Resonant Ultrasound Spectroscopy (RUS) for measurement of Stiffness Tensor of Anisotropic and Attenuative Materials

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Consultative Committee for Acoustics, Ultrasound, and Vibration
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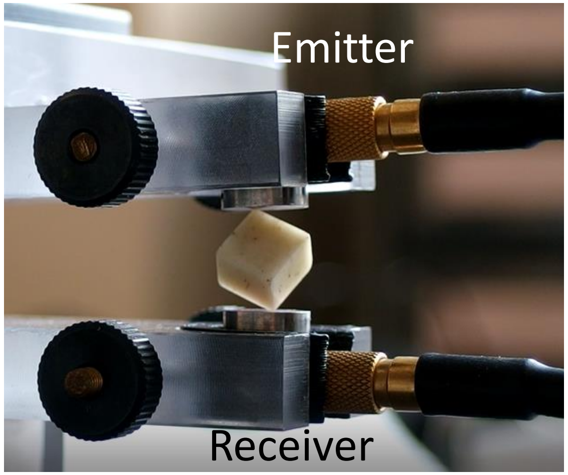
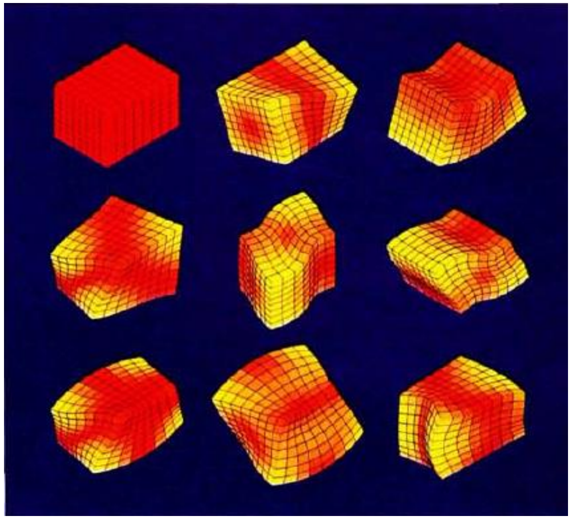
What is Resonant Ultrasound Spectroscopy (RUS) ?

The use of *piezoelectric transducers* to excite ultrasonic *natural frequencies* of a sample, and determine the complete *stiffness tensor* in one measurement



Boundary conditions of sample are supposed to be « free-free »



« Resonant Ultrasound Spectroscopy »,
J. Maynard, Physics Today, January 1996, p. 26-31

Mean range for first ten modes :

10 mm cube of PMMA => 40/60 kHz
3 mm cube of steel => 450/700 kHz

Why using RUS ?

> *"The ultrasonic and elastic properties of materials are conventionally measured using [..] a* **pulse-echo** *technique with the transducer driven at resonance. Problems with the technique include* **transducer ringing, transducer-sample coupling**, *parallelism of sample faces,* **beam diffraction, and the necessity of remounting transducers in order to measure all of the elastic constants**. *[...]* **with samples that are only a fraction of a millimeter in size, conventional ultrasound measurements become difficult if not impossible**. *[...]* **nearly all of these problems may be avoided if a resonance technique is used, and all of the elastic constants may be determined with a single measurement."**

