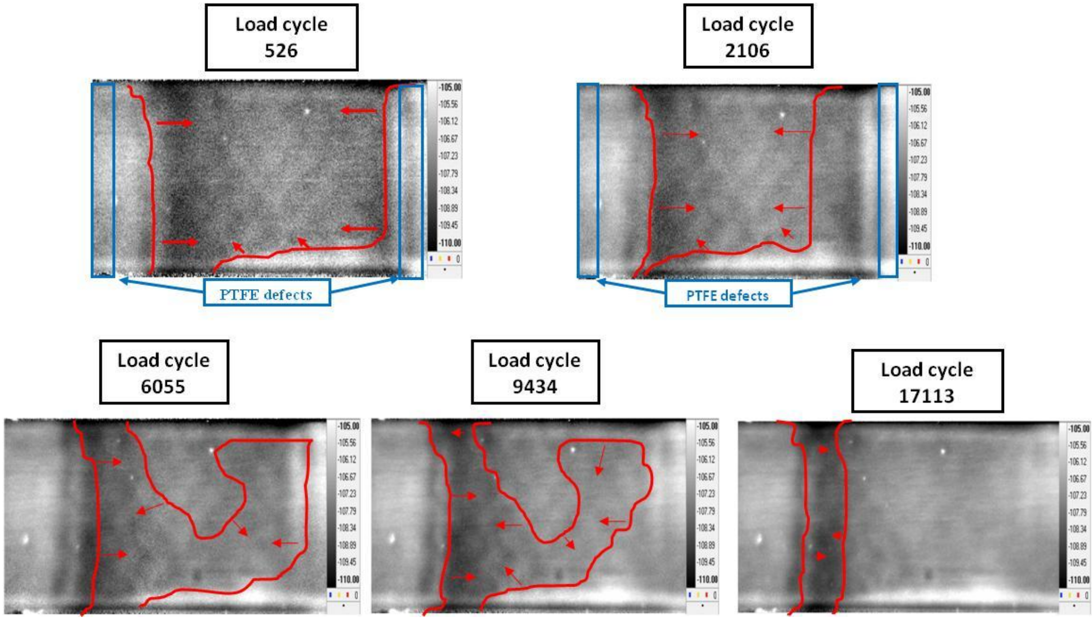
Advanced NDT



✓DISPERSION MONITORING

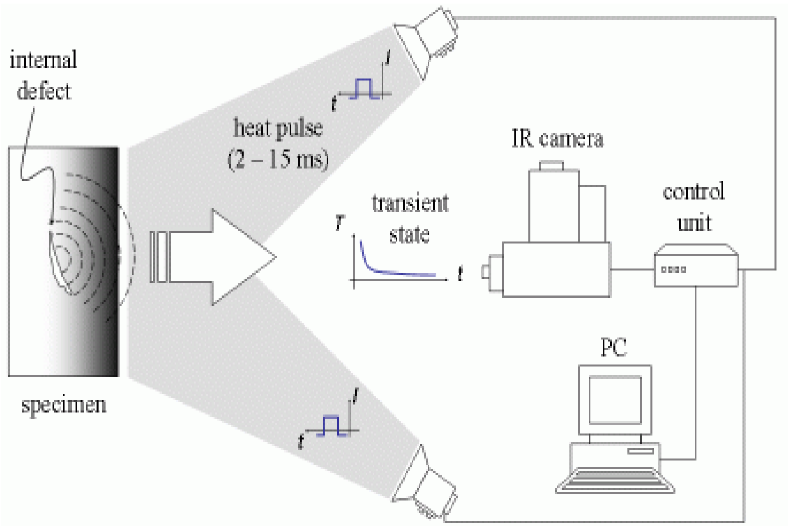


✓REPAIR EFFICIENCY MONITORING

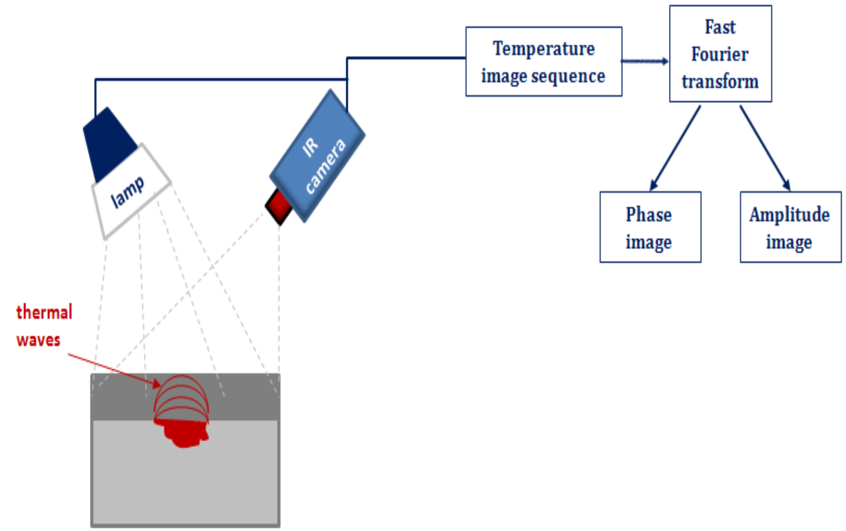


NDE: Thermography

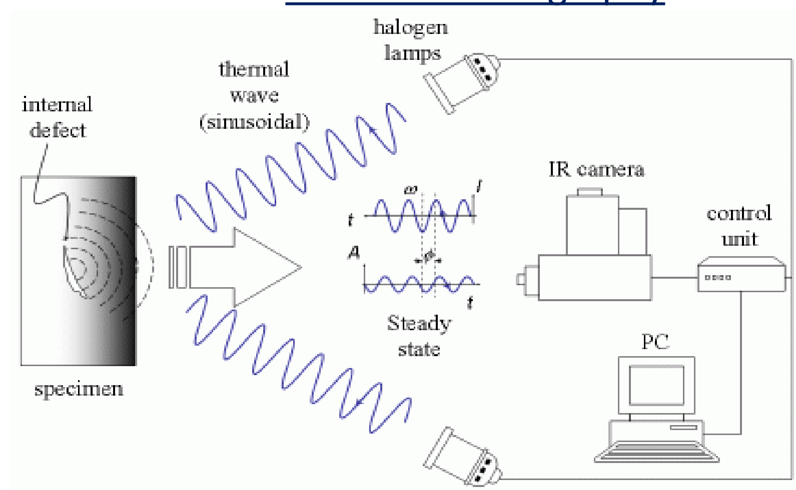
Pulsed thermography



Pulsed phase thermography



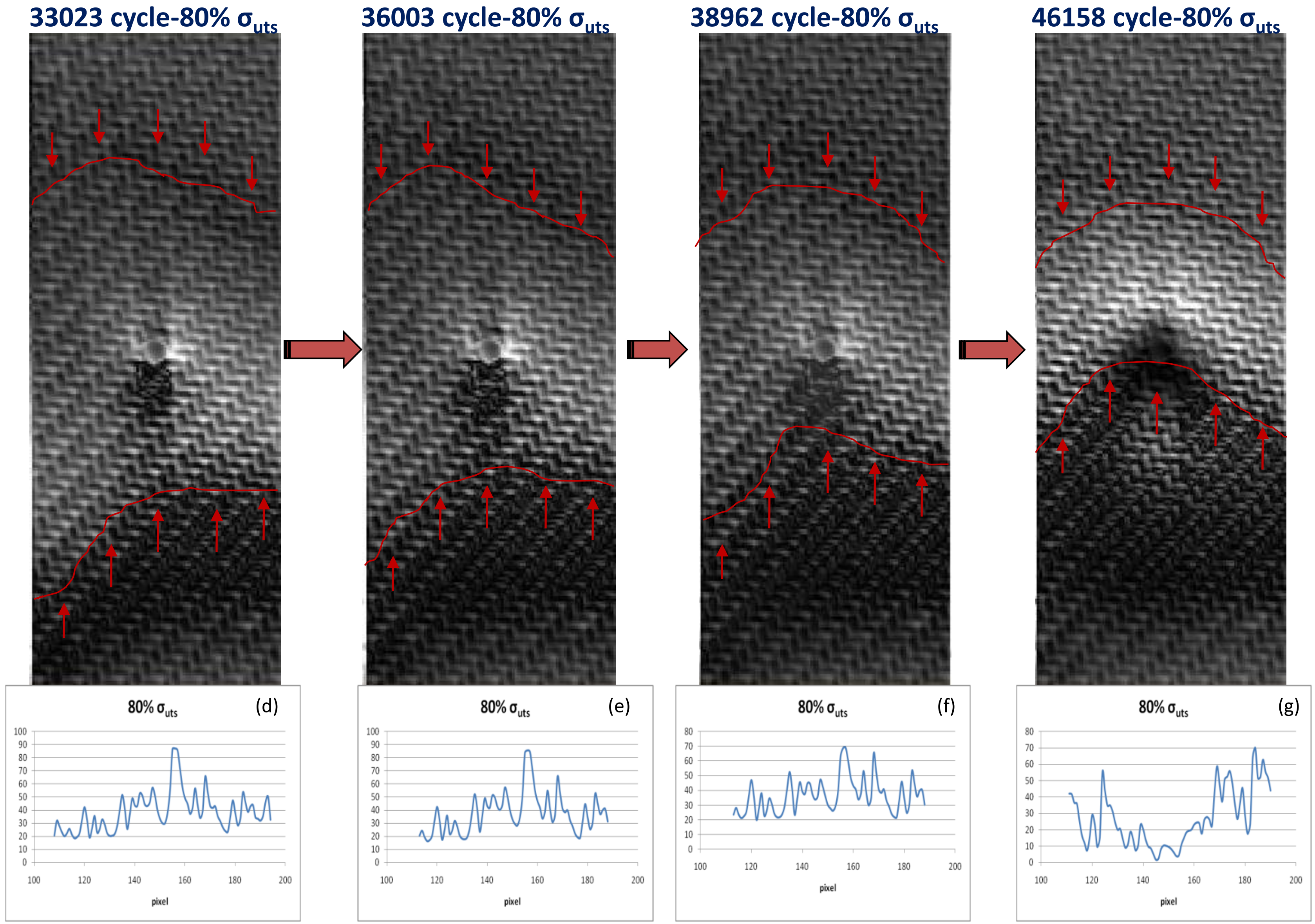
Lock-in thermography



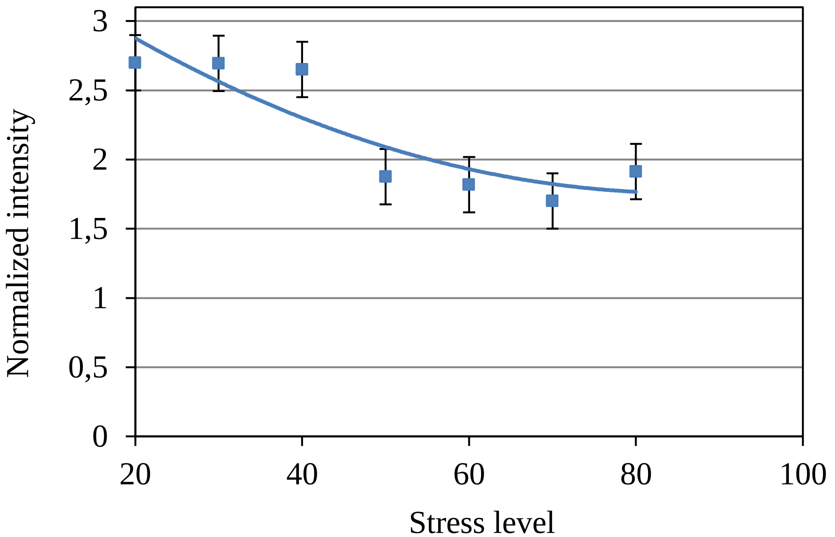
W. Ben Larbi, C. Ibarra-Castanedo, M. Klein , A. Bendada, and X. Maldague, “Experimental Comparison of Lock-in and Pulsed Thermography for the Nondestructive Evaluation of Aerospace Materials”, Sixth International Workshop, Advances in Signal Processing for Non Destructive Evaluation of Materials (IWASPNDE), London, Ontario, Canada, 25-27 August, 2009.

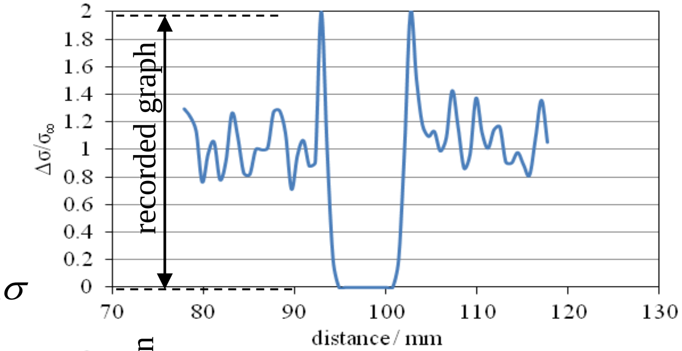
Scenario
(Combined NDT)

On-line lock-in thermography during fatigue loading testing

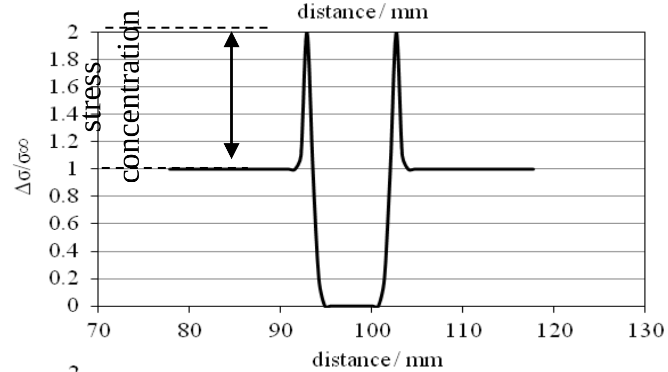


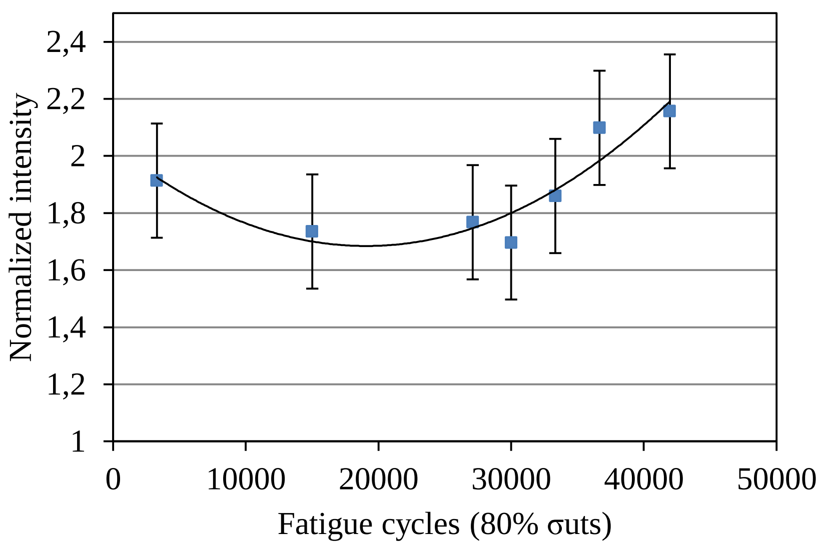
Stress concentrations at the notch



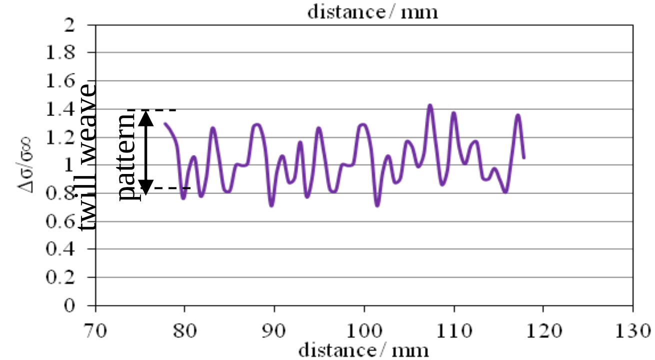


$$\Delta T = -\frac{\alpha}{\rho C_p} T \Delta\sigma$$





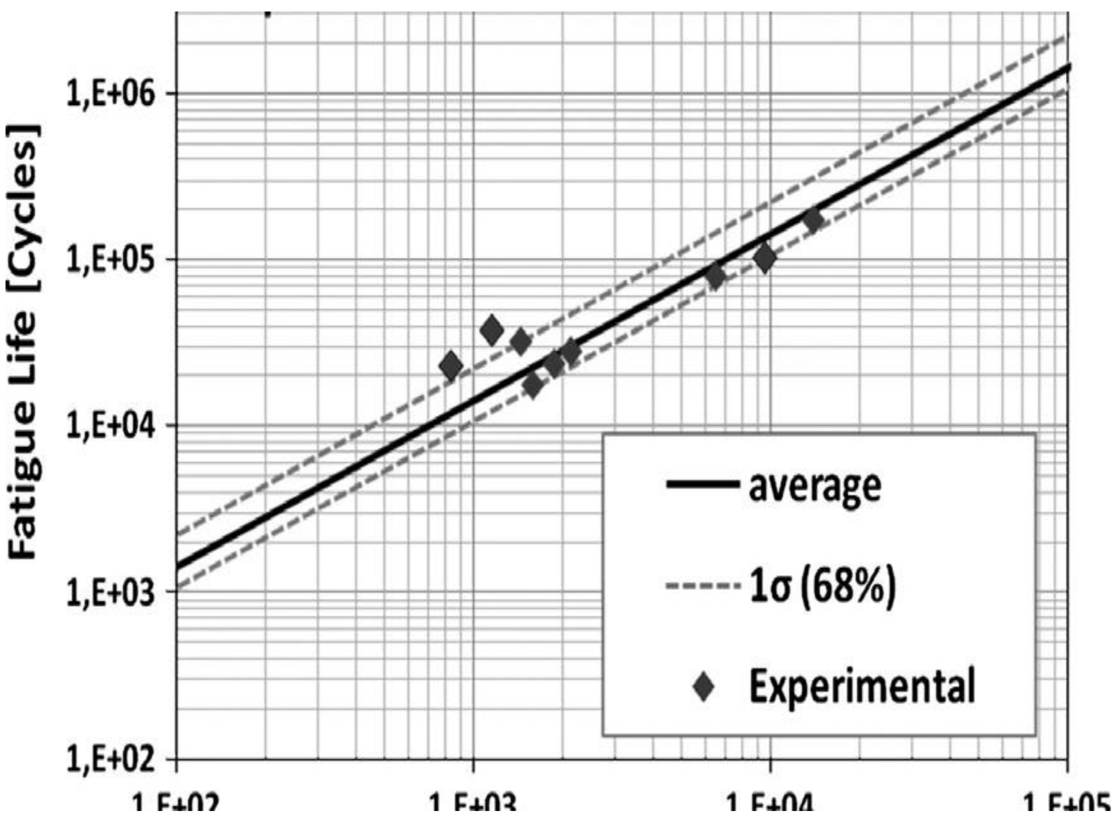
$$\frac{\Delta\sigma}{\Delta\sigma_\infty} = \frac{\Delta T}{\Delta T_\infty}$$

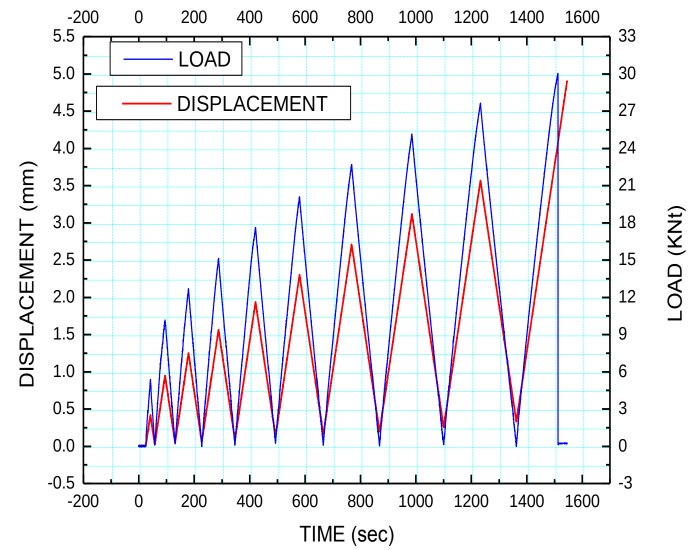


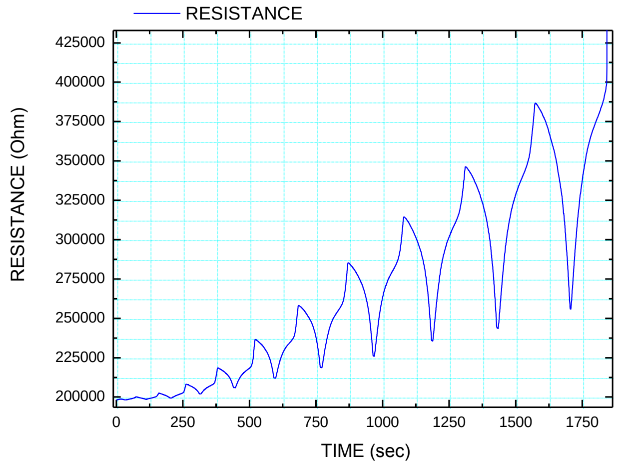
Electrical Resistance Monitoring

✓SELF SENSING

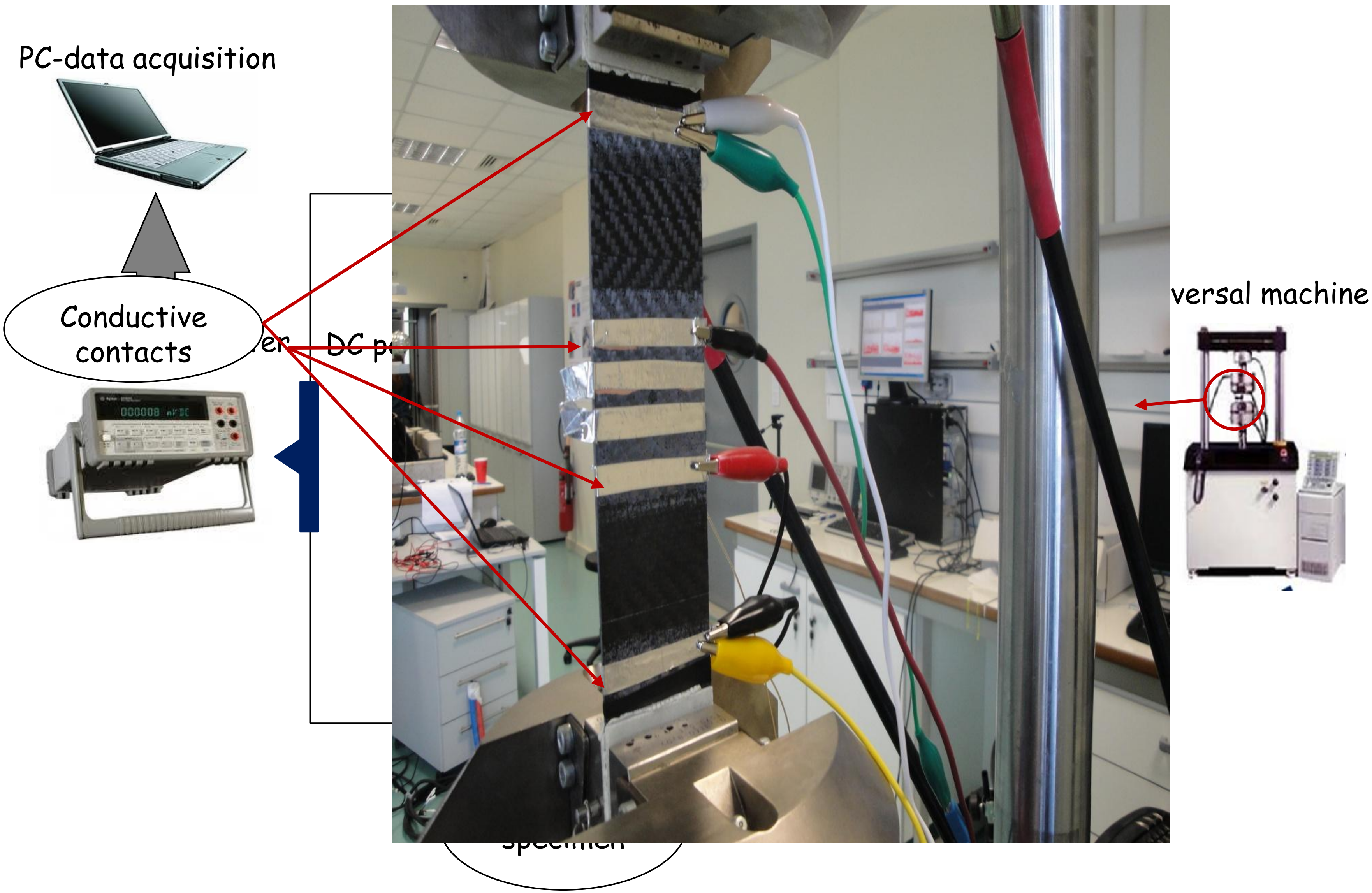
✓REMAINING LIFE FRACTION



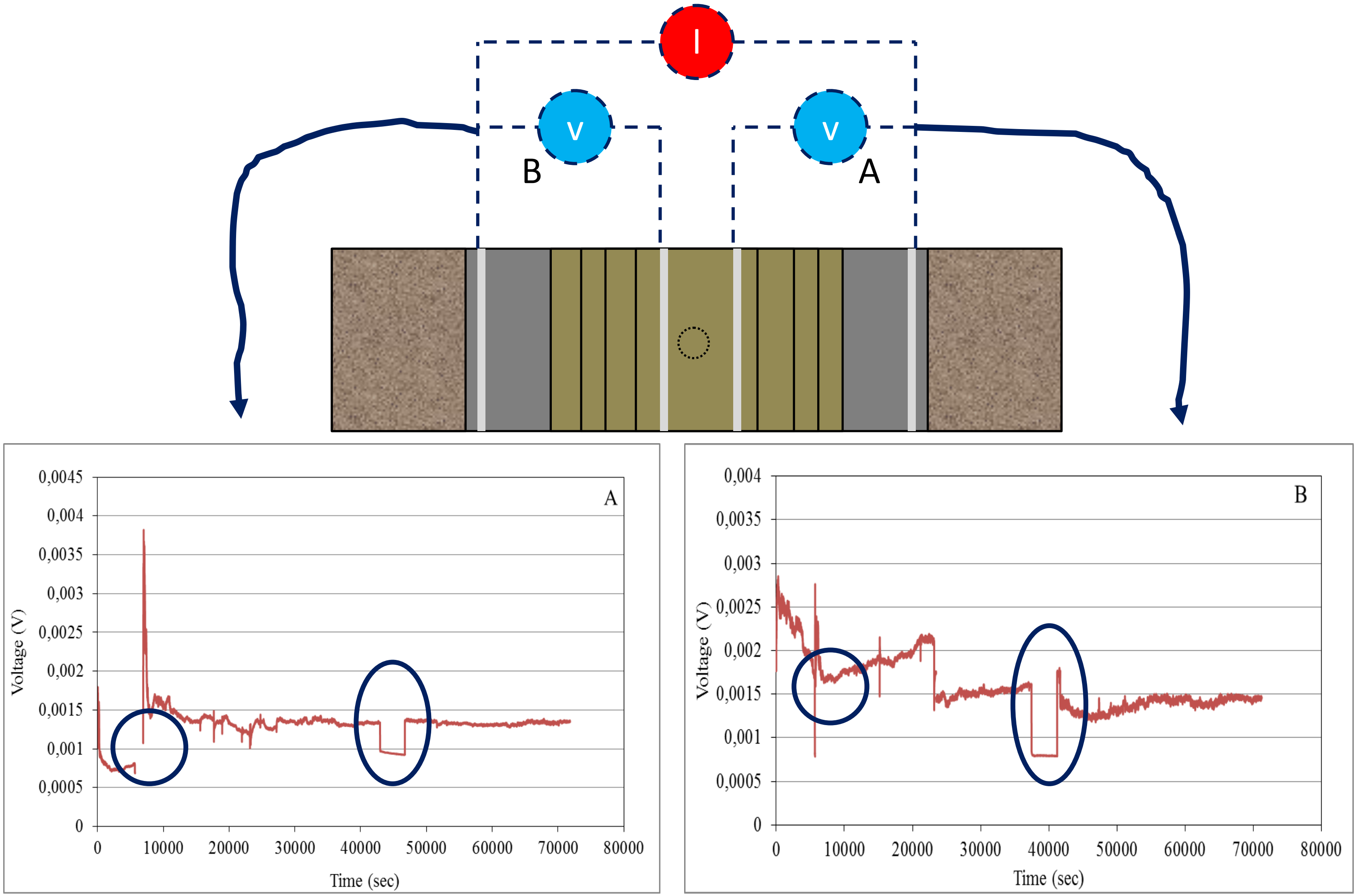




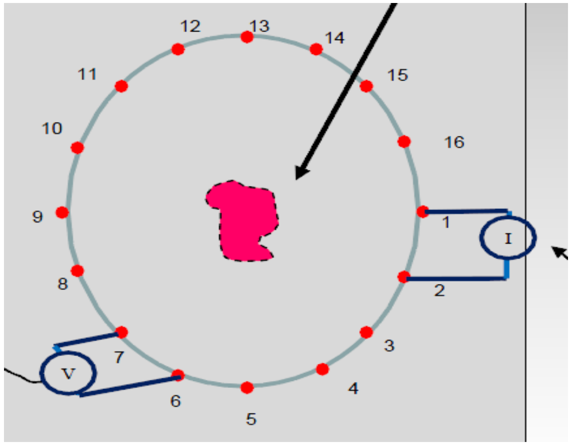
Electrical potential change monitoring

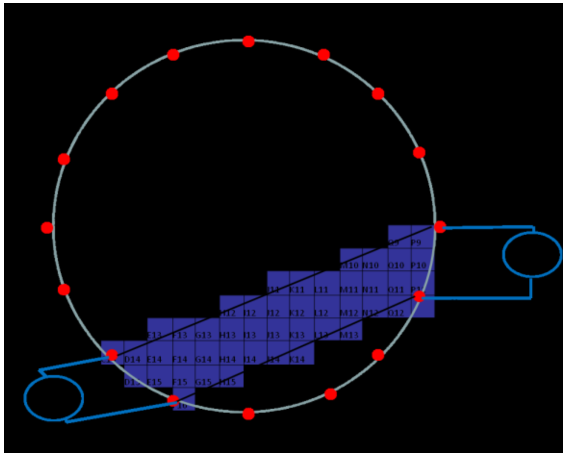


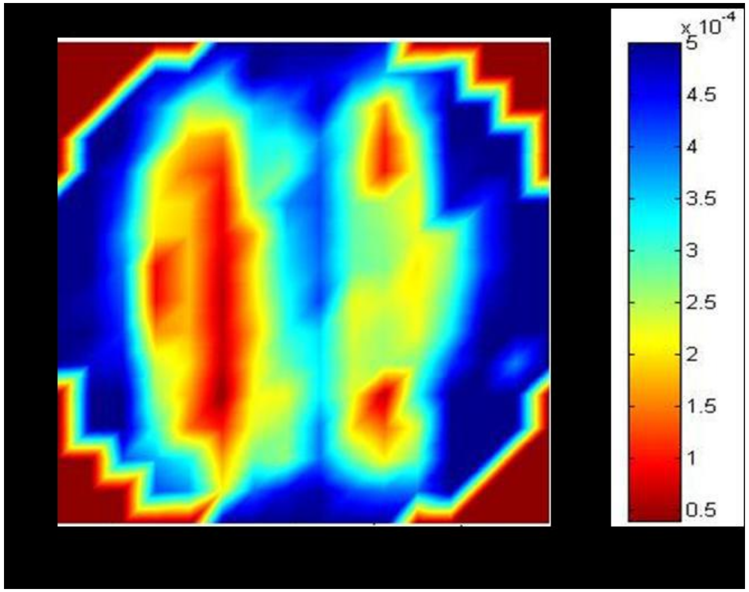
Electrical potential change monitoring



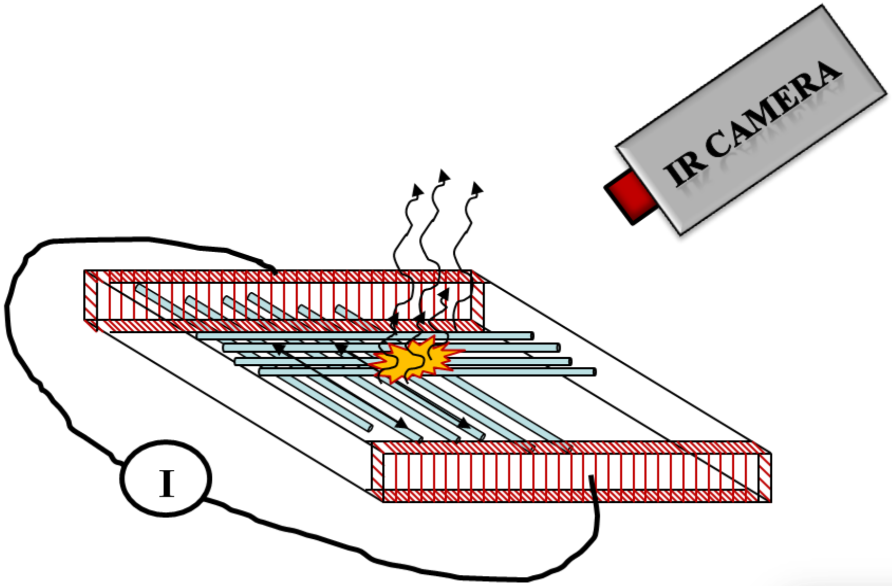
Electrical potential topography

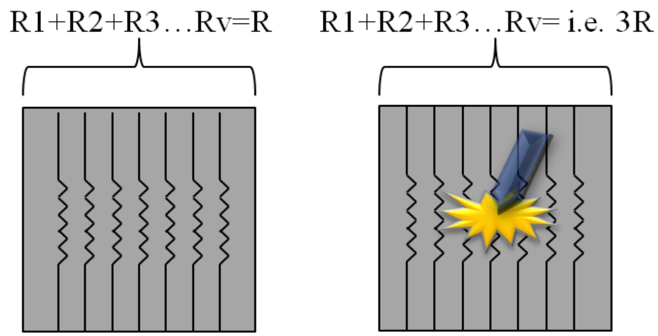




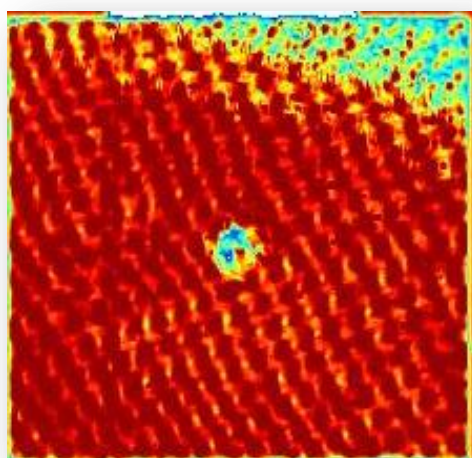


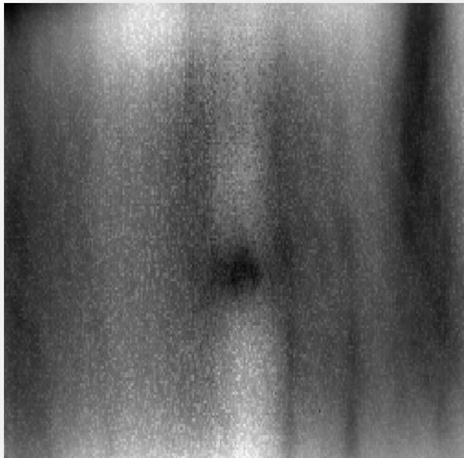
Current injection pulsed phase thermography





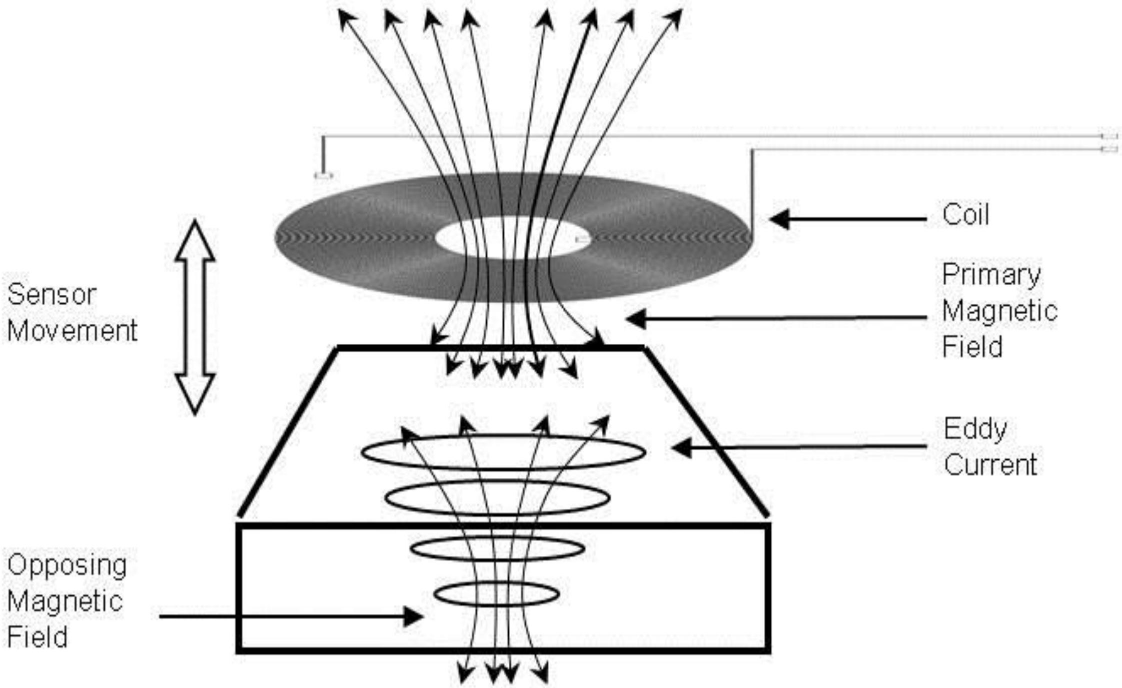
Case 1: Intact CFRP *Case 2: damaged CFRP*





