

GDR MIC (Composites) - Cours doctoraux: GDR MIC – 13 au 17 novembre 2017

Evaluation non destructive par ultrasons de matériaux composites.

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Définition d'un essai non destructif

Etudier l'état d'un milieu (solide, fluide, poreux, pièces individuelles, structures, assemblages ...) sans détérioration ou modification irréversible de celui-ci.

Les essais N.D. permettent de tester et de conserver autant d'échantillons que souhaité, contrairement aux essais destructifs qui consistent à tester, avec modification irréversible, N échantillons d'une famille et à établir une loi probabiliste sur l'état des pièces restantes (coûteux, risqué).

Deux familles d'essais non destructifs

- Contrôle Non Destructif

Détecter, positionner, identifier, dimensionner des défauts dans le milieu.

- Evaluation Non Destructive

Mesurer de façon indirecte des caractéristiques du milieu.

Quand utiliser les essais non destructifs ?

- **En fabrication**

Contrôle intégrité matière
Suivi de Process (chaîne de production)
Certification des produits par rapport normes

- **En utilisation**

Suivi de santé (health monitoring), vieillissement, durabilité,...
Expertise
Maintenance

Secteurs d'applications des essais non destructifs



- Industries de production

Métaux, polymères, céramiques, verres, **composites** à matrices organiques, métalliques ou céramiques, structures complexes, pièces de fonderie, etc...



- Industries du transport

Ferroviaire, Automobile, Naval, Aéronautique, Aérospatial



- Génie Civil - Ponts & Chaussées

Béton, béton armé, bâtiments divers, ...
Etat des routes, des ponts, des pistes d'atterrissage, ...



- Industries énergétiques

Stockage et transport de l'eau, du gaz, du pétrole, centrales nucléaires, etc ...



- Industries Agroalimentaires

Fruits, légumes, confiserie, ...



- Industries du bois et du papier

Planches, meubles, constructions en bois, cartons, feuilles, ...



- Médical

Contrôle de tout le corps humain (os, poumons, dents, ...) par échographie, radiographie, ...

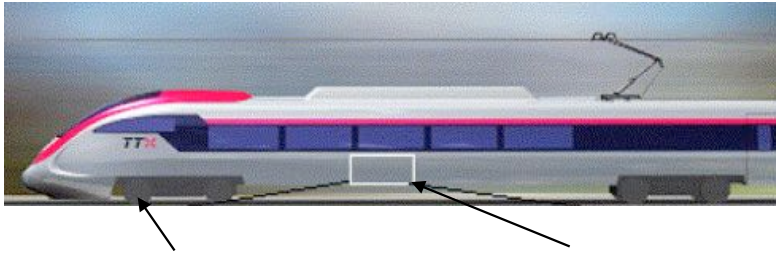


- etc ...

Types de défauts selon l'environnement

- **Matériaux solides**
Porosités, criques, micro-fissures, fissures, corrosion, taux de fibre/matrice non conforme, décohésions fibre-matrice, délaminages inter-plis, présence de nœuds, problèmes de cotation, de forme, ... chute des propriétés mécaniques...
- **Assemblages**
Problèmes liés aux rivets, soudures, serrages de boulon, collages (adhésion, cohésion), revêtements (peintures, dépôts chimiques...), ajustements, jeux,...
- **Structures et constructions**
Corrosion sur les rails de chemins de fer, dans les armatures de bétons armés, dans les canalisations de transports de fluides,... Fissuration des ponts, des bâtiments, des routes...
- **Nourriture**
Immaturité, pourriture, géométrie non conforme, présence de pépins, ... pour les fruits ou légumes
Trous, géométrie non conforme pour les confiseries (bonbons, sucettes, ...)
- **Corps humain**
Fêlure, fissures ou rupture d'un os – Ostéoporose – Eau dans les poumons – Malformation...
- **Sol terrestre**
Cavités cachées, nappes d'eau, composition des couches terrestres, fissures ...
- etc ...

De nombreux secteurs industriels utilisent les matériaux composites



*Brake lining made with metallic and/or ceramic -based composites to **reduce heating** but brake disks are metallic*

Car-body structure with aluminium honeycomb core and woven fabric carbon/epoxy face



*Carbon body structure ... **light, crash-test***



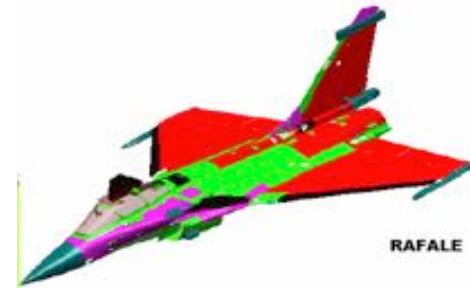
*Graphite, Kevlar, Carbon, Epoxy, PEEK
Maximal strength + minimal weight*



*Carbon or ceramic -based composites ... **light and fire-resistant***



*Glass, Carbon, Polyester, Epoxy
Light structures ... **No corrosion***



- Carbon / Epoxy
- Thermoplastic and others
- Aluminium alloys
- Titanium alloys



High performances + low weight



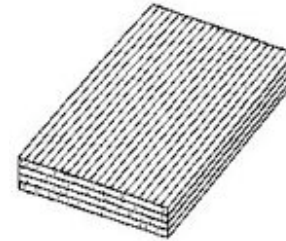
Typical composites used in many industries

Fibre reinforced composites:

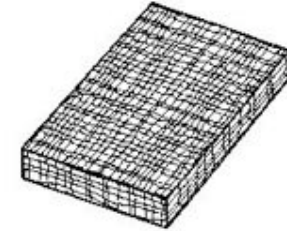
- **Fibres:** Glass, Carbon, Kevlar, Aramid, Boron, ...
- **Matrix:** Epoxy, Thermoplastic or thermosetting plastic, Ceramic, Metal, ...

Moulding methods:

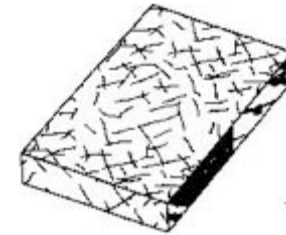
- Autoclave
- Resin Transfer moulding
- Vacuum or pressure bag moulding
- Filament winding
- etc...



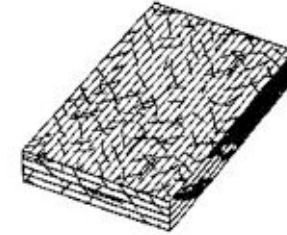
Continuous fibres



Woven fibres

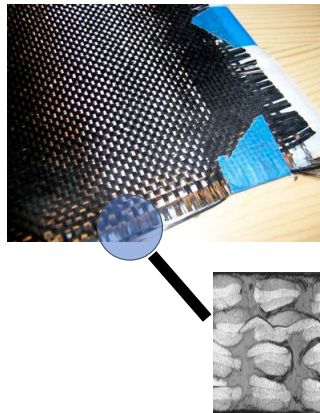


Chopped fibres

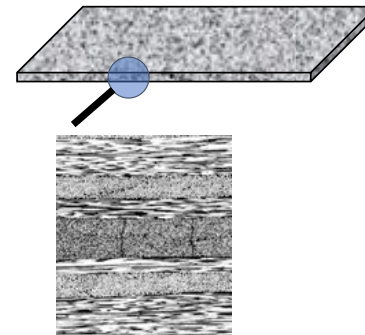
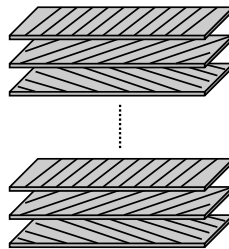


Hybrid composites

Elementary ply



Laminate = stacking of plies with various orientations

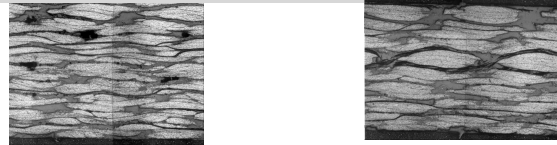


Sandwich structure



Possible defects in composites

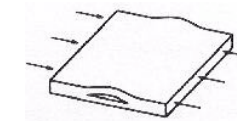
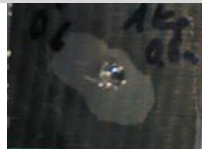
→ Moulding processes of composite materials may introduce **defects: porosity, fibre misalignment, disbonds ...**



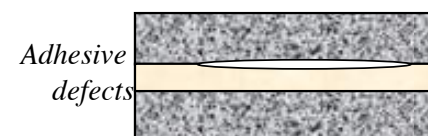
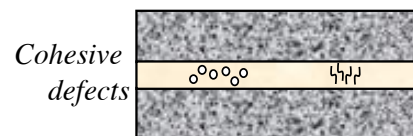
→ Composite materials are likely to have **properties changing with time = material ageing** due to **humid environments, mechanical loading** and **thermal changes** that may cause quite **uniform damaging** like **moisture content, matrix micro-cracking, fibre failure** and/or **fibre-matrix disbonding**, for example.



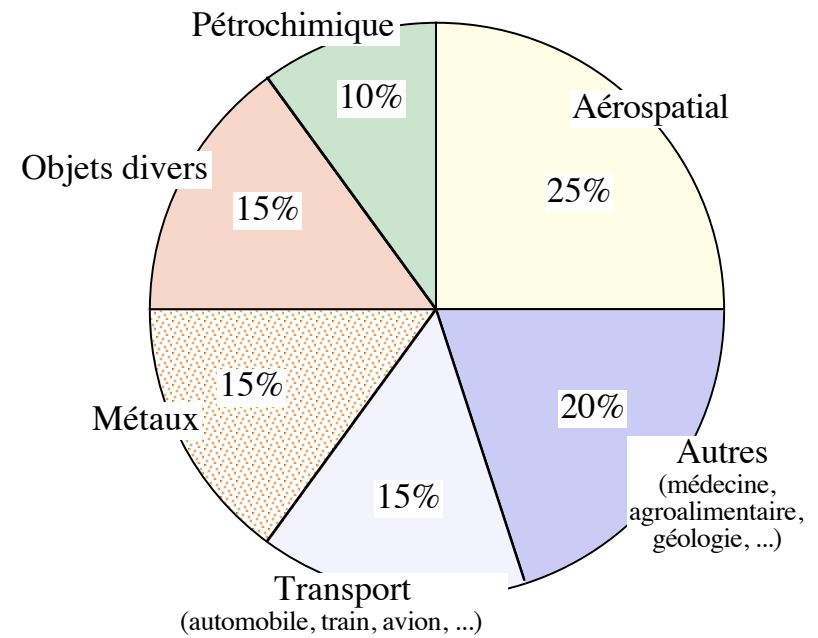
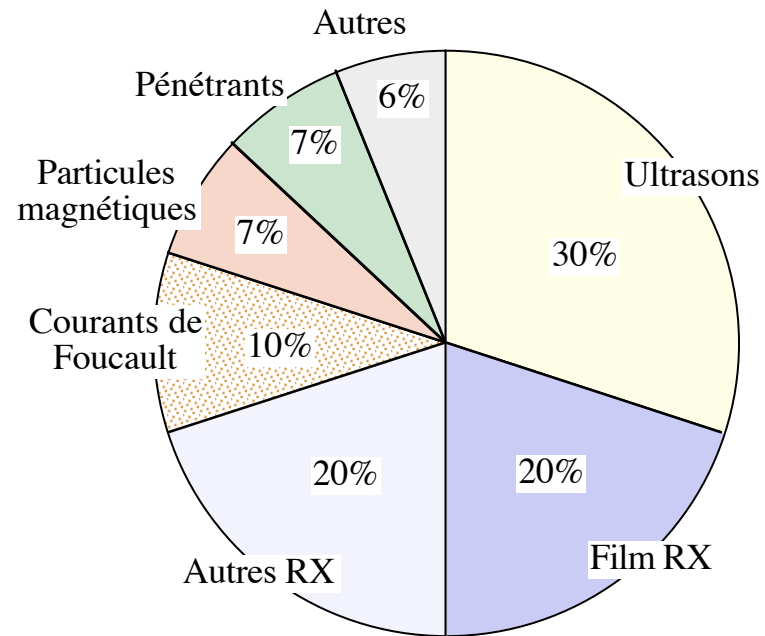
→ Corrosion disappears but **impacts** (lost bird, falling tool, ...) often cause **local defects** like **cracking thru thickness** and/or **delaminations** that may enlarge with time when structure is loaded.



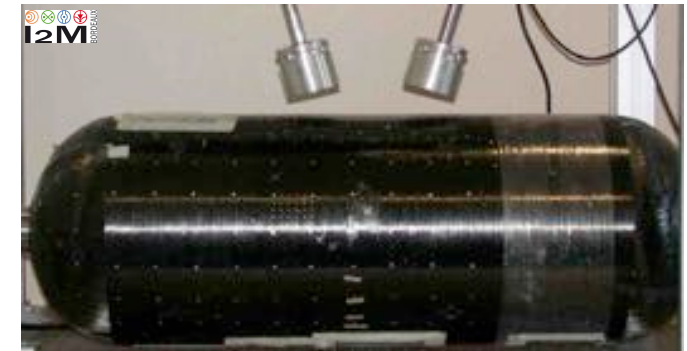
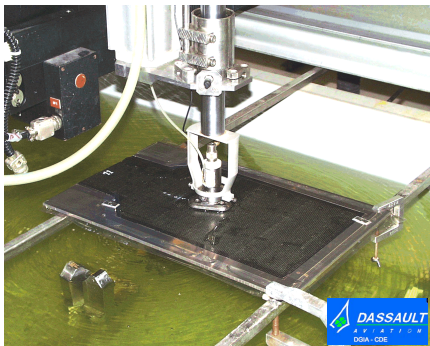
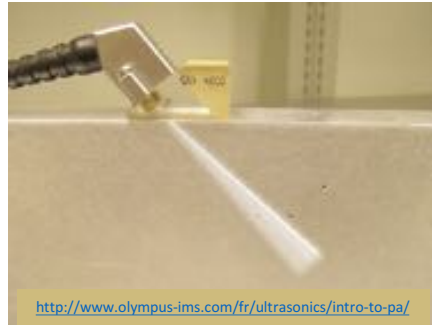
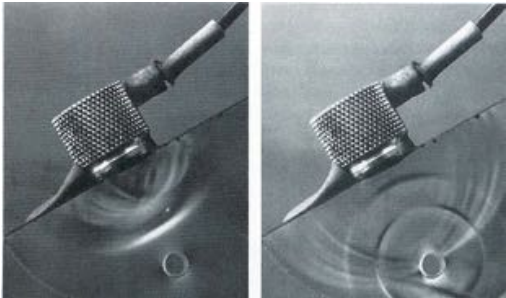
→ Composites are more and more **assembled** using **adhesive bonds** that may fail (cohesion or adhesion) at any time during in-service use, when structure is loaded.