JD Maynard - The Journal of the Acoustical Society of America, 1992

History

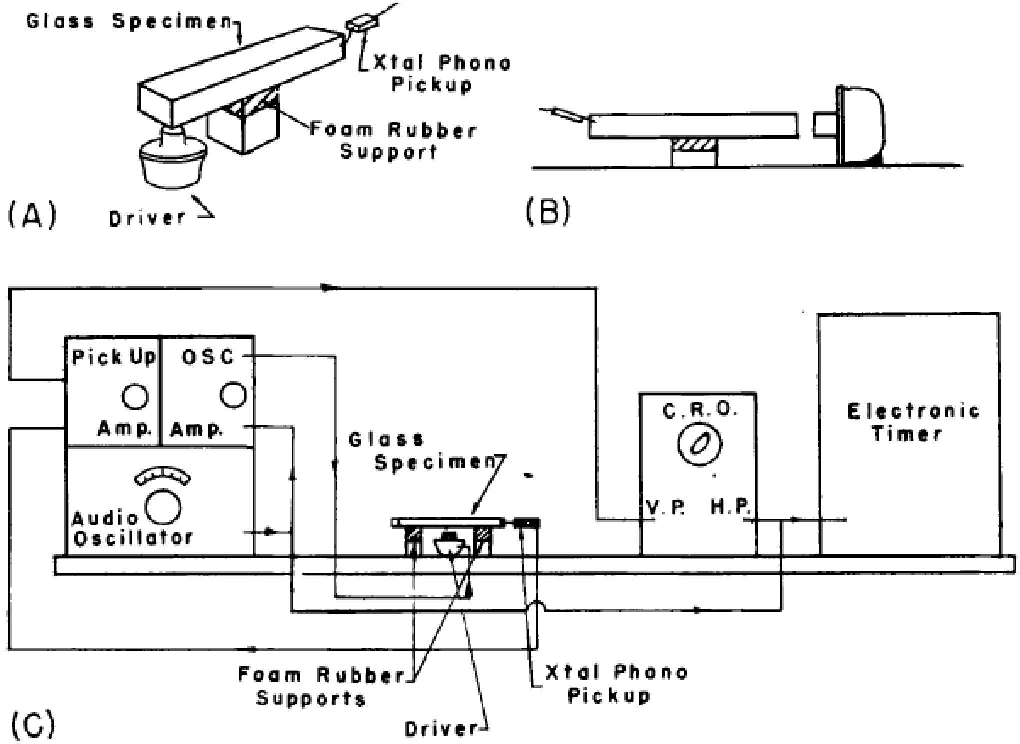


Fig. 1. Diagrammatic sketch of apparatus used for dynamic measurement. Position of driver and specimen (*A*) for torsional vibrations, (*B*) for longitudinal vibrations, and (C) for flectural vibrations.

"Elastic Moduli of Glasses by a Dynamic Method"
S. SPINNER, NBS, May ***1954***

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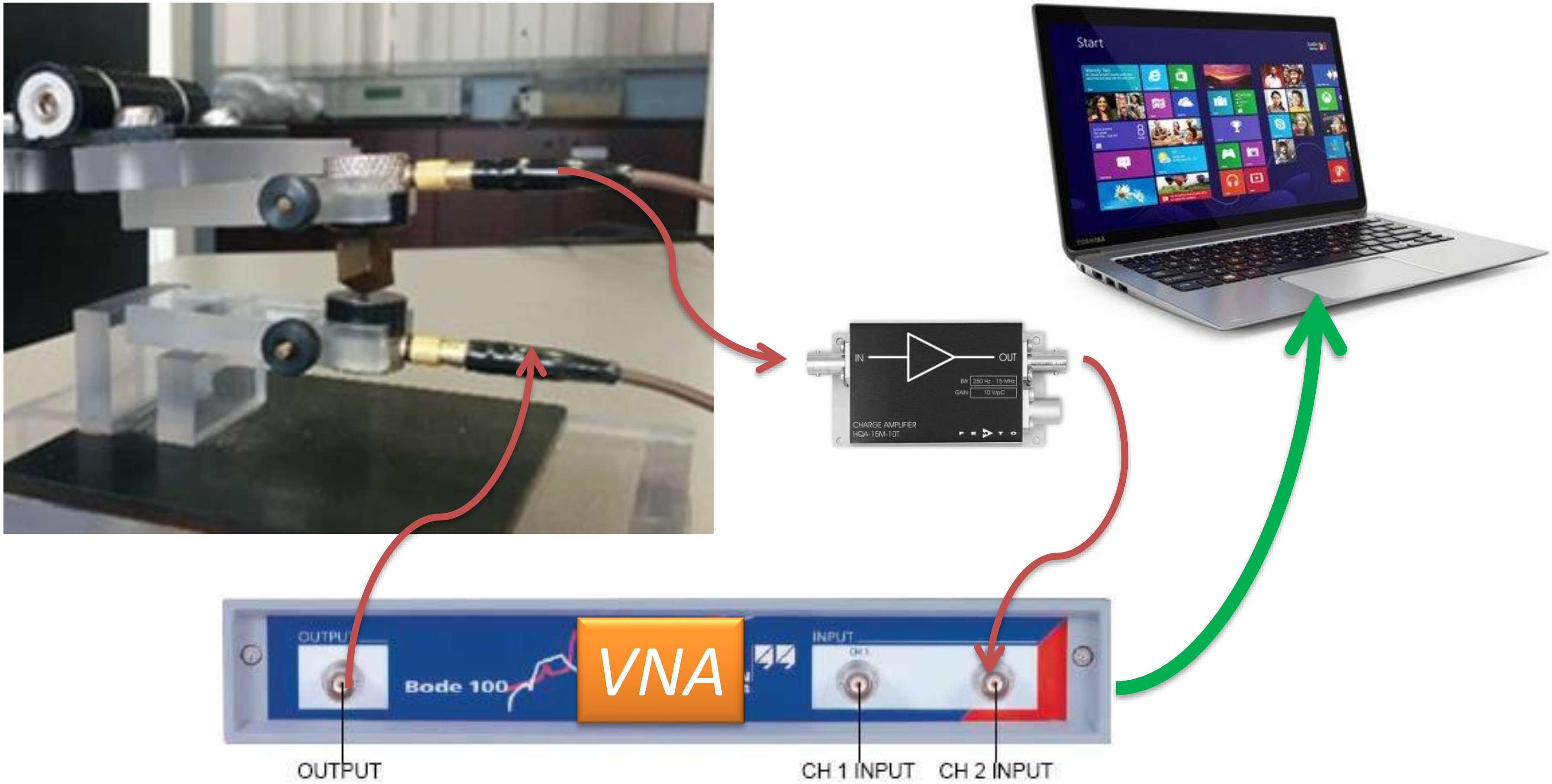
LIB set up