Impendance Spectroscopy

- Eddy current principle

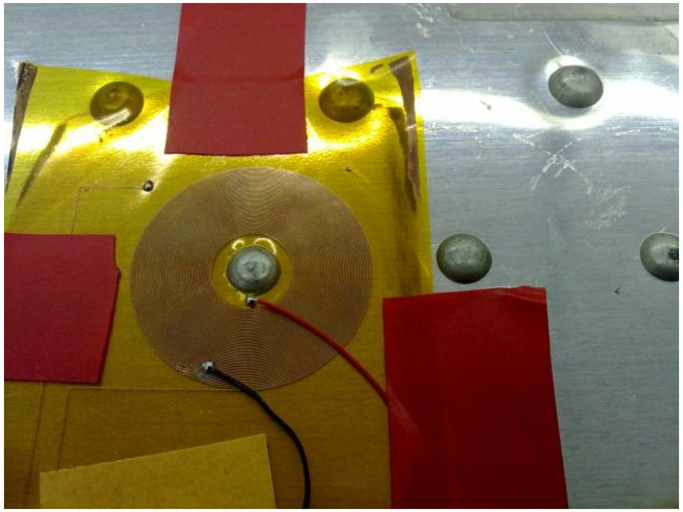


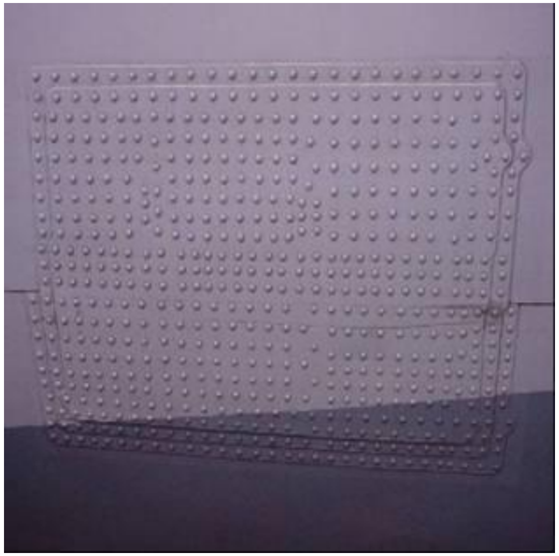
- Electric field: Faradays Law of induction: $\varepsilon = \frac{-d\Phi_m}{dt}$

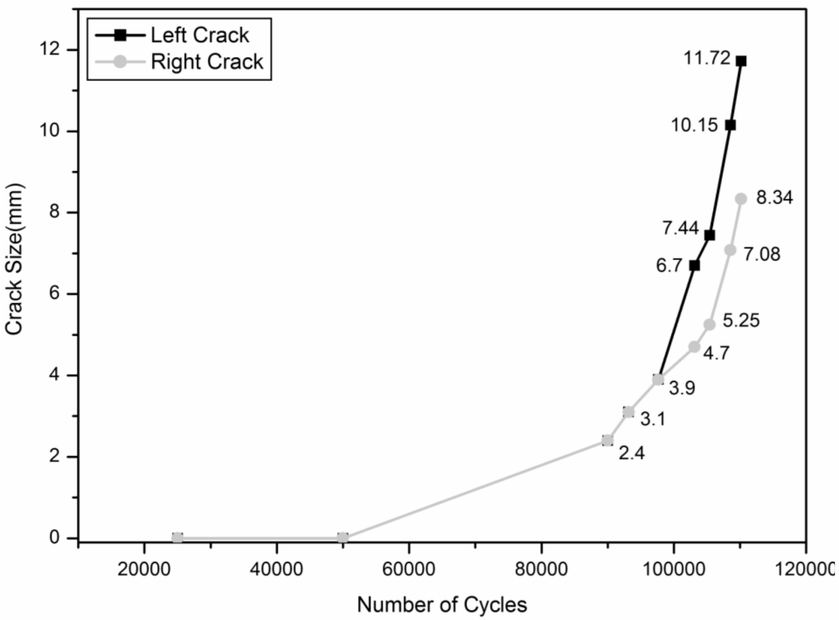
- Opposing magnetic field is responsible for the impedance change in the coil

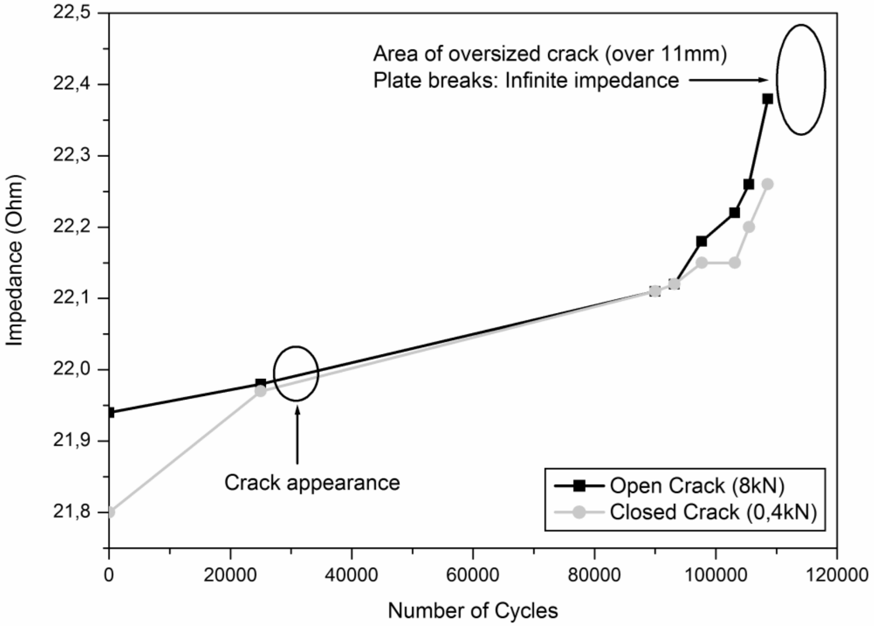
- Abnormalities in the near-surface depth of the conductive material will cause impedance discontinuities

Pitropakis et al, Proceeding of ETNDT5, Ioannina September 19-21 2011





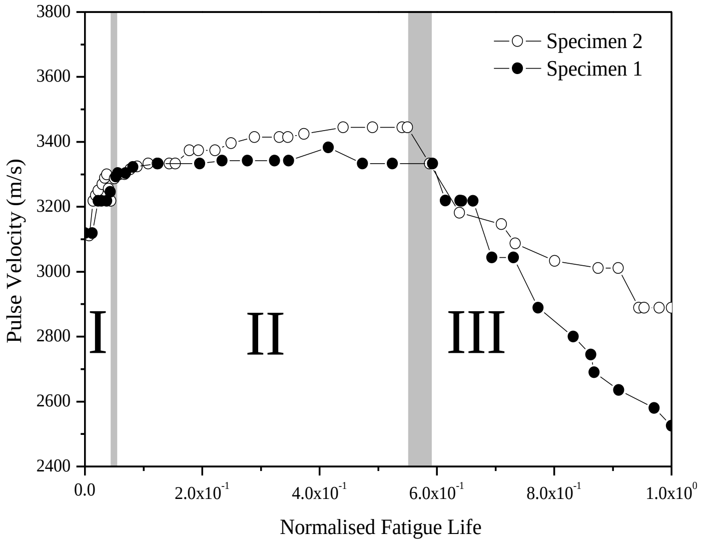


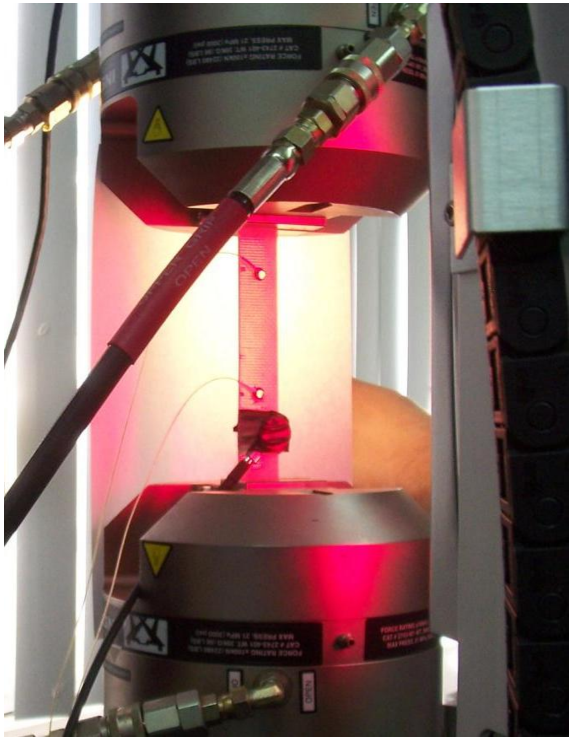


Pitropakis et al, Proceeding of ETNDT5, Ioannina September 19-21 2011

Structural Health Monitoring using acoustic methods

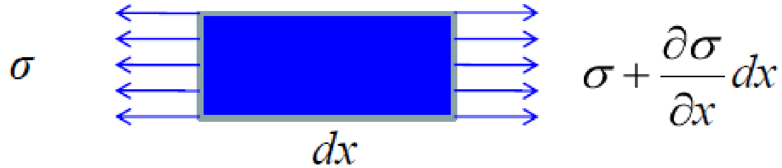
- Ultrasonics
- Acoustic Emission





Wave equation

Surface area A
density ρ



$$-\cancel{\sigma}\cancel{A} + \left(\cancel{\sigma} + \frac{\partial \sigma}{\partial x}\cancel{dx}\right)\cancel{A} = \rho \cancel{A}\cancel{dx}\frac{\partial^2 u}{\partial t^2}$$

$$\frac{\partial \sigma}{\partial x} = \rho \frac{\partial^2 u}{\partial t^2}$$

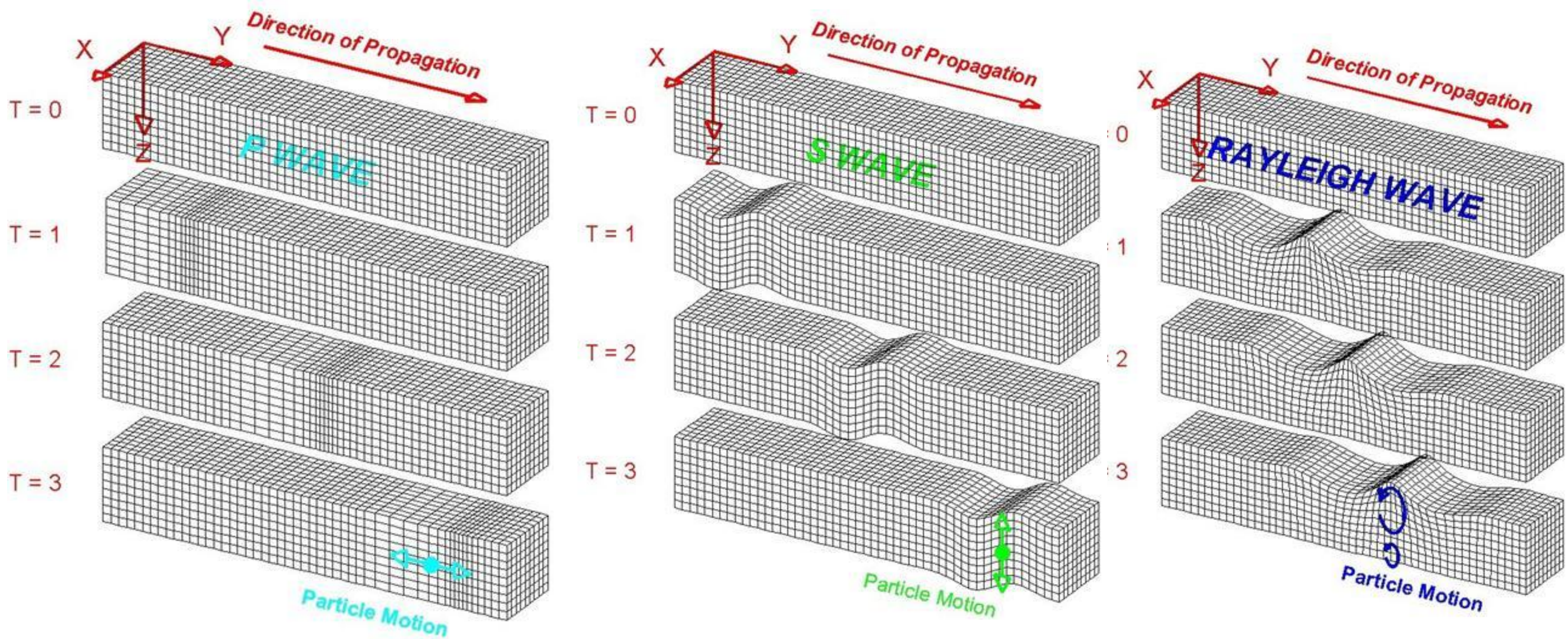
$$\sigma = \mathrm{E}\varepsilon \Leftarrow \varepsilon = \frac{\partial u}{\partial x}$$

$$\frac{\partial^2 u}{\partial x^2} = \frac{1}{C^2}\frac{\partial^2 u}{\partial t^2}$$

$$\frac{\partial}{\partial x}\left(\mathrm{E}\frac{\partial u}{\partial x}\right) = \rho \frac{\partial^2 u}{\partial t^2} \Rightarrow \mathrm{E}\frac{\partial^2 u}{\partial x^2} = \rho \frac{\partial^2 u}{\partial t^2} \Rightarrow \frac{\partial^2 u}{\partial x^2} = \frac{\rho}{\mathrm{E}}$$

$$\boxed{C = \sqrt{\frac{\mathrm{E}}{\rho}}}$$

Ultrasonic Waves



http://web.ics.purdue.edu/~braile/edumod/slinky/slinky.htm

Longitudinal

$$C_p = \sqrt{\frac{E \quad (1 - v)}{\rho(1 + v)(1 - 2v)}}$$

Shear

$$C_S = \sqrt{\frac{\mu}{\rho}} = \sqrt{\frac{E}{\rho \cdot 2(1 + \nu)}}$$

Rayleigh (surface)

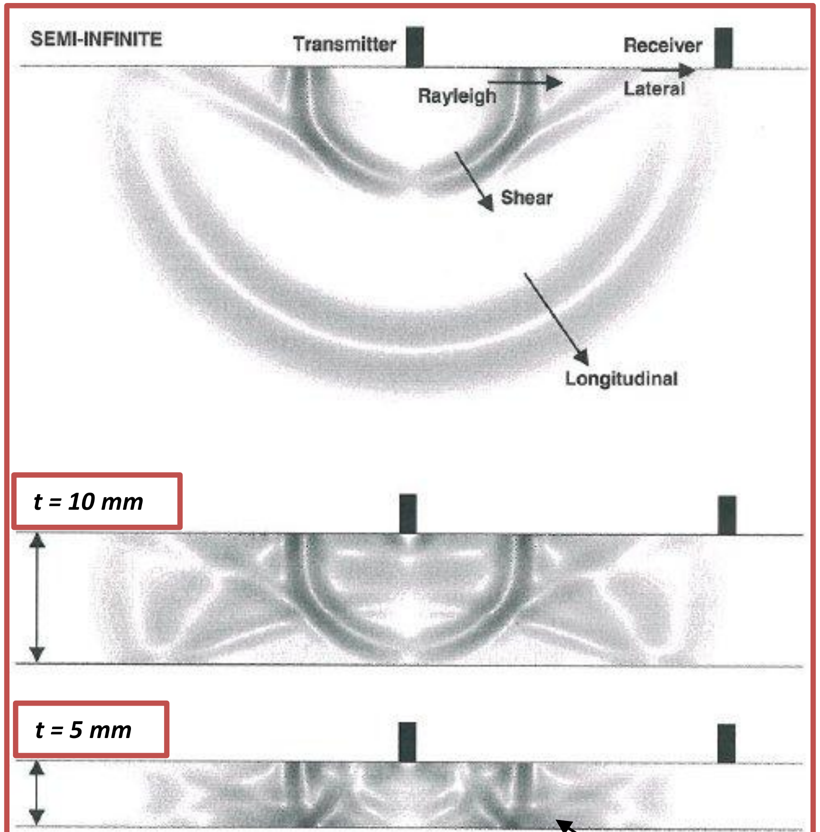
$$C_P = \frac{1 + \nu}{0.87 + 1.12\nu}\sqrt{\frac{2(1 - \nu)}{(1 - 2\nu)}}C_R$$

Generally $C_s \approx 0.61 C_P$, $C_R \approx 0.56 C_P$

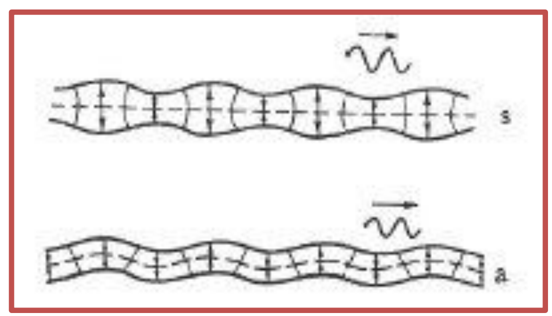
If we know ν and ρ we can estimate the elasticity modulus

Therefore, measuring the stress wave velocity estimation of the internal condition of the material can be done

Wave Modes in Different Geometries



- In infinite media there are only two types of waves: dilatational (P) and distortional (S).
- Semi-infinite media there are also Rayleigh and Lateral (Head) waves. Head waves produced by interaction of longitudinal wave with free surface.
- In double bounded media like plates there are also Lamb waves.



Symmetric

Antisymmetric

In thinnest plates only Lamb wave arrivals are visible.

Properties of Elastic Waves in Semi-Infinite Media

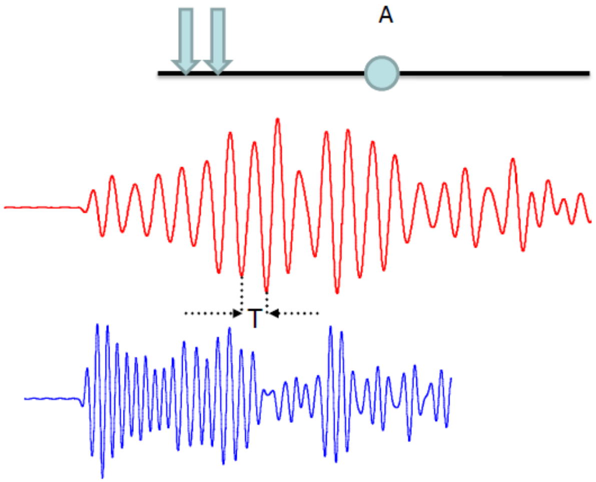
- Rayleigh waves carry 67% of total energy (for $\nu=0.25$).
- Shear 26%.
- Longitudinal 7%.

- Longitudinal and shear waves decay at a rate 1/r in the region away of the free surfaces.
- Along the surface they decay faster, at a rate $1/r^2$.
- Rayleigh waves decays much slower, at a rate of 1/sqrt(r).

“Dynamic Behavior of Materials” by M. Meyers

Wave attributes

•Frequency, f



Balance position

Low Frequency

Higher Frequency

(more cycles in a unit of time)

f=1/T

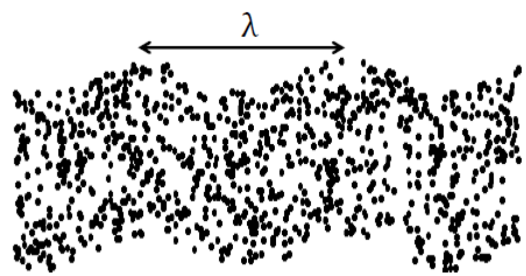
T (period)

When f>20 kHz, the stress waves are regarded as ultrasonics

8

•Wave length, λ

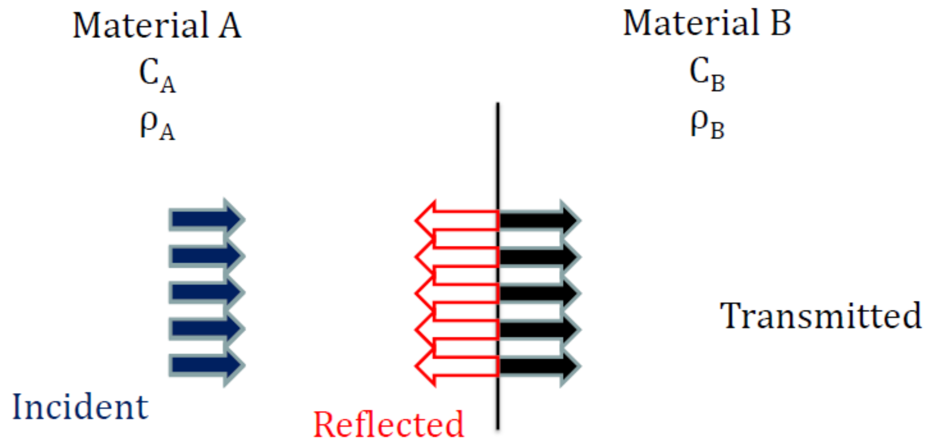
By taking a snapshot during propagation, the particle motion can be seen



$$C = \lambda \cdot f$$

C, wave speed(m/s)
λ, wavelength (m)
f, frequency (Hz [1/s])

Reflection and transmission



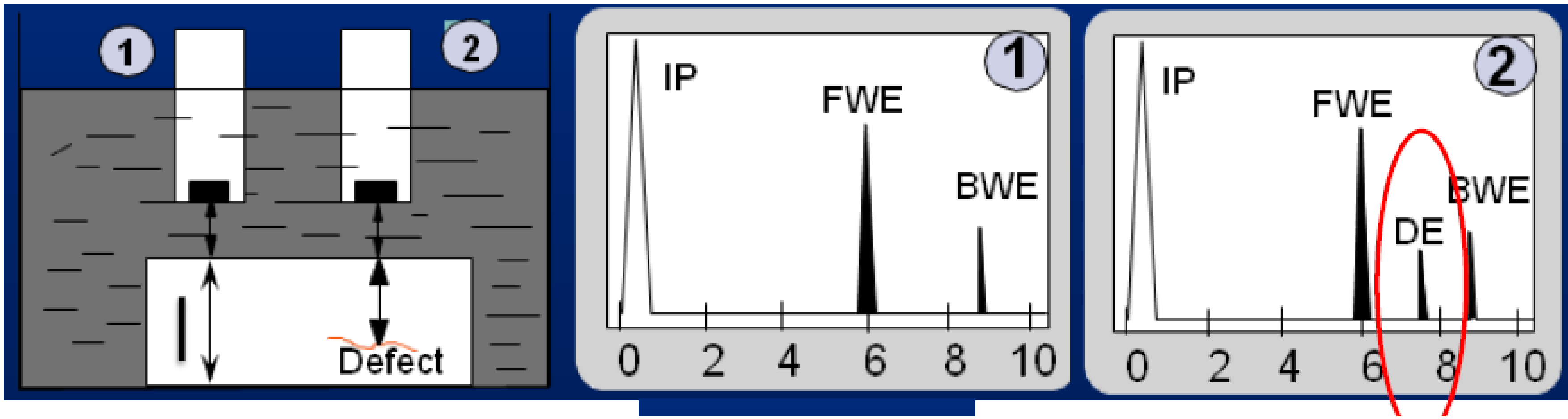
The proportion of energy reflected (or transmitted) depends on the "Acoustic Impedance, Z" of the materials

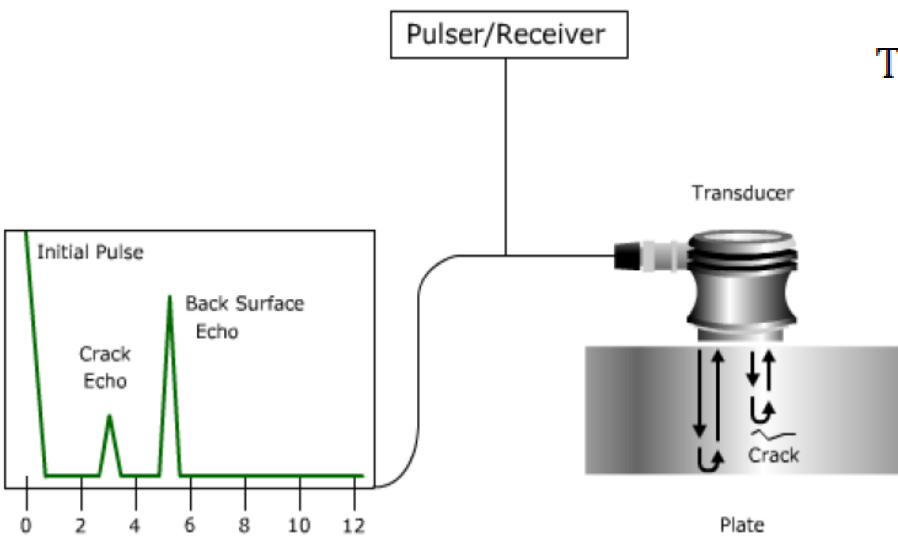
Z=ρC
C, wave speed
ρ, density

If the properties of the two materials are known, the reflection coefficient can be calculated...

$$r = \frac{Z_2 - Z_1}{Z_2 + Z_1} \qquad t = \frac{2Z_2}{Z_2 + Z_1}$$

Defect Location using ultrasonics

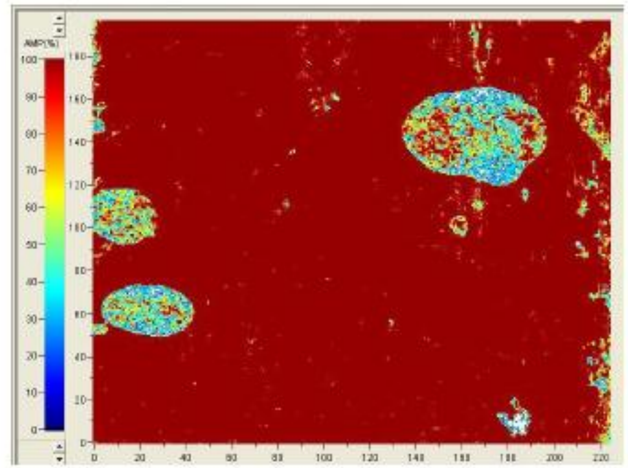


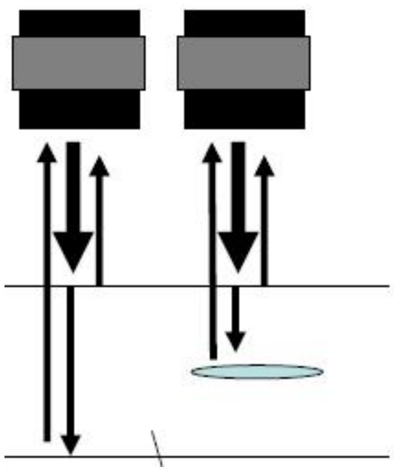


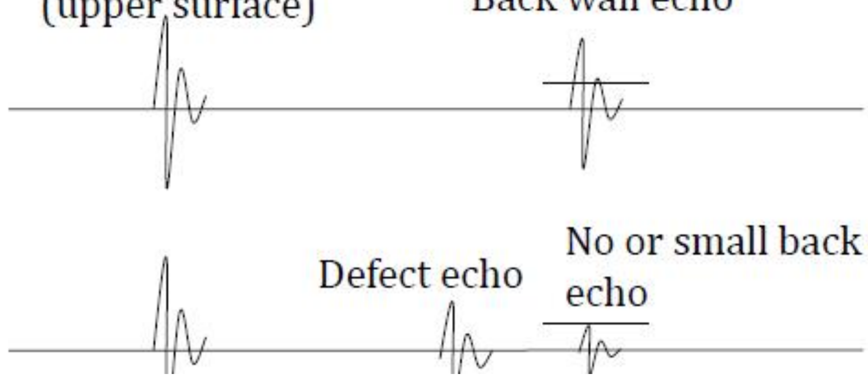
The received signal includes the reflection of the:

- First surface (Front wall echo)
- Second surface (back wall echo)
- Any other reflection due to dincontinuity.

C-Scan of composite plates

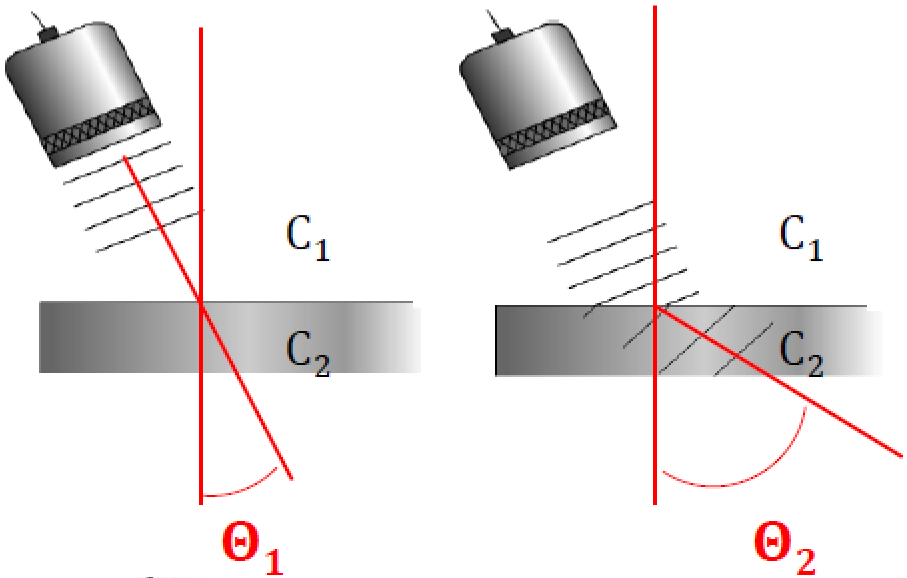




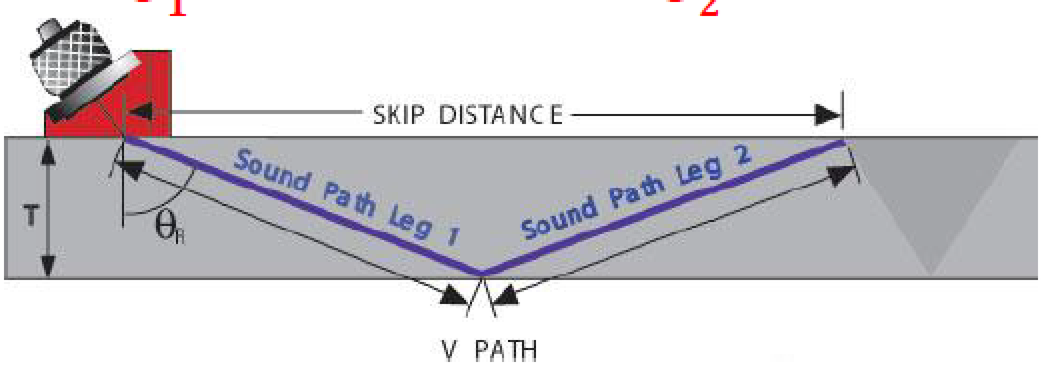


Snell's law

When an ultrasonic wave passes through an interface between two materials at an oblique angle, and the materials have different impedances, both reflected and refracted waves are produced.



$$\frac{C_1}{C_2} = \frac{\sin\theta_1}{\sin\theta_2}$$

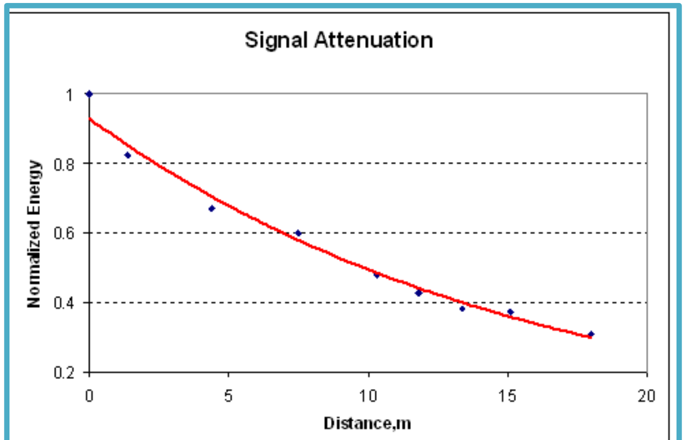


Very useful for welding inspection

Wave Propagation Effects

The following phenomena take place as AE waves propagate along the structure:

- **Attenuation**: The gradual decrease in AE amplitude due to energy loss mechanisms, from dispersion, diffraction or scattering.
- **Dispersion**: A phenomenon caused by the frequency dependence of speed for waves. Sound waves are composed of different frequencies hence the speed of the wave differs for different frequency spectrums.
- **Diffraction**: The spreading or bending of waves passing through an aperture or around the edge of a barrier.
- **Scattering**: The dispersion, deflection of waves encountering a discontinuity in the material such as holes, sharp edges, cracks inclusions etc.…

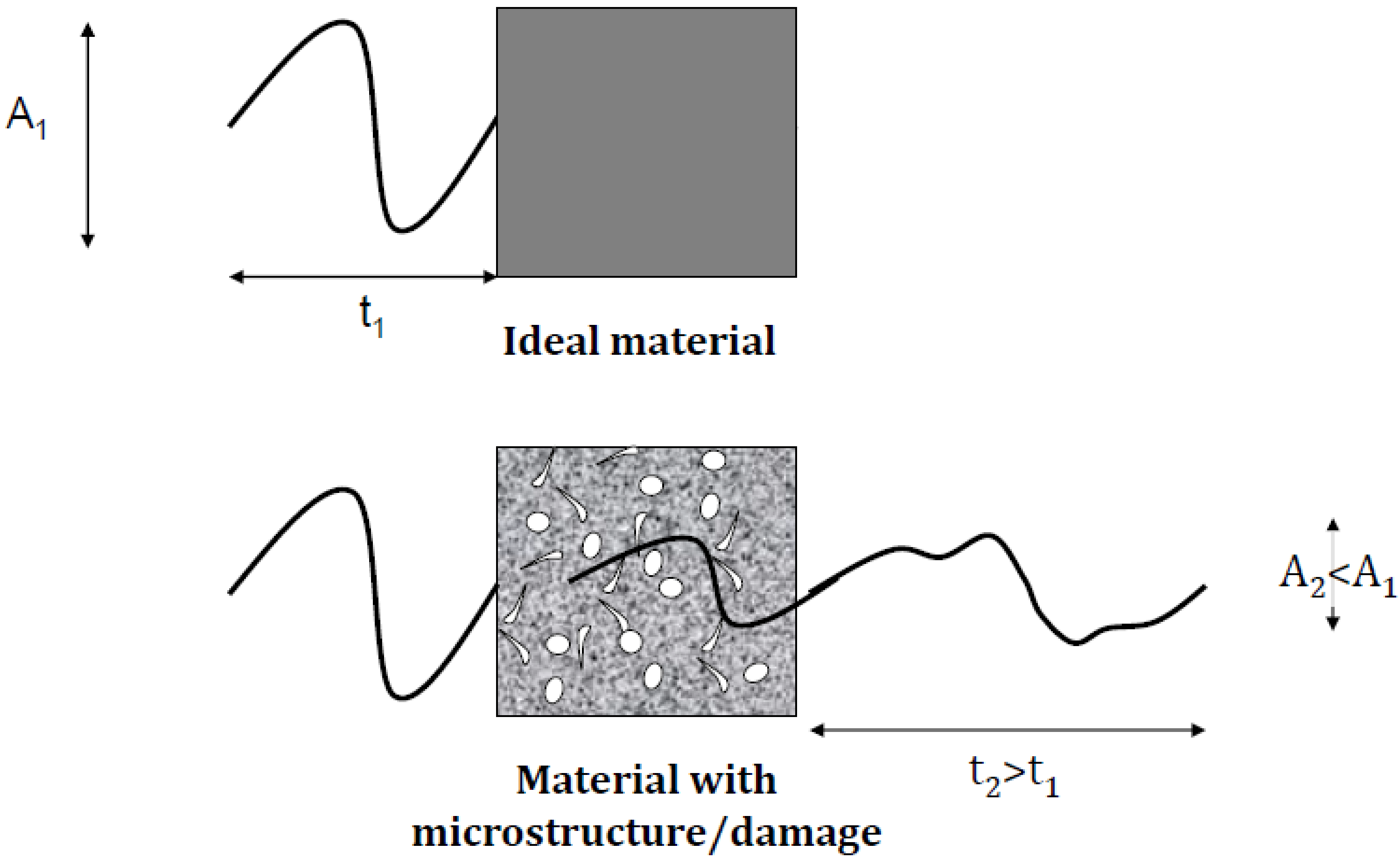


- Attenuation tests have to be performed on actual structures during their inspection.
- The attenuation curves allow to estimate amplitude or energy of a signal at a given distance from a sensor.

From www.muravin.com

Apart from the transit time which defines pulse velocity, thickness, position of defect or elastic modulus of the material, more advanced features are studied.