Principales techniques de CND-END

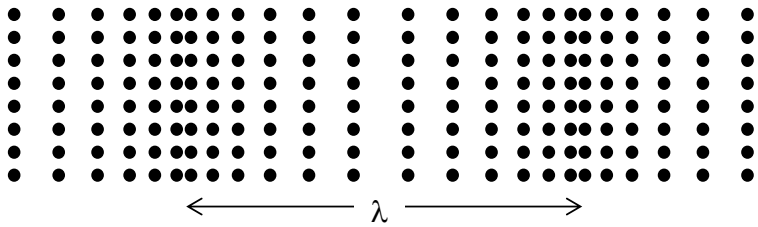


Couplage fluide souvent utilisé pour générer & détecter des ondes ultrasonores dans les solides à inspecter

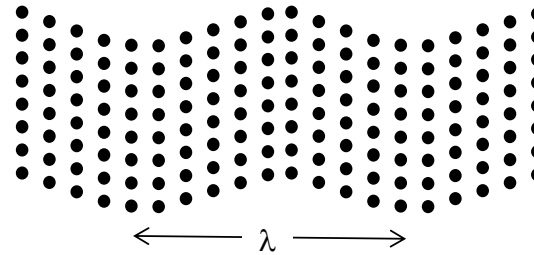


Ondes ultrasonores : deux modes fondamentaux de propagation dans les solides

Onde de compression (longitudinale ou P)



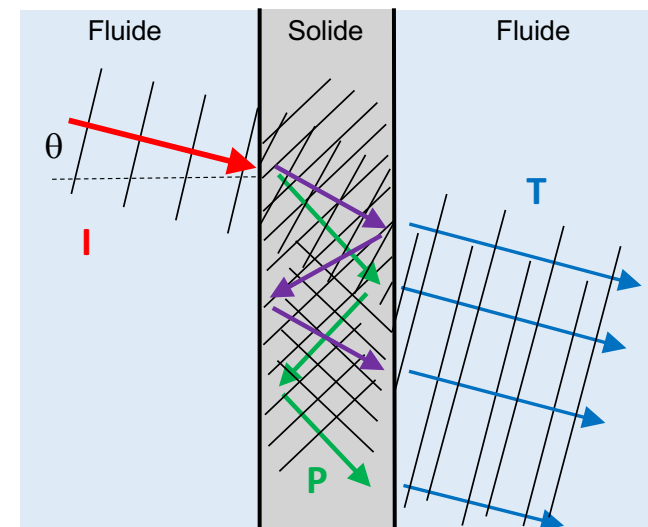
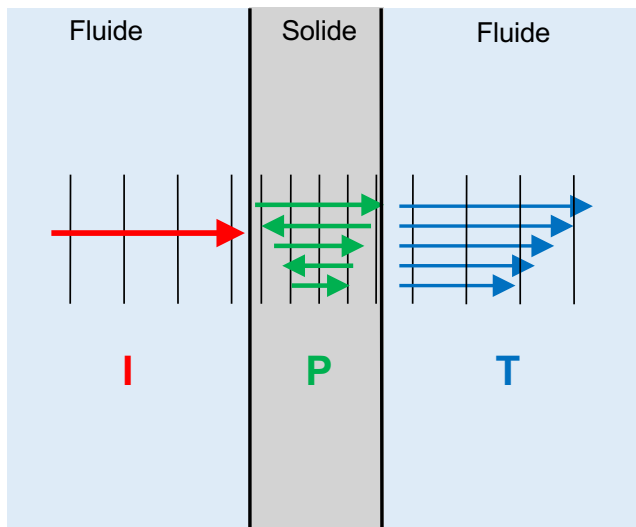
Onde de cisaillement (transversale ou S)



Remarque

Que des ondes de compression dans les fluides non visqueux (pas de cisaillement)

Configuration classique pour générer des ondes dans les solides

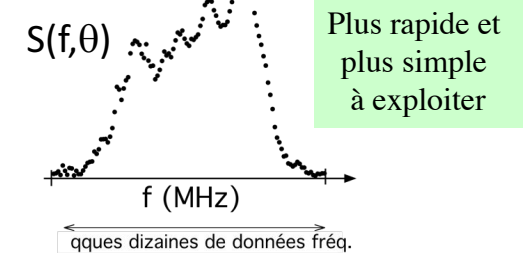
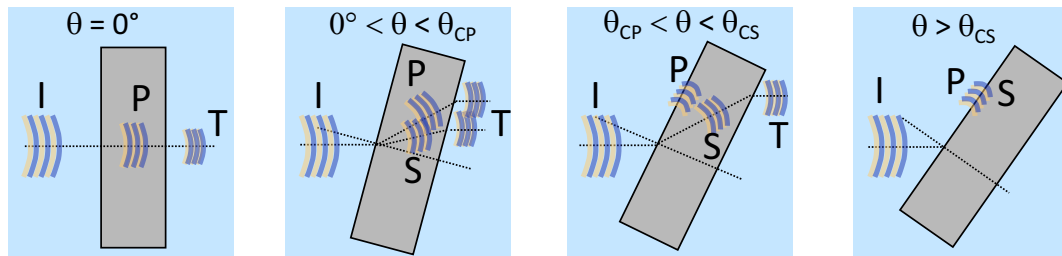
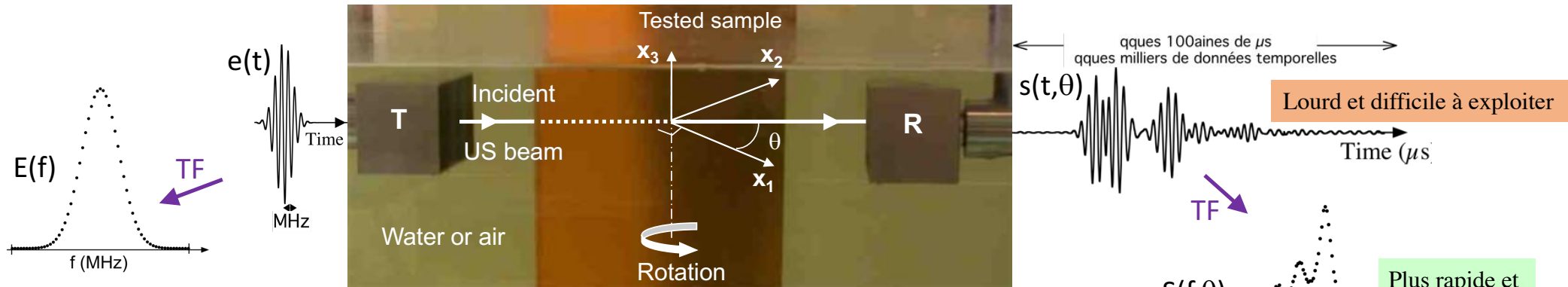


Evaluation Non Destructive

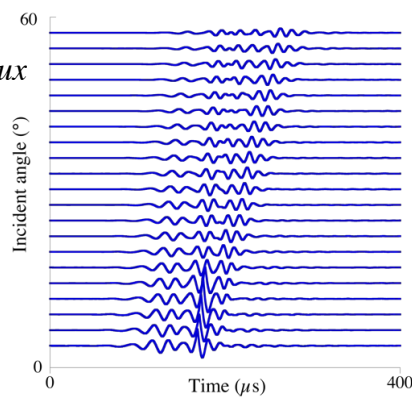
E.N.D.

- *Données d'entrées pour les simulations numériques :*
 - *Calculs de structures (bureaux d'études) ;*
 - *Développement ou optimisation de techniques avancées de CND-END ;*
- *Contrôle sur chaines de production (conformité des matériaux, cahiers des charges,...) ;*
- *Suivi de santé des matériaux et structures (endommagement, vieillissement, durabilité,...).*

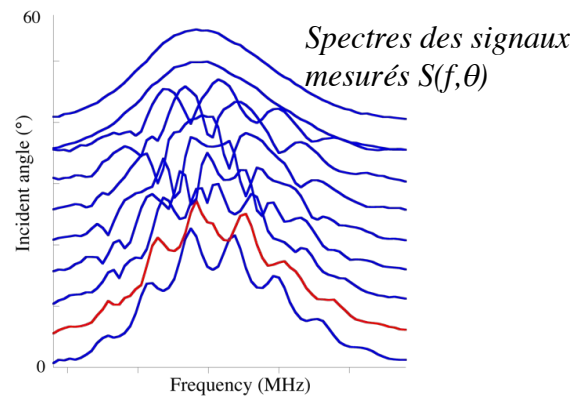
Procédé expérimental pour caractériser les propriétés mécaniques d'un matériau, par ultrasons



Ensemble de signaux mesurés $s(t, \theta)$



TF

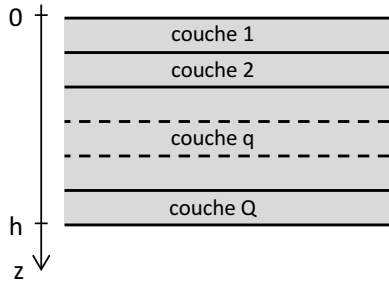


Coefficients de transmission expérimentaux

$$T^{\text{exp}}(f, \theta) = \frac{S(f, \theta)}{E(f)} = F(f, \theta, h^{1..Q}, \rho^{1..Q}, C_{ij}^{1..Q})$$

Modèle matriciel pour simuler numériquement la propagation des ultrasons dans un matériau stratifié, anisotrope et viscoélastique

Hypothèse : onde plane
Restriction : géométrie plane



Contraintes aux interfaces q-1 et q (surfaces de la couche q)

$$\begin{bmatrix} \mathbf{T}^{\zeta_{q-1}} \\ \mathbf{T}^{\zeta_q} \end{bmatrix} = \mathbf{Z}^q \begin{bmatrix} \mathbf{V}^{\zeta_{q-1}} \\ \mathbf{V}^{\zeta_q} \end{bmatrix} = \begin{bmatrix} \mathbf{Z}_{11}^q & \mathbf{Z}_{12}^q \\ \mathbf{Z}_{21}^q & \mathbf{Z}_{22}^q \end{bmatrix} \begin{bmatrix} \mathbf{V}^{\zeta_{q-1}} \\ \mathbf{V}^{\zeta_q} \end{bmatrix}$$

Vitesses particulières aux interfaces q-1 et q (surfaces de la couche q)

Matrice d'impédance de la couche q (expressions analytiques des ondes P et S) : $\mathcal{F}(h^q, \rho^q, C_{ij}^q, f, \theta)$

Matrice d'impédance pour les q+1 premières couches (démarche récursive)

$$\mathbf{Z}^{1..q+1} = \begin{bmatrix} \mathbf{Z}_{11}^{1..q} + \mathbf{Z}_{12}^{1..q} \mathbf{z} \mathbf{Z}_{21}^{1..q} & -\mathbf{Z}_{12}^{1..q} \mathbf{z} \mathbf{Z}_{22}^{q+1} \\ \mathbf{Z}_{21}^{q+1} \mathbf{z} \mathbf{Z}_{21}^{1..q} & \mathbf{Z}_{22}^{q+1} - \mathbf{Z}_{21}^{q+1} \mathbf{z} \mathbf{Z}_{12}^{q+1} \end{bmatrix}$$

où $\mathbf{z} = (\mathbf{Z}_{11}^{q+1} - \mathbf{Z}_{22}^{1..q})^{-1}$ et $\mathbf{Z}_{ij}^{1..q}$ (i, j = 1,2) sont les sous matrices d'impédance des q premières couches.

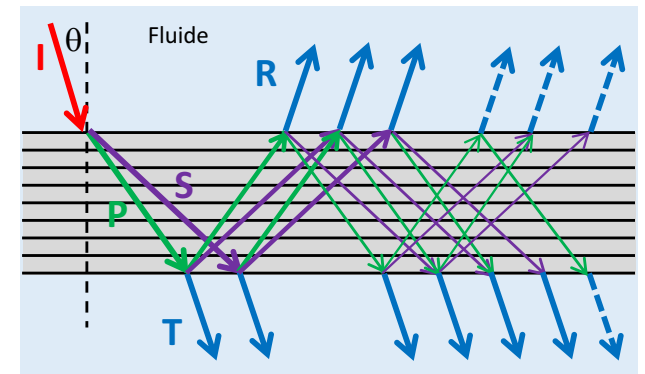
La matrice d'impédance $\mathbf{Z}^{1..Q}$ d'un multicouche constitué de Q couches permet de relier les contraintes appliquées aux surfaces externes (z=0 et z=h) avec les vitesses particulières en ces mêmes surfaces :

$$\begin{bmatrix} \mathbf{T}^{\zeta_0} \\ \mathbf{T}^{\zeta_Q} \end{bmatrix} = \mathbf{Z}^{1..Q} \begin{bmatrix} \mathbf{V}^{\zeta_0} \\ \mathbf{V}^{\zeta_Q} \end{bmatrix} = \begin{bmatrix} \mathbf{Z}_{11}^{1..Q} & \mathbf{Z}_{12}^{1..Q} \\ \mathbf{Z}_{21}^{1..Q} & \mathbf{Z}_{22}^{1..Q} \end{bmatrix} \begin{bmatrix} \mathbf{V}^{\zeta_0} \\ \mathbf{V}^{\zeta_Q} \end{bmatrix}$$

Si le multicouche est immergé dans un fluide, l'écriture des conditions aux limites aux interfaces fluide / solide (en z=0 et z=h) permet d'exprimer le coefficient de transmission (T) pour une onde plane monochromatique sous une incidence θ :

$$T = \frac{2 \Lambda S_{21}^{33}}{(S_{11}^{33} + \Lambda)(S_{22}^{33} - \Lambda) - S_{21}^{33} S_{12}^{33}} = F(f, \theta, h^{1..Q}, \rho^{1..Q}, C_{ij}^{1..Q})$$

avec $\Lambda = \frac{\cos \theta}{i \omega \rho_f V_f}$, où ρ_f est la densité du fluide, V_f la célérité dans le fluide, θ l'angle d'incidence et ω la pulsation.



Optimisation des modules de rigidité

Minimisation de la fonctionnelle :
$$\delta = \sum_{\theta_{\min}^{Exp}}^{\theta_{\max}^{Exp}} \sum_{f_{\min}^{Exp}}^{f_{\max}^{Exp}} \left[\left| T^{Th}(f, \theta, h, \rho, C_{ij}) \right| - \left| T^{Exp}(f, \theta) \right| \right]^2$$

Deux cas de figures

Plaque faite d'un seul matériau homogène à l'échelle des fréquences de travail

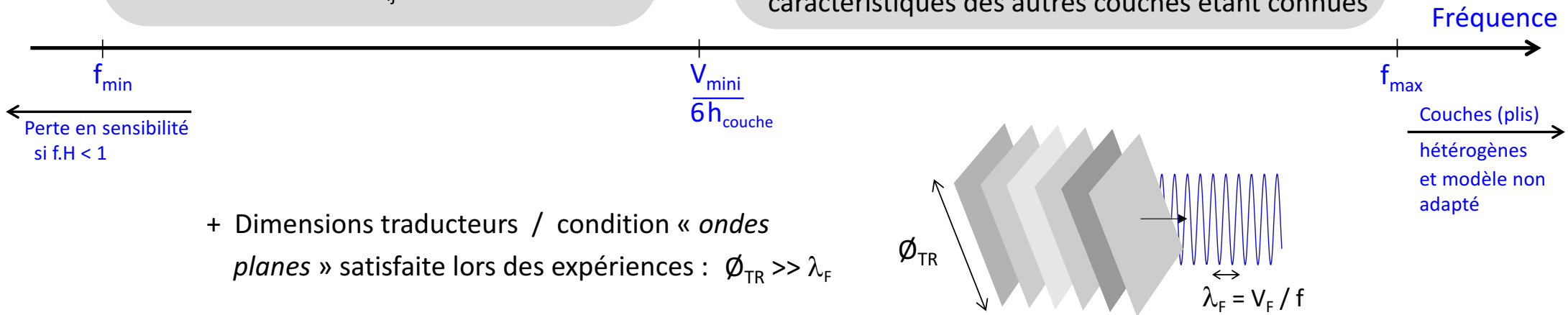


Optimisation des C_{ij} du matériau

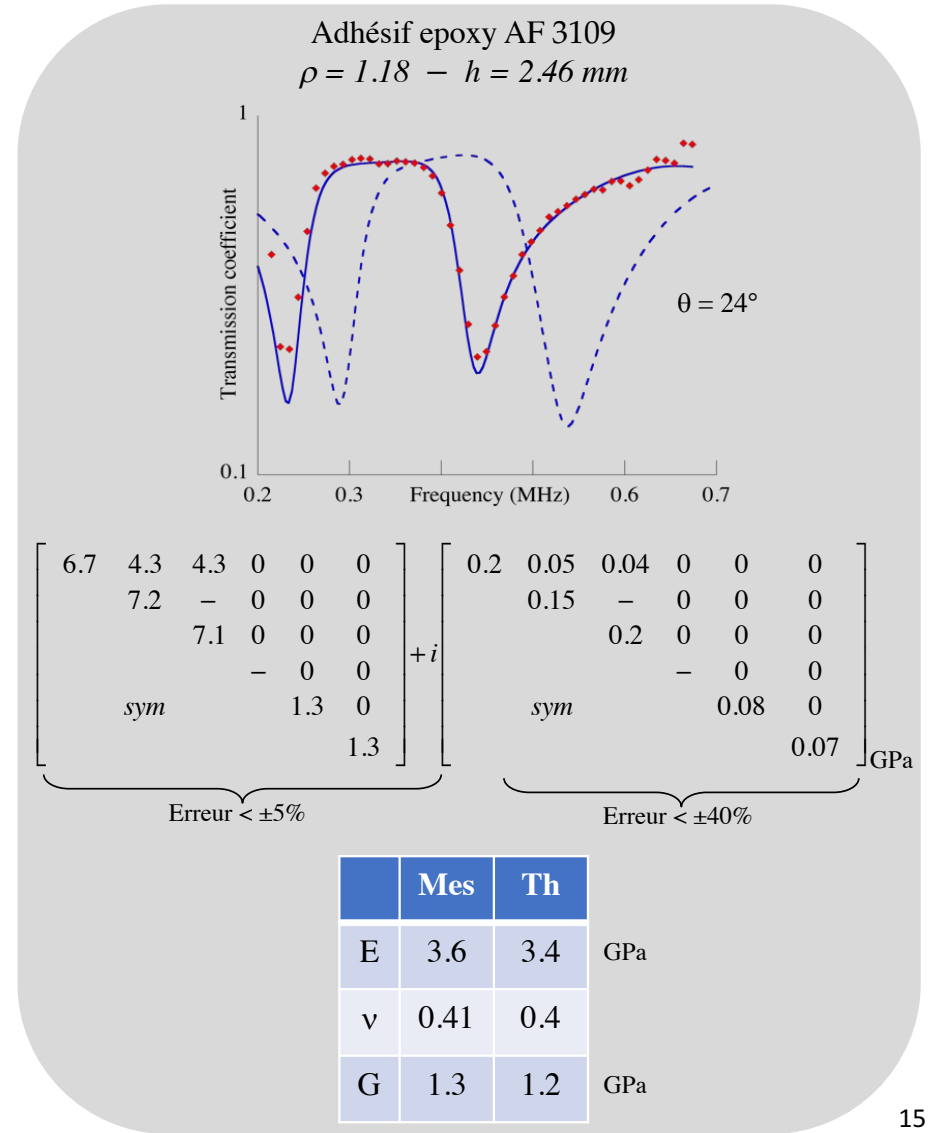
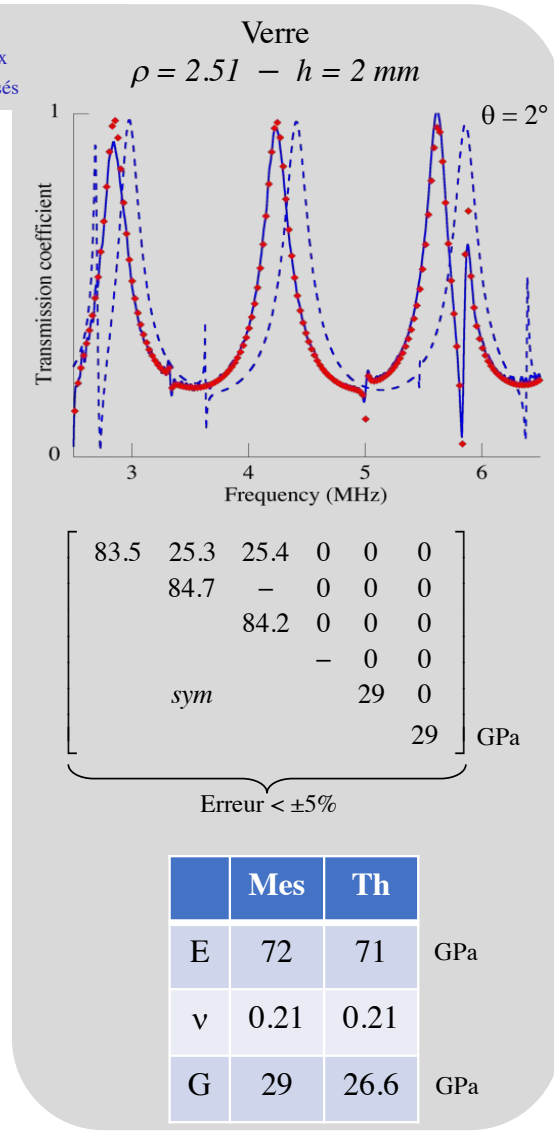
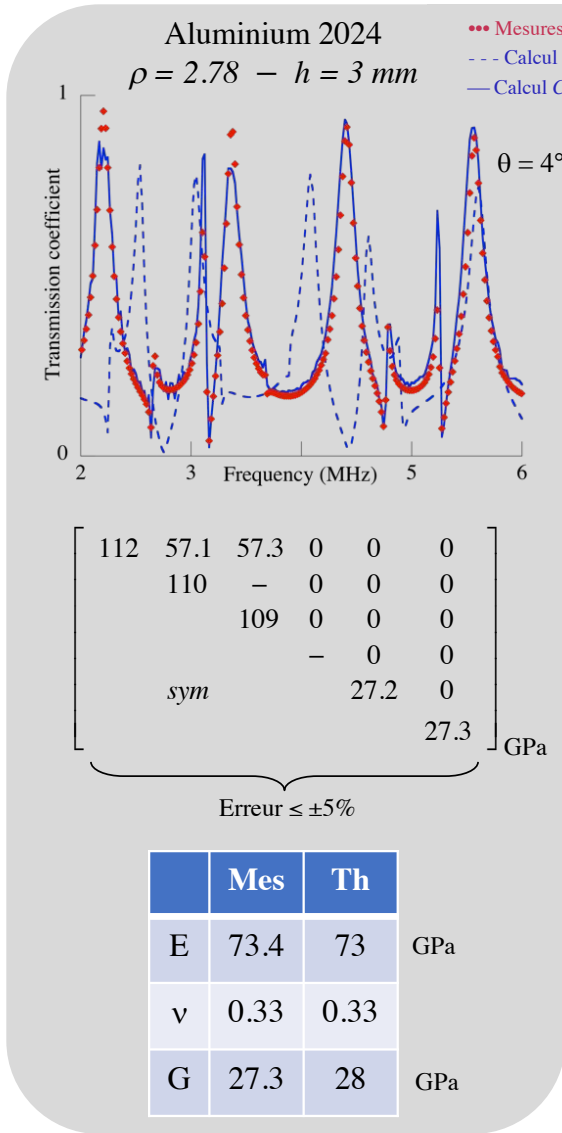
Plaque vue comme un empilement de plusieurs matériaux chacun étant homogène à l'échelle des fréquences de travail, mais plaque hétérogène



Optimisation des C_{ij} d'une couche (pli, colle,...), les caractéristiques des autres couches étant connues



Exemples de caractérisation des propriétés mécaniques de matériaux isotropes

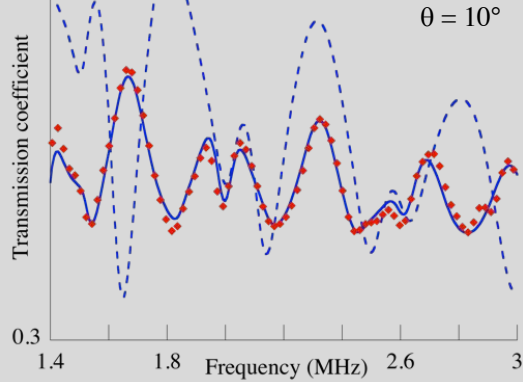
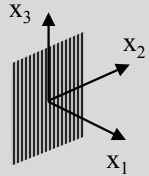


Exemples de caractérisation des propriétés mécaniques de matériaux composites

Carbone Epoxy UD composite

$\rho = 1.58$ - $h = 4.82$ mm

••• Mesures
 --- Calcul C_{ij} initiaux
 — Calcul C_{ij} optimisés



$\theta = 10^\circ$

$$\begin{bmatrix} 14.3 & 7.4 & 8.2 & 0 & 0 & 0 \\ & 13.7 & 7.5 & 0 & 0 & 0 \\ & & 156 & 0 & 0 & 0 \\ & & & 5.8 & 0 & 0 \\ & sym & & & 5.2 & 0 \\ & & & & & 3.4 \end{bmatrix} + i \begin{bmatrix} 0.4 & 0.1 & 0.2 & 0 & 0 & 0 \\ & 0.4 & 0.1 & 0 & 0 & 0 \\ & & 5 & 0 & 0 & 0 \\ & & & 0.08 & 0 & 0 \\ & sym & & & 0.6 & 0 \\ & & & & & 0.12 \end{bmatrix} \text{ GPa}$$

Erreur < $\pm 10\%$ Erreur < $\pm 50\%$

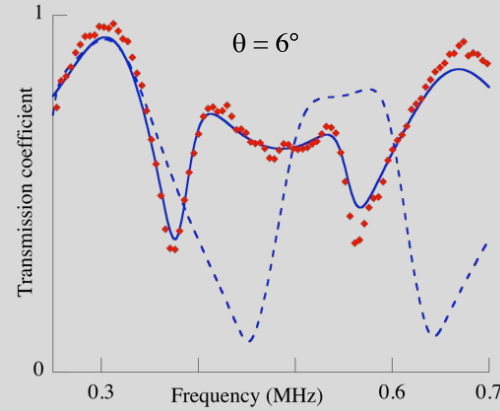
GPa	Mes	Th
E_1	10.2	-
E_2	9.8	-
E_3	150	146

	Mes	Th
ν_{12}	0.5	-
ν_{13}	0.4	-
ν_{23}	0.33	-

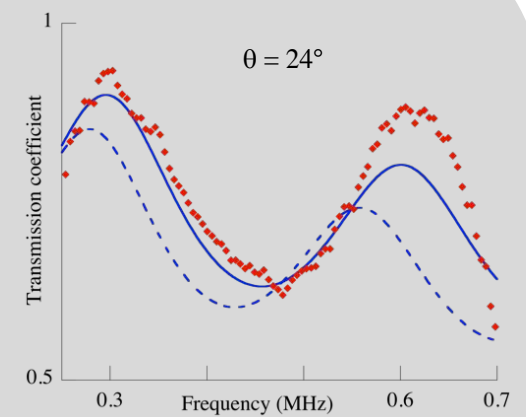
GPa	Mes	Th
G_{12}	3.4	-
G_{13}	5.3	-
G_{23}	5.8	5.2

Carbone Epoxy [0/45/90/-45]_{3s} composite

$\rho = 1.58$ - $h = 4.54$ mm



$\theta = 6^\circ$



$\theta = 24^\circ$

$$\begin{bmatrix} 13.0 & 8.7 & 7.8 & 0 & 0 & 0 \\ & 69 & - & 0 & 0 & 0 \\ & & 74 & 0 & 0 & 0 \\ & & & - & 0 & 0 \\ & sym & & & 3.8 & 0 \\ & & & & & 3.9 \end{bmatrix} + i \begin{bmatrix} 0.2 & 0.02 & 0.03 & 0 & 0 & 0 \\ & 6 & - & 0 & 0 & 0 \\ & & 6 & 0 & 0 & 0 \\ & & & - & 0 & 0 \\ & sym & & & 0.2 & 0 \\ & & & & & 0.16 \end{bmatrix} \text{ GPa}$$

Erreur $\leq \pm 25\%$ Erreur \uparrow

Milieu hétérogène car fréquence trop élevée

Si fréquence abaissée, TC pas suffisamment sensible aux C_{ij}

car plaque mince → Limites de la méthode

Prédiction des propriétés mécaniques de matériaux composites stratifiés

Simulation numérique
de l'expérimentation
avec des conditions idéales
BF (milieu homogène)
et un stratifié très épais
(grand nombre de super-couches
Ex.: [0/45/90/-45]_{20s})

C_{ij} mesurés pour plaque UD assimilés aux C_{ij} de chaque pli

$$\begin{bmatrix} 14.3 & 7.4 & 8.2 & 0 & 0 & 0 \\ & 13.7 & 7.5 & 0 & 0 & 0 \\ & & 156 & 0 & 0 & 0 \\ & & & 5.8 & 0 & 0 \\ sym & & & & 5.2 & 0 \\ & & & & & 3.4 \end{bmatrix} + i \begin{bmatrix} 0.4 & 0.1 & 0.2 & 0 & 0 & 0 \\ & 0.4 & 0.1 & 0 & 0 & 0 \\ & & 5 & 0 & 0 & 0 \\ & & & 0.08 & 0 & 0 \\ sym & & & & 0.6 & 0 \\ & & & & & 0.12 \end{bmatrix}$$



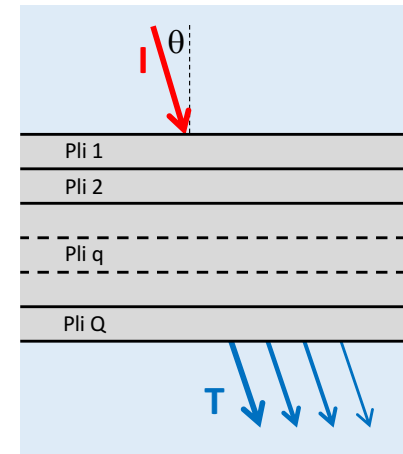
Matrice d'impédance $Z^{1..Q}$ du multicouche constitué de Q couches

$$\begin{bmatrix} \mathbf{T}^{\xi_0} \\ \mathbf{T}^{\xi_Q} \end{bmatrix} = \mathbf{Z}^{1..Q} \begin{bmatrix} \mathbf{V}^{\xi_0} \\ \mathbf{V}^{\xi_Q} \end{bmatrix} = \begin{bmatrix} \mathbf{Z}_{11}^{1..Q} & \mathbf{Z}_{12}^{1..Q} \\ \mathbf{Z}_{21}^{1..Q} & \mathbf{Z}_{22}^{1..Q} \end{bmatrix} \begin{bmatrix} \mathbf{V}^{\xi_0} \\ \mathbf{V}^{\xi_Q} \end{bmatrix}$$



Coefficient de transmission en ondes planes

$$T^{ExpSimul}(f, \theta, \varphi^{1..Q}, h^{1..Q}, \rho^{1..Q}, C_{ij}^{1..Q})$$

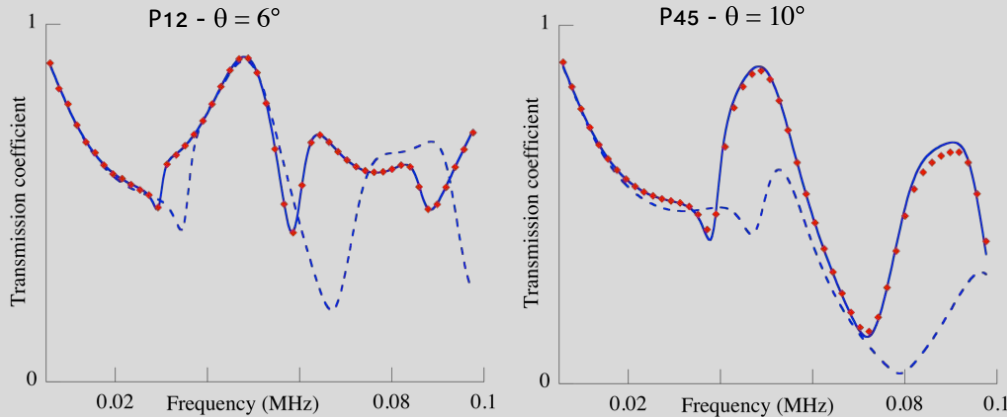


Optimisation des C_{ij} équivalents : $\sum \sum \left[\left| T^{ExpSimul}(f, \theta, \varphi^{1..Q}, h^{1..Q}, \rho^{1..Q}, C_{ij}^{1..Q}) \right| - \left| T^{th}(f, \theta, \varphi, H, \rho, C_{ij}^{EQUIV}) \right| \right]^2$