Dispersion - Attenuation



Acoustic Emission

ASTM-E610-82:

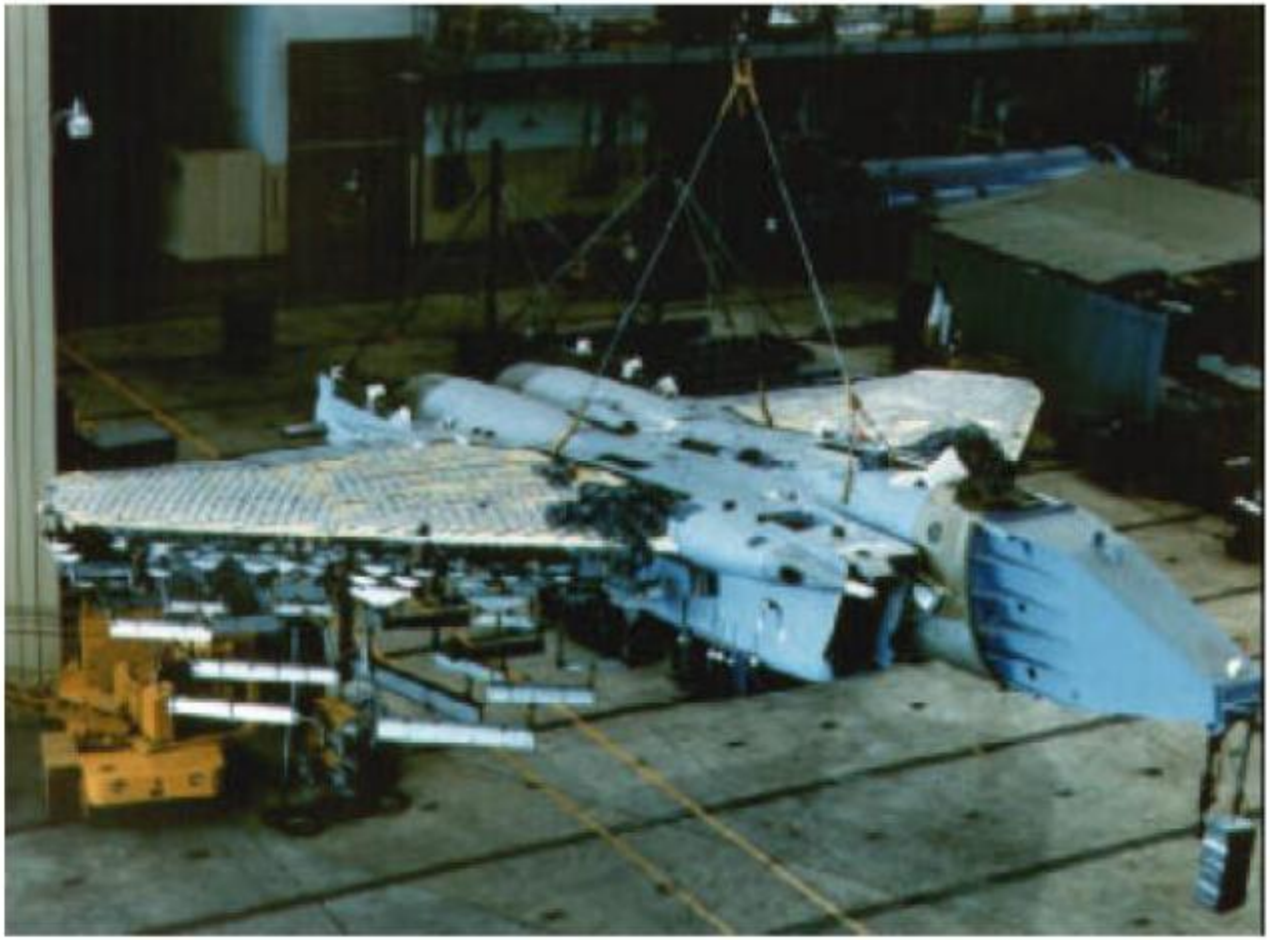
Acoustic Emissions (AE) are the transient elastic waves generated by the rapid release of energy from localized sources within the material.

In real-life

The sound we hear when breaking a wooden stick or tearing a piece of paper or throwing an ice-cube into warm water. If we bend a plastic ruler, individual fibers start breaking and produce audible sounds, which become stronger and more intense as the bending increases, giving us a 'warning' of when the ruler is about to break.

The presence of acoustic emission presupposes the presence of a stress field

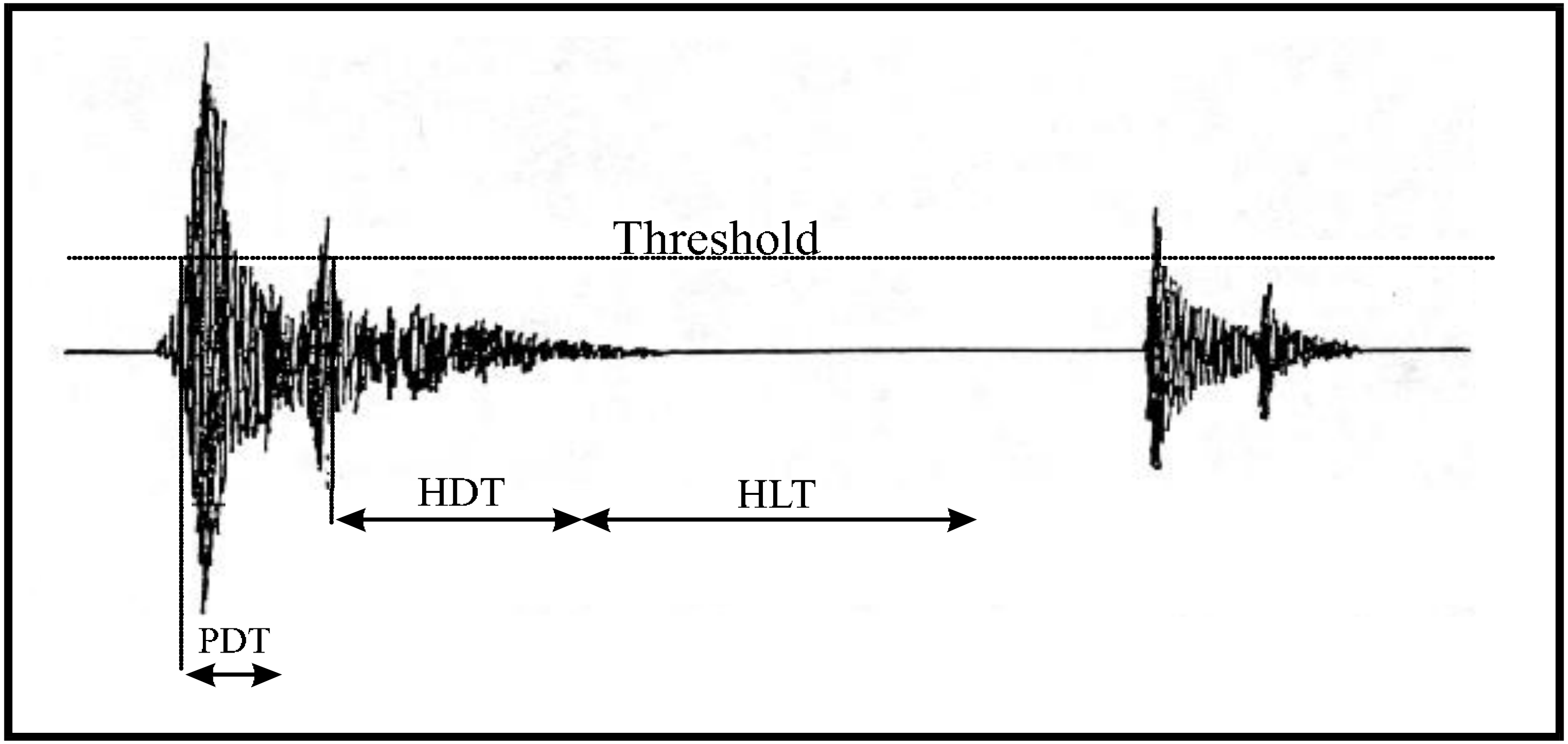
AE inspection in aircrafts



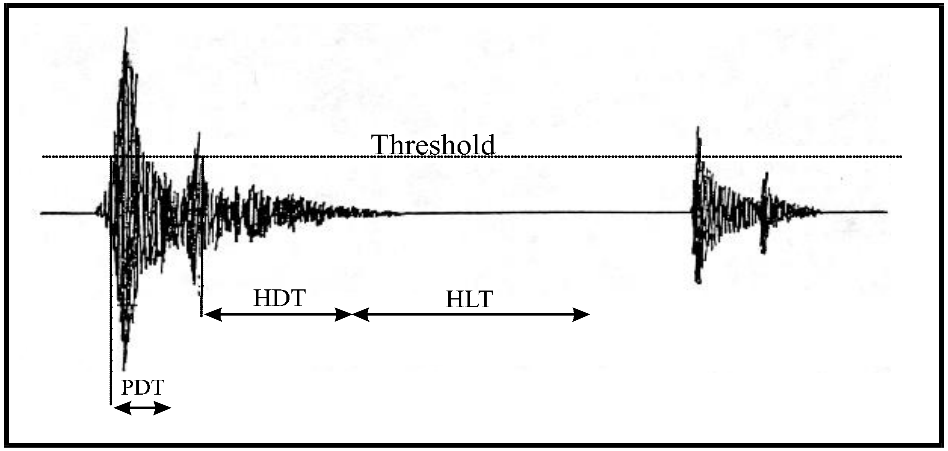
A fighter aircraft being readied for a full scale fatigue test with AE monitoring

R.D. Finlayson et al., Insight 43(3) 2001

Acoustic Emission: Noise or plethora of information?



Acoustic Emission: Descriptors



Hits: Measure of activity

Amplitude: The peak voltage of the AE hit. It is useful as a measure of intensity, key to delectability (attenuation) and the failure characterization.

Energy: The area between the hit's voltage curve and the time axis. This feature serves as measure of activity.

Counts: The number of times that the voltage has exceeded the threshold. This feature serves also as measure of activity.

Duration: The time period between the first and the last threshold crossings. Useful for signal qualification and noise rejection.

Rise Time: The time period between the first threshold crossing and the peak voltage . Useful for signal qualification and noise rejection.

Counts to Peak: The number of counts that occurred within the rise time. Signal qualification and spectral information.

Classification of AE

AE classes: material and mechanical

AE source mechanism size: macro- and micro-scopic

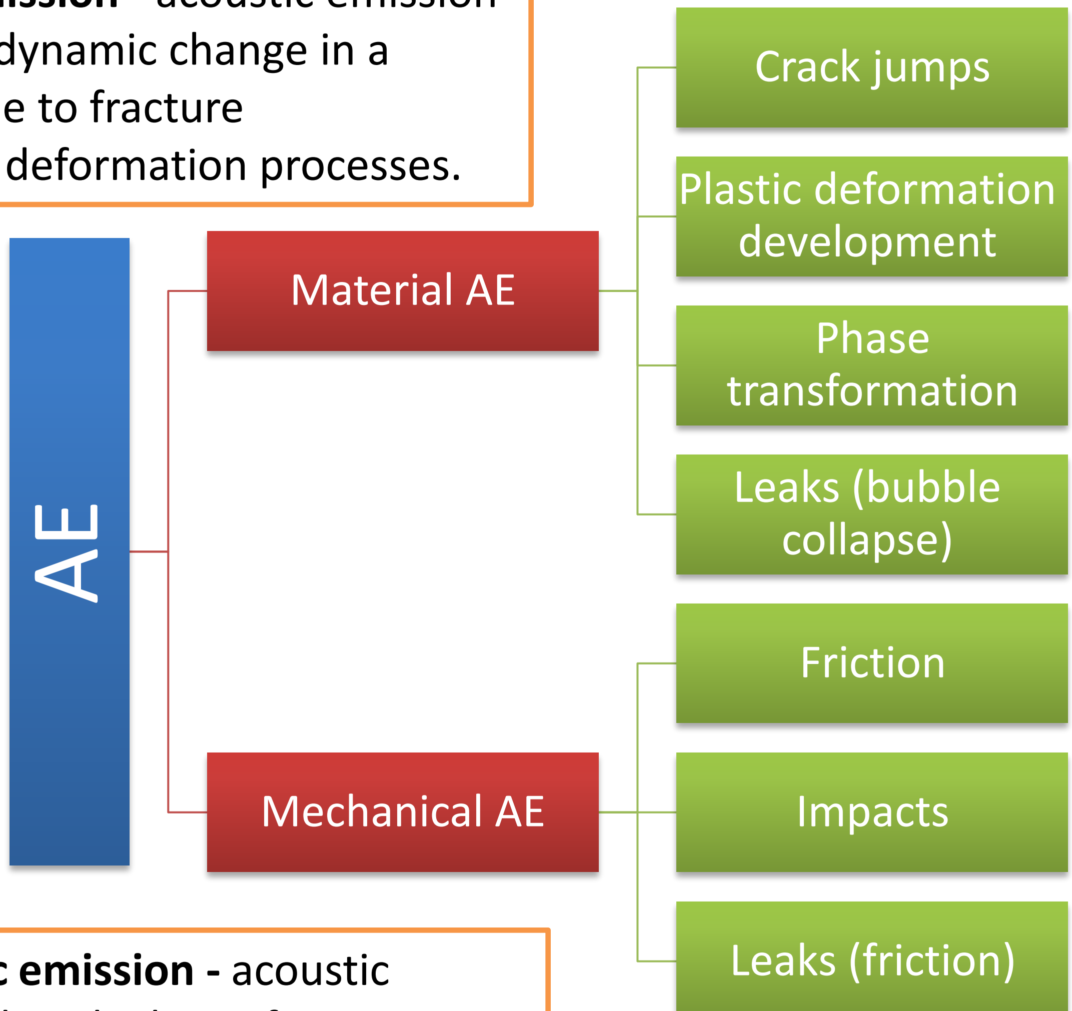
AE types: burst and continuous.

Significance/occurrence: primary and secondary.

From: www.muravin.com

Classes and Mechanisms of Acoustic Emission

Material acoustic emission - acoustic emission generated by a local dynamic change in a material structure due to fracture development and/or deformation processes.



Mechanical acoustic emission - acoustic emission generated by a leakage, friction, impact or other sources of mechanical origin.

From: www.muravin.com

Primary vs. Secondary AE

Primary AE	Secondary AE
Crack jump	Crack surface friction
Plastic deformation	Inclusion breakage in the process zones
Crack growth	Corrosion layer fracture in corrosion fatigue cases

Source Mechanisms in Composites

Matrix cracking, Fiber fracture, Delamination, Fiber pullout, Friction.

AE Effects

- Kaiser effect is the absence of detectable AE at a fixed sensitivity level, until previously applied stress levels are exceeded.
- Dunegan corollary states that if AE is observed prior to a previous maximum load, some type of new damage has occurred. The *dunegan corollary* is used in proof testing of pressure vessels.
- Felicity effect is the presence of AE, detectable at a fixed predetermined sensitivity level at stress levels below those previously applied. The felicity effect is used in the testing of fiberglass vessels and storage tanks.

$$\text{felicity ratio} = \frac{\text{stress at onset of AE}}{\text{previous maximum stress}}$$

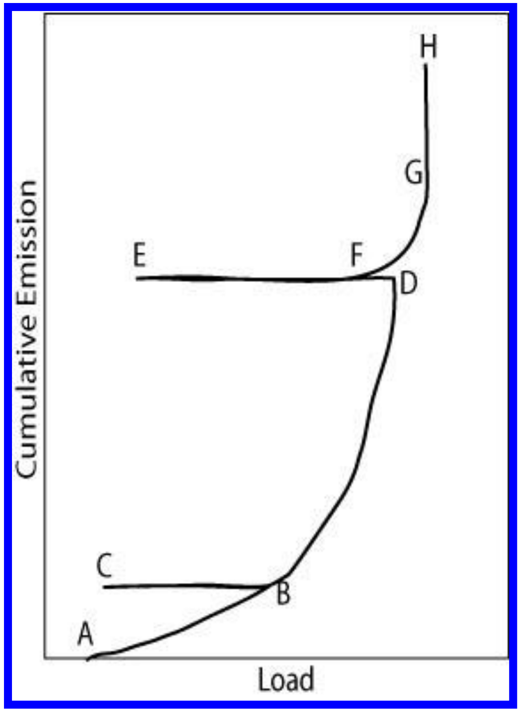
ASTM E1316: 2010

Kaiser effect—the absence of detectable acoustic emission at a fixed sensitivity level, until previously applied stress levels are exceeded.

Discussion—Whether or not the effect is observed is material specific. The effect usually is not observed in materials containing developing flaws.

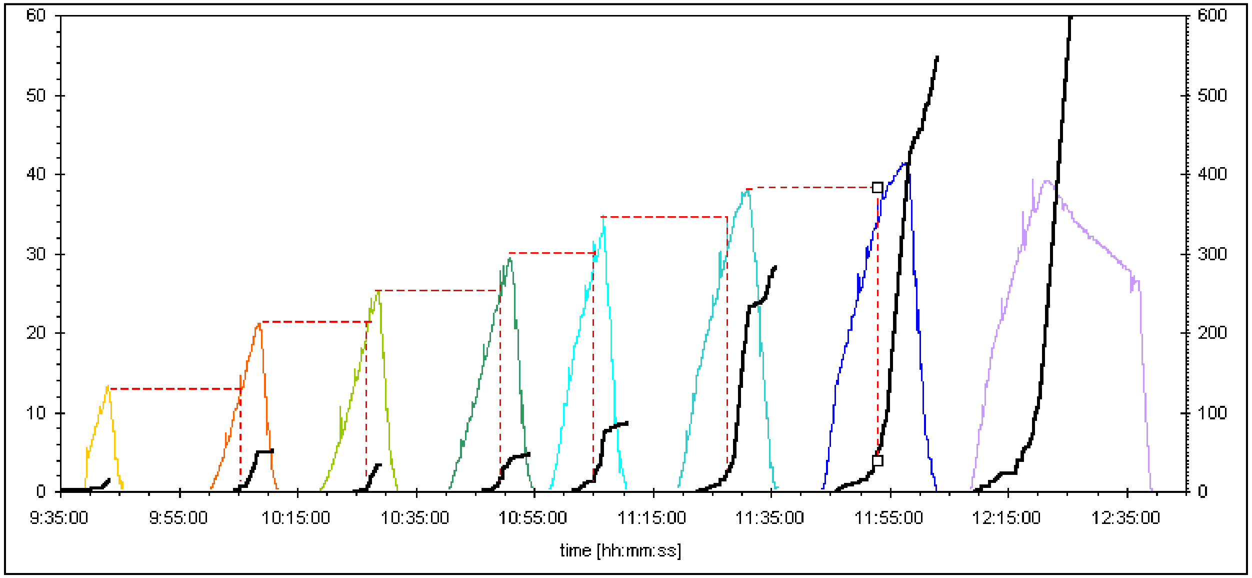
Kaiser effect *(BCB)*

Felicity effect *(DEF)*



Kaiser Effect

- The immediately irreversible characteristic of AE resulting from an applied stress at a fixed sensitivity level.
- If the effect is present, there is an absence of detectable AE until previously applied stress levels are exceeded.

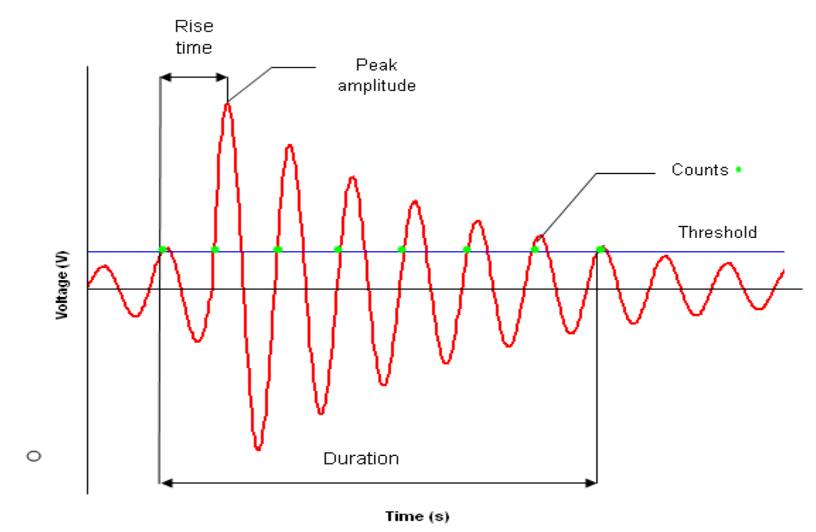


Example of the Kaiser Effect in a cyclically loaded concrete specimen. Thick black lines represents AE activity, thin lines the loads and dashed lines the Kaiser Effect.

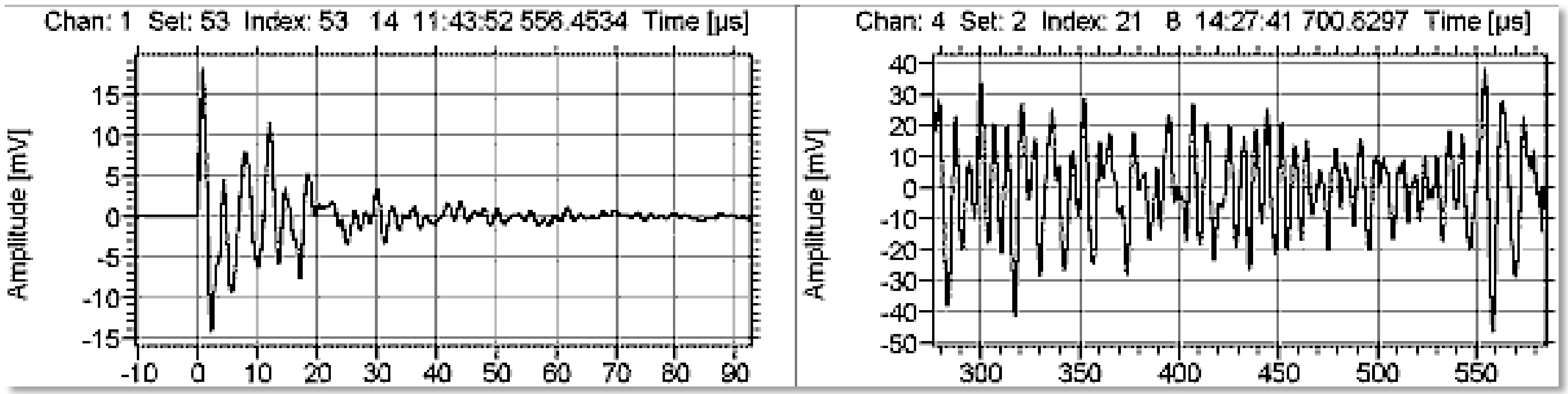
http://www.ndt.net/ndtaz/content.php?id=476

AE Types: Burst and Continuous AE Signals

Burst AE is a qualitative description of the discrete signal's related to individual emission events occurring within the material.

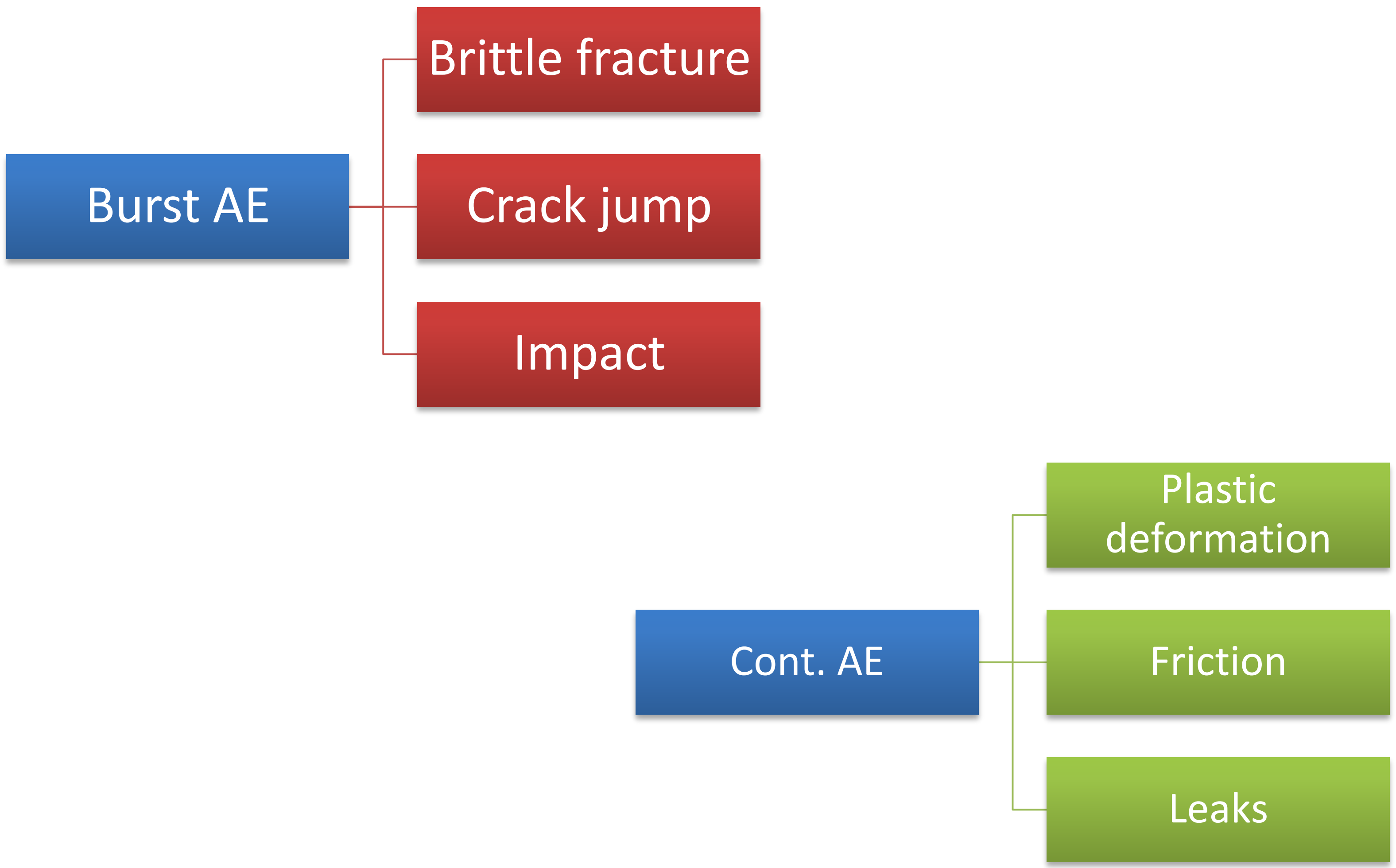


Continuous AE is a qualitative description of the sustained signal produced by time-overlapping signals.



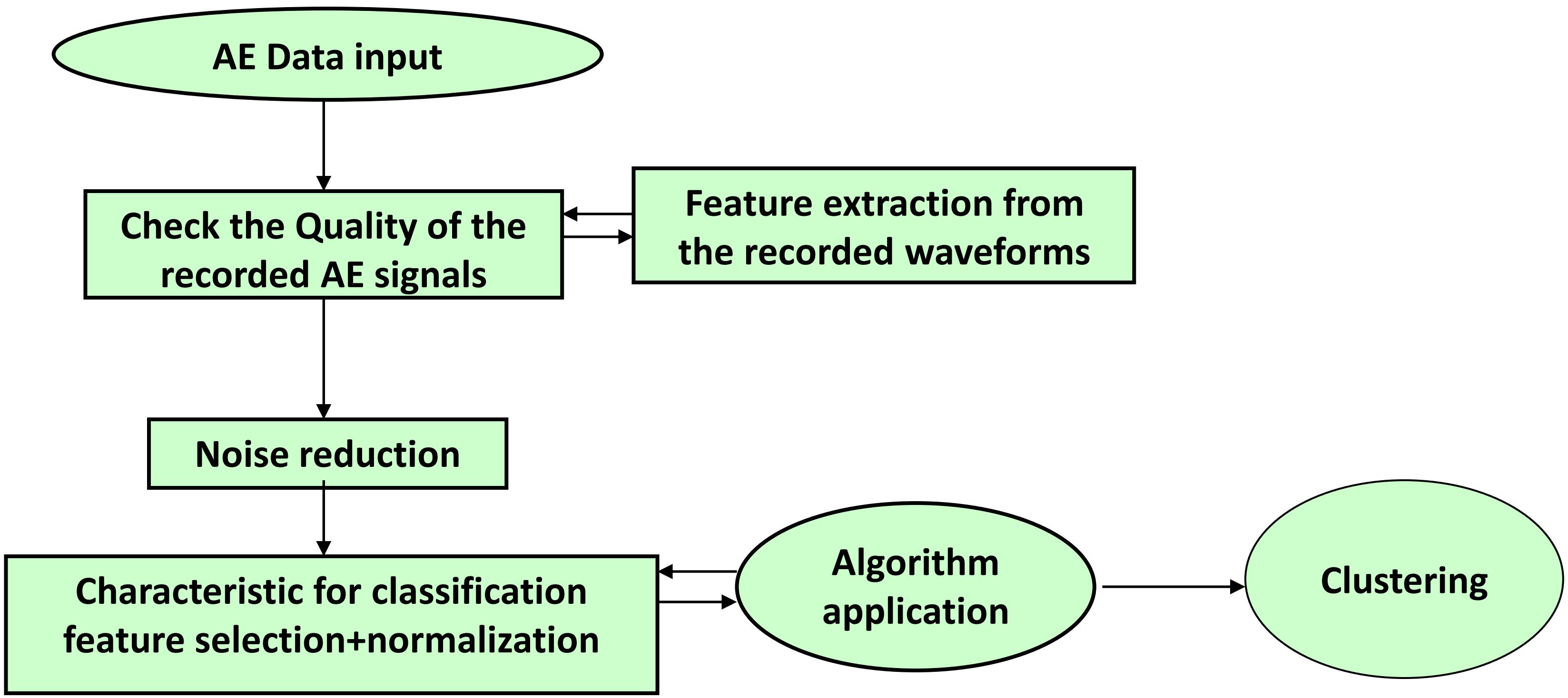
From www.muravin.com

Some Mechanisms of Burst and Cont. AE

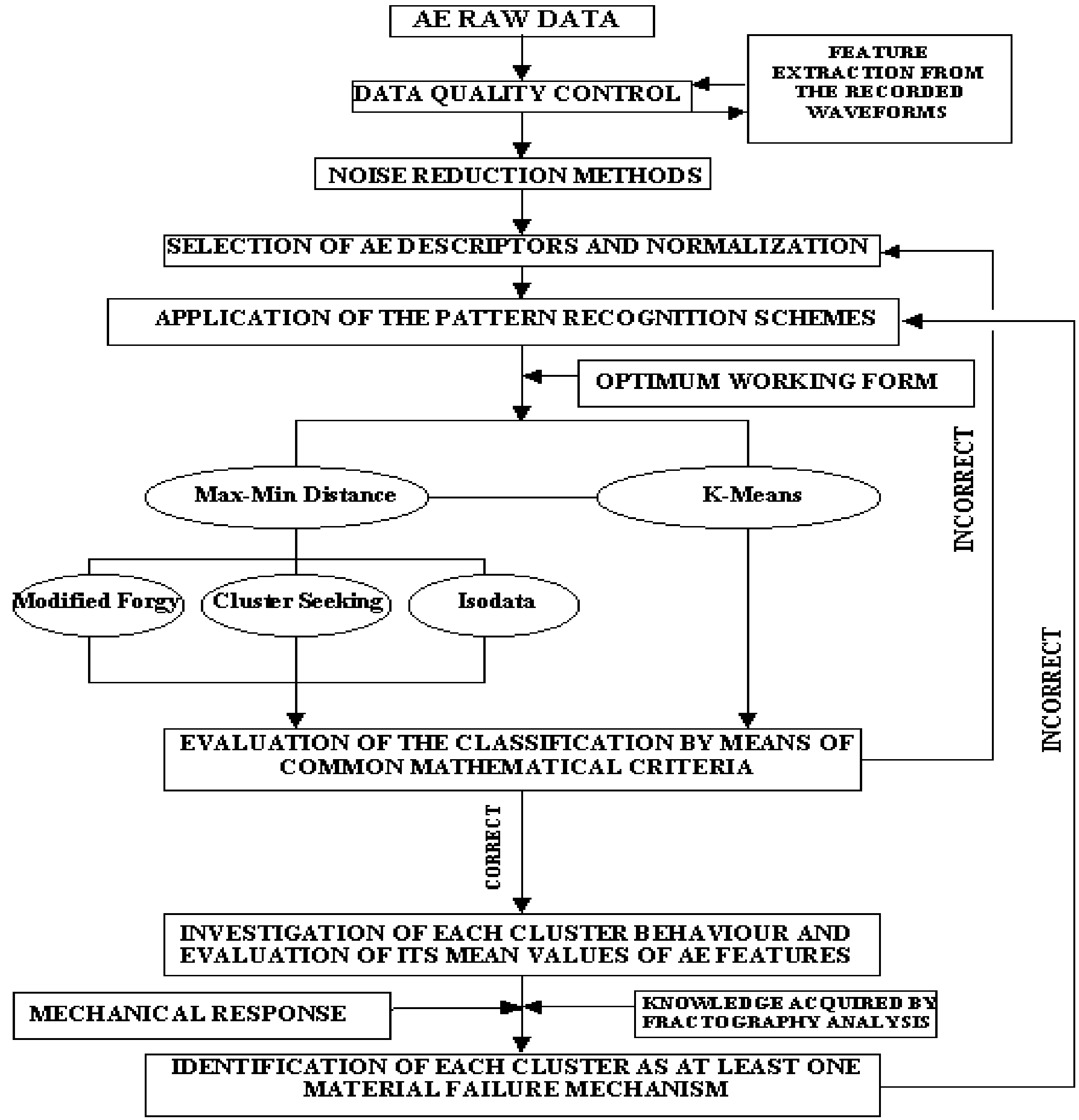


Acoustic Emission: Pattern Recognition algorithm

Acoustic emission data enter a PR scheme in the form of pattern vectors: $X=[x_1\ x_2 \ldots x_n]^T$. The components of this vector are AE features such as Duration, Counts, Amplitude, Energy etc. of the recorded AE hits.

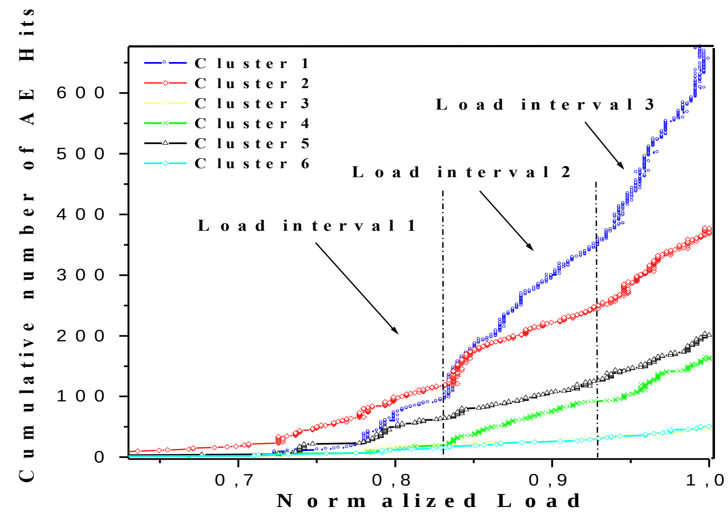


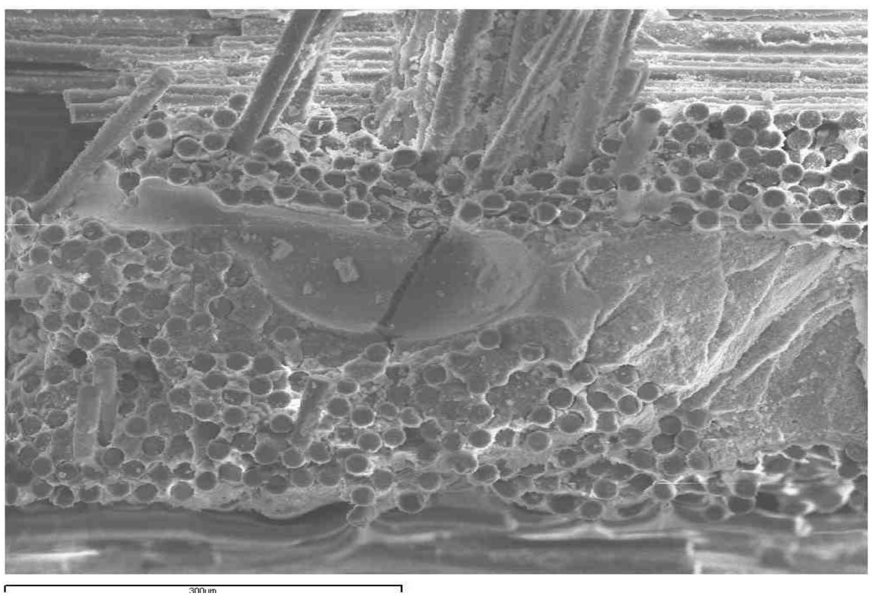
Acoustic Emission: Pattern Recognition algorithm

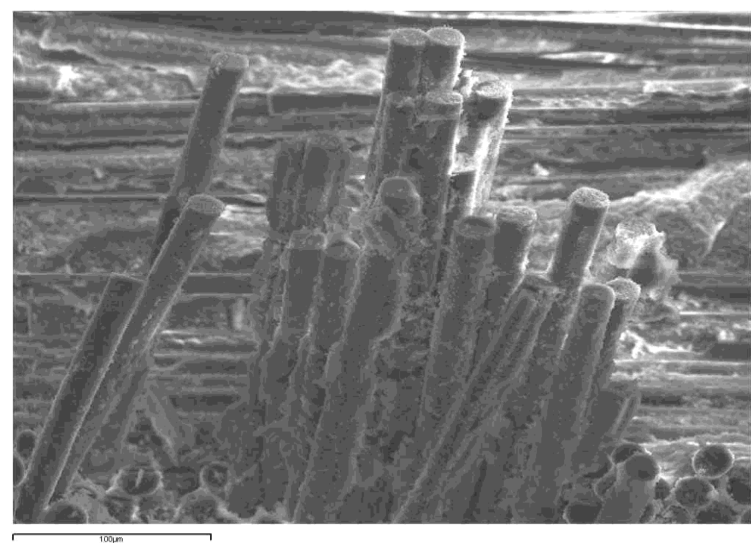


Acoustic Emission: Damage Mode identification

Cluster 1: matrix cracking
Cluster 2: stochastic fibre failure
Cluster 5: fibre/matrix debonding-interface disruption
Cluster 4: fibre pullout- destruction of the woven structure
Clusters 3,6: reverberation/reflection phenomena, noise, minor friction events





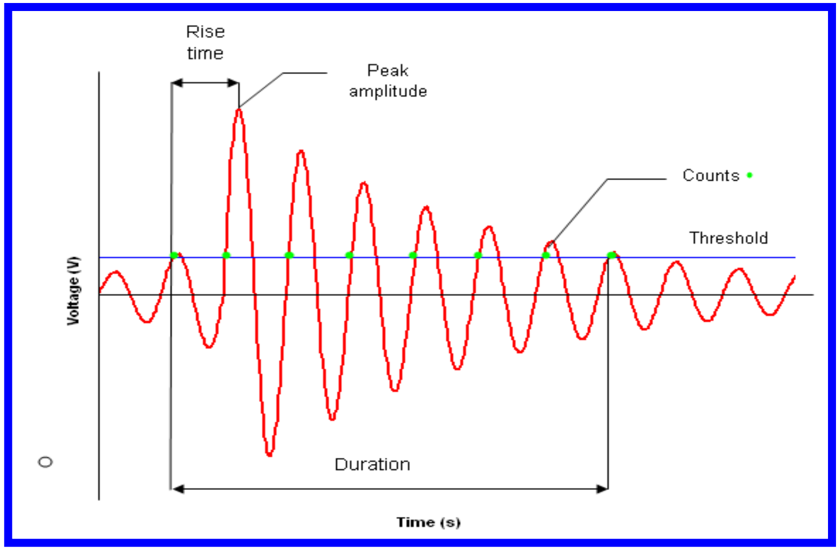


Acoustic Emission Source Location

- Time difference based on Time of Arrival locations.
- Cross-correlation time difference location.
- Zone location.
- Attenuation based locations.
- Geodesic location.