Exemples de simulation ondes dans empilements stratifiés + caractérisation des propriétés mécaniques équivalentes

Carbone Epoxy [0/45/90/-45]_{20s}
 $\rho = 1.58$ - $h = 30.24$ mm

••• Mesures simulées
 --- Calcul C_{ij} équivalents initiaux
 — Calcul C_{ij} équivalents optimisés



14.3	7.8	7.8	0	0	0	0.4	0.13	0.1	0	0	0
	67.9	24.5	0	0	0		2	0.18	0	0	0
		69.1	0	0	0			2	0	0	0
			21.6	0	0				0.14	0	0
				4.1	0					0.28	0
					4.1						0.28

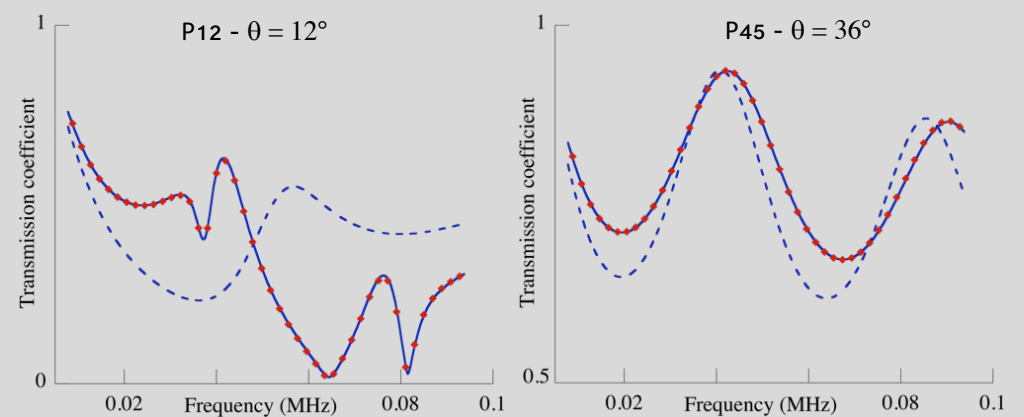
Erreur < ±1%

Erreur < ±20%

Modules de l'ingénieur déduits des C_{ij} optimisés :

$E_1 = 13$ - $E_2 = 57.3$ - $E_3 = 58.3$ GPa
 $\nu_{12} = 0.37$ - $\nu_{13} = 0.38$ - $\nu_{23} = 0.32$
 $G_{12} = 4.1$ - $G_{13} = 4.1$ - $G_{23} = 21.6$ GPa

Carbone Epoxy [45/-45/90/0/45/.../0/90/-45/45]_{6s}
 $\rho = 1.57$ - $h = 38.1$ mm
 24 plis



14.3	6.8	6.8	0	0	0	0.25	0.2	0.2	0	0	0
	57.2	19.7	0	0	0		2.1	1.6	0	0	0
		57.2	0	0	0			2.2	0	0	0
			18.8	0	0				0.3	0	0
				4.4	0					0.07	0
					4.4						0.07

Erreur < ±1%

Erreur < ±20%

Modules de l'ingénieur déduits des C_{ij} optimisés :

$E_1 = 13.1$ - $E_2 = 49$ - $E_3 = 49$ GPa
 $\nu_{12} = 0.33$ - $\nu_{13} = 0.33$ - $\nu_{23} = 0.30$
 $G_{12} = 4.4$ - $G_{13} = 4.4$ - $G_{23} = 18.8$ GPa

Characterization of Cohesive and Adhesive Properties of Adhesive Bonds Using Transmitted Ultrasonic Waves

Emmanuel SIRYABE, Mathieu RENIER, Anissa MEZIANE, Michel CASTAINGS
I₂M – CNRS UMR 5295, University of Bordeaux, FRANCE

JOCELYNE GALY

IMP – UMR 5223 CNRS – INSA Lyon, Villeurbanne Cedex, FRANCE

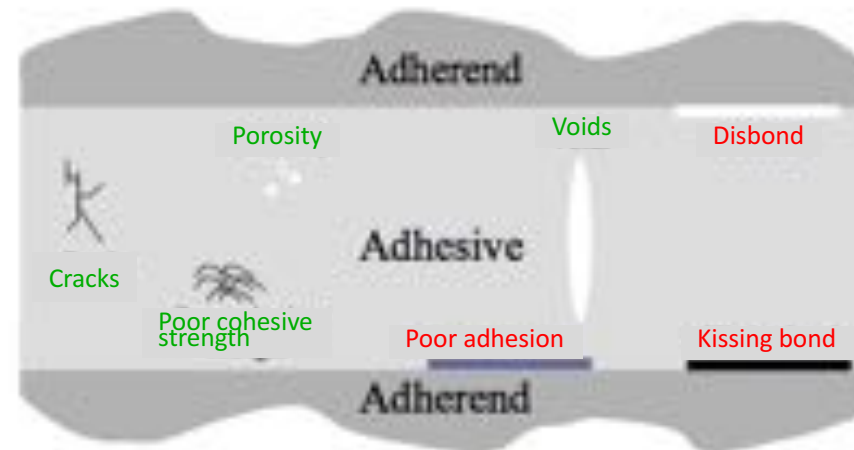
Project ISABEAU (Innovating for Structural Adhesive Bonding Evaluation and
Analysis with Ultrasounds) supported by French ANR, AIRBUS-DS and SAFRAN

Context: Replacement of screwing or welding assemblies by adhesive assemblies

→ *Light weight, uniform stress distribution along the bonded zones*

2 classes of defects:

- *Cohesive defects*
- *Interface defects*



NDE methods are required to distinguish between those two classes of defects and to evaluate mechanical weaknesses of the adhesive layer and/or of the interfaces.

Objectives & strategy

➤ Objectives:

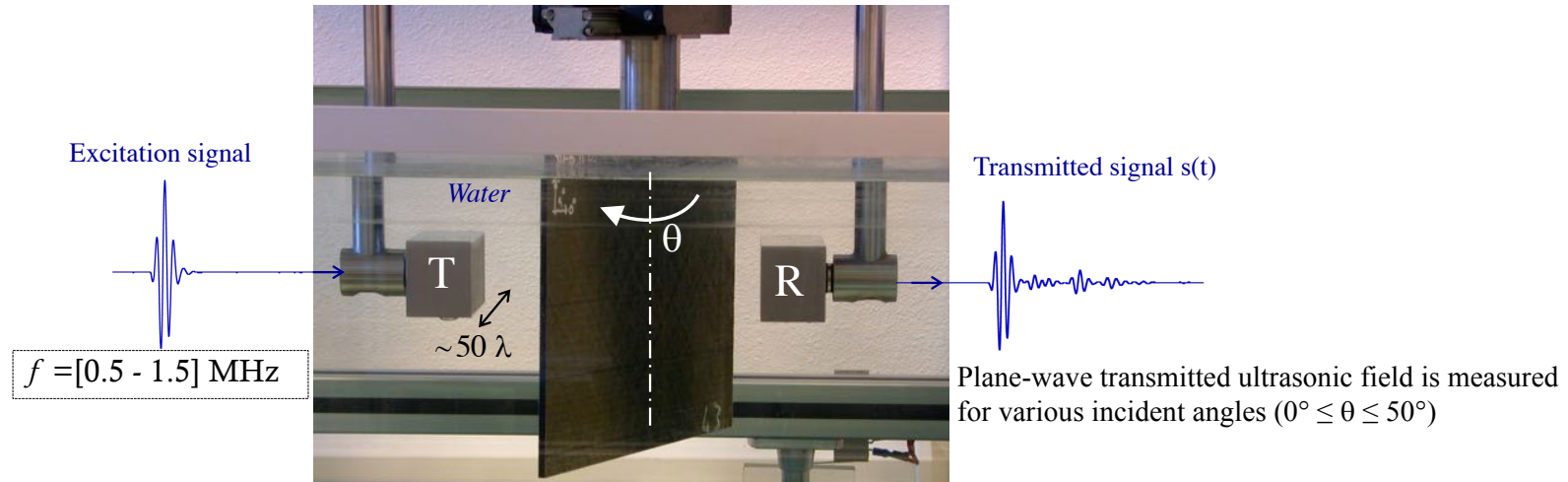
- 1) To infer mechanical properties of the adhesive layer between 2 substrates : cohesive properties;
- 2) To study the influence of surface treatment and incomplete cross-linking on that estimation of mechanical properties of the adhesive layer;
- 3) To infer mechanical properties of interphases connecting substrates to adhesive: adhesion properties

Zone of 1 or few μm thick

➤ Approach:

- Assemblies made of Aluminium substrates and Epoxy adhesive (isotropic & homogeneous materials);
- Immersion measurements of plane-wave transmitted ultrasonic field (PWTUF) for:
 - a) Optimization of C_{ij} of adhesive layer without the assumption of isotropic medium
+ investigation of the influence of the preparation of substrates' surfaces on these C_{ij} ;
 - b) Optimization of two stiffness's k_L and k_T for mechanical properties of interphases.

Measurements of (visco)elastic *moduli* using the plane-wave transmitted ultrasonic field (PWTUF) method



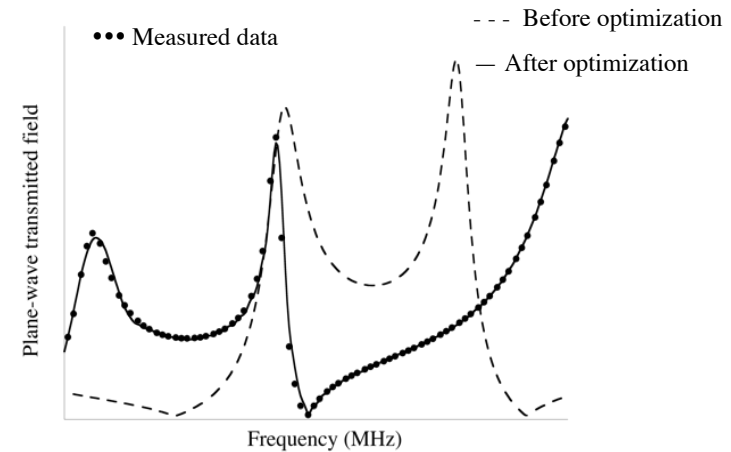
Transmitted acoustic fields are computed;

Assumptions: incident plane wave, homogeneous, anisotropic, visco-elastic material, perfect fluid.

$$|A_T^{Th}(f, \theta, C_{ij})| = |T^{Th}(f, \theta, C_{ij}) \cdot A_I^{Exp}(f)|$$

Optimization routine (Simplex method) is used to infer complex $C_{11}, C_{22}, C_{12}, C_{66}$ (in P_{12} plane) of material to be characterized.

Example of PWTUF for one given incident angle

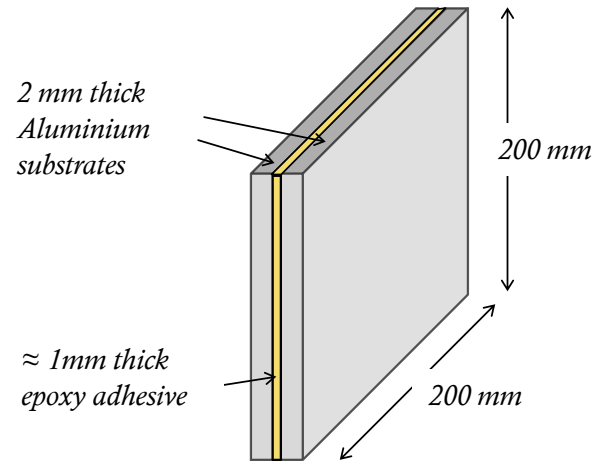


Characterization of components of the assembly from individual plate-like samples

<i>Aluminium</i>	<i>Epoxy (fully cured)</i>	<i>Epoxy (80% cured)</i>
$h_{alu} = 2 \text{ mm} \pm 3\%$	$h_{adhesive} = 2.92 \text{ mm} \pm 3\%$	$h_{adhesive} = 2.96 \text{ mm} \pm 3\%$
$C_{11} = 106 \text{ GPa}$	$C_{11} = 7.77 + i 0.23 \text{ GPa}$	$C_{11} = 7.56 + i 0.16 \text{ GPa}$
$C_{22} = 109 \text{ GPa}$	$C_{22} = 7.75 + i 0.30 \text{ GPa}$	$C_{22} = 7.52 + i 0.25 \text{ GPa}$
$C_{66} = 26 \text{ GPa}$	$C_{66} = 1.71 + i 0.10 \text{ GPa}$	$C_{66} = 1.53 + i 0.06 \text{ GPa}$
$C_{12} = 55 \text{ GPa}$	$C_{12} = 4.35 + i 0.07 \text{ GPa}$	$C_{12} = 4.25 + i 0.05 \text{ GPa}$
$\Delta C'_{ij} \approx \pm 2\%$	$\Delta C'_{ij} \approx \pm 2\% \quad \pm 3\% \leq \Delta C''_{ij} \leq \pm 10\%$	$C'_{ij} \approx \pm 2\% \quad \pm 5\% \leq \Delta C''_{ij} \leq \pm 16\%$

- Isotropy found for all materials;
- C_{ij} of 80% cured Epoxy are slightly $<$ C_{ij} of fully cured Epoxy.

Manufacturing of adhesively-bonded tri-layer assemblies & characterization process of cohesive properties of adhesive layer



Plane-wave transmitted ultrasonic field is measured
for various incident angles ($0^\circ \leq \theta \leq 35^\circ$)

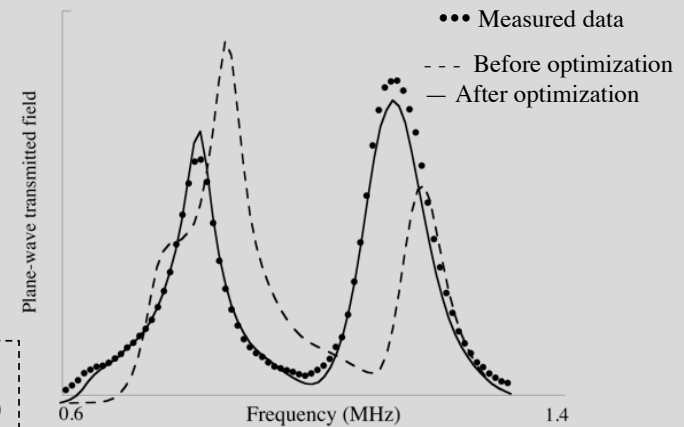
Optimization routine is used to infer C_{ij} of adhesive layer

Aluminium properties are known

h & ρ for adhesive are known

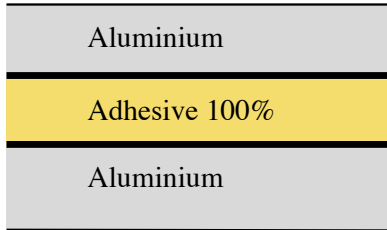
Interphases are supposed to be nominal

Inverse problem
(Simplex method)

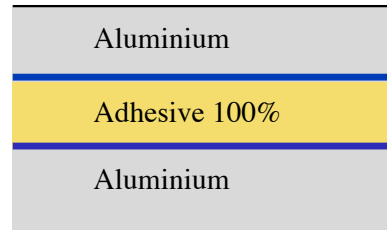


Manufacturing of adhesively-bonded tri-layer assemblies & characterization process of cohesive properties of adhesive layer

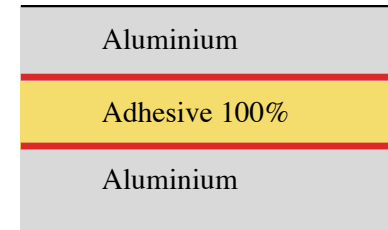
1st set of assemblies with totally cured epoxy (100%)



DSSi100

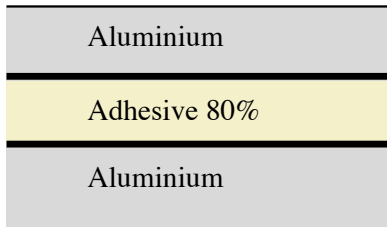


DS100

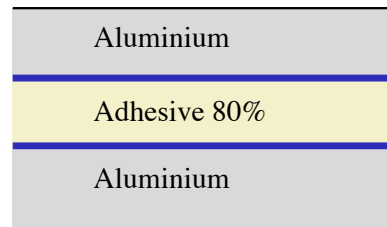


D100

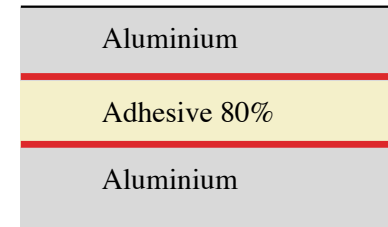
2nd set of assemblies with partially cured epoxy (80%)



DSSi80



DS80



D80

DSBSi = Degreasing + Sand-blasting
+ Silane

DSB = Degreasing + Sand-blasting

D = Degreasing

a priori nominal adhesion

a priori intermediate adhesion

a priori (very) weak adhesion

Evaluation of mechanical *moduli* of the adhesive layer and investigation of the influence of the preparation of substrates' surfaces on this evaluation

Assembly	100% cured Epoxy			80% cured Epoxy		
	DSSi100 Nominal	DS100 Intermediate	D100 Weak	DSSi80 Nominal	DS80 Intermediate	D80 Weak
Thickness (mm)	0.98	0.87	0.96	1.09	0.78	0.75
C_{11} (GPa)	7.9	6.6	5.7	7.4	6.2	5.5
C_{22} (GPa)	7.1	6.8	6.9	7.0	6.8	6.8
C_{66} (GPa)	1.8	1.6	1.4	1.7	1.4	1.3
C_{12} (GPa)	4.6	4.3	4.1	4.4	4.1	3.9
$\beta = \beta_1 + \beta_2$	0	0.23	0.45	0	0.26	0.45

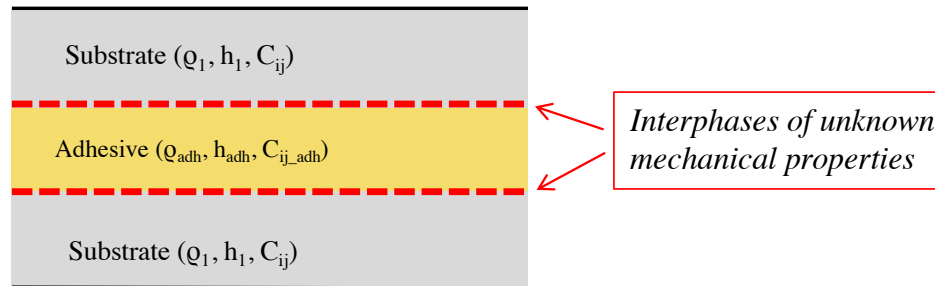
$$\beta_1 = \frac{C_{22}^{app} - C_{11}^{app}}{C_{22}^{app}}$$

$$\beta_2 = \frac{C_{12}^{app} - (C_{11}^{app} - 2C_{66}^{app})}{C_{12}^{app}}$$

- ✓ Changes in apparent C_{ij} of adhesive layers of various samples > measurements errors $\Delta C_{ij} \approx 3\%$;
- ✓ Measured C_{ij} of adhesive layers in DSSi samples are close to those obtained for individual epoxy plates;
- ✓ Measured C_{ij} of adhesive layers show apparent anisotropy (β), which increases as Aluminium surfaces are not well prepared (weak interphases), for both series of samples (100% & 80% cure adhesive). This was confirmed by numerical simulations of experiments & optimization process from simulated signals;
- ✓ Cohesive defect (incomplete curing DSSi80) leads to uniform change in all elastic *moduli* and respect of isotropy if interphases are nominal => Cohesive defect has a different signature than adhesion defect !!

Estimation of mechanical stiffnesses k_L and k_T of interphases

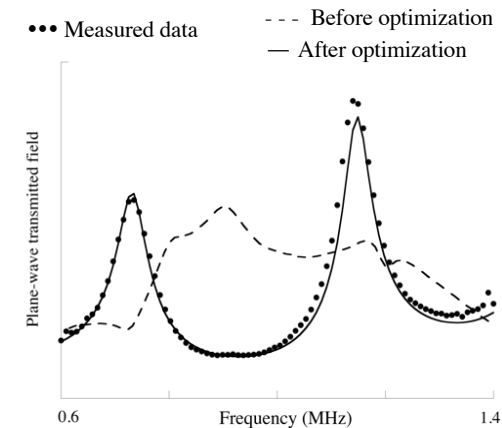
ρ and C_{ij} of substrates and adhesive are known, and given values previously measured on individual plate samples – Thicknesses are also known (or measured separately)



- ✓ Numerical sensitivity study of ultrasonic PWTUF to k_L and k_T changes
 \Rightarrow optimal incident angles: $\theta_{inc} = 0^\circ$ to optimize k_L and $\theta_{inc} = 21^\circ$ to optimize k_T (for this structure)

- ✓ Use of previously measured PWTUF ($\theta_{inc} = 0^\circ$ and $\theta_{inc} = 21^\circ$ only)
 + optimization routine \Rightarrow numerical values of k_L and k_T of interphases to be characterized

Inverse problem (Least square method)



Numerical estimation of interfacial stiffnesses

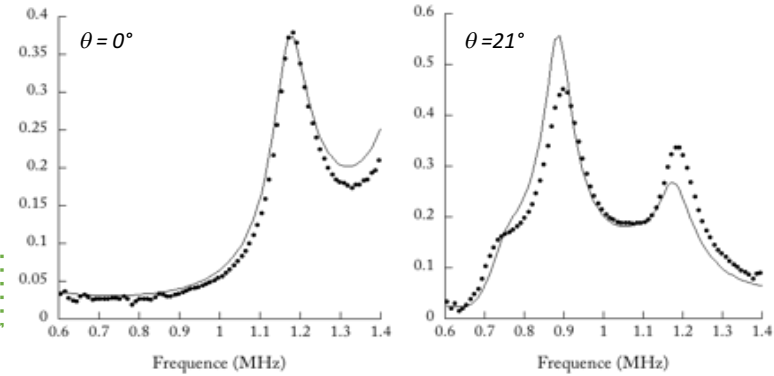
1st set of assemblies with totally cured epoxy (100%)

	<i>DSSi100</i>	<i>DS100</i>	<i>D100</i>
k_L (PPa/m)	2.08	0.25	0.10
k_T (PPa/m)	1.00	0.10	0.02

$$k_{L_D100} \approx k_{L_DSSi100}/21 \quad \& \quad k_{T_D100} \approx k_{T_DSSi100}/50$$

$$1 \text{ PPa/m} = 10^{15} \text{ Pa/m}$$

Measured (•) and simulated with optimized stiffness's (—)
PWTUF for assembly DSSi100

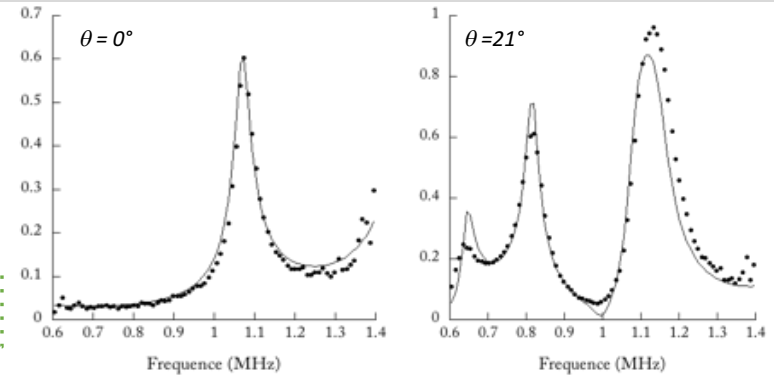


2nd set of assemblies with partially cured epoxy (80%)

	<i>DSSi80</i>	<i>DS80</i>	<i>D80</i>
k_L (PPa/m)	2.09	0.23	0.13
k_T (PPa/m)	1.00	0.10	0.02

$$k_{L_D80} \approx k_{L_DSSi80}/15 \quad \& \quad k_{T_D80} \approx k_{T_DSSi80}/50$$

Measured (•) and simulated with optimized stiffness's (—)
PWTUF for assembly DSSi80

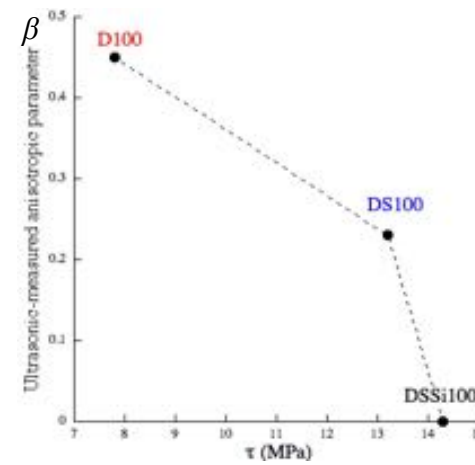
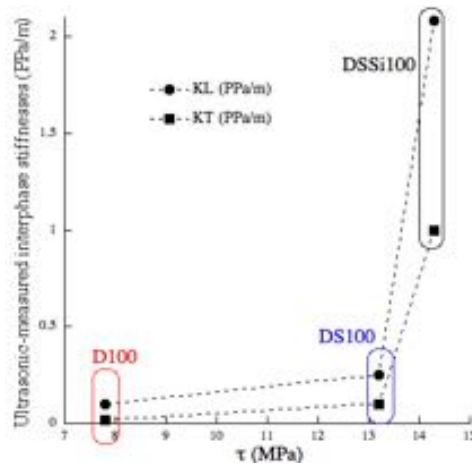
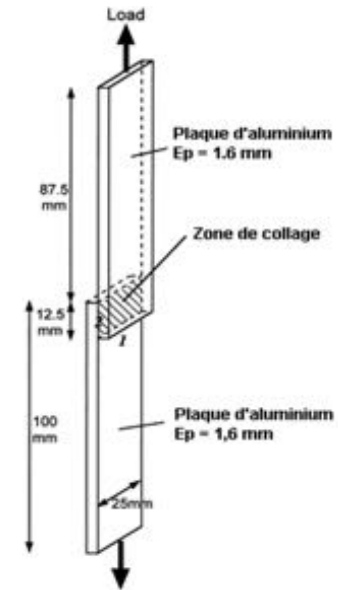


k_T decreases more significantly than k_L as interphases get degraded, independently of adhesive curing state !

Comparison with destructive mechanical tests

- Standard shear lap-joint tests:
- NF-EN 2243-1
- Adhesive thickness $\sim 300 \mu\text{m}$

	<i>DSSi100</i>	<i>DS100</i>	<i>D100</i>
τ (MPa)	14.3 ± 1.1	13.2 ± 1.3	7.8 ± 0.7
k_L (PPa/m)	2.08	0.25	0.10
k_T (PPa/m)	1.00	0.10	0.02
β	0	0.23	0.45



J. Galy, N. Ylla, J. Moysan and A. El Mahi, Conference paper, Thermosets, (2015).

- ⇒ Mechanical strength (τ) decreases as interphases get degraded. Not in the same proportion than the ultrasonically-measured k_L , k_T or β
- ⇒ However, as τ versus k_L , k_T or β are monotonic plots, then the ultrasonically-measured quantities could be good indicators of **level of adhesion at interphases**.

Conclusions and Outlooks

Conclusions

- PWTUF measurements + optimization process allow elastic *moduli* of adhesive layer or stiffness coefficients of interphases between adhesive and adherents to be inferred;
- An **uncured state of the adhesive** layer is revealed by **low values of the estimated elastic *moduli***, but that adhesive layer is still viewed as an **isotropic medium** (if interphases are of good quality);
- A **weak adhesion** is revealed by **low values of the elastic *moduli*** and an **apparent anisotropy** of the adhesive layer;
- If adherents' and adhesive properties are known, then **stiffness coefficients of interphases, k_L & k_T** , can be assessed. Their values are sensitive (but not in the same proportion) to the surface treatment of the substrates and therefore **good indicators of the quality of bonded surfaces**;
- Ultrasonically-measured data have been successfully compared with mechanical destructive tests and monotonic plots link both types of quantities, confirming that **apparent anisotropy** and **stiffness coefficients** are **good indicators of the adhesion level**.

Outlooks

- To investigate the effect of **one imperfect surface** only on PWTUF measurements and on associated evaluation process of **cohesive and adhesion properties**;
- To use ultrasonic **beams** of small sizes (collimated or focused beams) in order to **locally characterize adhesive bonds**;
- To extend this study to adhesively-bonded **composite** panels.

Contrôle Non Destructif

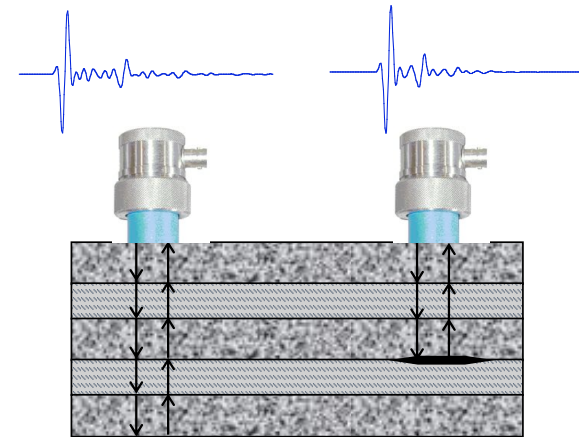
C.N.D.

- *Détection, localisation, quantification, imagerie de discontinuités ;*
- *Contrôle en sortie de chaînes de production (conformité des matériaux, cahiers des charges,...) ;*
- *Suivi de santé des matériaux et structures (endommagement, durabilité,...).*

Ultrasonic NDT of composite structures

Not so easy to control as metals because:

- Ply orientation causes **anisotropy** which makes waves **speed** to depend on **direction of propagation**
- Matrix is often **viscoelastic** and causes **absorption** of ultrasounds
- Medium is heterogeneous and **multi-reflections** at interfaces cause ultrasound **attenuation** or make **signals complex**



Pulse-Echo contact Technique / Manual ultrasonic testing



- Easy to use +
- Slow point-by-point technique
- Coupling-dependent
- Remove and clean-up gel after testing -

Laser Ultrasonics

- **Non-contact technique** based on conversion of electromagnetic energy into elastic energy and *vice-versa* +
- Slower and more **expensive** than conventional systems on flat components -
- **Less sensitive to probe alignment than conventional US techniques** → **significant advantage for curved components** +
- Low signal levels ⇒ **retro-reflective coating** sometimes **used** to cover the structure ... not convenient for large areas -
- **Need to scan tested structures** ($\approx 10\text{m}^2/\text{h}$ with $12.5 \times 12.5\text{mm}$ pixel size, using **rotating mirrors** to deflect laser beams) -
- Used by US aerospace manufacturers for **inspection of complex composite components used in modern aircrafts**

UltraOptec (Canada)

Laser Ultrasonic Inspection System (LUIS)



Many research centres developing Laser -based ultrasounds:

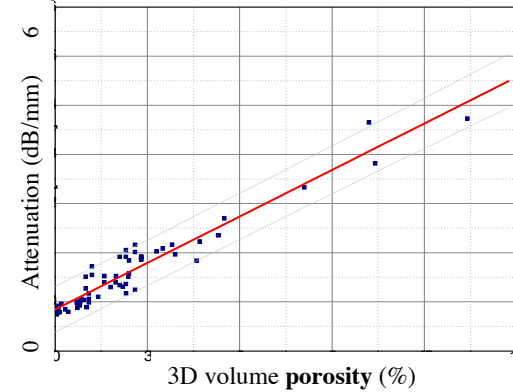
- Bremen Institute of Applied Beam Technology (Germany)
- Industrial Materials Institute (Canada)
- Università Politecnica delle Marche (Italia)
- Institute of Acoustics of Tongji Univ., (Shanghai, China)
- I₂M University Bordeaux (France)
- etc...