Time of Arrival Evaluation

- Most of existing location procedures require evaluation of time of arrival (TOA) of AE waves to sensors.
- TOA can detected as the first threshold crossing by AE signal, or as a time of peak of AE signal or as a time of first motion. TOA can be evaluated for each wave mode separately.



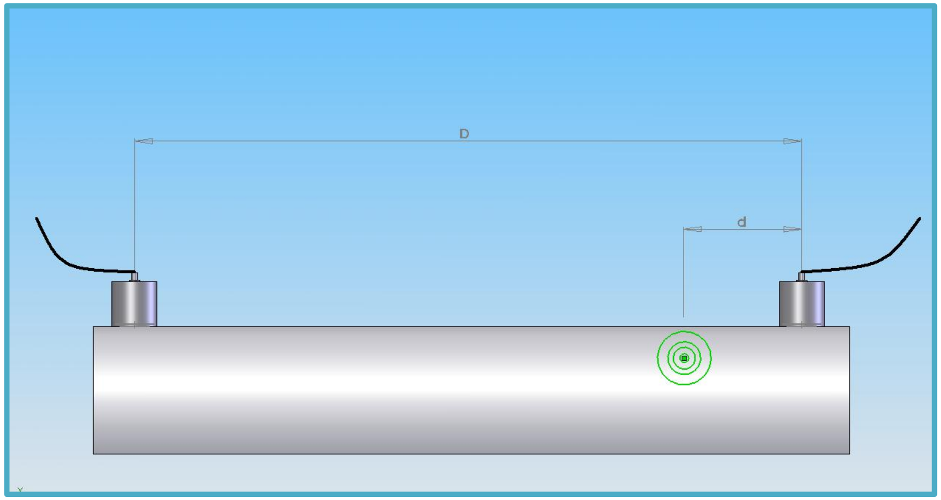
Effective Velocity

- Another parameter necessary for time difference location method is effective velocity.
- Effective velocity can be established experimentally with or without considering different wave propagation modes.
- When propagation modes are not separated, the error in evaluation of AE source location can be significant. For example, in linear location it can be about 10% of sensors spacing.
- Detection of different wave modes arrival times separately and evaluation of their velocities can significantly improve location accuracy. Nevertheless, detection and separation of different wave modes is computationally expensive and inaccurate in case of complex geometries or under high and variable background noise conditions.

Material	Effective velocity in a thin rod [m/s]	Shear [m/s]	Longitudinal [m/s]
Brass	3480	2029	4280
Steel 347	5000	3089	5739
Aluminum	5000	3129	6319

Linear Location

- Linear location is a time difference method commonly used to locate AE source on linear structures such as pipes, tubes or rods. It is based on evaluation of time difference between arrival of AE waves to at least two sensors.
- Source location is calculated based on time difference and effective wave velocity in the examined structure. Wave velocity usually experimentally evaluated by generating artificially AE at known distances from sensors.



$$d = \frac{1}{2}\left(D - \Delta T \cdot V\right)$$

d = distance from first hit sensor

D = distance between sensors

V = wave velocity

Two Dimensional Source Location

For location of AE sources on a plane minimum three sensors are used. The source is situated on intersection of two hyperbolas calculated for the first and the second sensors detected AE signal and the first and the third sensor.

$$\Delta t_{1,2} V = R_1 - R_2$$

$$Z = R_2 \sin\theta$$

$$Z^2 = R_1^2 - (D - R_2)^2$$

$$\rightarrow R_2^2 \sin^2\theta = R_1^2 - (D - R_2\cos\theta)^2$$

$$R_2^2 = R_1^2 - D^2 + 2D\cos\theta$$

$$R_1 = \Delta t_{1,2} V + R_2$$

$$\Rightarrow R_2 = \frac{1}{2}\frac{D^2 - \Delta t_{1,2}^2 V^2}{\Delta t_{1,2} V + D\cos\theta}$$

D – distance between sensor 1 and 2

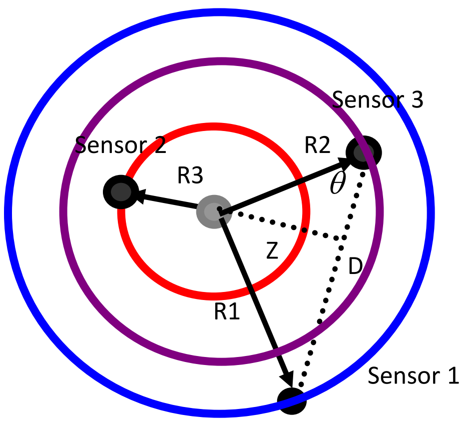
R_1 – distance between sensor 1 and source

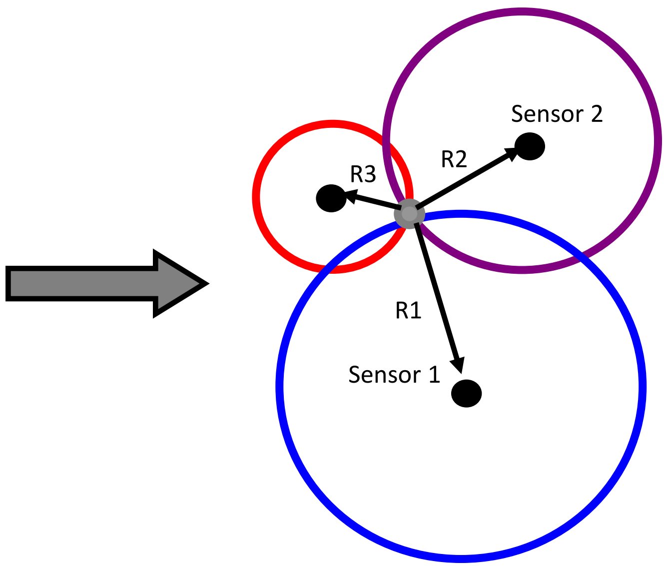
R_2 – distance between sensor 2 and source

$\Delta t_{1,2}$ – time differance between sensor 1 and 2

θ – angle between lines R_2 and D

Z – line perpendicular to D





Over-determined Source Location

- Generally, it is necessary 2 sensors for linear, 3 sensors for 2D and 4 sensors for 3D locations.
- When more sensors detect AE wave from a source than necessary it is possible to use this information to improve location accuracy by error minimization optimization methods.

$$\chi^2 = \sum (\Delta t_{i,obs} - \Delta t_{i,calc})^2$$

Chi Squared error function that minimized in over-determined source location.

$$\Delta t_{i,calc} = \left(\sqrt{(x_i - x_s)^2 + (y_i - y_s)^2} - \sqrt{(x_1 - x_s)^2 + (y_1 - y_s)^2} \right) \frac{1}{V}$$

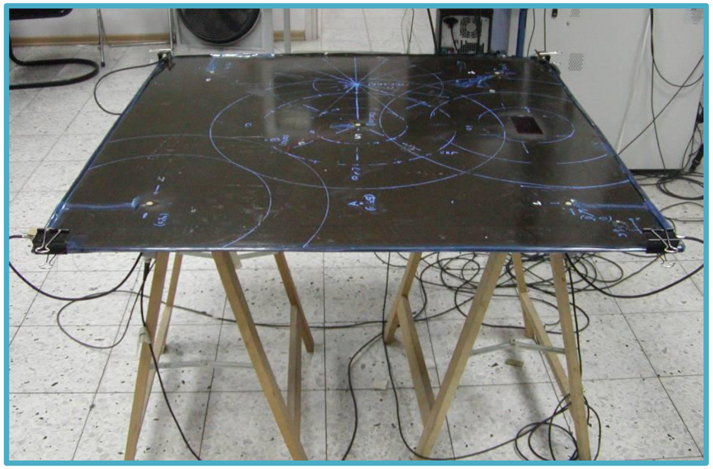
$\Delta t_{i,calc}$ = The calculated time difference between the i sensor and the first hit sensor, where x_s and y_s are the unknown coordinates of the source.

$\Delta t_{i,obs}$ = The observed time difference

Location in Anisotropic Materials

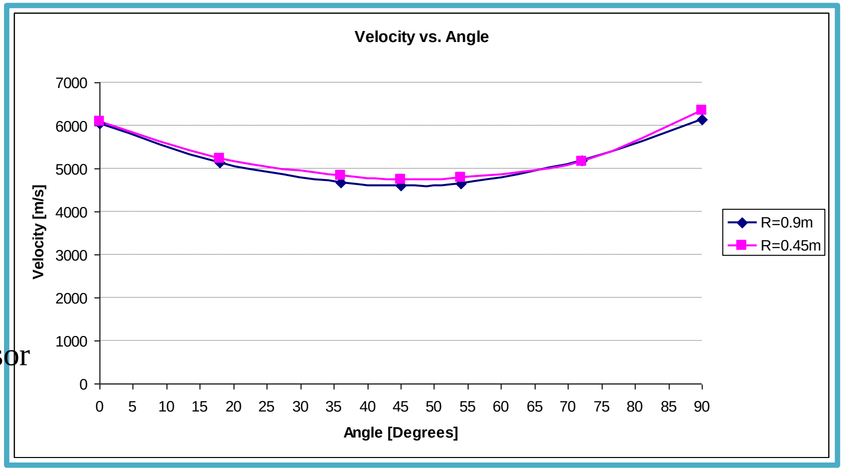
- In anisotropic materials, the velocity of wave propagation is different in different direction.
- In order to achieve appropriate results in source location it is necessary to evaluate velocity profile as a function of propagation direction and incorporate this into the calculation of time differences as done in the example of the composite plate.

R=0.9m		R=0.45m	
Angle [Degrees]	Velocity [m/s]	Angle [Degrees]	Velocity [m/s]
0	6035	0	6101
18	5137	18	5224
36	4671	36	4843
45	4600	45	4741
54	4649	54	4784
72	5182	72	5164
90	6141	90	6345



$$\Delta t_{i,calc} = \frac{\sqrt{(x_i - x_s)^2 + (y_i - y_s)^2}}{v_{\theta,i}} - \frac{\sqrt{(x_1 - x_s)^2 + (y_1 - y_s)^2}}{v_{\theta,1}}$$

$\Delta t_{i,calc}$ = The time difference recorded by the i sensor relative to the first hit sensor



Other Location Methods

- Cross-correlation based Location
- Zone location
- Geodesic Location
- FFT and wavelet transforms are be used to improve location by evaluation of modal arrival times.
- Cross-correlation between signals envelopes.
- There are works proposing use of neural network methods for location of continuous AE.

Case study 1: ANISOTROPIC DAMAGE MODELLING OF COMPOSITE MATERIALS USING ULTRASONIC STIFFNESS MATRIX MEASUREMENTS

Paipetis A, et al Advanced Composites Letters. 2005;14(3):85-94

Introduction - Scope of work

- Oxide/Oxide composites in gas turbine engine applications
- Application of advanced material characterisation techniques
  - ¤ Periodic exposure to a simulating environment
  - ¤ Stiffness matrix identification from ultrasonic velocity measurements
- Damage evolution modelling
- Damage evolution simulation

Ultrasonic Stiffness Measurements

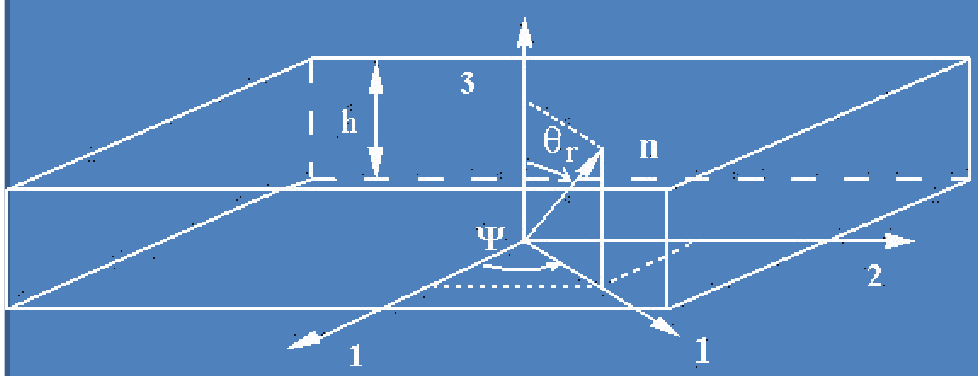
Wave propagation tensor: $\Gamma_{\mathbf{ij}} = C_{ilkj}\, n_l\, n_k$

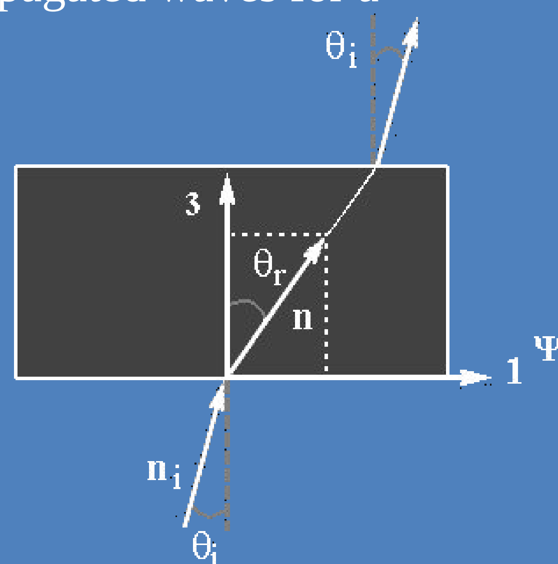
where C_{ilkj} $\rightarrow$ elasticity tensor

n_k (k=1,2,3) $\rightarrow$ propagation direction vector components

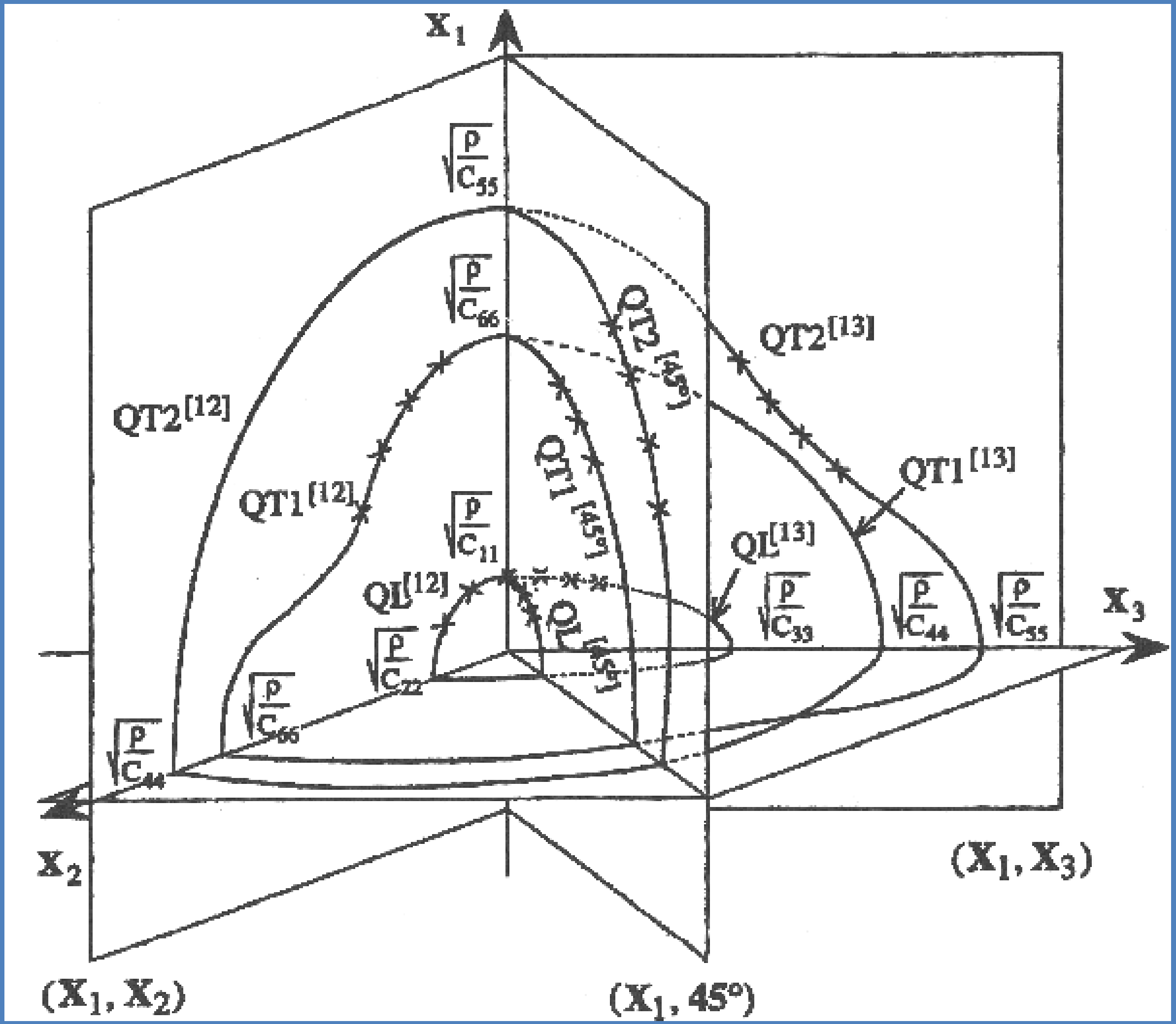
Wave propagation equation (Christoffel): $\det(\Gamma_{ij} - \rho V^2 \delta_{ij}) = 0$

eigenvalues $\rightarrow$ phase velocities of the three propagated waves for a given propagation direction **n**





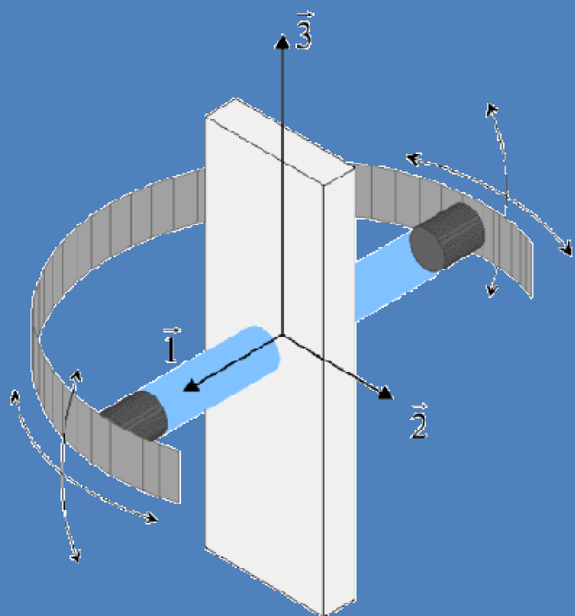
THEORETICAL BEHAVIOUR OF AN ORTHOTROPIC MATERIAL

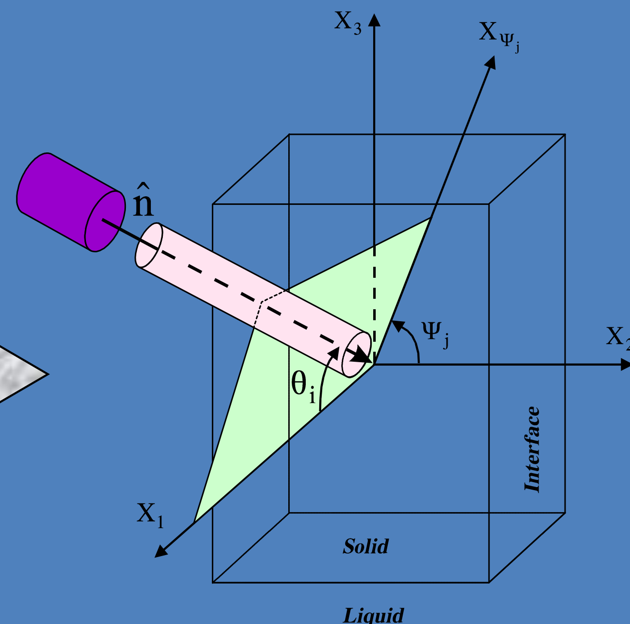


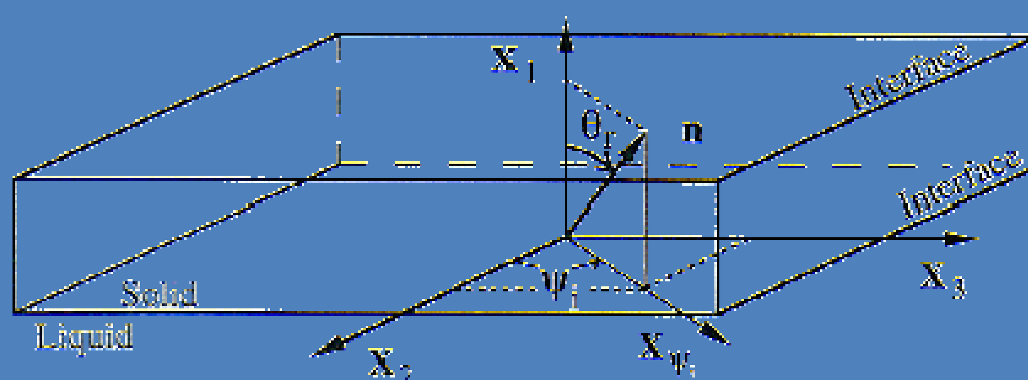
Slowness Curves

S. Baste, et al.

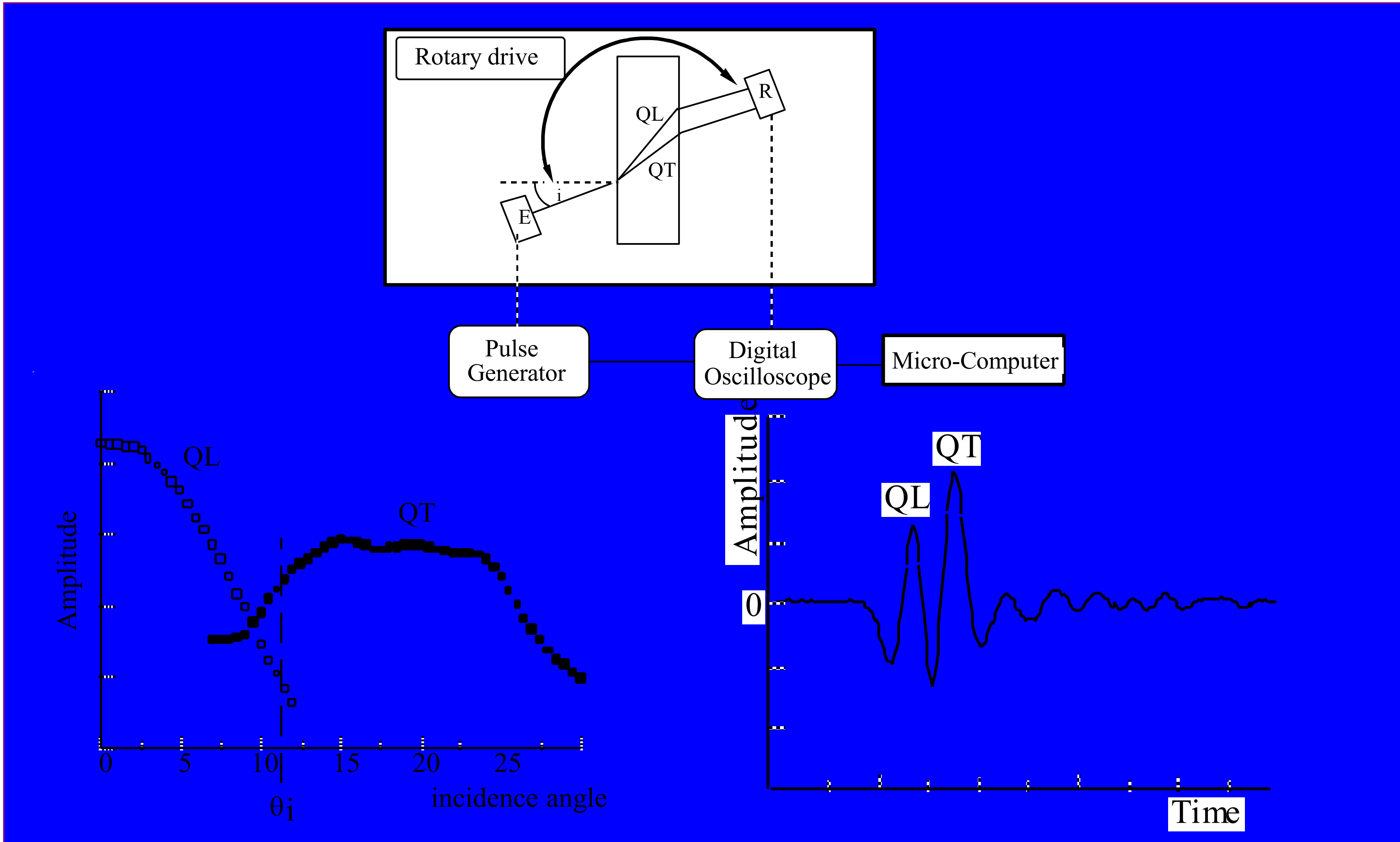
STIFFNESS MEASUREMENT PROCESS







ULTRASONIC MONITORING



Ultrasonic Stiffness Measurements

Propagation velocities

⇓ Least square regression analysis (minimization of the residuals of the wave propagation equations for the complete set of measurements)

The components of the elasticity tensor

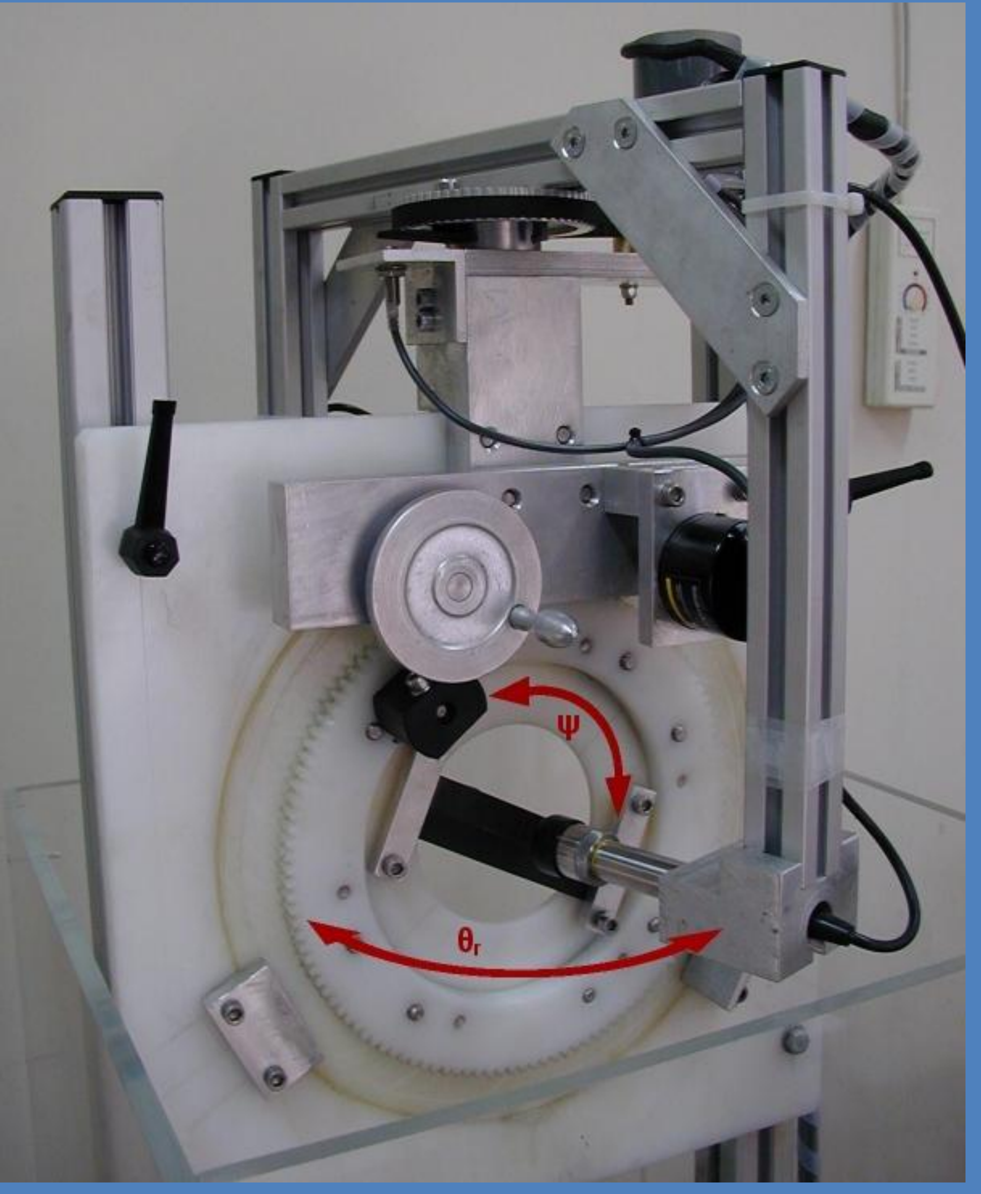
$$F(C_{ij}) = \sum_{p=1}^{N} \left\{ f_p \left(\lambda_p(n), C_{ij} \right) \right\}^2$$

where p = 1 to N, N is the total number of measurements of a range of incident angles θ_i, each corresponding to a different propagation direction **n**, and $\lambda_p = \rho_b V_p^2$

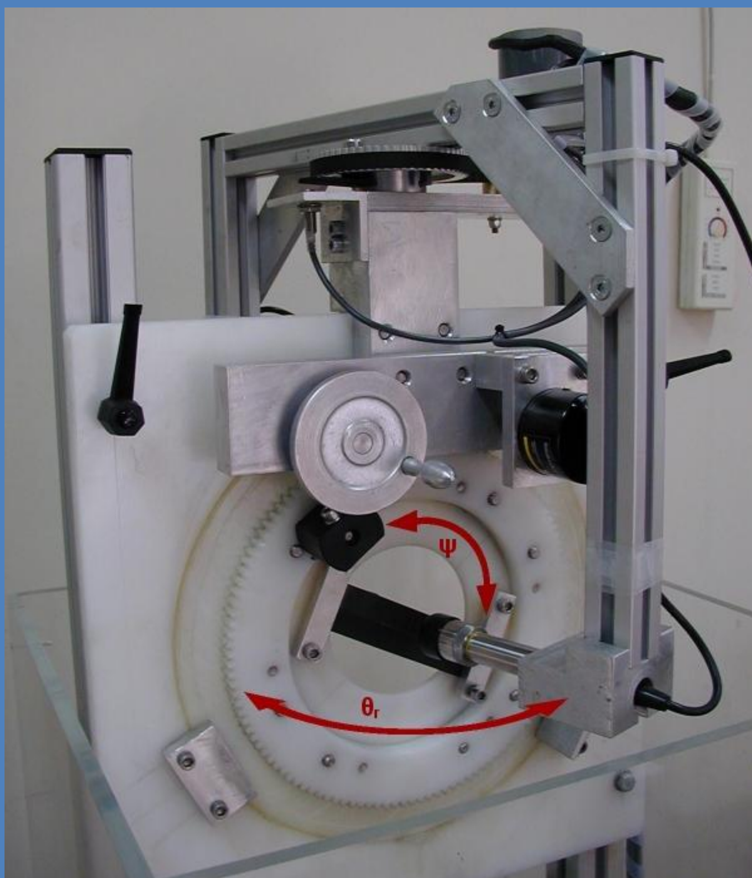
Material
Oxide /Oxide Composites

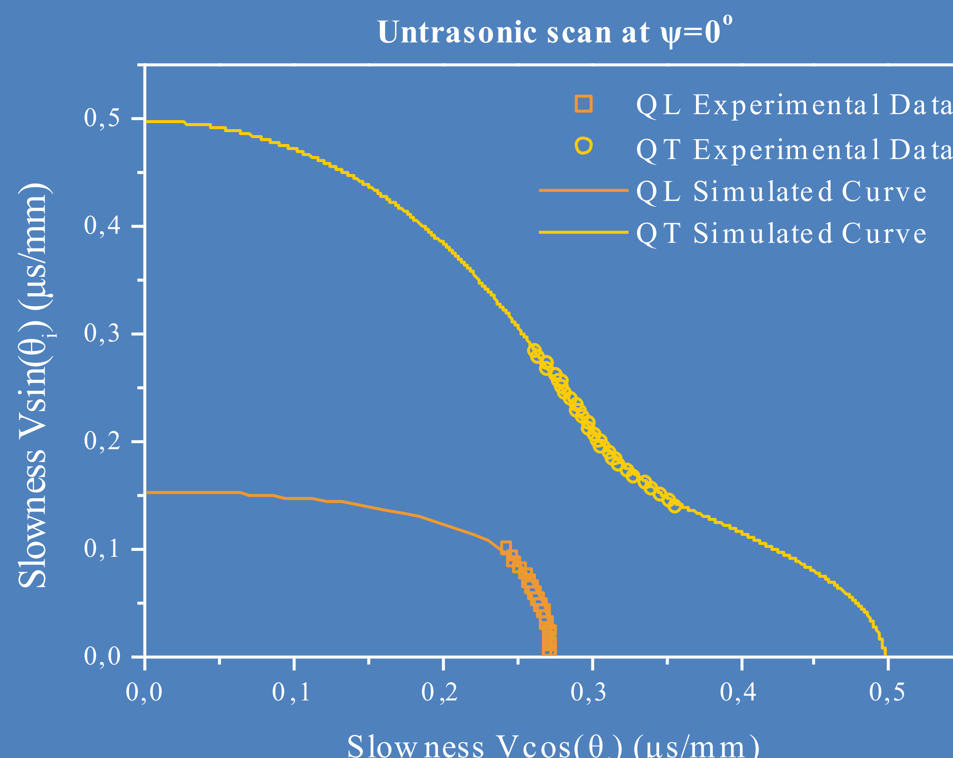
- mullite matrix NEXTEL 720 (3000 denier) fibre reinforced composite with a fugitive fibre/matrix carbon interface applied by sol/gel technique manufactured by EADS/Dornier GmbH .
- The composite was manufactured using a symmetric 0˚/90˚ fibre lay-up configuration with the polymer infiltration process (PIP). The final fibre content is 41%.
- An 150x150 mm2 was manufactured as above. Specimens were cut from the plate using a heavy duty diamond saw.