Through-transmission ultrasonic technique

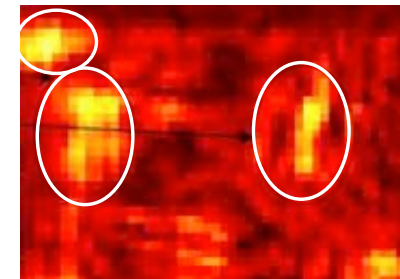
Water jet



Ultrasonic Attenuation coefficient
Sensor 1MHz, 4mm jet of water, squirted

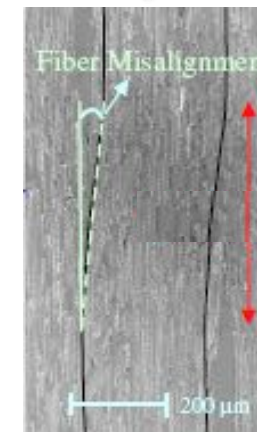


Detection of delaminations



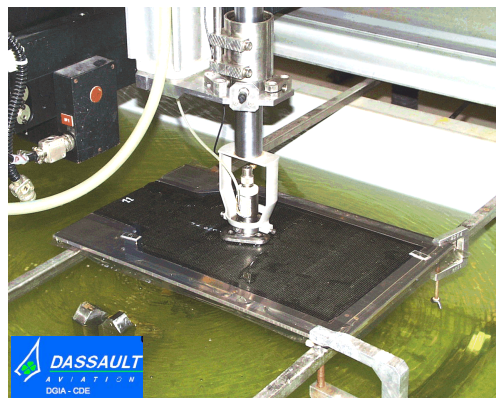
<http://www.wavelength-ndt.com/composites.htm>

Detection of fibres misalignment

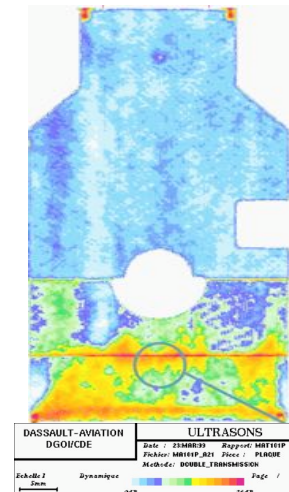


J. Cho, Scripta Materialia 56, 2006

Pulse-echo ultrasonic technique / Immersion tank



US cartography of porous zone of RTM



- Large choice of frequencies
depending on size of searched defect
- Point-by-point scanning techniques
- Need to dismantle tested pieces

+

-

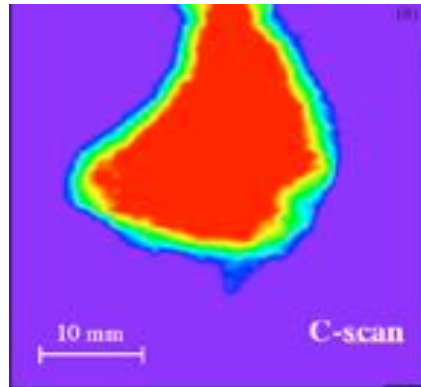
Imaging impact damages for composite materials

Ex.: Carbon Kevlar composite plates - 3 mm

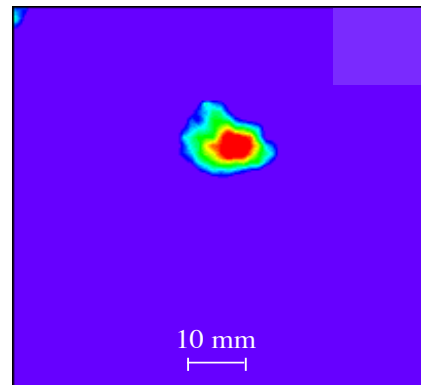
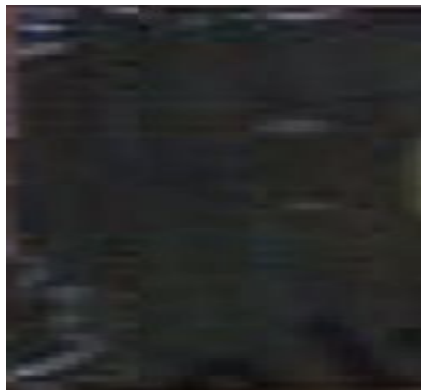
Photography

Ultrasound scan - 250 kHz

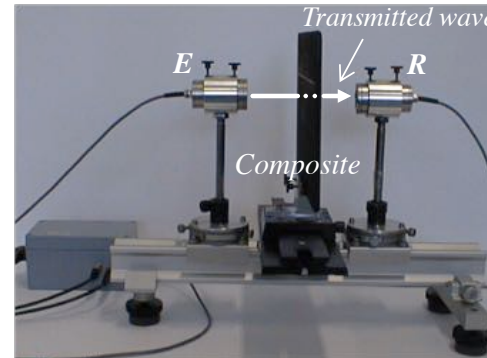
High impact



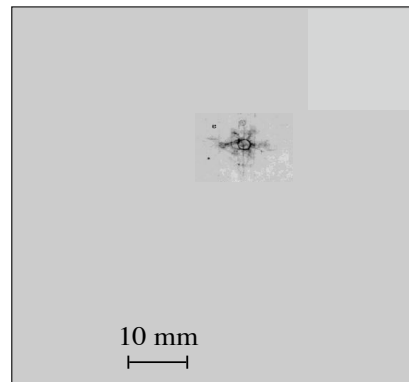
Low impact



Air-coupled transducers



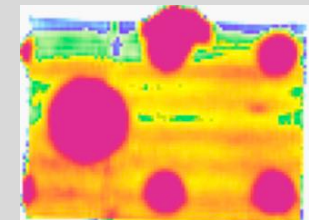
RX measurements



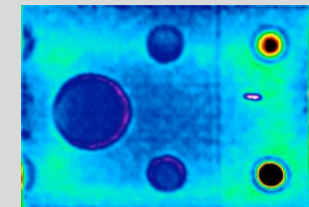
Contact-less ultrasonic scan of sandwich composite component



US C-scan

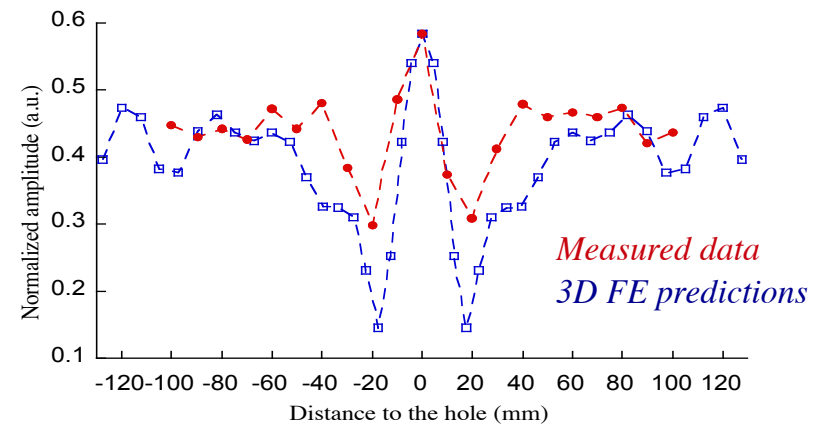
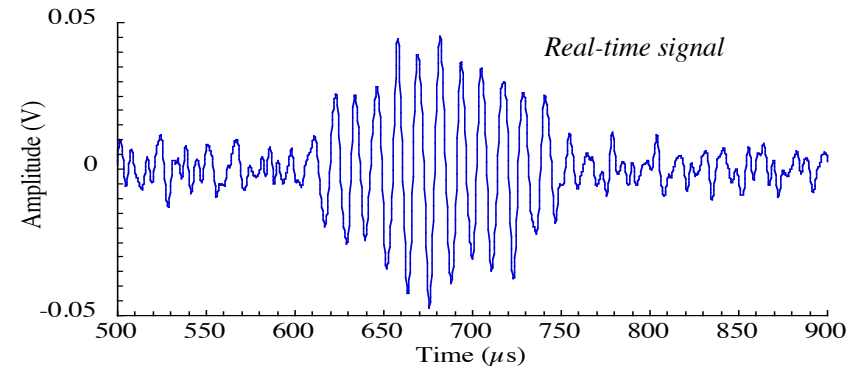
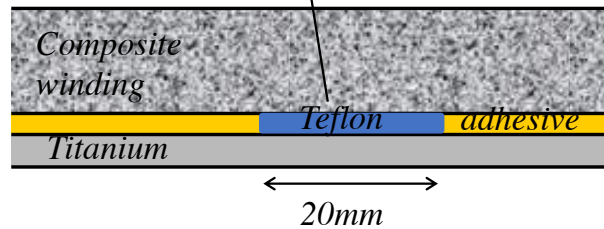
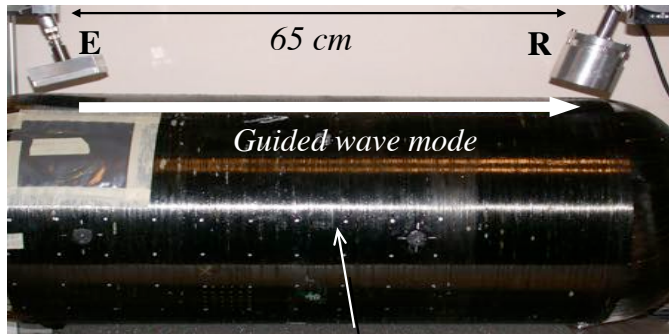


Thermography

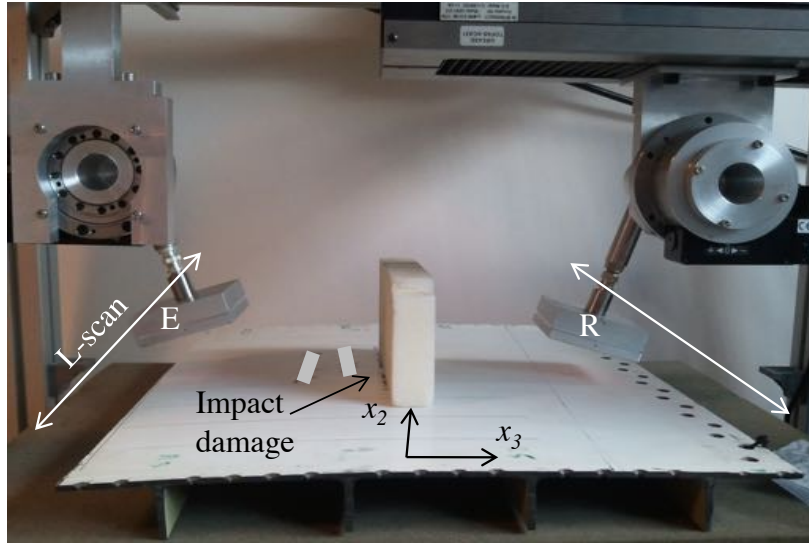


Air-coupled ultrasonic transducers for contact-less and single-sided detection and localisation of defects in complex composite structures

e.g. High pressure composite tank (ARIANE) = Titanium liner + adhesive layer + Carbon Epoxy winding

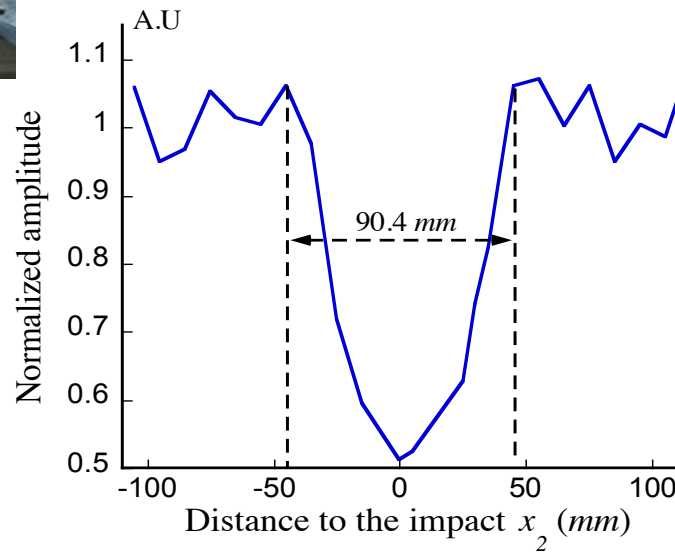


Inspection of aeronautic composite structures using guided waves generated and detected by single-sided access, ultrasonic, air-coupled transducers

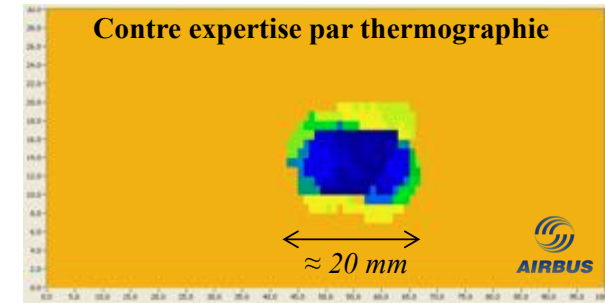
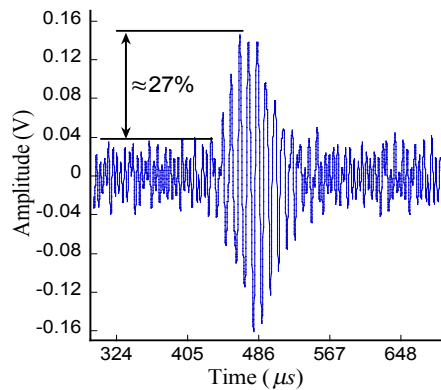


Air-coupled ultrasonic transducers
 Frequency = 100 kHz
 Angles = $\pm 15^\circ$
 Guided mode = A_0 along x_3
 Path of propagation = 300 mm over 3 stiffeners
 Scanning along x_2

U-shaped measured distribution of A_0 amplitude reveals presence of impact damage.