Ultrasonic Stiffness Measurements Setup

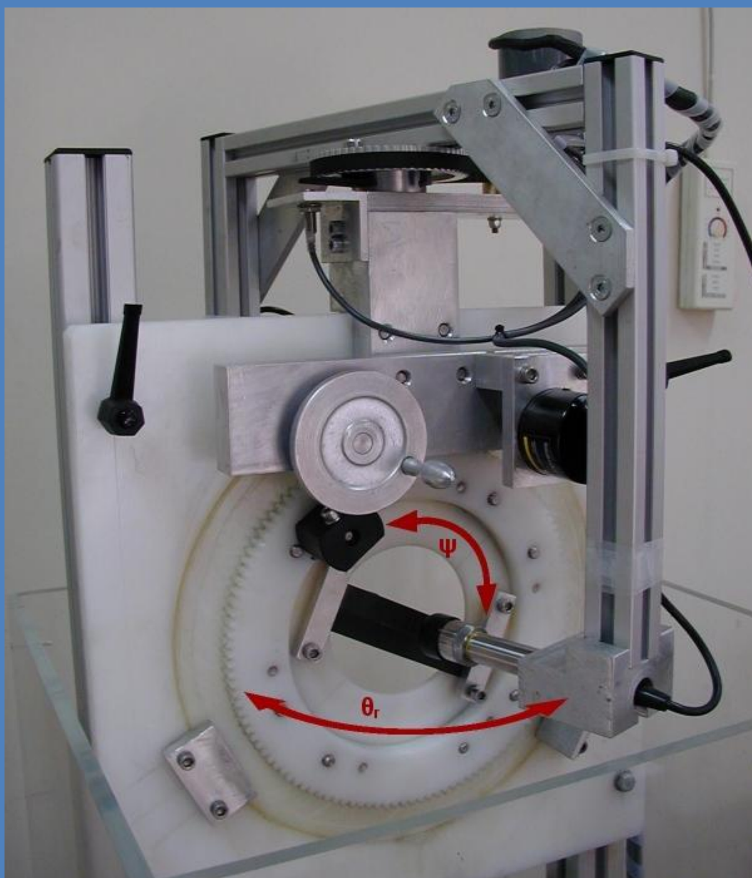


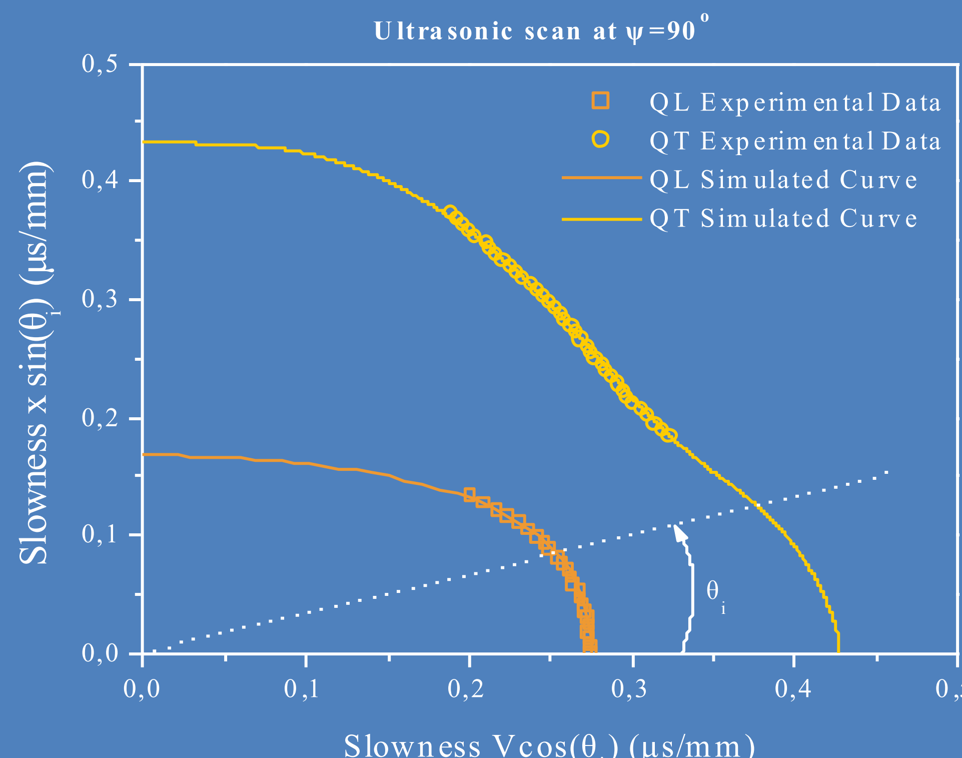
Ultrasonic Stiffness Measurements



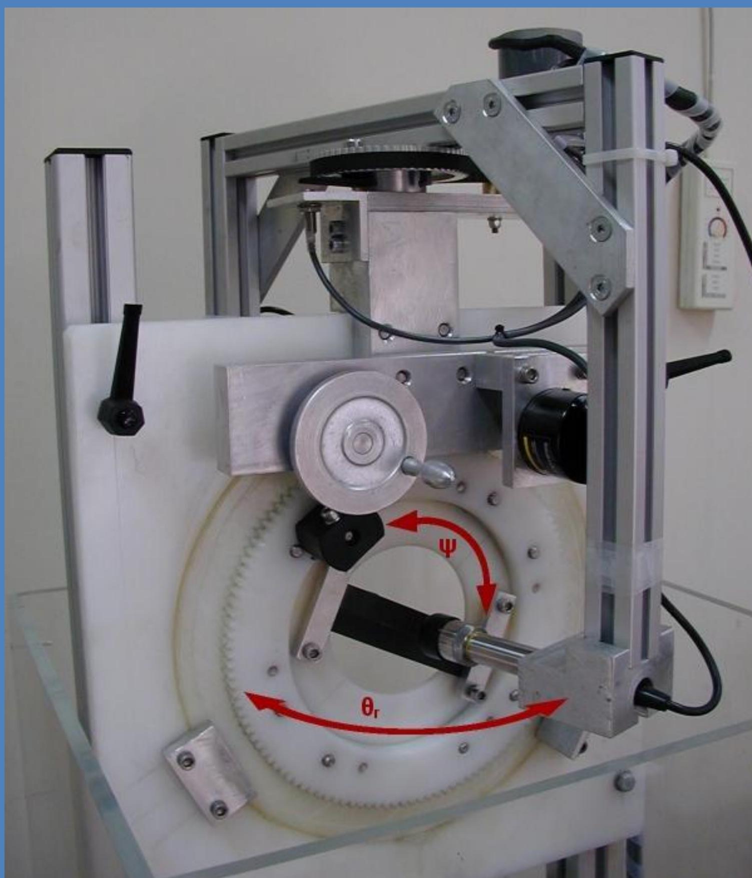


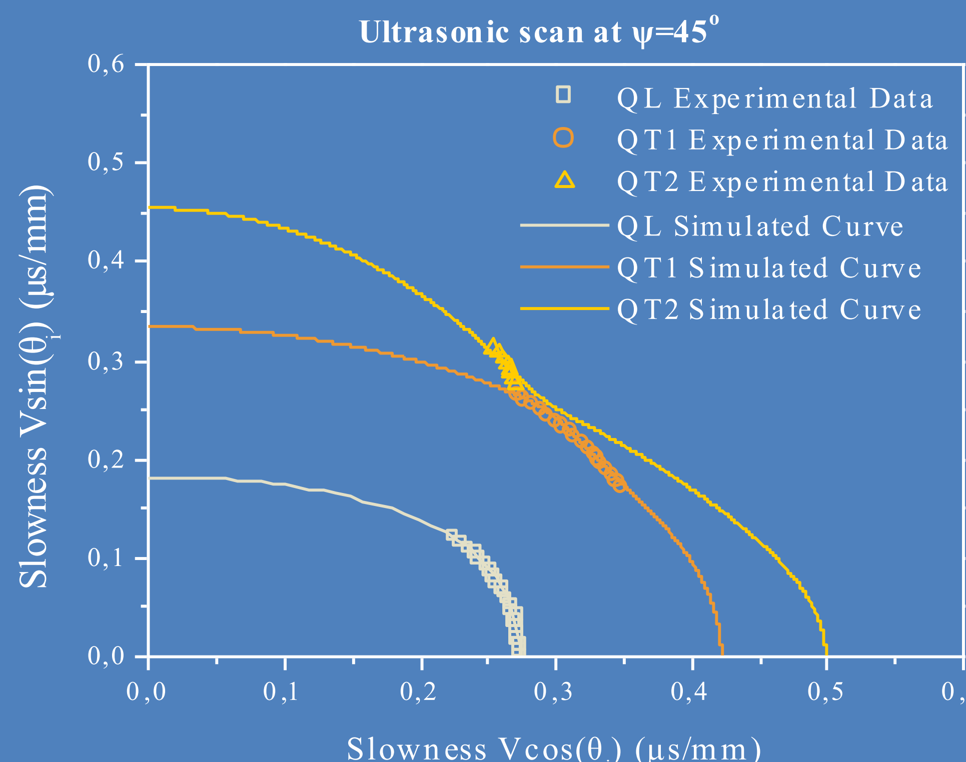
Ultrasonic Stiffness Measurements





Ultrasonic Stiffness Measurements



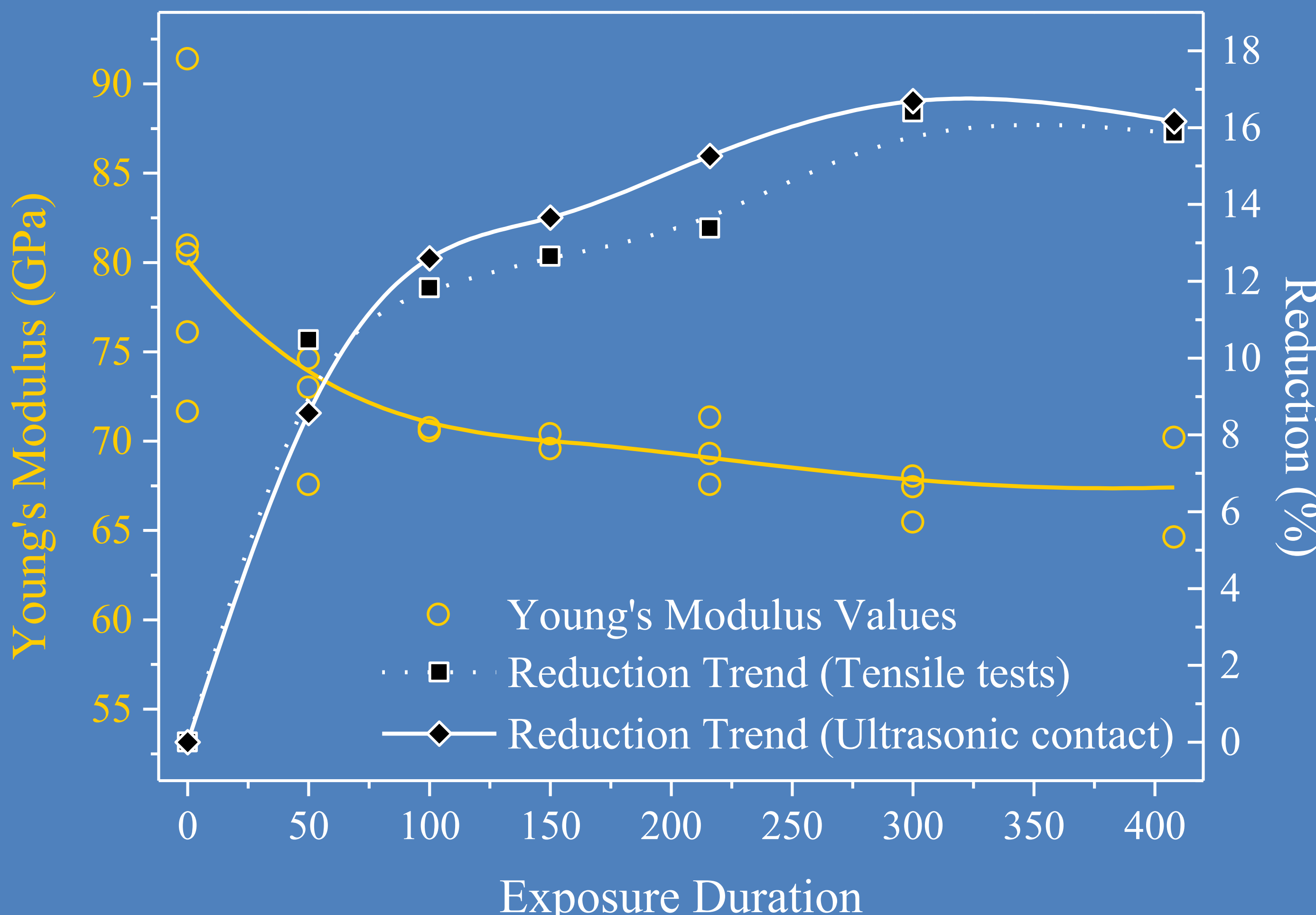


ULTRASONIC METHOD RESULTS

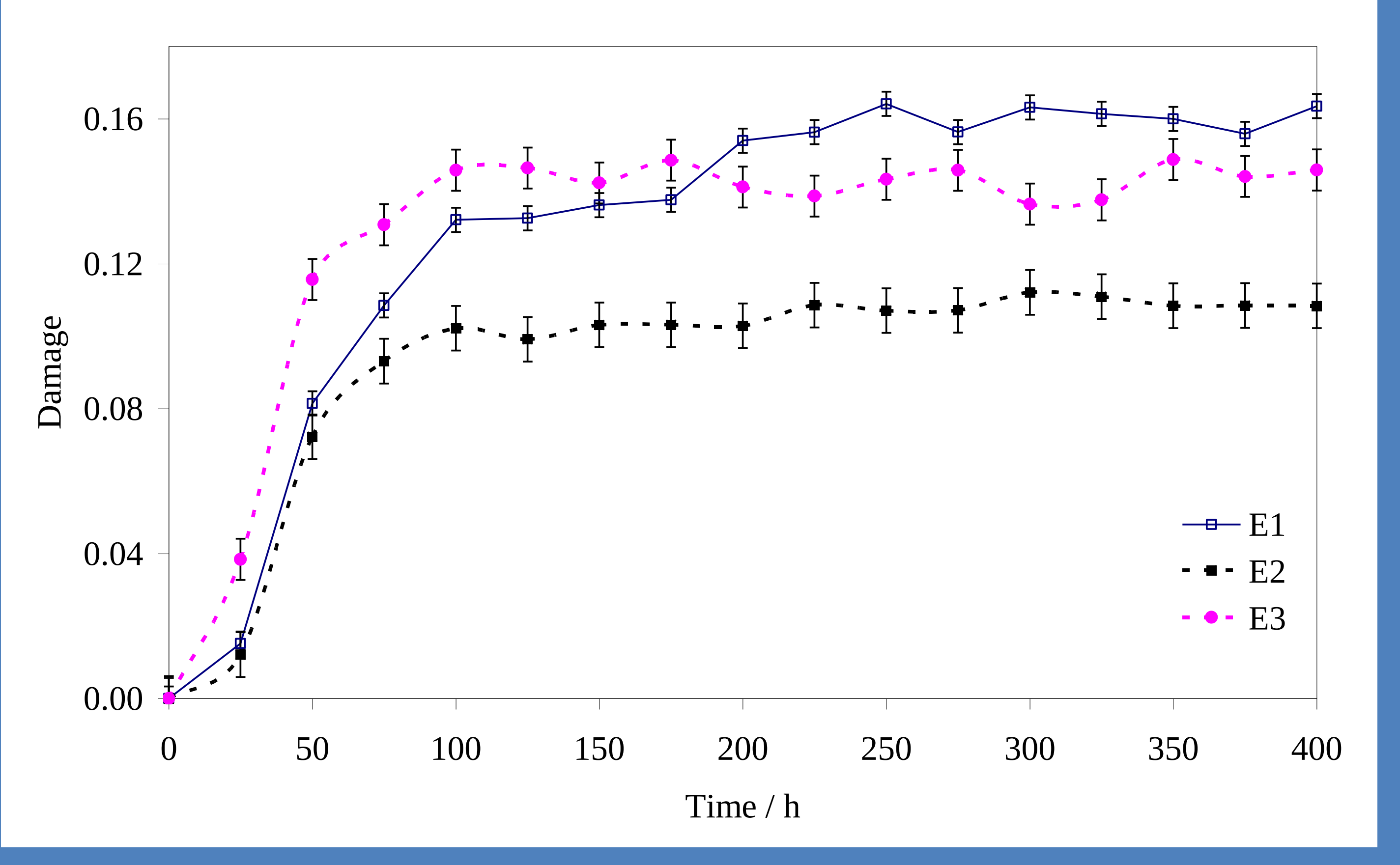
Material	Thickness (mm)	Density (kg/m^3)	Water Temperature (°C)	Used Frequency (MHz)	Coordinate System	$\theta_{critical}$ (deg)	$\theta_{critical}$ (deg)	$\theta_{critical}$ (deg)
Al_2O_3/Al_2O_3	2.56	2480	23	5	Off Axes	29.3	20.2	25.9
						X_1-X_2	X_1-X_3	X_1-X_{45}

Stiffness (GPa)		Matrix	Confidence Intervals (90%)	Error (%)	Measurement Quality	Engineering		Constants	Units
C_{11}	=	33.28	±0.13		High	E_1	=	30.36	GPa
C_{22}	=	85.88	±1.15	0.33	High	E_2	=	62.42	GPa
C_{12}	=	12.14	±0.19		High	E_3	=	71.23	GPa
C_{66}	=	13.95	±0.11		High	G_{12}	=	14.93	GPa
C_{33}	=	100.83	±3.85		Low	G_{13}	=	9.97	GPa
C_{13}	=	16.21	±0.64	0.29	High	G_{23}	=	13.95	GPa
C_{55}	=	9.97	±0.47		Low	ν_{12}	=	0.07	-
C_{23}	=	47.95	±2.25		Low	ν_{13}	=	0.127	-
C_{44}	=	**14.93**	±3.58	0.46	Low	ν_{23}	=	0.452	-

Experimental Results



Damage evolution for E1, E2 and E3: Master curves and standard deviation



Damage Evolution Modelling

For the deterioration of E_1, E_2, E_3 it can be assumed that there is no coupling, therefore a scalar damage function can be independendly defined for each modulus:

$$\frac{d\omega}{dt} = A \cdot f^{m}(\sigma, \varepsilon) \cdot (1 - \omega)^{-n}$$

where

ω : the damage function

f(σ,ε) : the stress strain state function of the material

A,m,n : material constants

Rate of damage proportional to damage

$$\frac{d\omega}{dt} = A \cdot (1 - \omega)$$

Deterioration is asymptotically approaching a value S^{∞}_{ij} A deterioration function can be defined as:

$$R(t) = \frac{E(t) - E_{\infty}}{E_0 - E_{\infty}}$$

At any given time t: 1<R(t)<0

and by definition R(t)=1-ω so eq.(1) becomes:

$$\frac{dR}{dt} = A \cdot R(t)$$

Integrating for boundary conditions ω=0 and t=0 we obtain for the reduction:

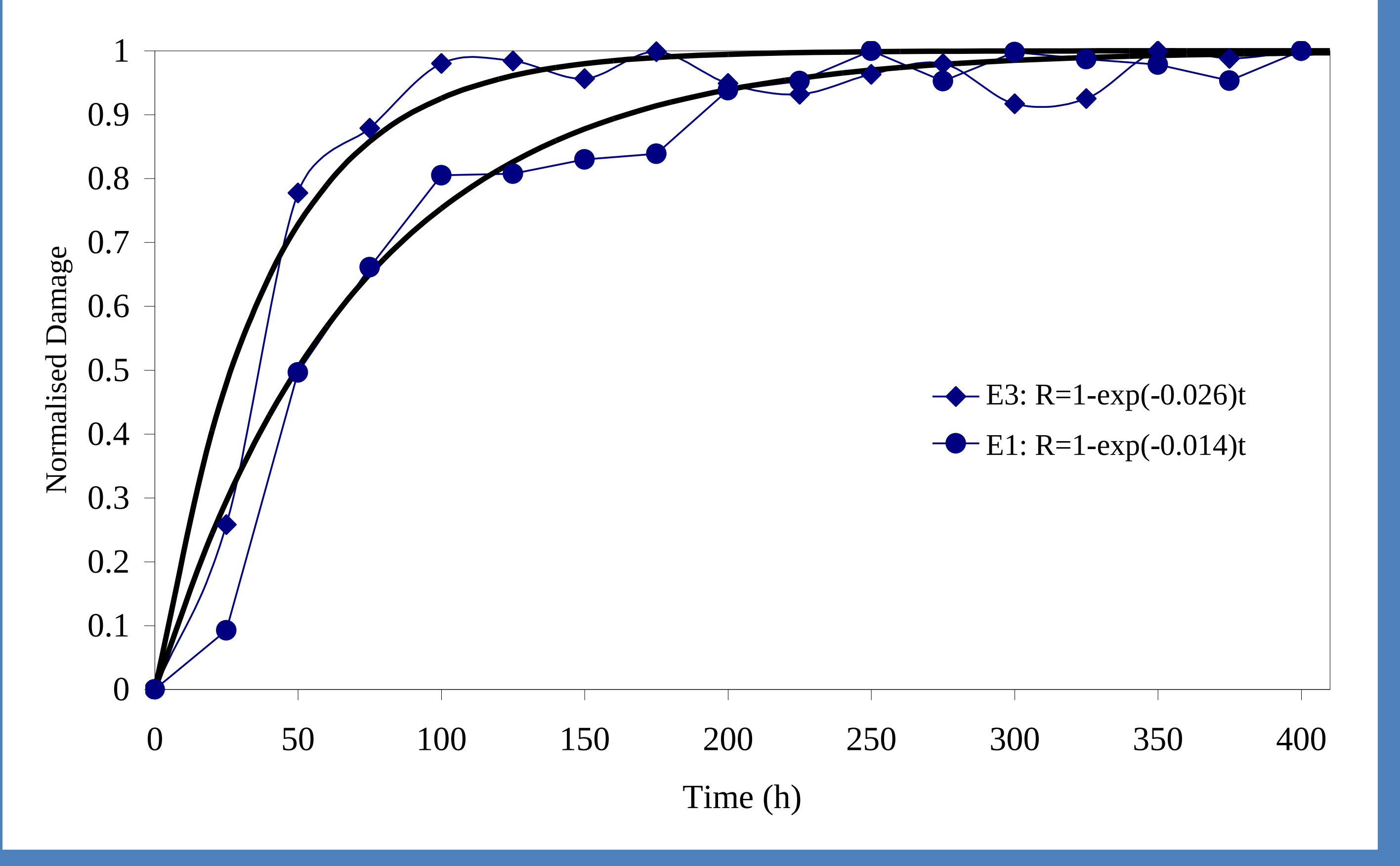
$$\boxed{R(t) = e^{-At}}$$

Or equivalently for the damage:

$$\boxed{D(t) = 1 - e^{-At}}$$

Damage Evolution Modelling

E_1 & E_3



Markov process simulation

- If we regard the damage accumulation observations as a Markov process, then the typical form of the process for a discrete function is:

$$x_{v+1} = x_v + \kappa\,(\sigma - x_v)$$

v the states of the process as a function of the quantity in interest (exposure time)
κ the damage increase rate
σ the standard deviation of the measurable quantity x (an elastic constant)

Markov process simulation

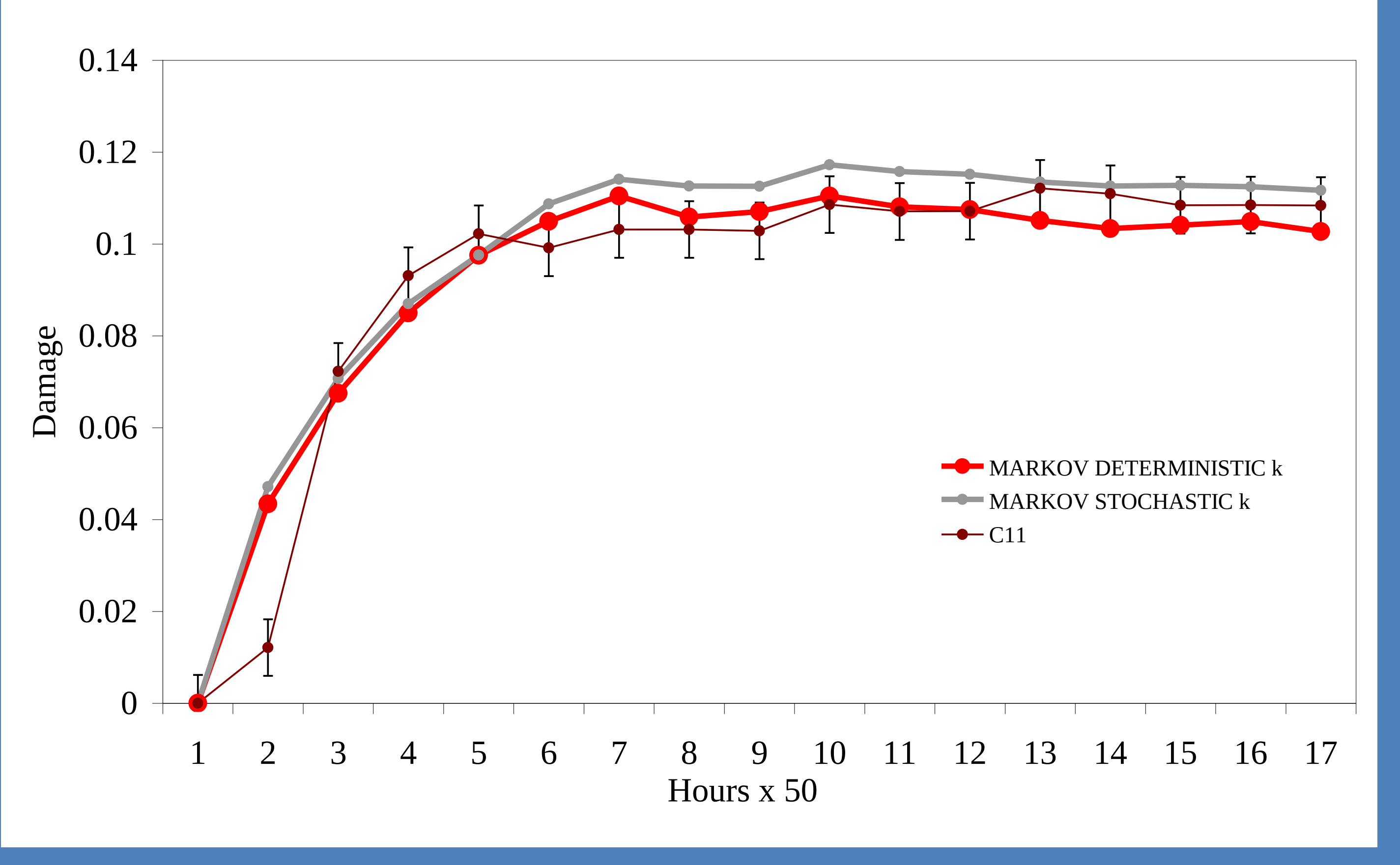
- The stochasticity of the system is introduced by an error function with a mean value of *U*:

$$x_{v+1} = x_v + \kappa (\sigma - \chi_v) + U e_{v+1}$$

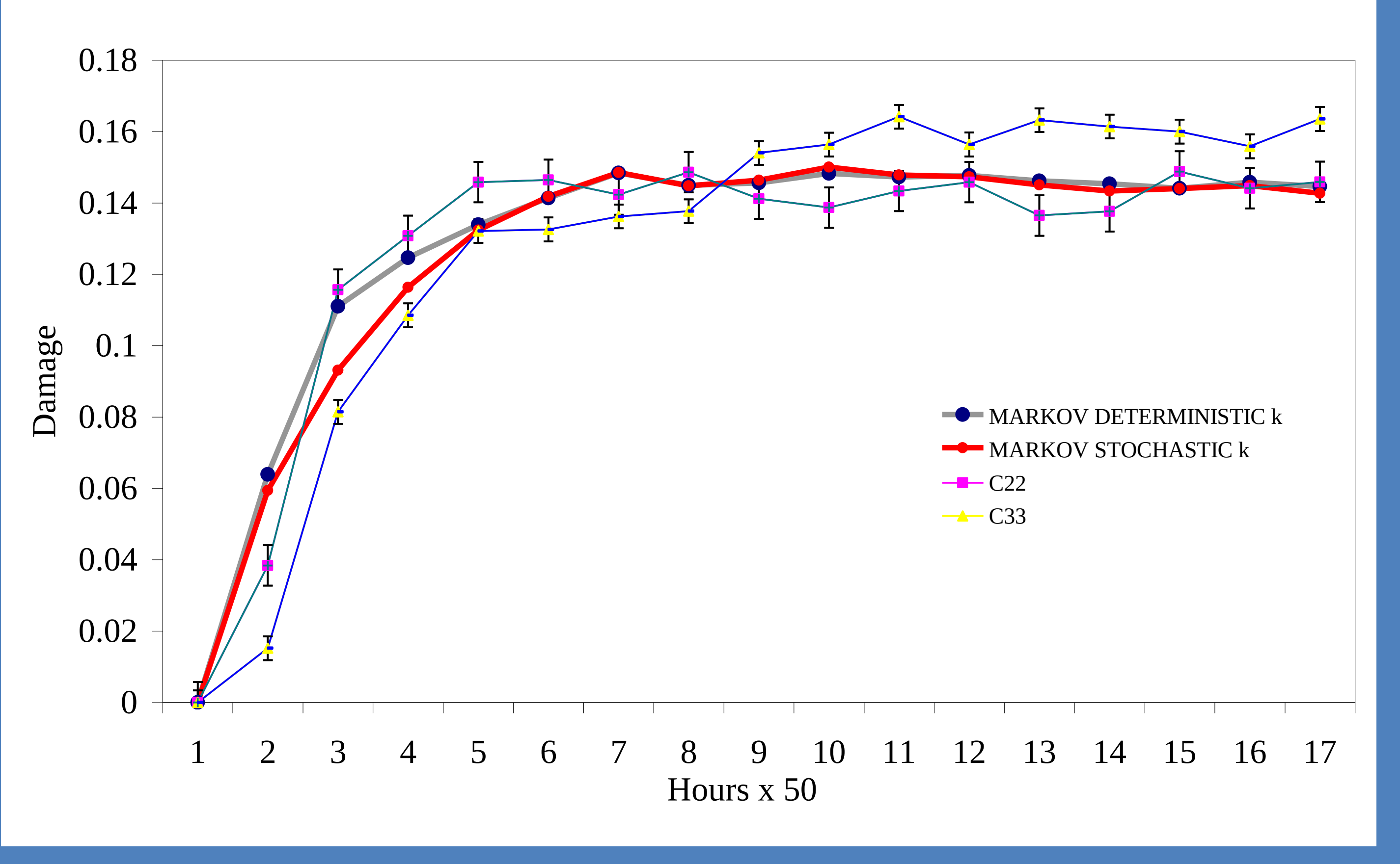
e_{v+1} is a random variable following a normal distribution (0,1).
κ may also be stochastic with an added error function ie. the damage development is of a stochastic nature regarding the time evolution of the elastic constants of the material.

- For κ known time function,the system becomes non stationary.

Markov process simulation for C_{11}



Markov process simulation for C_{22} & C_{33}



Conclusions

- The degradation of the mechanical properties of a novel Al_2O_3/Al_2O_3 composite under thermal exposure was identified by means of ultrasonic measurements
- Experimental results were validated by comparison with conventional tensile tests
- A damage evolution modelling scheme was applied and exponential decay functions that accurately describe the variation of the moduli of elasticity were determined.
- A stochastic damage accumulation model was employed using Weibull distributions and discrete time Markov chain models to yield modulus probability distributions
- Finally, a simulation of the stiffness degradation process is presented.

Case Study II: Monitoring of resin curing and hardening by ultrasound.

Aggelis DG, Paipetis AS. Construction and Building Materials. 2012;26(1):755-60

The goal…

A **problem** in the manufacturing of composite materials is the **monitoring** of the **curing process**

Distinguish **different stages** of the **structural formation**

Provide **adequate** conditions for proper **epoxy impregnation**

...the goal

- Curing monitoring efficiency in epoxy systems provides a **measurement** of the **structural state** of the **epoxy/composite system**

✓Subjected : load bearing conditions

aggressive environments

- Lots of methods allow for the **off-line estimation (e.g. Differential scanning calorimetry)** of the **curing degree** and few for the **on-line monitoring (e.g. dielectric spectroscopy)** of the **curing process**

The principle…
Setting and hardening monitoring system

✓Epoxies viscous liquids in room temperature conditions
✓Slight change in viscosity after hardener addition

➡ Epoxy viscosity depends on:

- temperature
- time

➢**Viscosity decreases** with temperature until macromolecules start to form
➢The polymerization leads to **a rapid increase in viscosity**
➢The **rate of chemical reaction** is **not linearly dependent** with **time** as polymerization reaches maximum or when the polymer freezes to a glassy state
➢**Post curing** leads to **increased cross linking** and **enhanced stiffness** of the epoxy system

… the principle
Setting and hardening monitoring abilities

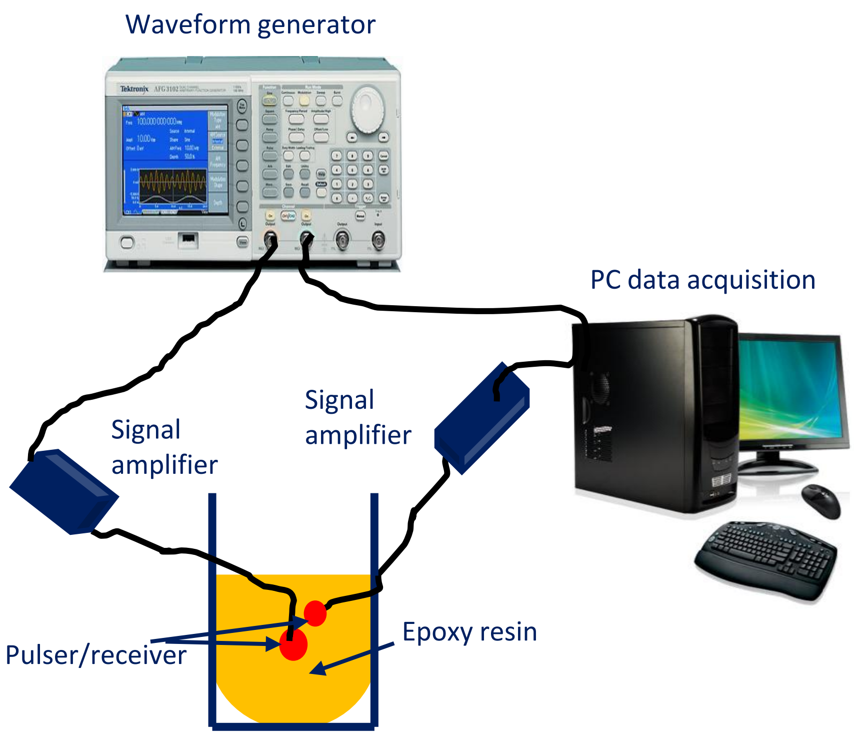
✓Proposed **setting and hardening monitoring** system is based on:

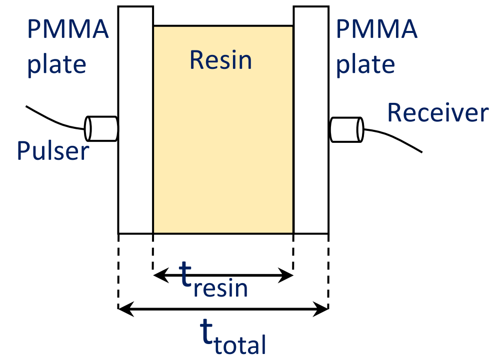
- the **wave propagation** properties (**viscosity** and **stiffness**) of the **time dependent** epoxy system

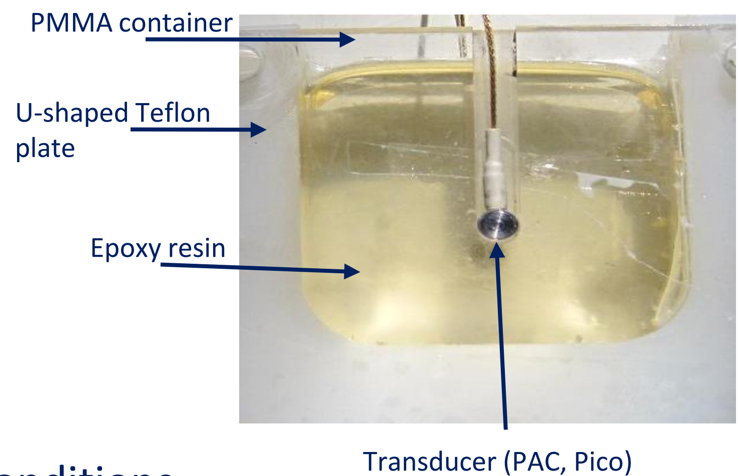
Purpose of this study:

“Contribution to the understanding of the wave propagation in epoxy during curing, with the aim to provide an ultrasound based curing monitoring system”

Experimental setup

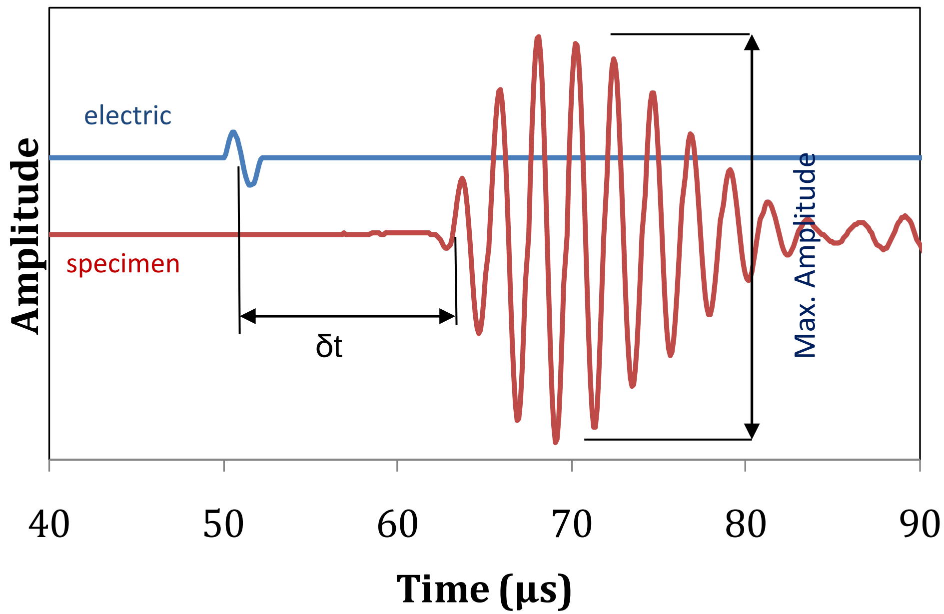


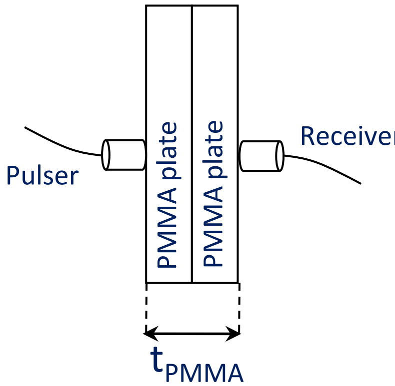




- ✓Distance between sensors : 20mm
- ✓Sampling rate 10MHz
- ✓Ultrasonic gel to enhance acoustic coupling conditions
- ✓Electric signal : 1 cycle of 500kHz
- ✓5 min interval for a 15hrs period of time

Experimental protocol



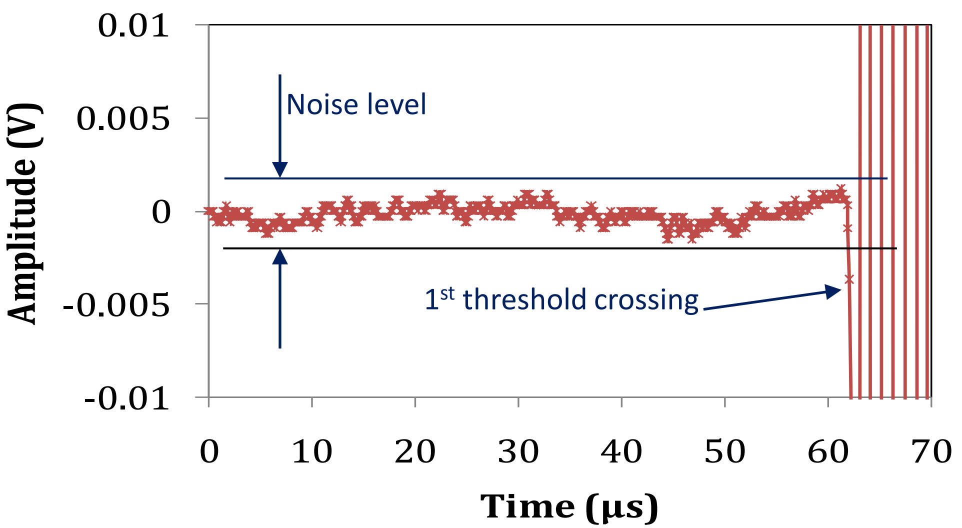


- Transit time (excluded) between PMMA plates : 5,2 µs

- Pulse velocity is measured by the time delay between the received signal through the sample and the electric pulse directly fed from the generator to the acquisition board
- Transmission is measured by the maximum voltage of the received waveform

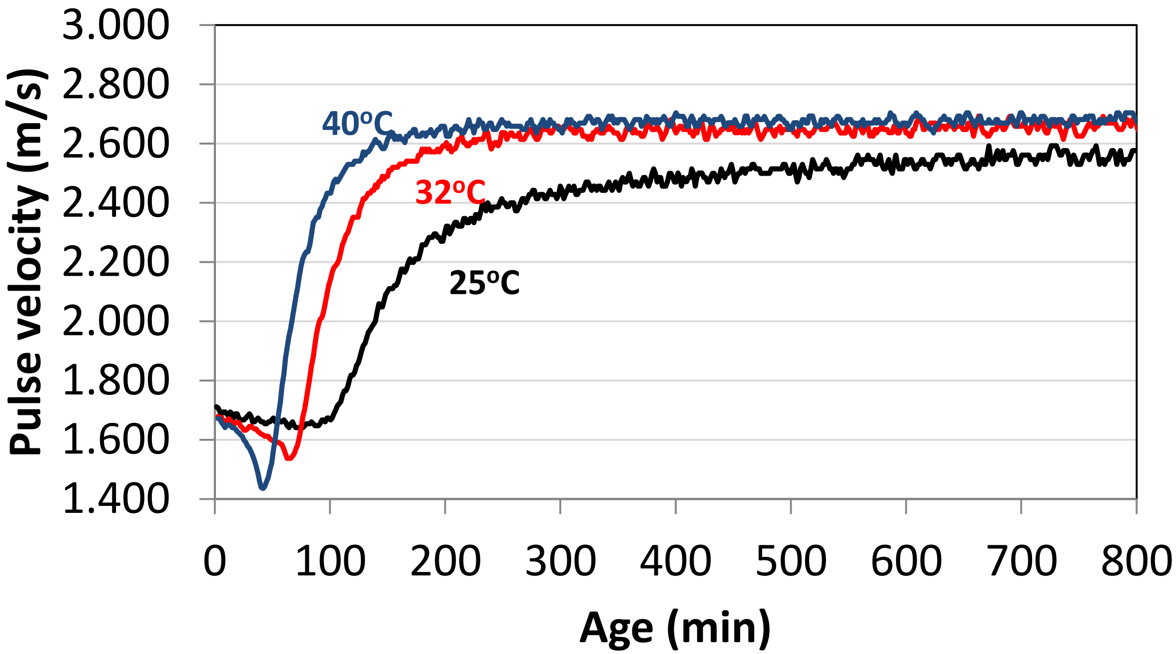
..experimental protocol

Threshold crossing algorithm (Matlab) processed the waveforms



- The onset is measured by a threshold crossing algorithm
- Threshold equal to 1,2 times the max amplitude recorded during the 50µs period of the pre-trigger
- No need to enhance signal to noise ratio
- Sampling rate of 0,1µs results in a **standard error 0,7 %**

Results



➢Velocity measured after a week in pulse-echo mode measured at 2730m/s due to completed polymerization

Sample at 25 °C

➢Initial pulse velocity 1700m/s > 1500m/s (water)

➢Sharp increase for the 1^{st} 70min

➢Steady increase until 180min

➢At 800min velocity increase converges to 2600m/s

➢55% increase of velocity vs. initial measurement

Sample at 40 °C

➢Shorter initial decrease

➢Small decrease

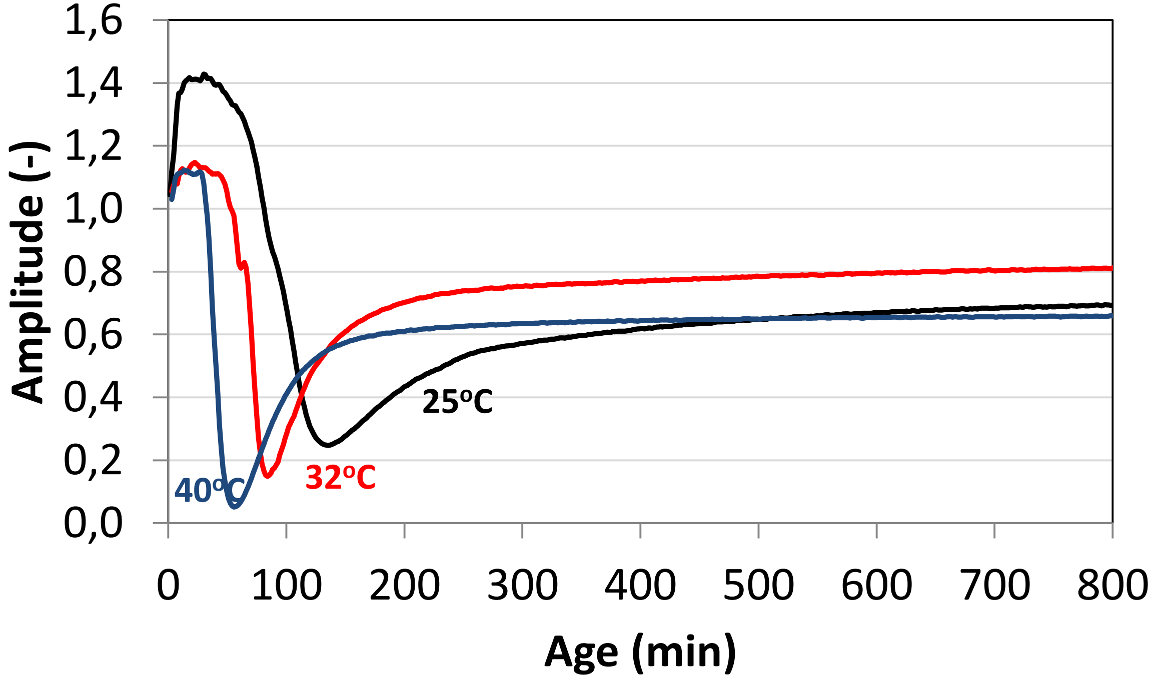
➢Steady increase until 90min

➢Final velocity reached much earlier

30 mm

Resin

..results

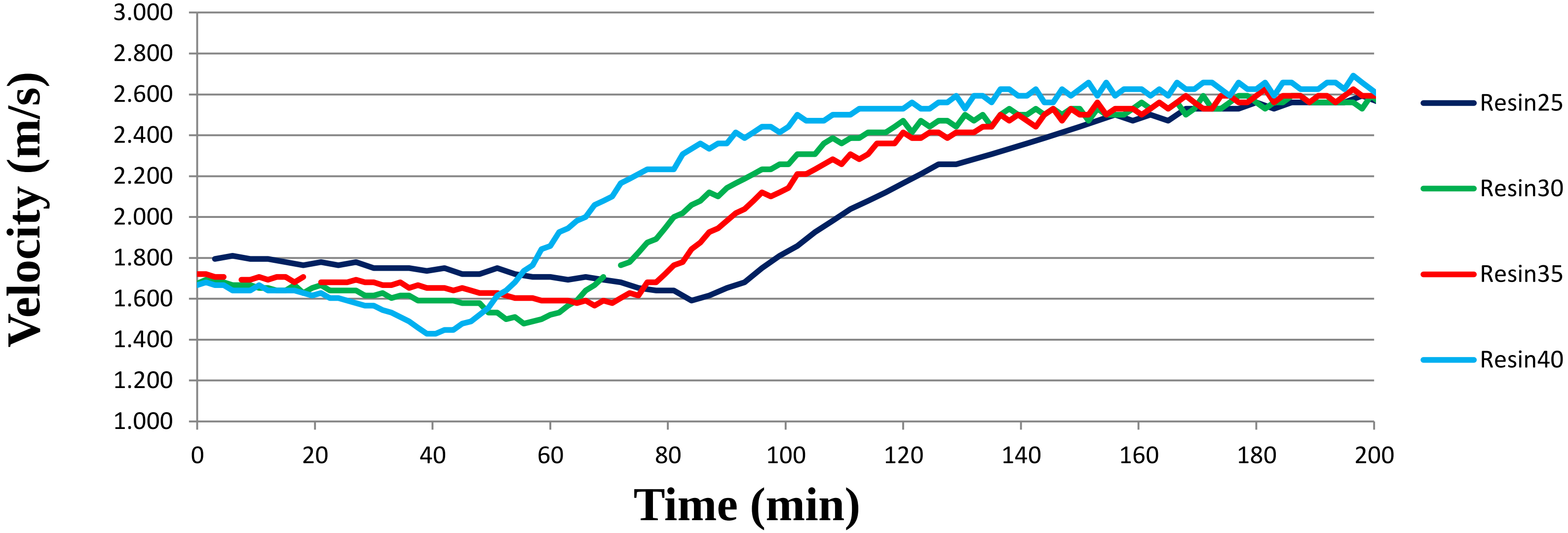


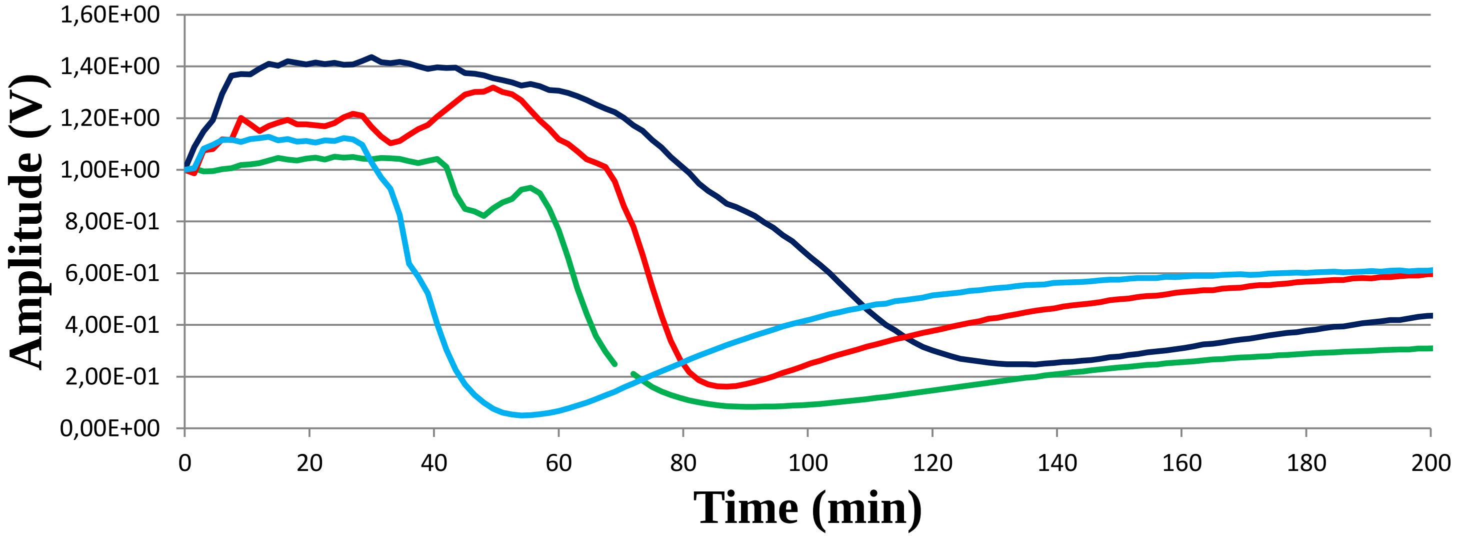
Sample at 32 ⁰C

Sample at 40 ⁰C

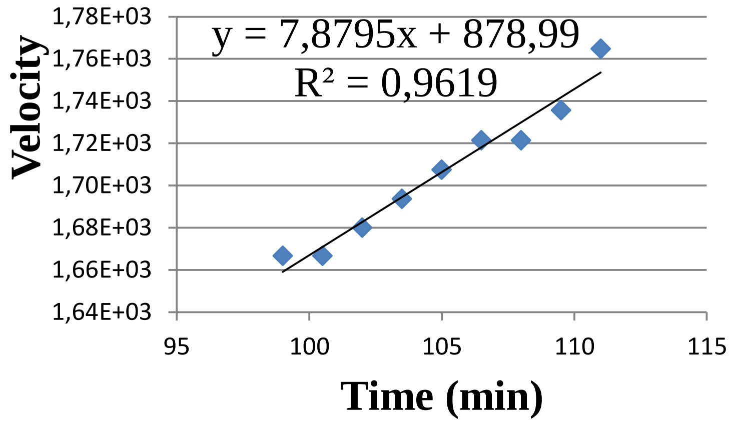
- Similar initial amplitude but smaller increase
- Amplitude plateau from 20 to 50min
- Rapid decrease in less than 80min
- Amplitude decreases rapidly until the 130min
- Then amplitude increase with decreasing rate
- Amplitude increase with decreasing rate

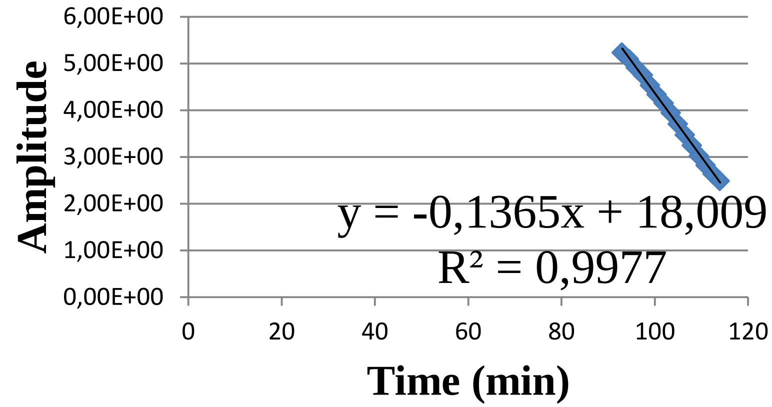
Temperature vs velocity and amplitude



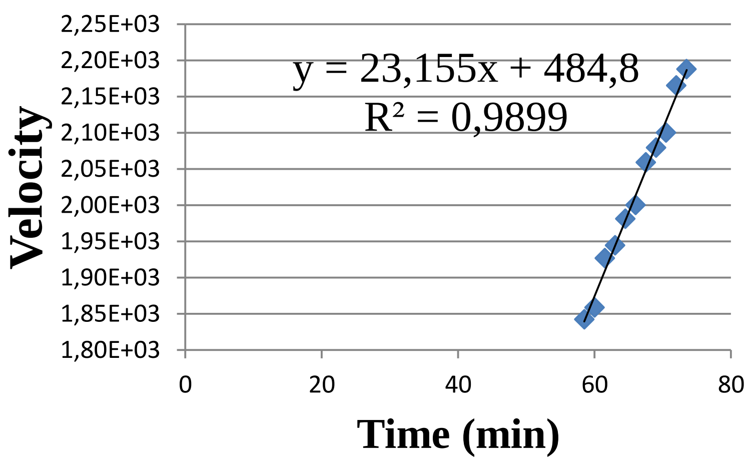


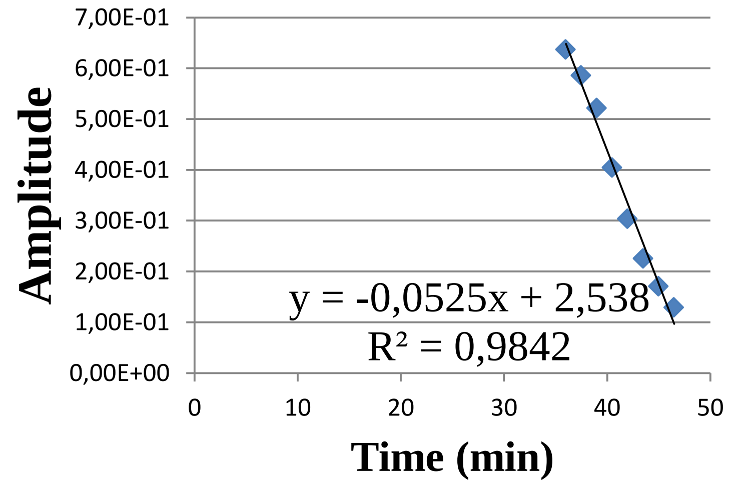
Rates of velocity and Amplitude change





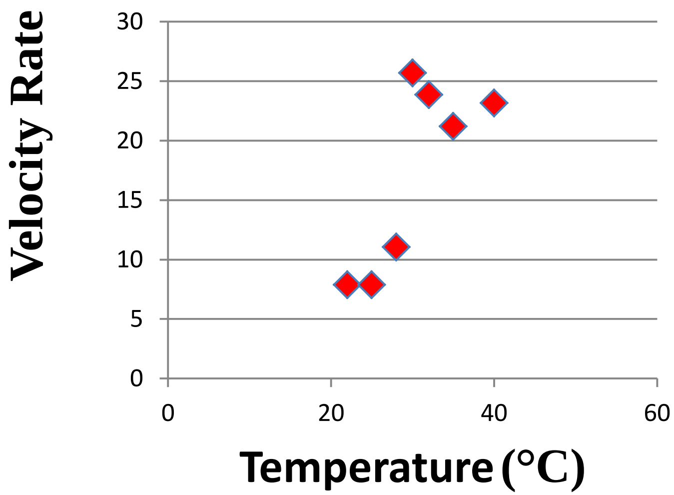
Στους 25°C

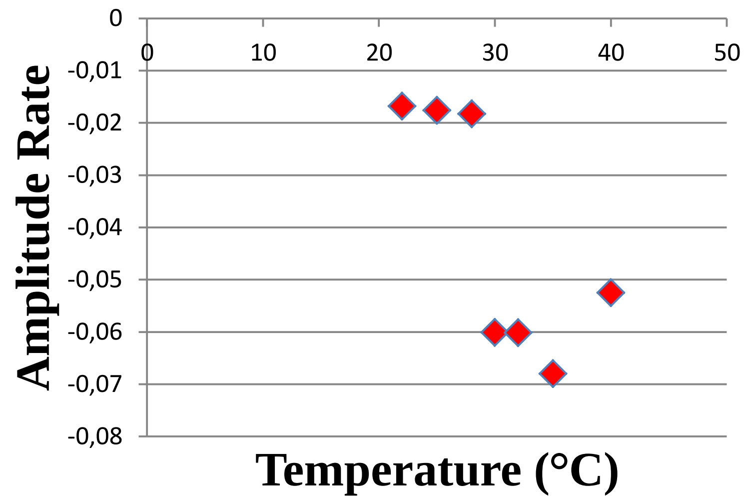




At 40°C

...Results...





α/α	**Velocity Rate**	Amplitude Rate
Specimen 1 (22 °C)	7,87	-0,016
Specimen 2 (25 °C)	7,88	-0,017
Specimen 3 (28 °C)	11,06	-0,0183
Specimen 4 (30 °C)	25,69	-0,0601
Specimen 5 (32 °C)	23,85	-0,0602
Specimen 6 (35 °C)	21,20	-0,068
Specimen 7 (40 °C)	23,15	-0,0525

Stiffness

For 3D propagation:

$$c = \sqrt{\frac{E\,(1-\nu)}{\rho\,(1+\nu)(1-2\nu)}}$$

α/α	E(GPa)
Specimen (22 °C)	5,49
Specimen (25 °C)	5.48
Specimen (28 °C)	5.44
Specimen (30 °C)	5.53
Specimen (32 °C)	5.82
Specimen (35 °C)	5.40
Specimen (40 °C)	5.40
Specimen (35 °C-100kHz)	5.91
Specimen (35 °C-1MHz)	5.40

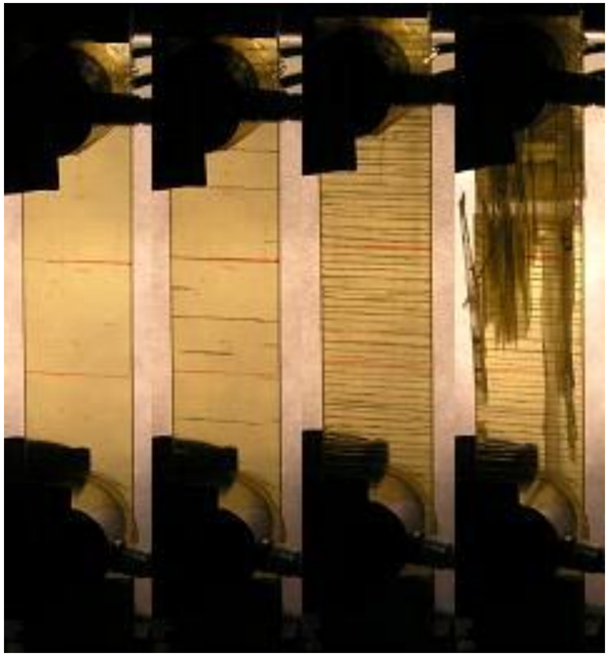
Conclusions

- Pulse velocity increase indicates an increase of the stiffness of the material.
- As polymerization proceeds, material becomes stiffer thus velocity increases tending asymptotically to a maximum.
- Exothermic reaction of the polymerization process leads to global increase in temperature as well as a decrease in viscosity. This is shown as an initial increase in amplitude and decrease in velocity.
- As macromolecular chains start to form, viscosity is increasing and amplitude starts to decrease. Afterwards the gradual stiffening of the material leads to amplitude increment similarly to velocity.
- The fluid nature of the material governs the measurements at early curing times and the stiff nature the completion of the curing process.
- Ultrasonic monitoring provides information on the rate of curing and completion of the reaction.
- Lastly, combined measurements of velocity and amplitude shed light in the transformation process of the epoxy allowing for the study of the individual mechanisms.

- CASE STUDY 3: Load induced degradation in cross ply laminates

Katerelos DTG, Paipetis A, Loutas T, Sotiriadis G, Kostopoulos V, Ogin SL. In situ damage monitoring of cross-ply laminates using acoustic emission. Plastics, Rubber and Composites. 2009;38(6):229-34
Aggelis DG, Barkoula NM, Matikas TE, Paipetis AS. Acoustic structural health monitoring of composite materials : Damage identification and evaluation in cross ply laminates using acoustic emission and ultrasonics. Composites Science and Technology. 2011.

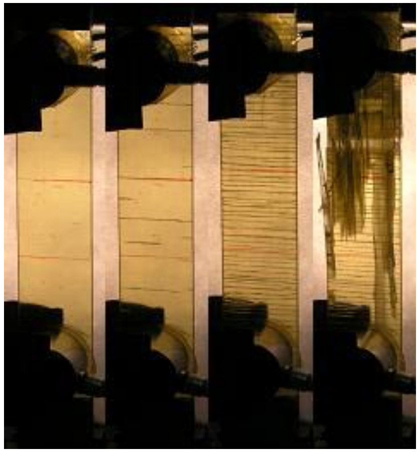
Motivation



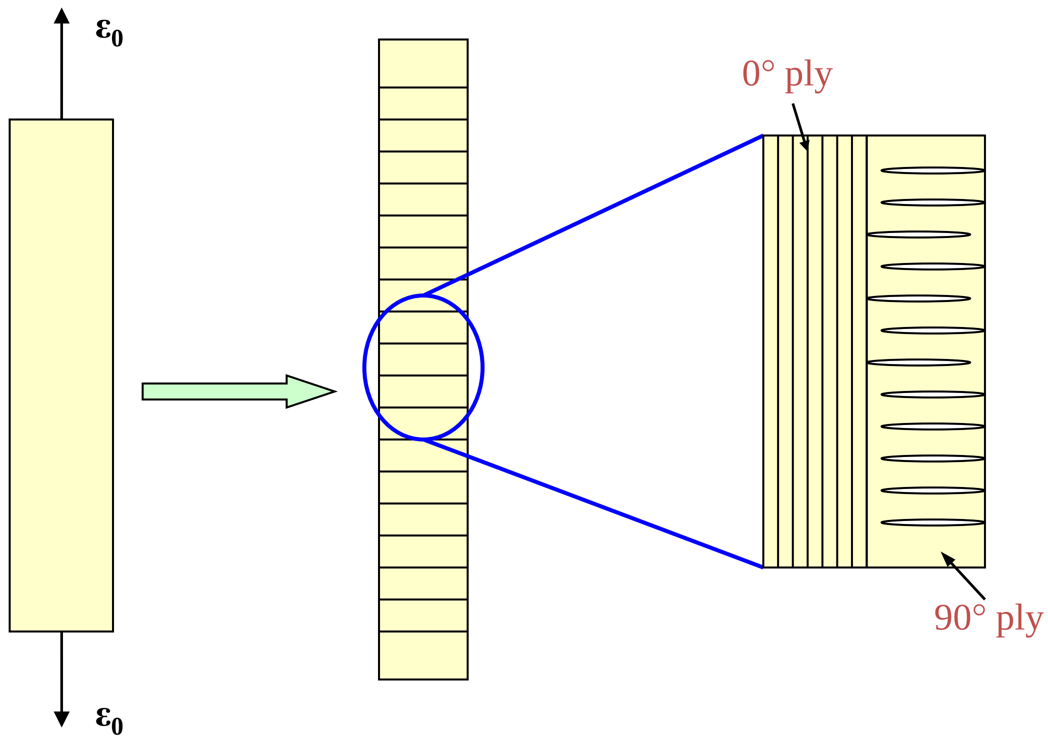
- Motivation: The identification and classification of the damage mechanisms in composite laminates using Acoustic Methods

Outline

- Three case studies: monotonic loading, step loading, fatigue loading
- Damage identification using unsupervised Data Clustering
- Detailed study of AE activity and correlation with macroscopic activity
- Wave propagation characteristics
  - Simulation **&** Experimental verification
- Conclusions



Failure of Cross Ply Laminates

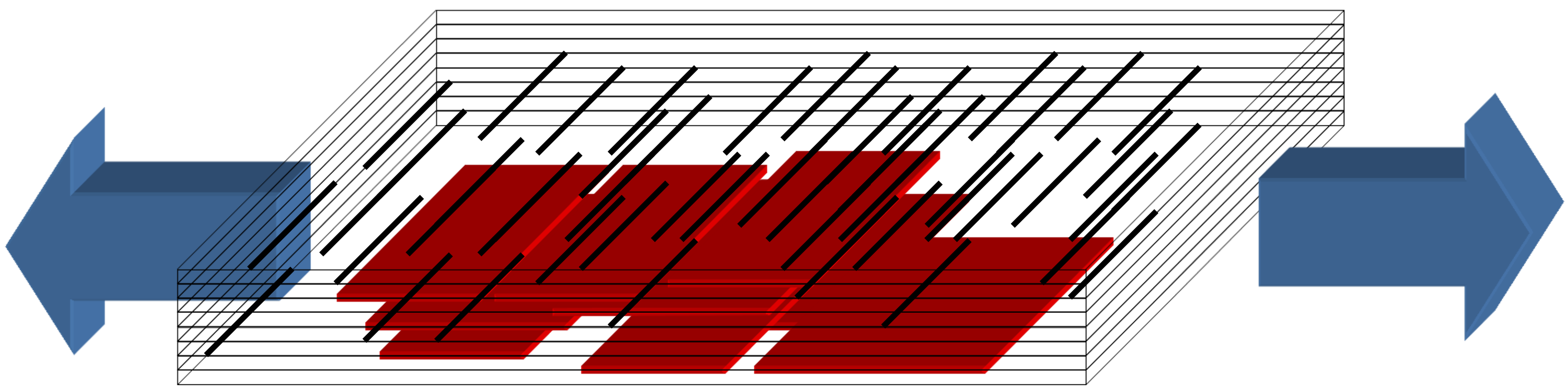


Failure process

(i) transverse cracking (mode I)

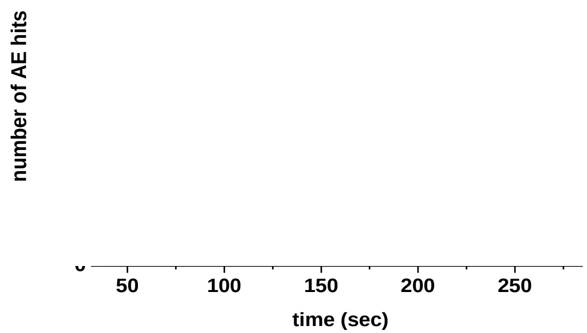
(iia) Delaminations (mode II) vs. (iib) fibre fracture (mode I)

Transverse matrix cracking, I

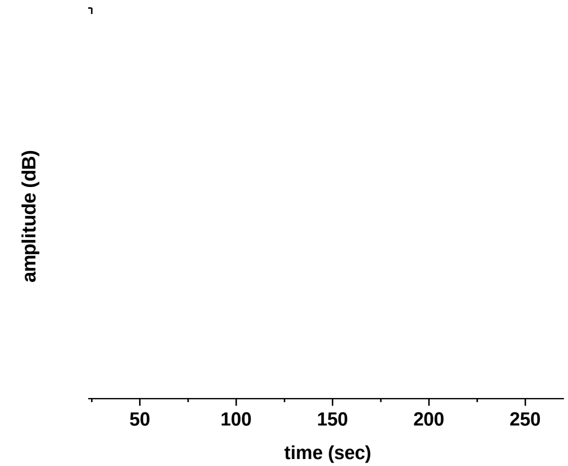


Delaminations due to elasticity mismatch
between the different layers, II

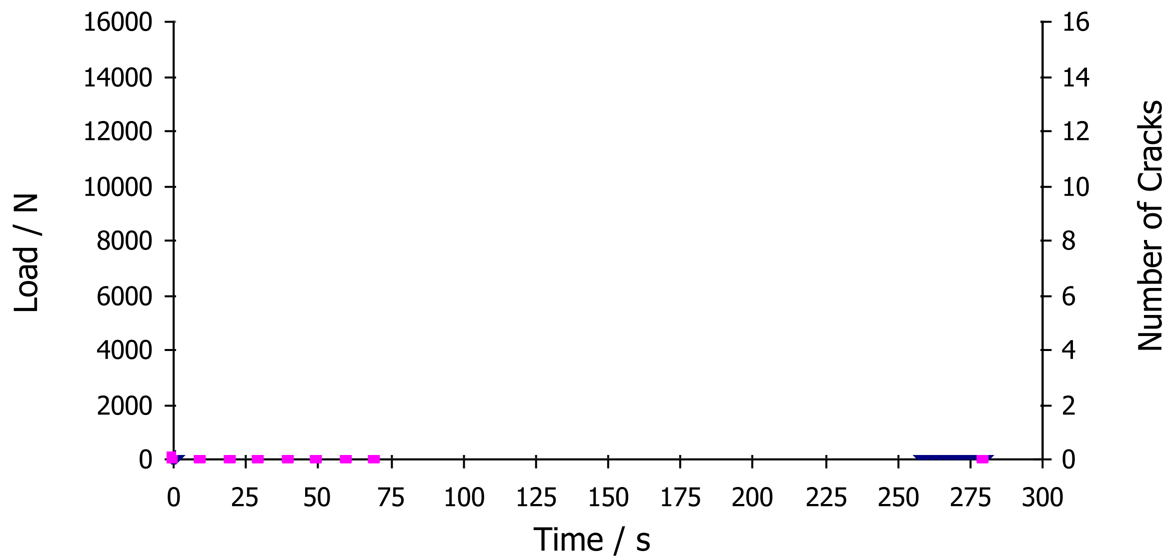
RESULTS



number of AE hits
50
100
150
200
250
time (sec)

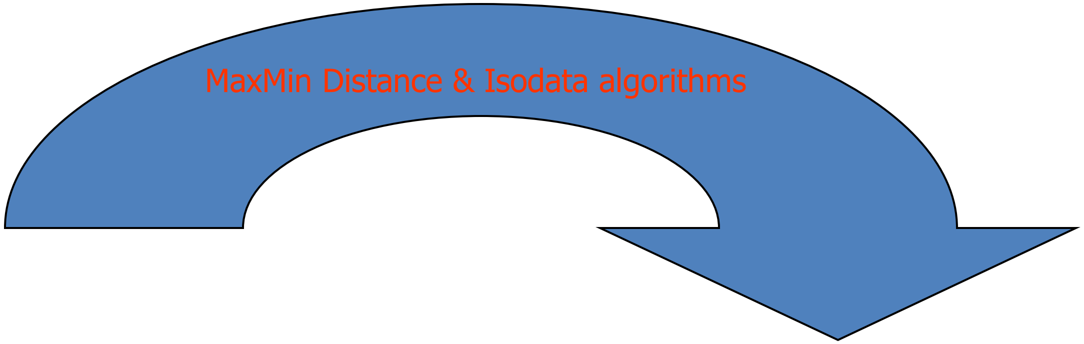


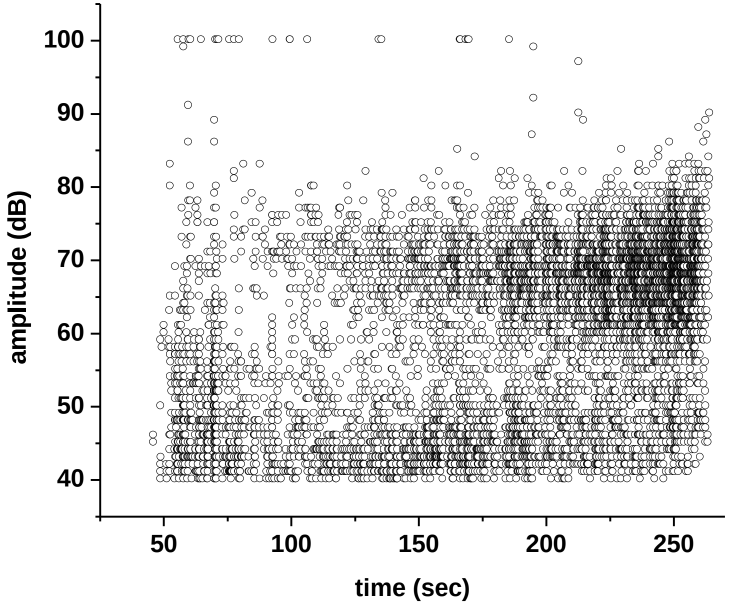
amplitude (dB)
50
100
150
200
250
time (sec)

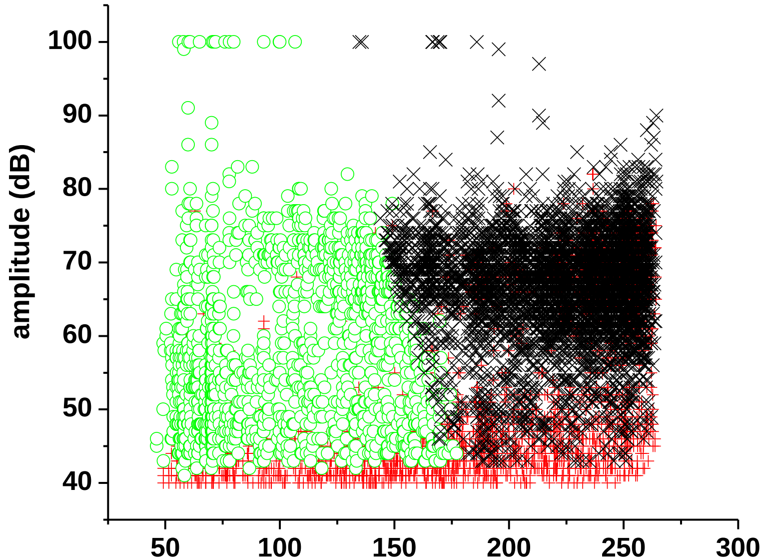


Load / N
16000
14000
12000
10000
8000
6000
4000
2000
0
Number of Cracks
16
14
12
10
8
6
4
2
0
0
25
50
75
100
125
150
175
200
225
250
275
300
Time / s