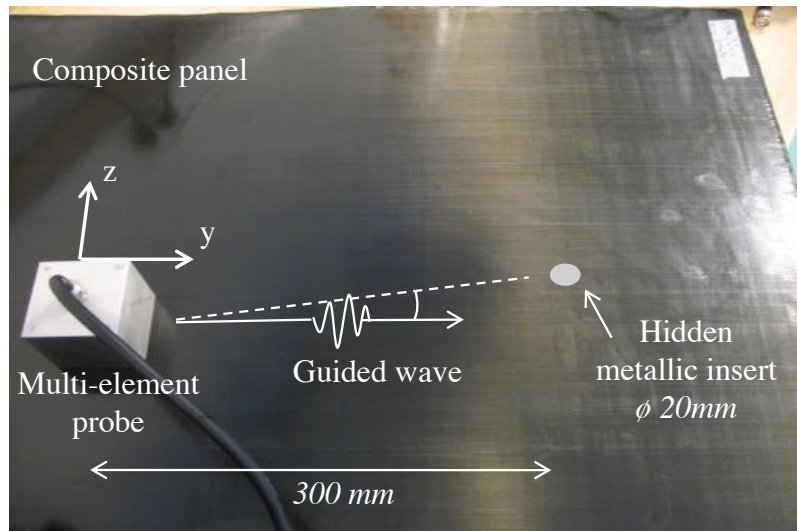
Real-time A_0 waveform generated and detected by air-coupled transducers



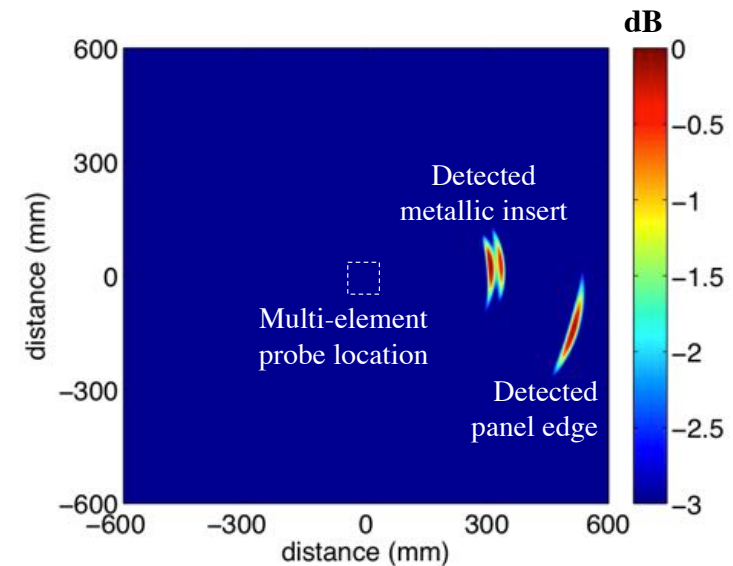
$$\begin{aligned}
 W_{\text{signal_drop}} &\approx 90 \text{ mm} \\
 W_{\text{transducers}} &= 70 \text{ mm} \\
 \Rightarrow W_{\text{damage}} &= W_{\text{signal_drop}} - W_{\text{transducers}} \\
 &\approx 20 \text{ mm}
 \end{aligned}$$

Inspection of large composite structures using guided waves generated and detected by remote ultrasonic multi-element probe

Ultrasonic multi-element probe is gel-coupled at a fixed position, and electronically driven by multi-channel device to inspect remote (eventually non accessible) zones of large composite panel.



Photography of experimental set-up



Ultrasonic image of panel.



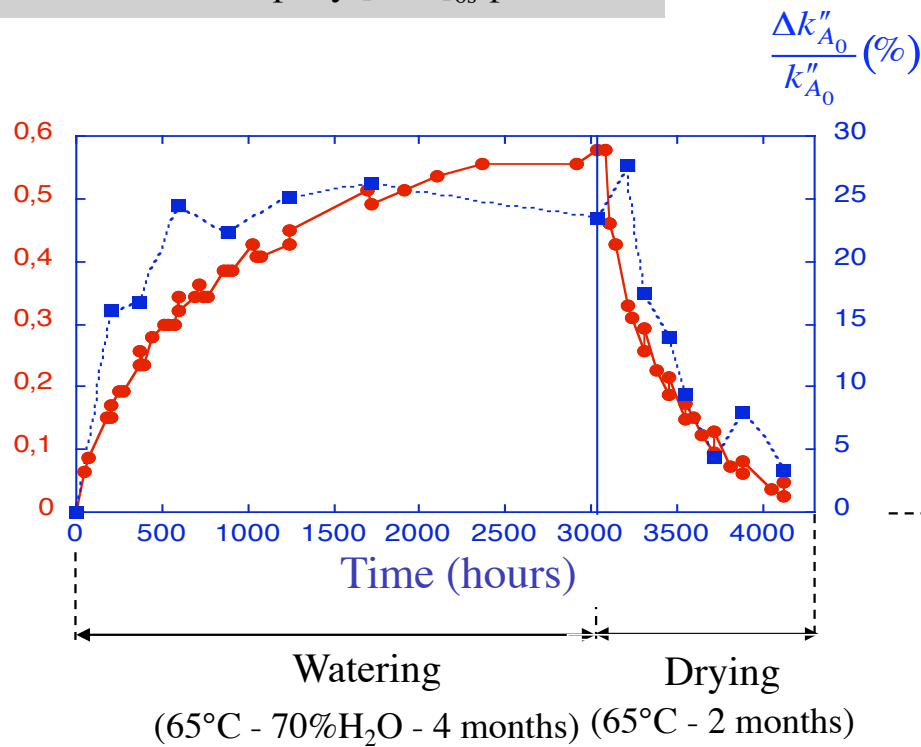
Merci pour votre attention.



Autres techniques US d'E.N.D.

Air-coupled guided waves - Composite materials ageing

Ex.: Health monitoring of moisture content in 5 mm Carbone Epoxy [0/90]_{6s} plates



Inverse problem

↓

≈ 30% change in imaginary part of Coulomb modulus



=



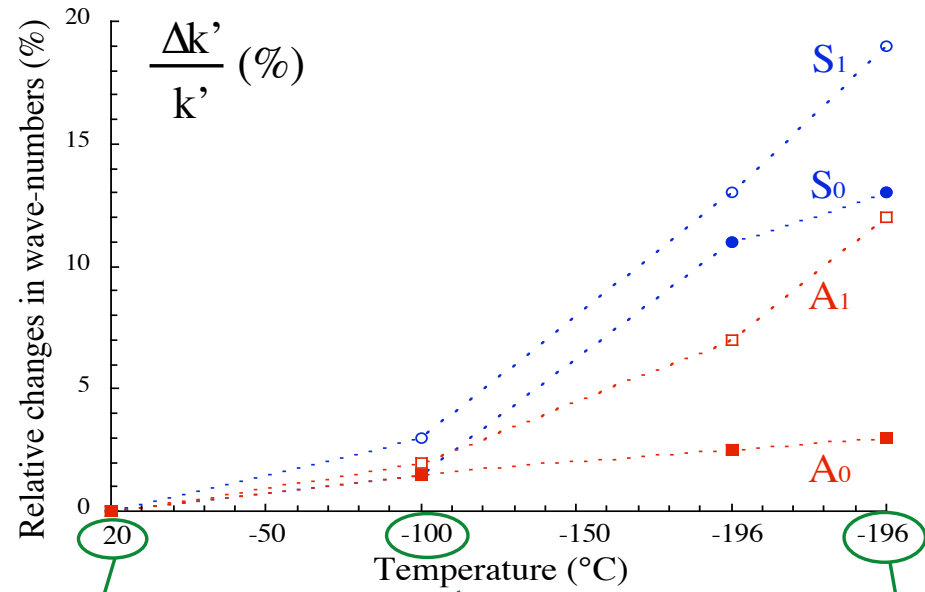
+



+



Health monitoring of micro-cracking in 16.5 mm thick $[0/+60/-60]_{11s}$ Carbone Epoxy laminate



Micrographies

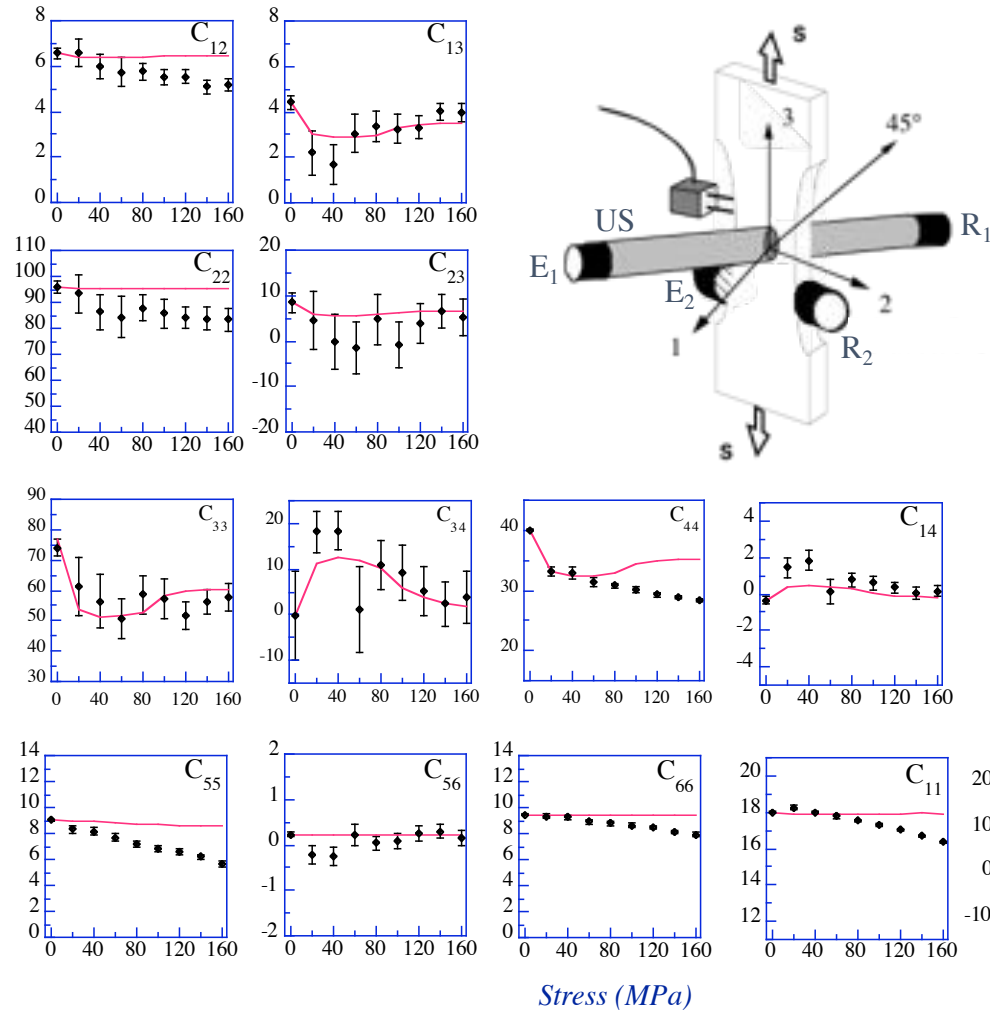


Inverse problem \approx 10 to 20% decrease of C'_{ij}

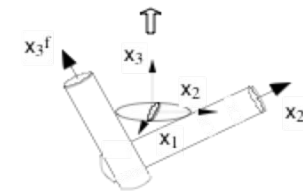
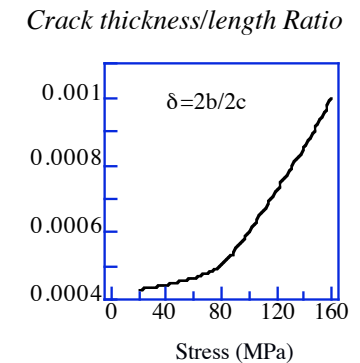
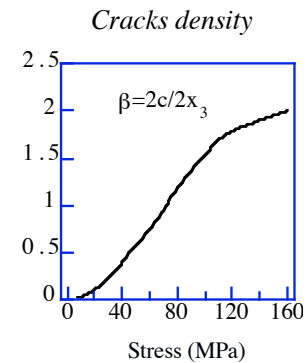
Time of flight immersion technique

Characterization of material anisotropy induced by cracks in composites under load

••• : Experimental evaluation of complete material stiffness (GPa) versus loading (Mpa)

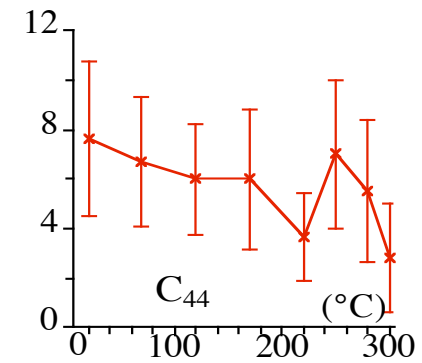
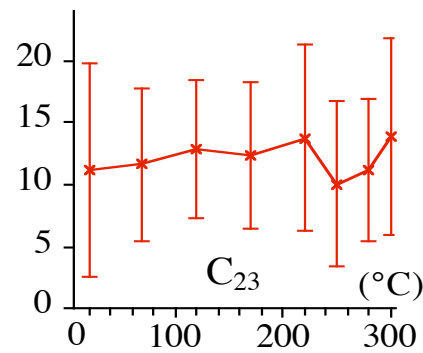
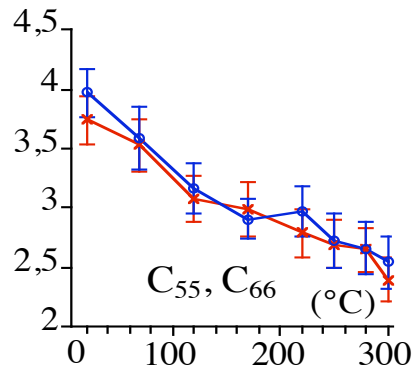
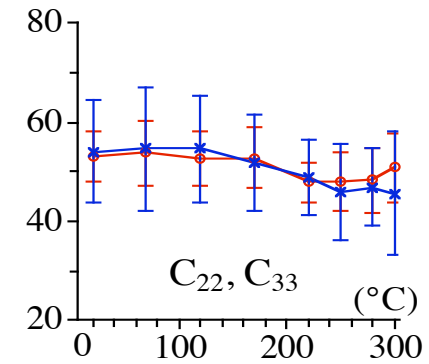
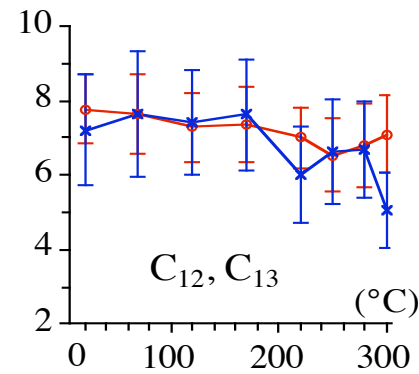
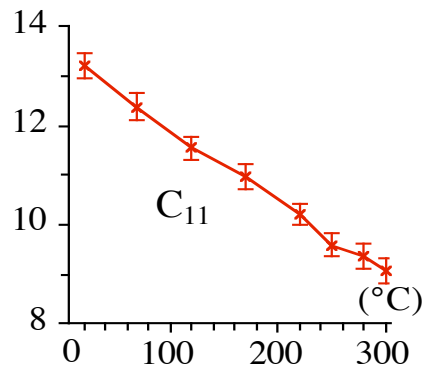
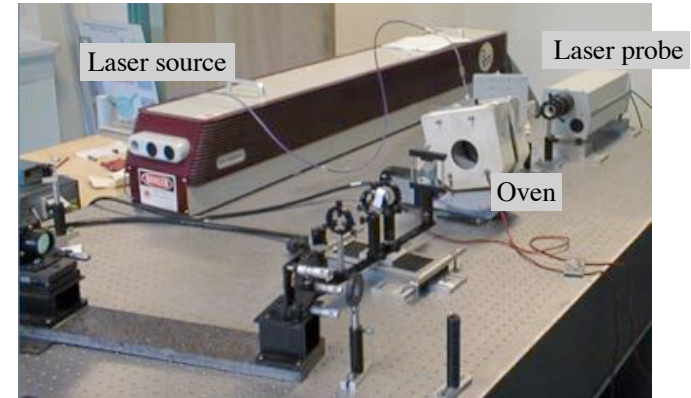


Constitutive law built-in for anisotropic materials with tilted cracks and used for predicting changes in material stiffness versus loading: —

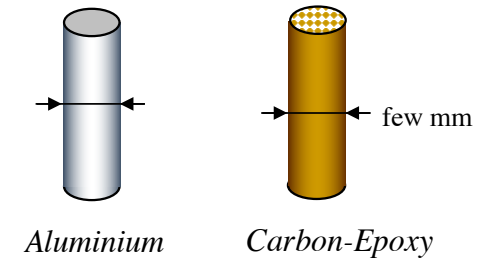
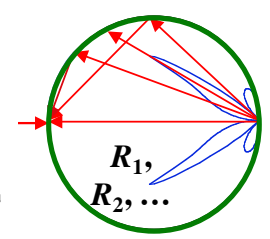
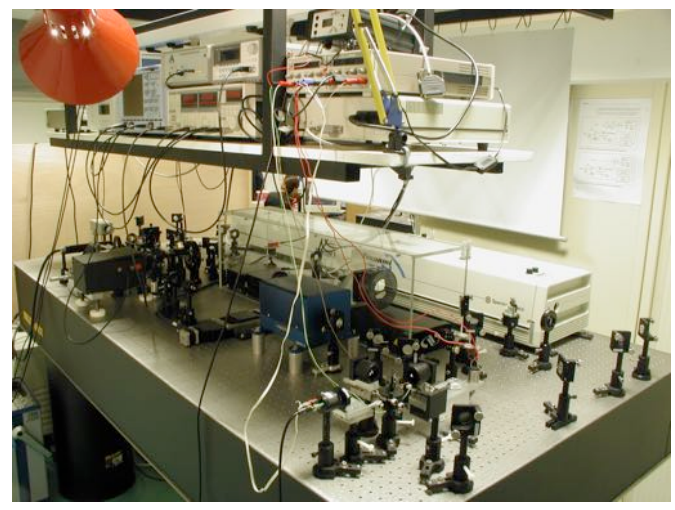
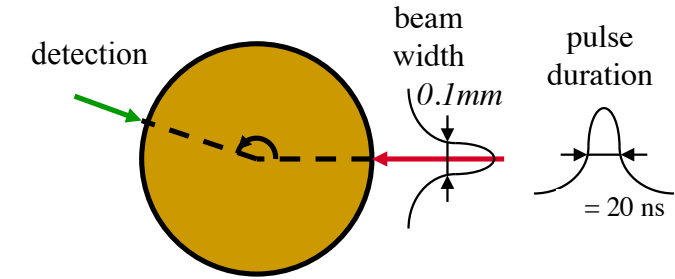
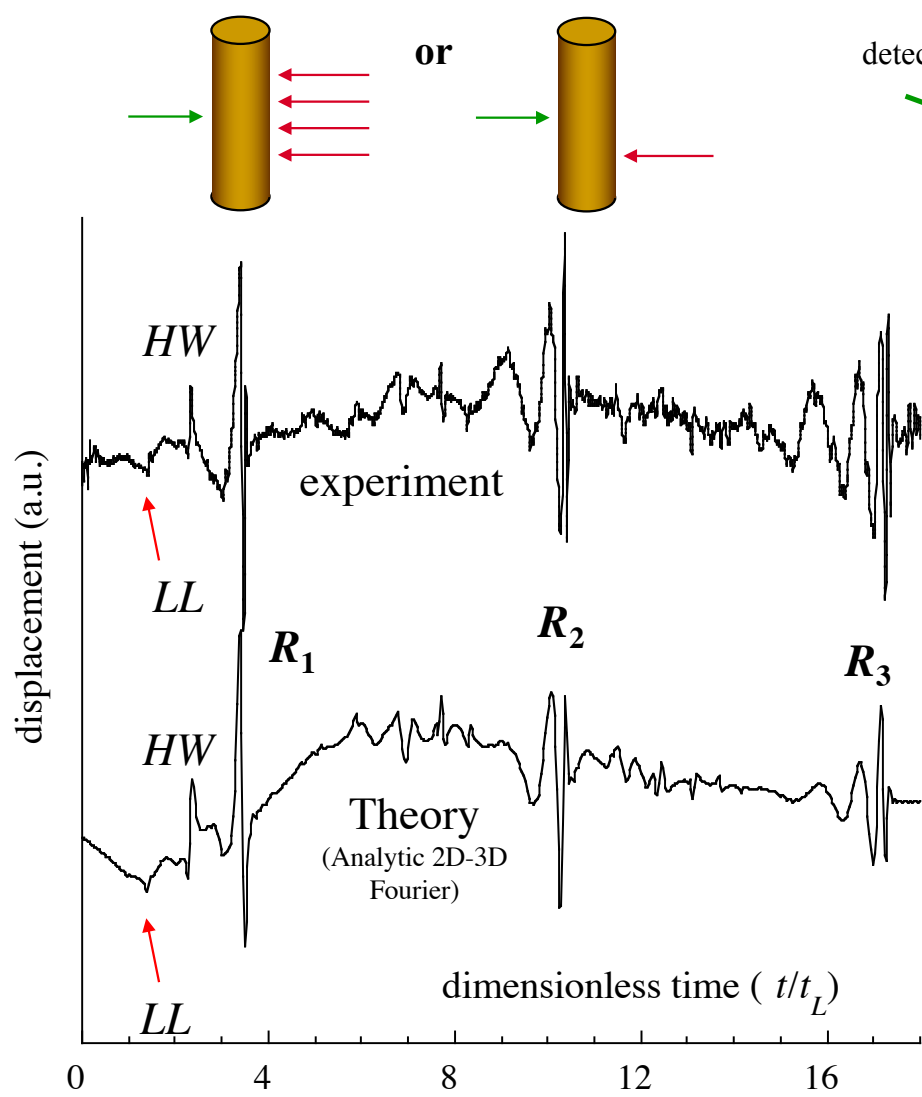


Laser-US for characterization of the anisotropic degradation produced by temperature

Ex. : Measured elastic moduli (GPa) of 2D woven composite during temperature increase / DGA-SNECMA 1998

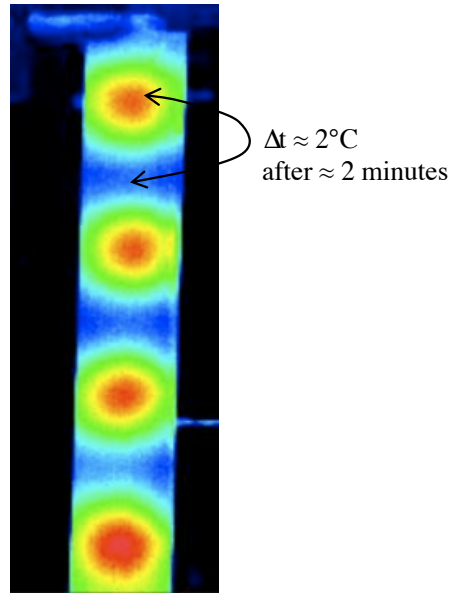
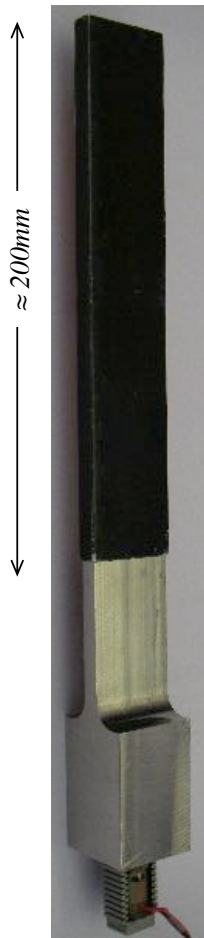


Laser-US for wave propagation in small isotropic or anisotropic cylinders

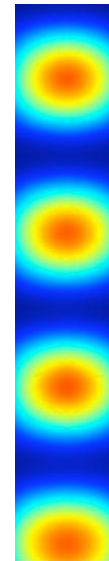


Thermosonics ... Viscoelastic propagation of standing waves with energy dissipation causing heating
 ... Measurements + **Linear FE-based model**

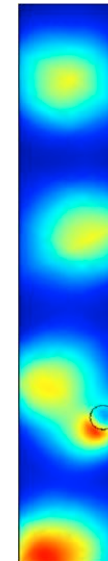
Ex.: Preliminary results on 5mm Perspex plate



Measurements



*FE modelling
No defect*



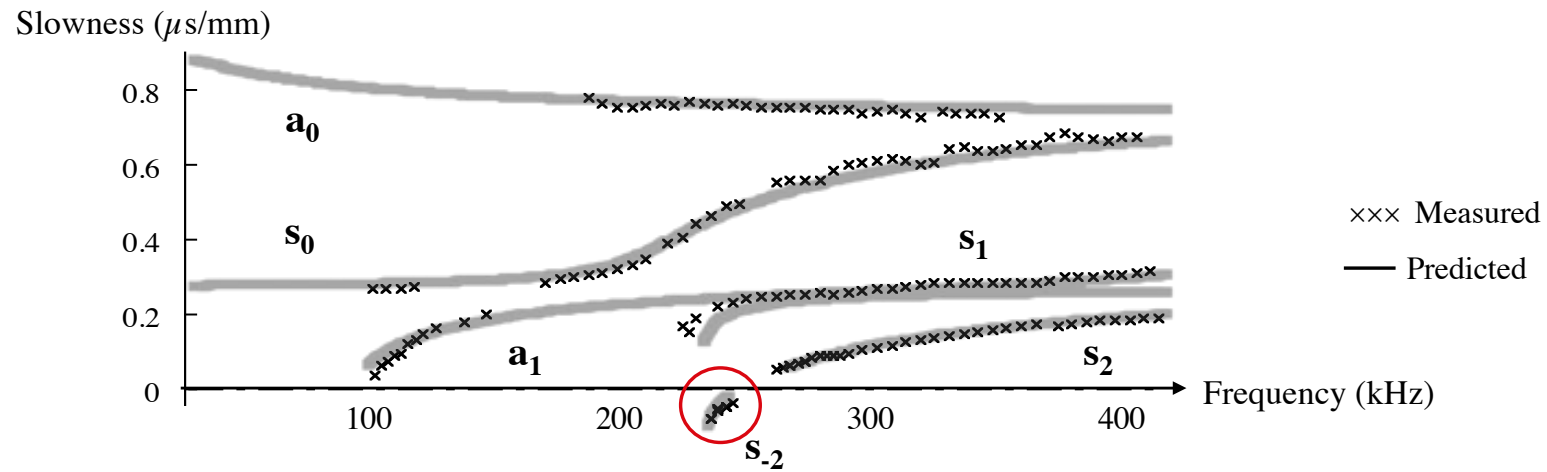
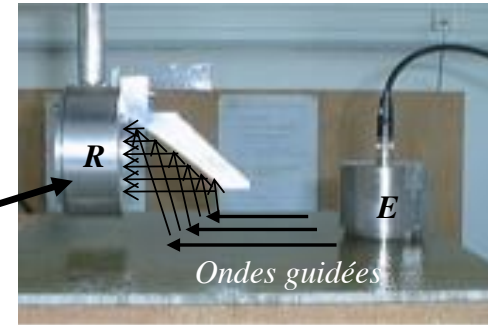
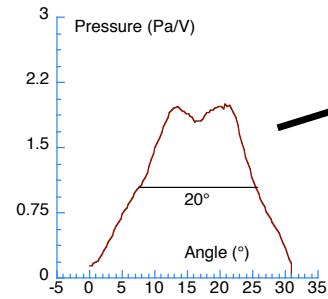
*FE modelling
Defect - Linear effects*

PROJECT:

- To perform measurements on composite materials
- To **characterize thermal properties, viscoelasticity** and/or **defects** using FE linear model
- To develop non-linear model in order to further **characterize defects** producing non-linear phenomena

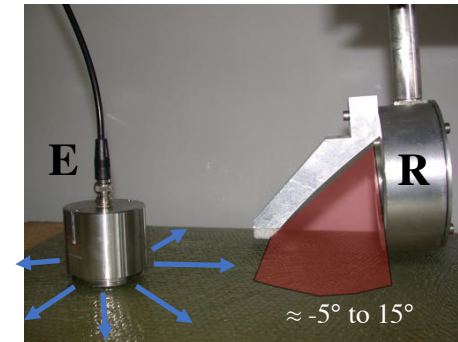
Air-coupled guided waves - Material characterization

Ex. : Crossed-ply Carbone Epoxy - 5 mm

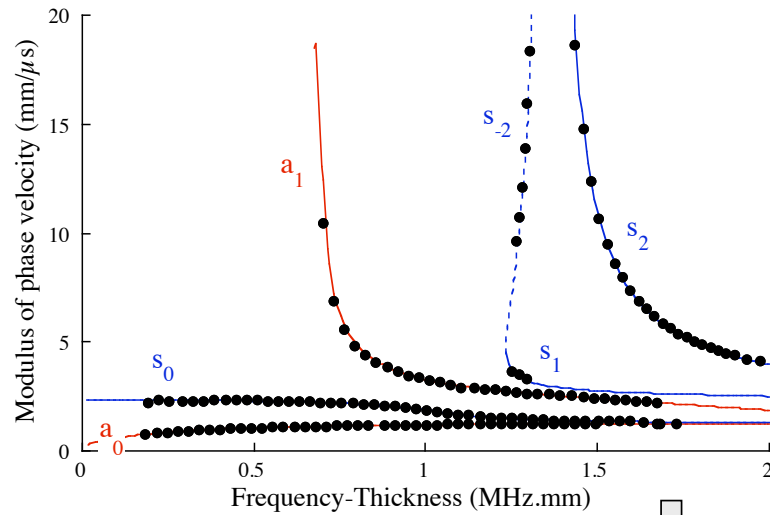


C_{11}	C_{12}	C_{22}	C_{66}	C_{13}	C_{33}	C_{55}	GPa
14 ± 0.3	8.2 ± 0.2	32 ± 3	3.5 ± 0.1	8.1 ± 0.2	30 ± 3	3.4 ± 0.1	

Examples of measured negative phase velocities



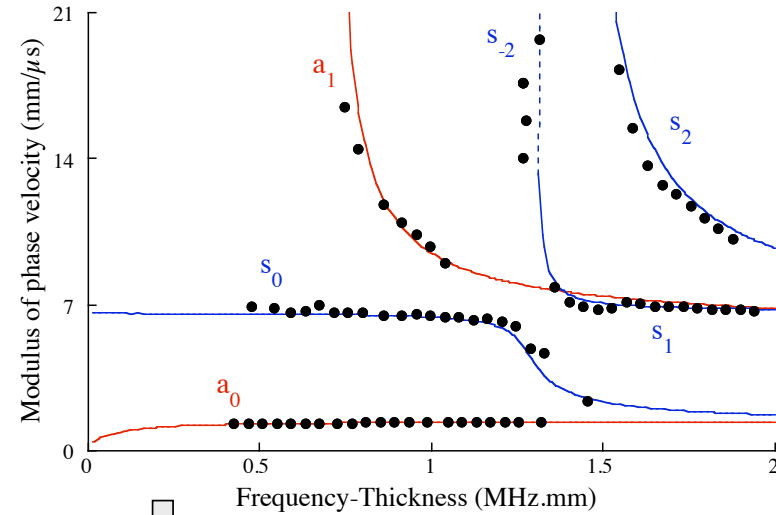
Ex. : 3.9 mm Plexiglas plate



→ Optimised C_{ij} in plane
of propagation (in GPa)

$$\begin{aligned} C_{11} &= 8.6 \pm 0.3 \\ C_{22} &= 8.7 \pm 0.3 \\ C_{12} &= 4.2 \pm 0.2 \\ C_{66} &= 2.2 \pm 0.1 \end{aligned}$$

Ex. : 4.2 mm $[0^\circ/45^\circ/90^\circ/-45^\circ]_{6s}$ Carbon-Epoxy plate



$$\begin{aligned} C_{11} &= 11.8 \pm 0.7 \\ C_{22} &= 75 \pm 4 \\ C_{12} &= 7.3 \pm 0.4 \\ C_{66} &= 3.1 \pm 0.2 \end{aligned}$$