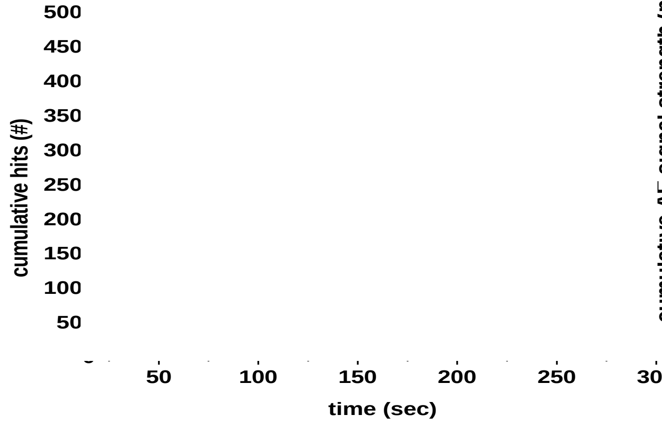
DATA CLUSTERING

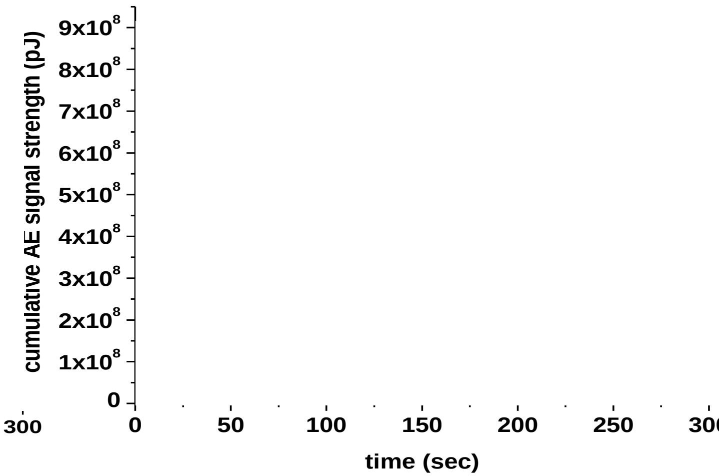


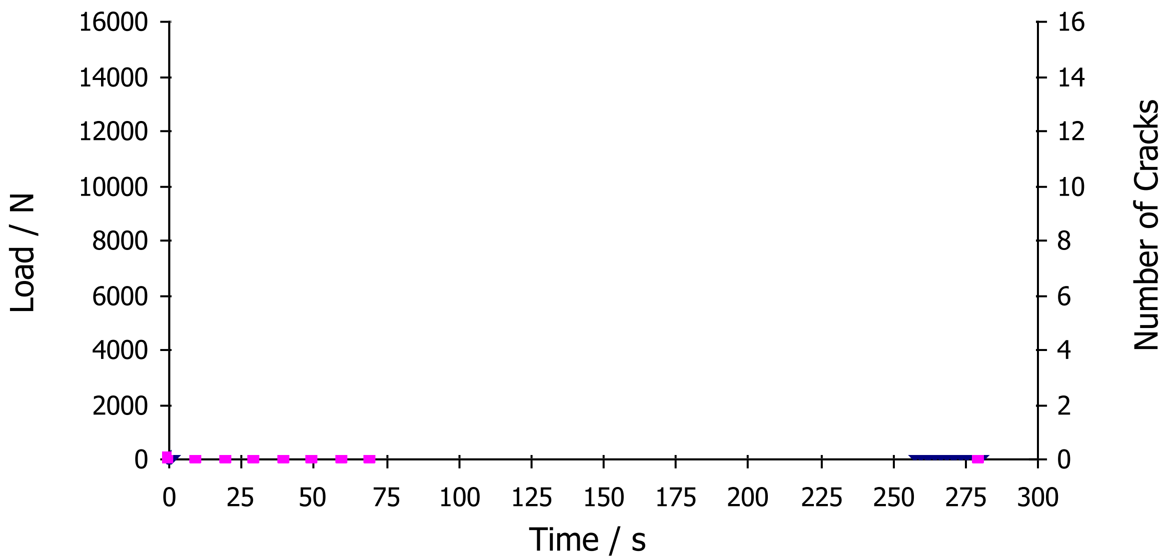




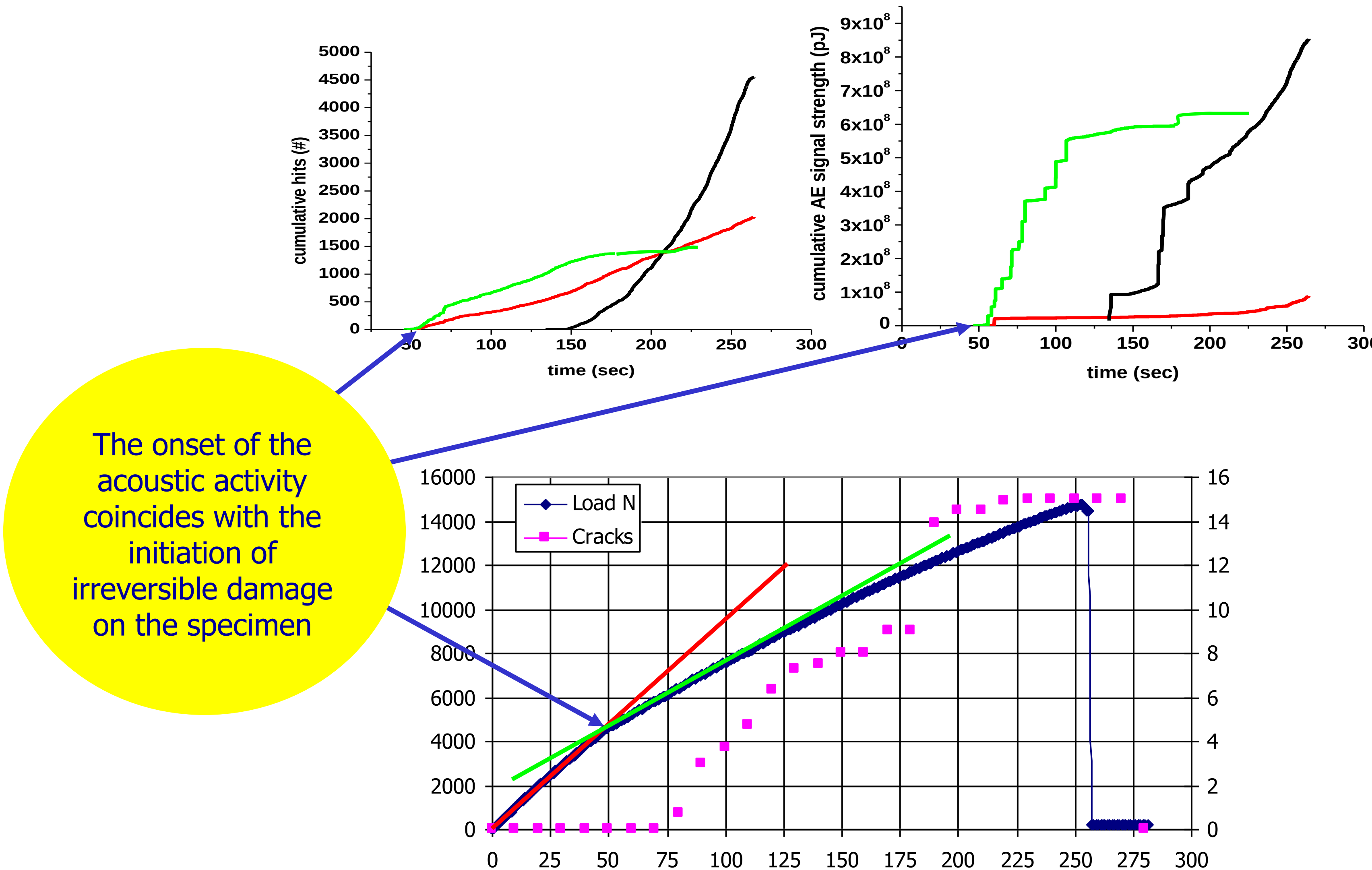
RESULTS


cumulative hits (#)
500
450
400
350
300
250
200
150
100
50
50
100
150
200
250
300
time (sec)


cumulative AE signal strength (pJ)
9x10^8
8x10^8
7x10^8
6x10^8
5x10^8
4x10^8
3x10^8
2x10^8
1x10^8
0
0
50
100
150
200
250
300
time (sec)

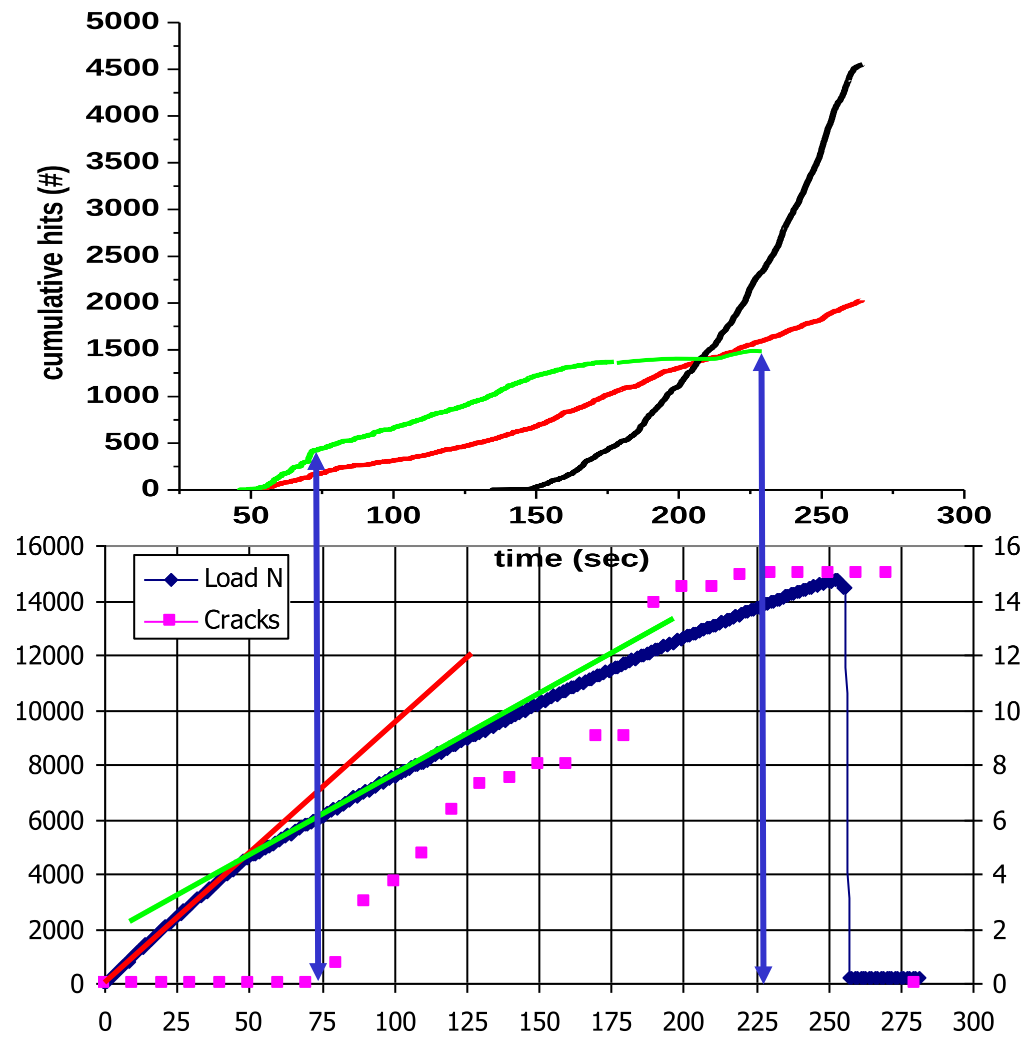

Load / N
16000
14000
12000
10000
8000
6000
4000
2000
0
Number of Cracks
16
14
12
10
8
6
4
2
0
0
25
50
75
100
125
150
175
200
225
250
275
300
Time / s

Acoustic Emission: Onset of acoustic activity



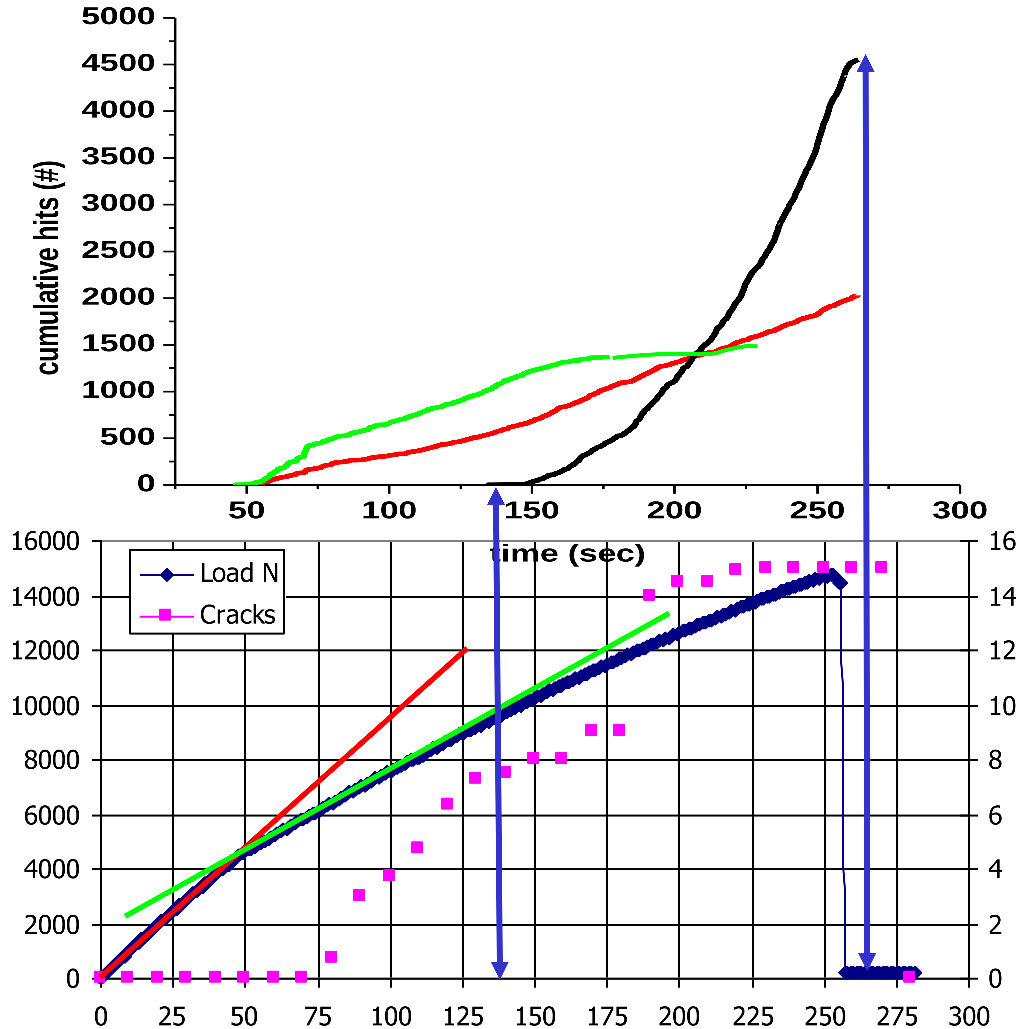
Acoustic Emission:
Onset of acoustic activity

The green mode follows the transverse cracking of the specimen



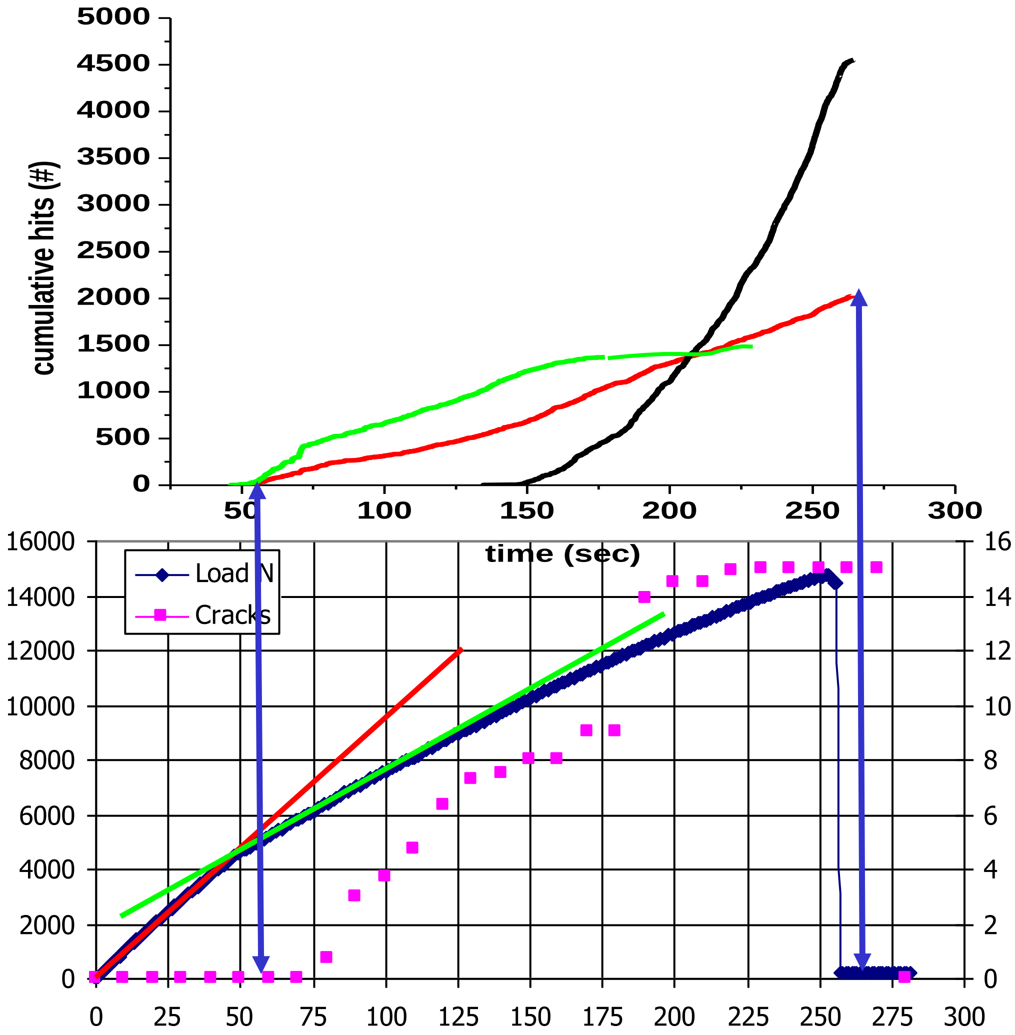
Acoustic Emission:
Onset of acoustic activity

The black mode starts prior to the end of transverse cracking and becomes dominant until failure



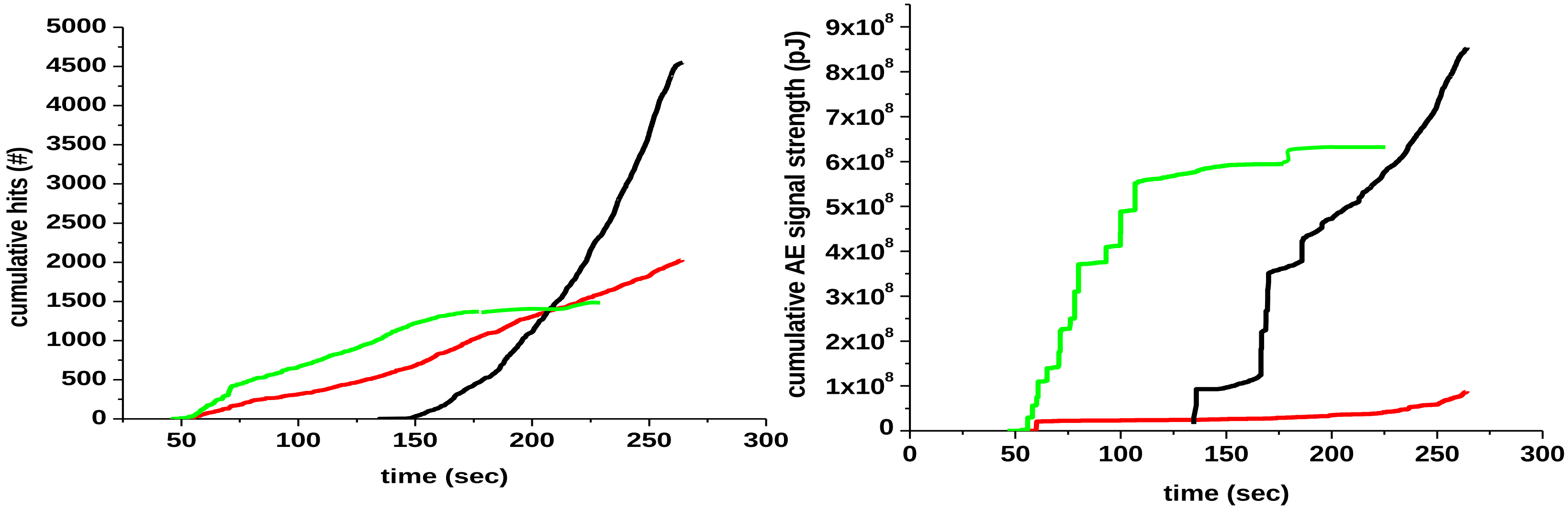
Acoustic Emission:
Onset of acoustic activity

The red mode is active throughout the damage development until failure

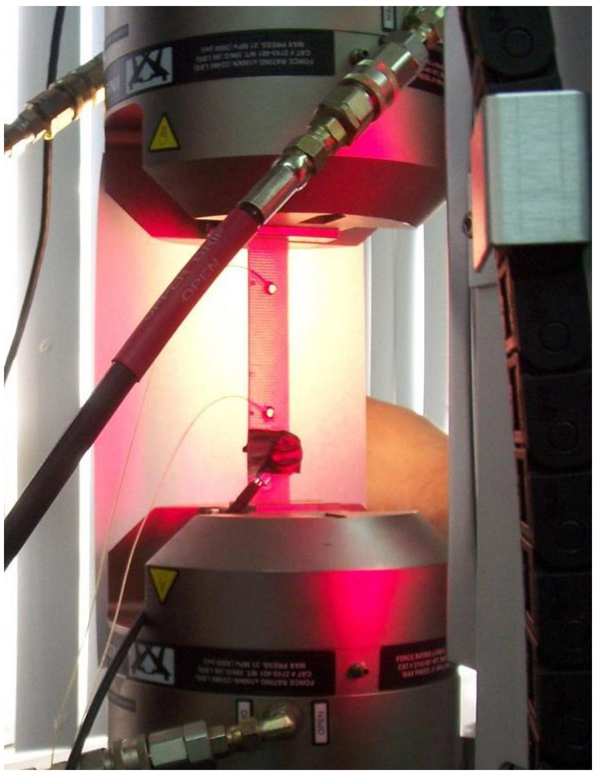


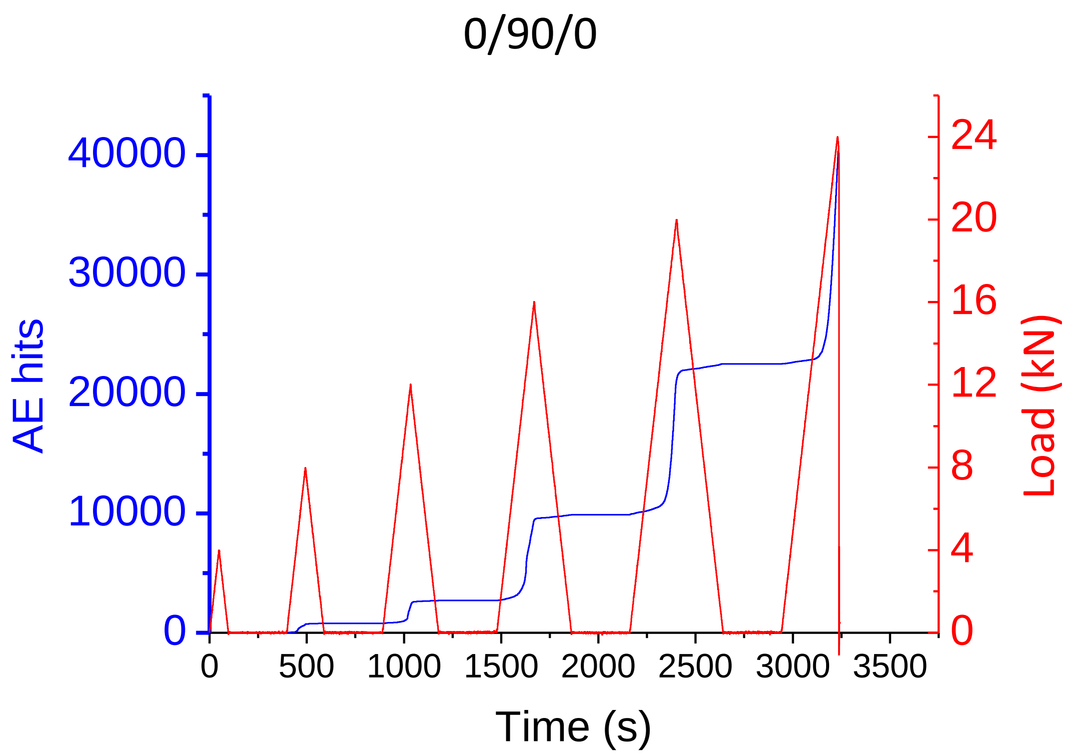
Acoustic Emission:
Damage Mode identification

Cluster 1: transverse cracking
Cluster 2: interfacial/ interlaminar failure
Cluster 3: longitudinal fibre failure

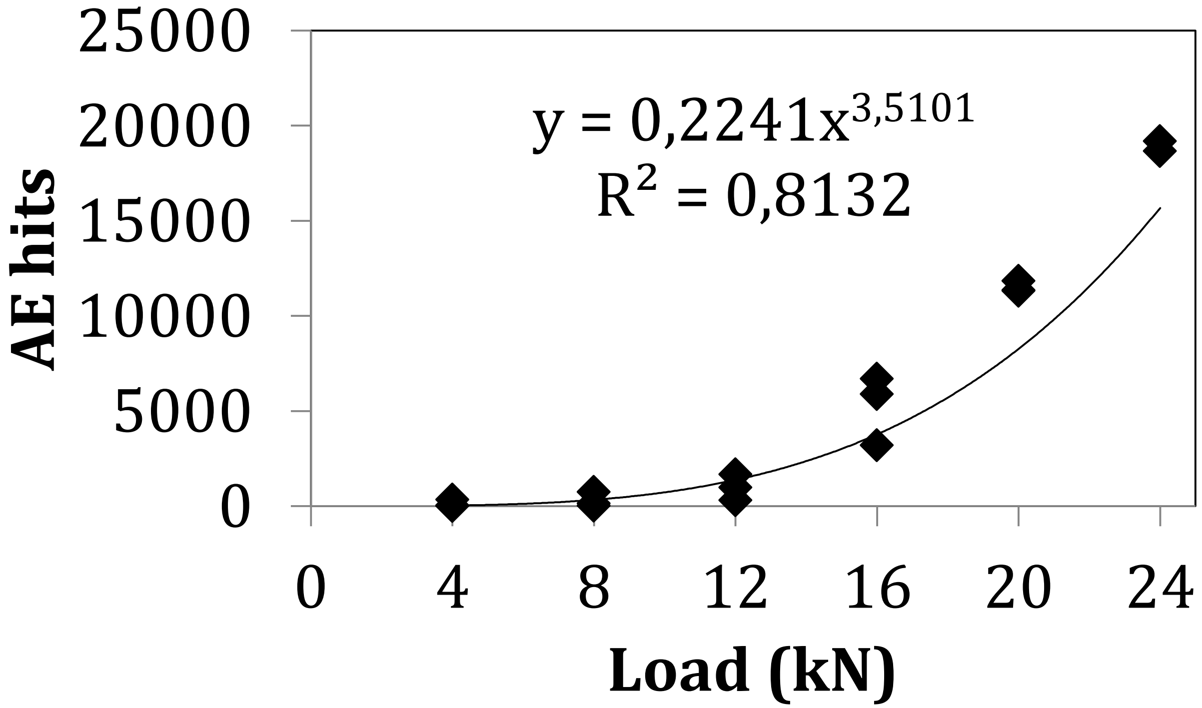


Application of AE indices in GFRPs under step loading



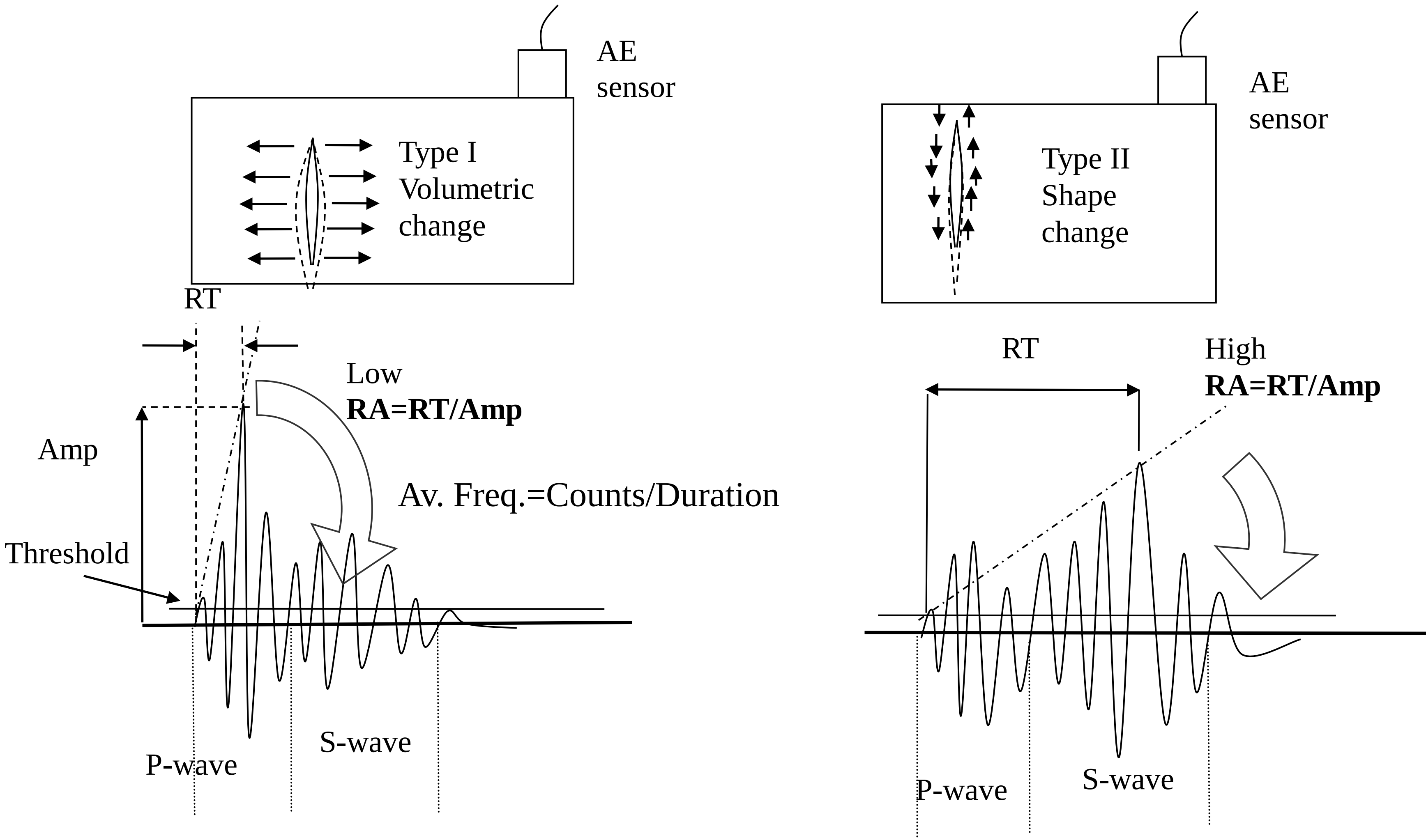


Total AE Activity vs load

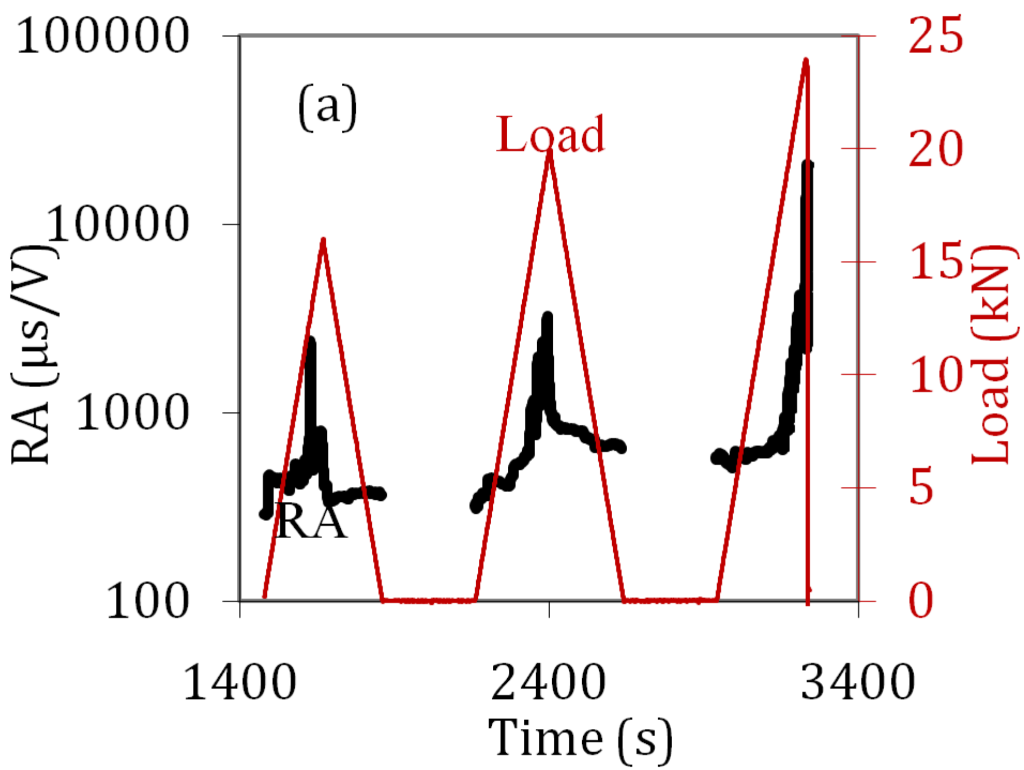


The number of the acquired AE signals correlates with the sustained load.

Tensile vs. shear cracks

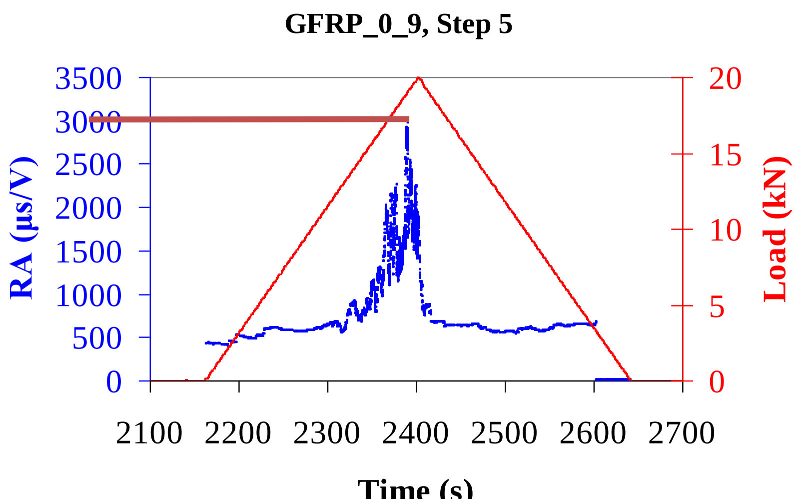


Damage mode conversion vs. loading history

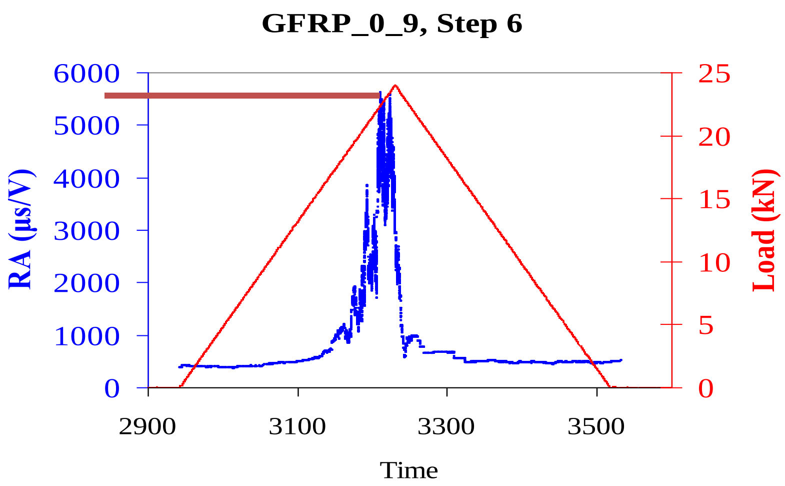


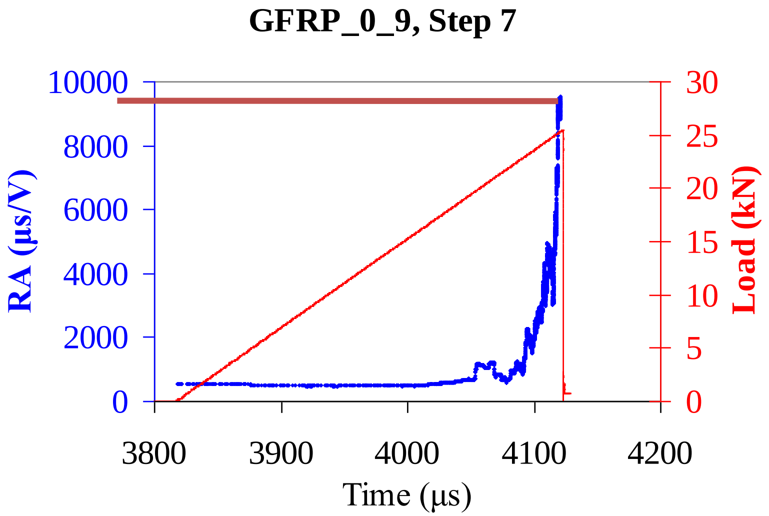
RA value as a transient feature increases with load increase. It also increases for successive load steps. It indicates the higher amount of delaminations over matrix cracking.

Detailed Analysis of AE signals
RA value

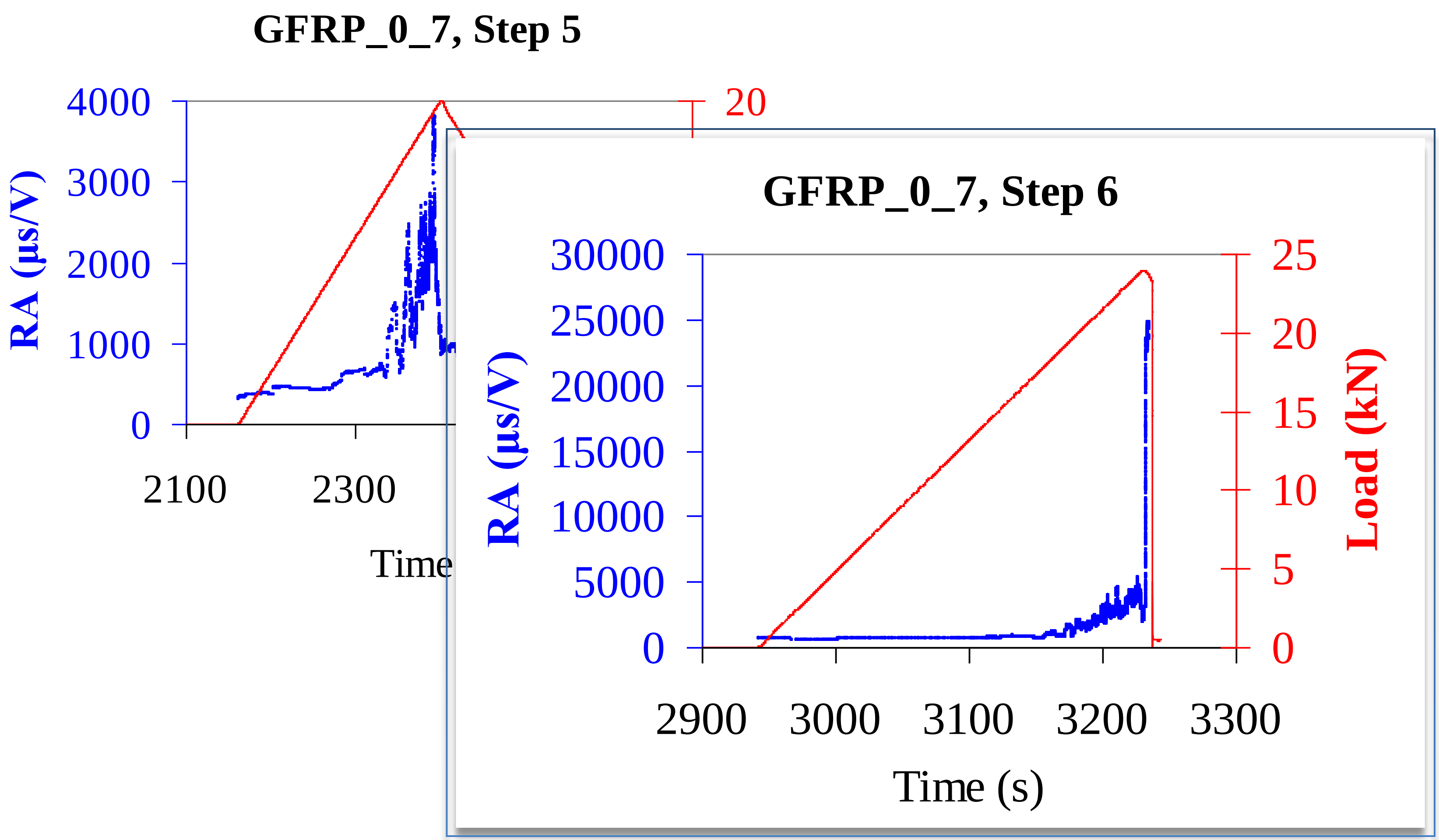


- As the load increases, the RA value increases (moving average of 500 hits)
- During unloading it drops to approx. 500 and stays constant
- For the successive steps, the maximum RA increases

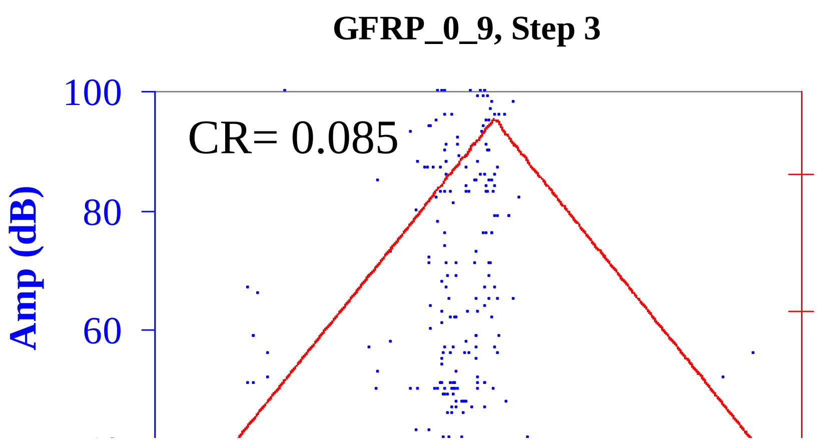


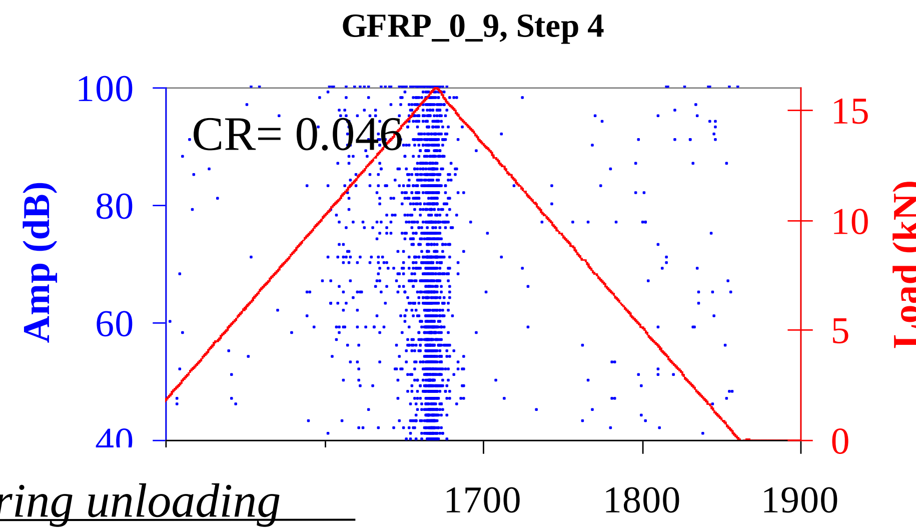


Detailed Analysis of AE signals

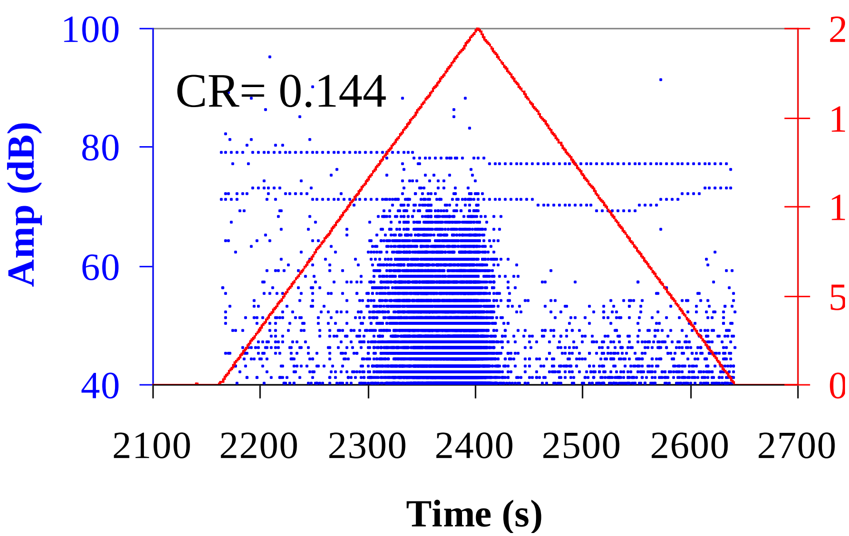


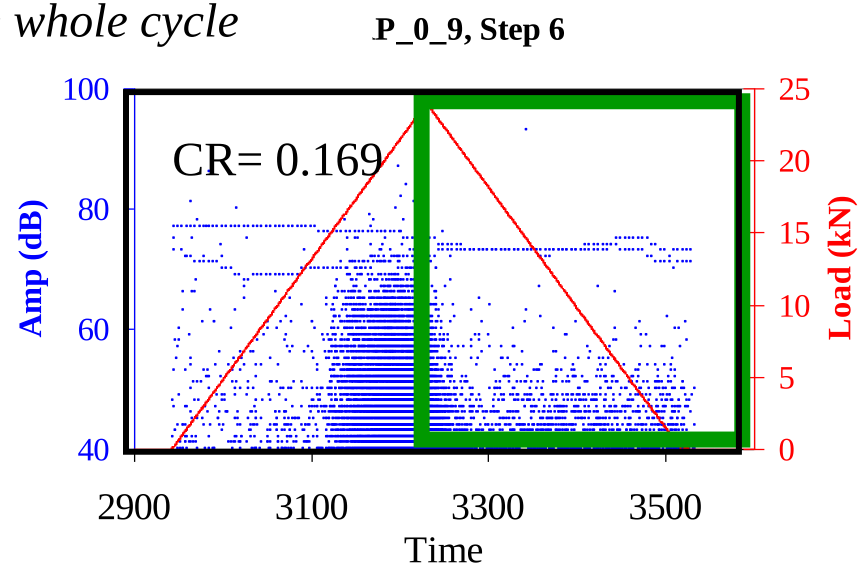
AE during loading and unloading





$$\textit{Calm Ratio} = \frac{\textit{Number of AE hits during unloading}}{\textit{Number of AE hits the whole cycle}}$$





AE during loading and unloading

In AE literature the value of 0.05 is a rule of thumb to distinguish between intermediate and heavy damage

For all 3 specimens the Calm ratio obtained its maximum value at the step before failure

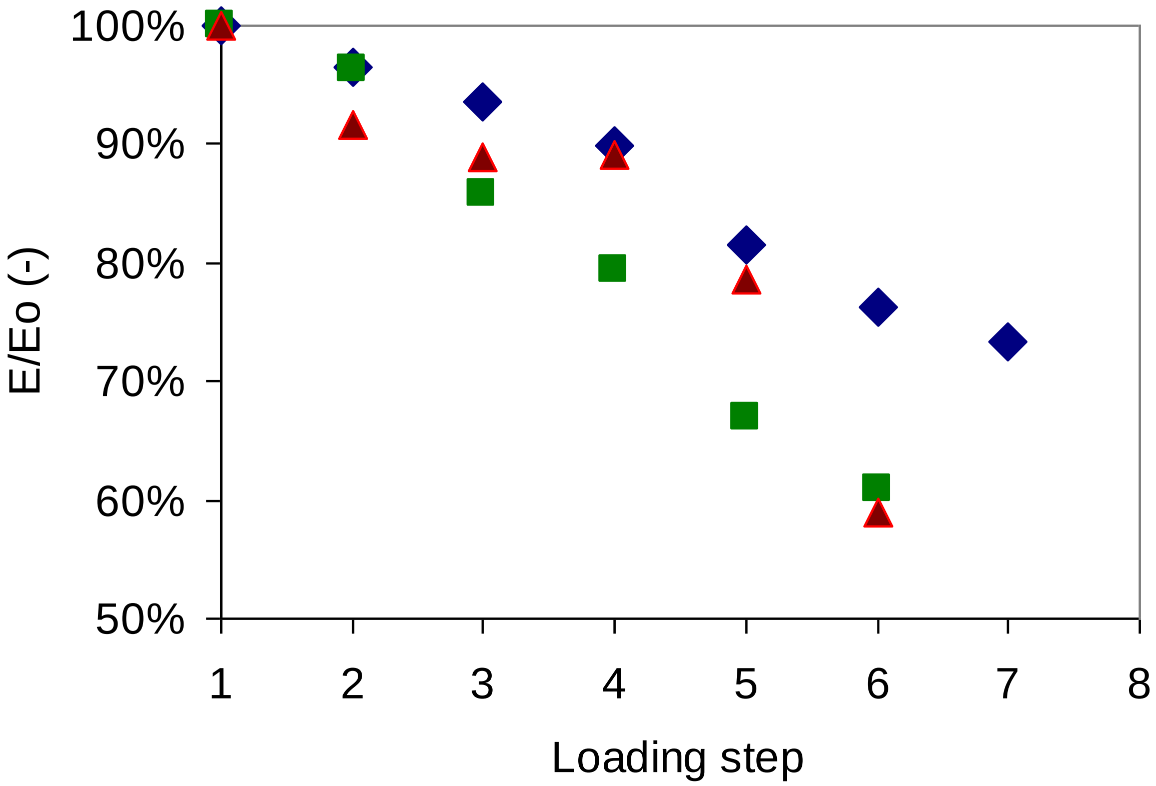
0.169

0.157

0.115

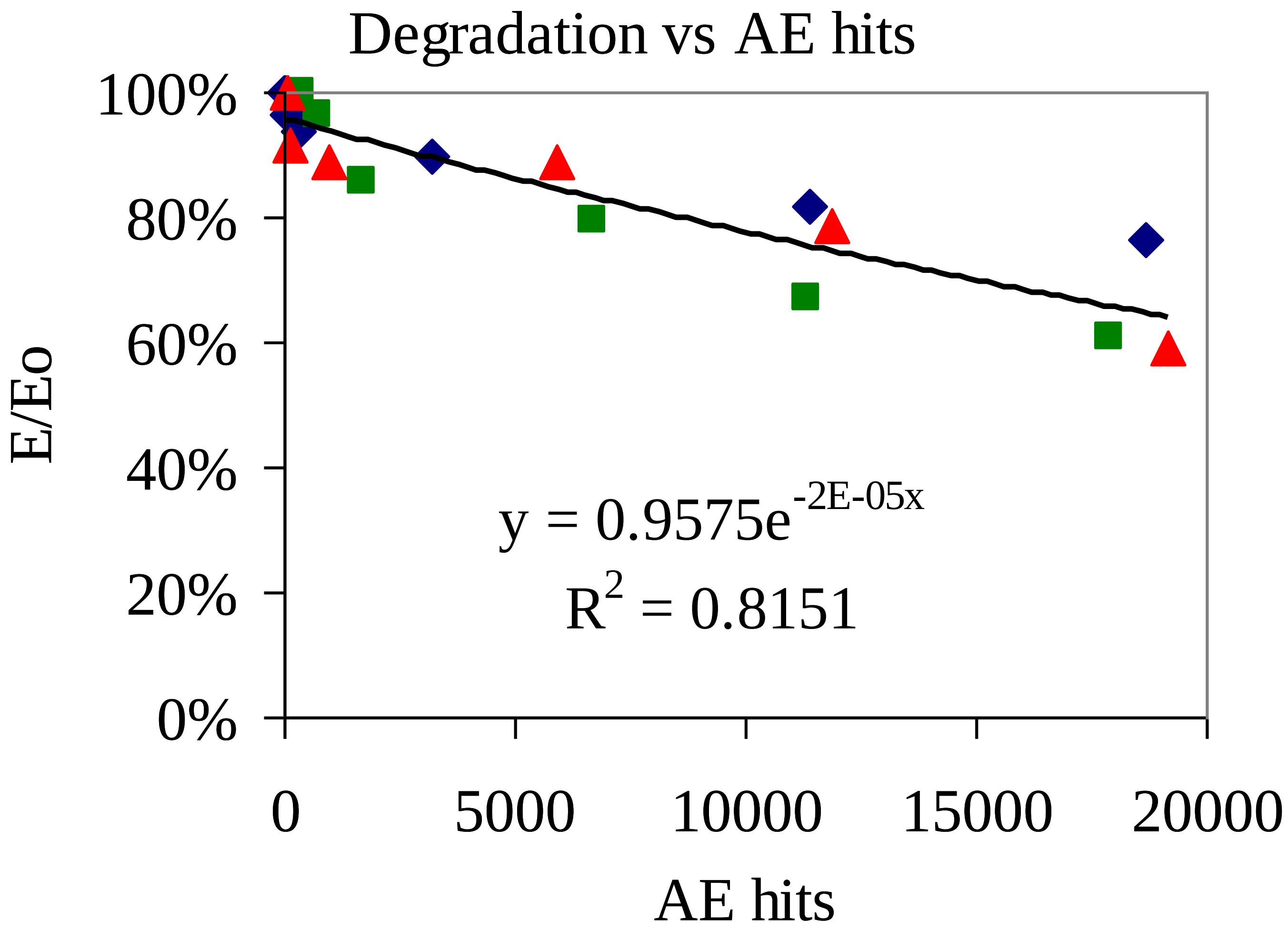
ie. their structural health had been severely compromised

Relative Stiffness loss vs. Load steps

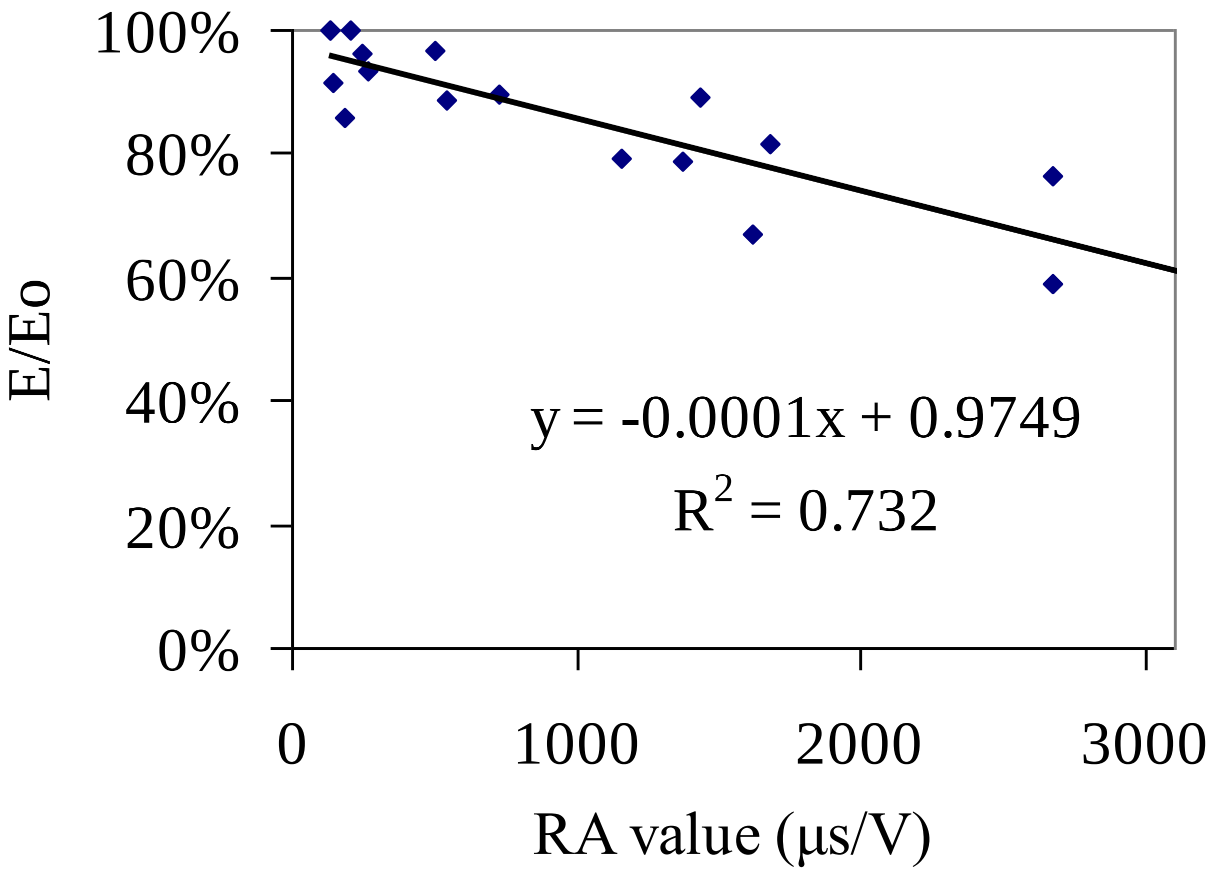


Relative Stiffness loss vs.

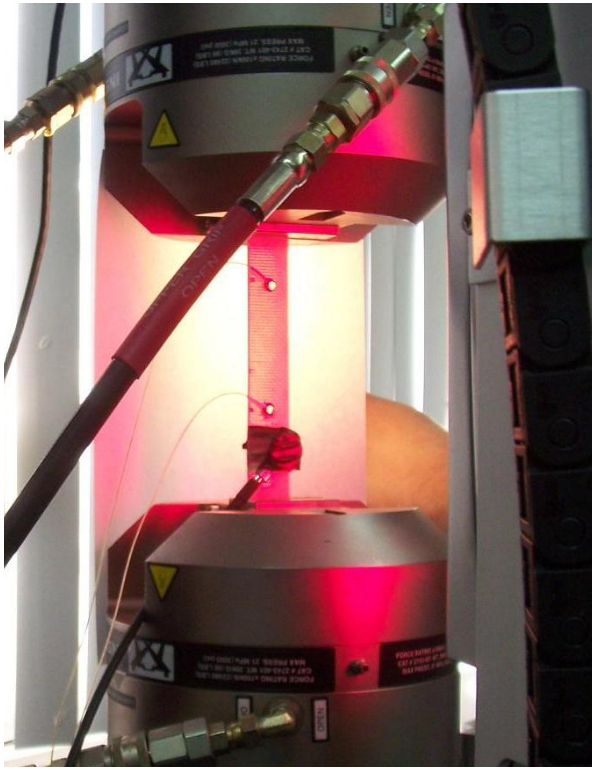
AE hits (for each step)



Relative Stiffness loss vs. mean RA (for each step)



Acoustic monitoring of GFRPs under Fatigue loading



Frequency 5 Hz

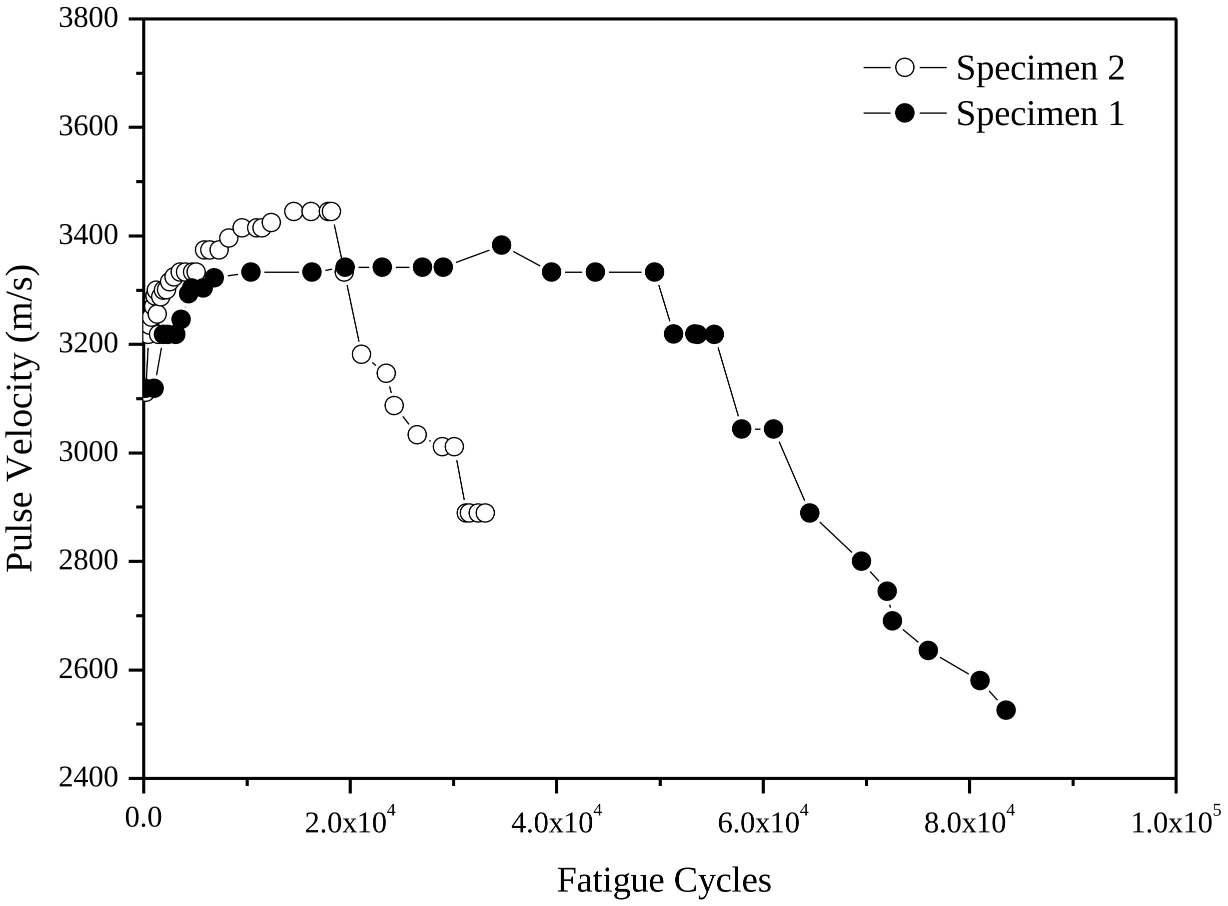
R=0.1

3 stress levels

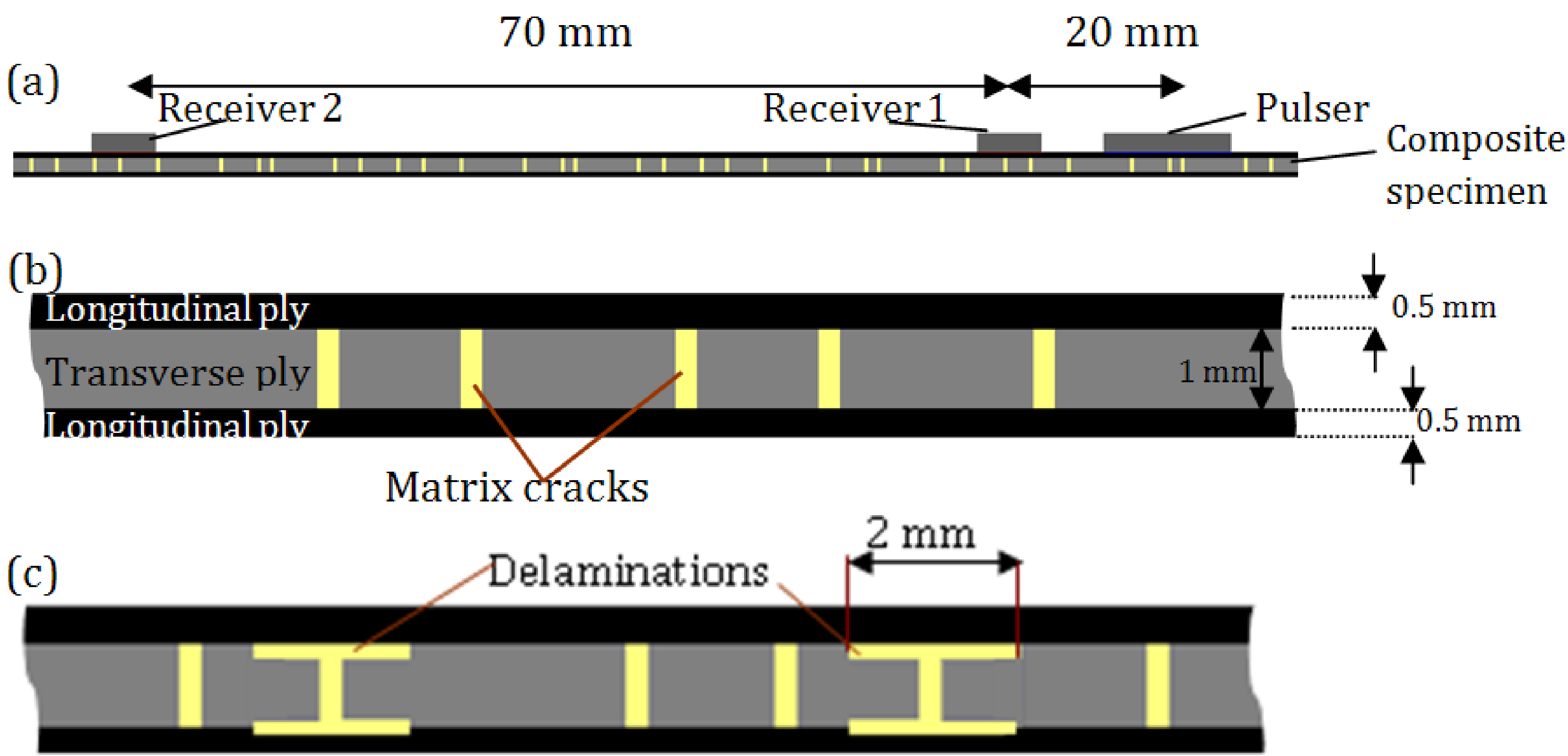
The pulser (R15, PAC) emits a tone burst of ten electric cycles of 200 kHz every 10s.

The pico sensors record the emitted signal.

Pulse velocity vs. N

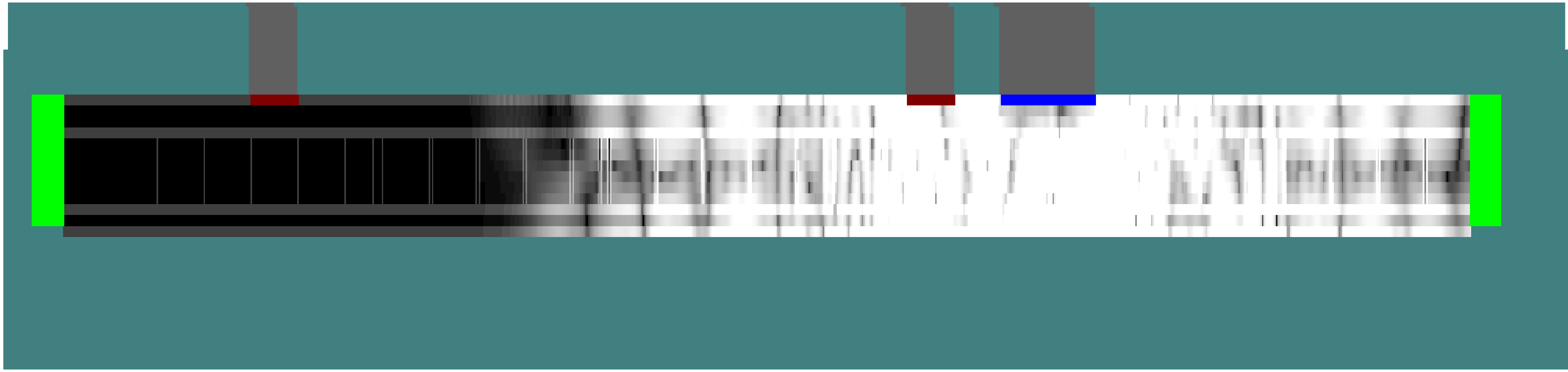


Simulation of wave propagation



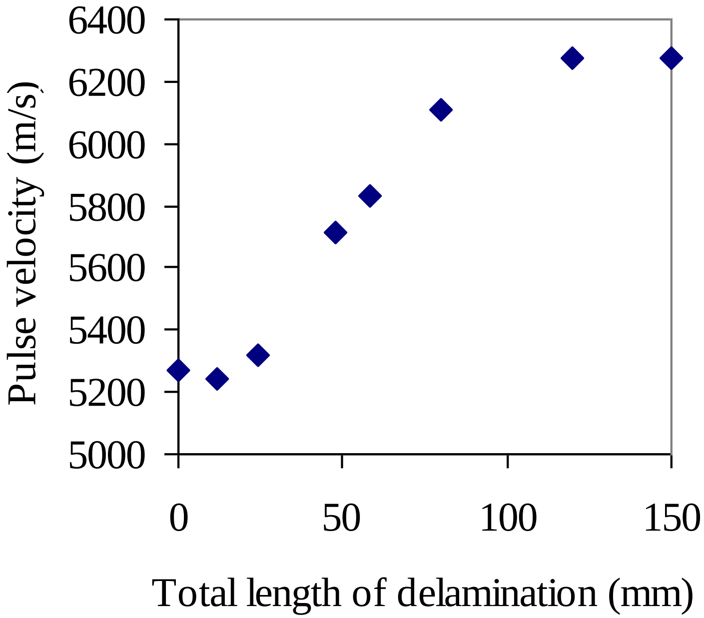
E_L/E_{tr}=10, Wavelength =10 mm

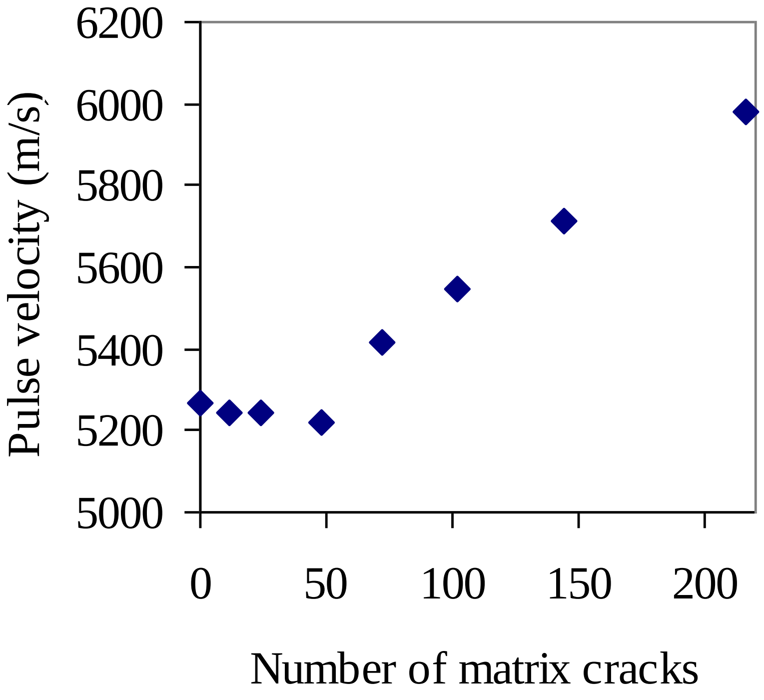
Simulation of wave propagation



Simulation vs. number of matrix cracks

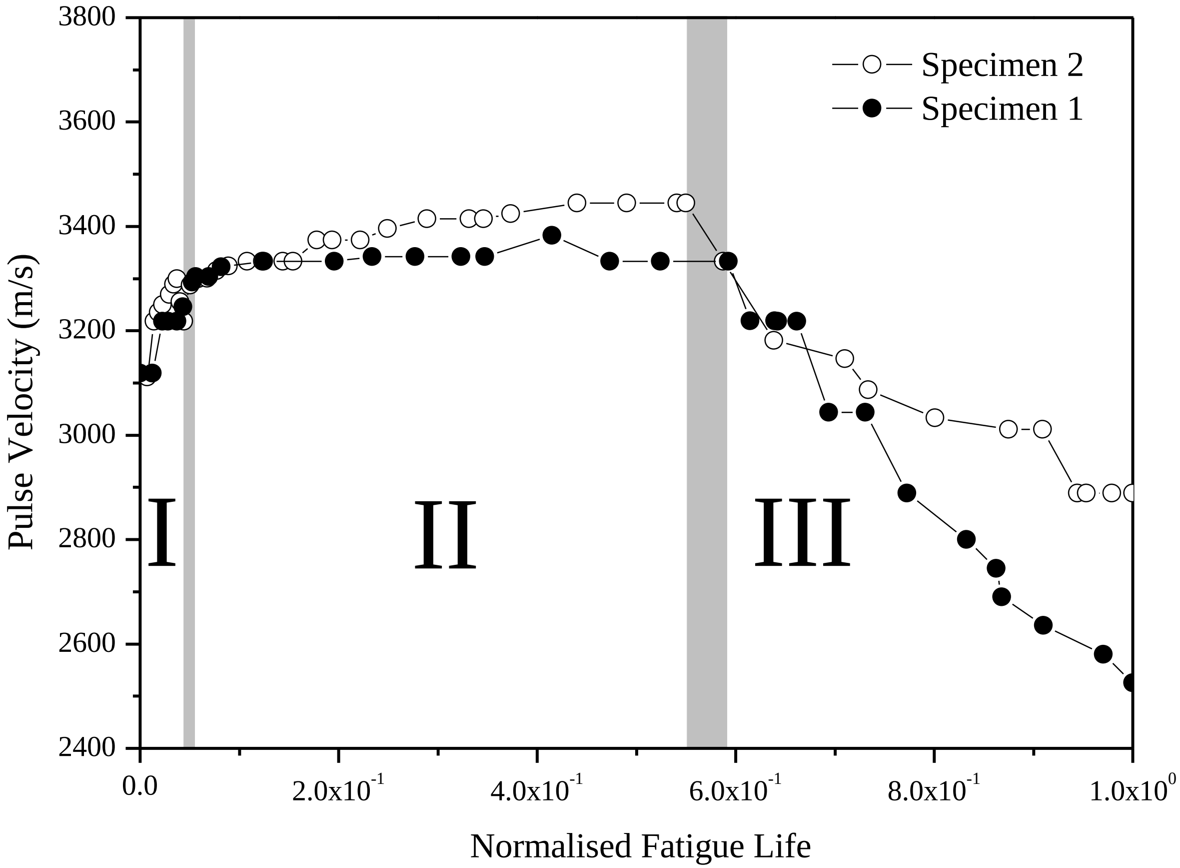
(excitation 10 cycles of 500 kHz)





Velocity increases with the number of matrix cracks and delminations as the top stiff layer becomes progressively more isolated

Pulse velocity vs. life fraction



Conclusions

- AE was successfully to identify and classify damage
- The pattern recognition algorithm successfully identified three major damage modes which were linked to distinct failure processes.
- AE parameters correlate well with damage modulus degradation and load (number of hits, RA, Energy)
- Wave propagation measurements were used to identify the distinct damage entities and correlated to the remaining life time of the composite
- Wave propagation behaves differently than other homogeneous materials: transmission and velocity may increase with accumulation of damage due to isolation of top layer.