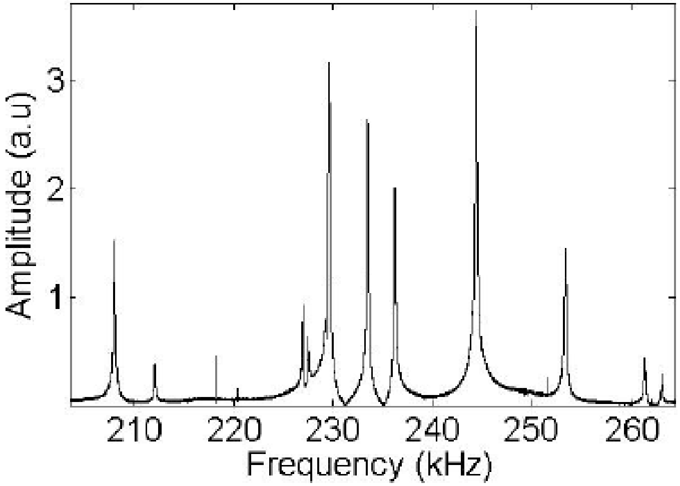
(2017)

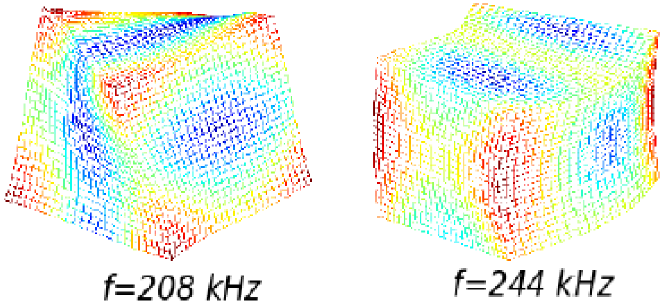


Overview of RUS to determine the stiffness tensor

1. Measurement of free vibration spectrum
   → resonance frequencies f_k^{exp}
2. Forward model
   → $f_k^{mod}(C_{ij})$ (semi-analytical Rayleigh-Ritz method)
3. Optimization problem
   → stiffness tensor terms C_{ij}
   (Gauss-Newton and gradient methods):

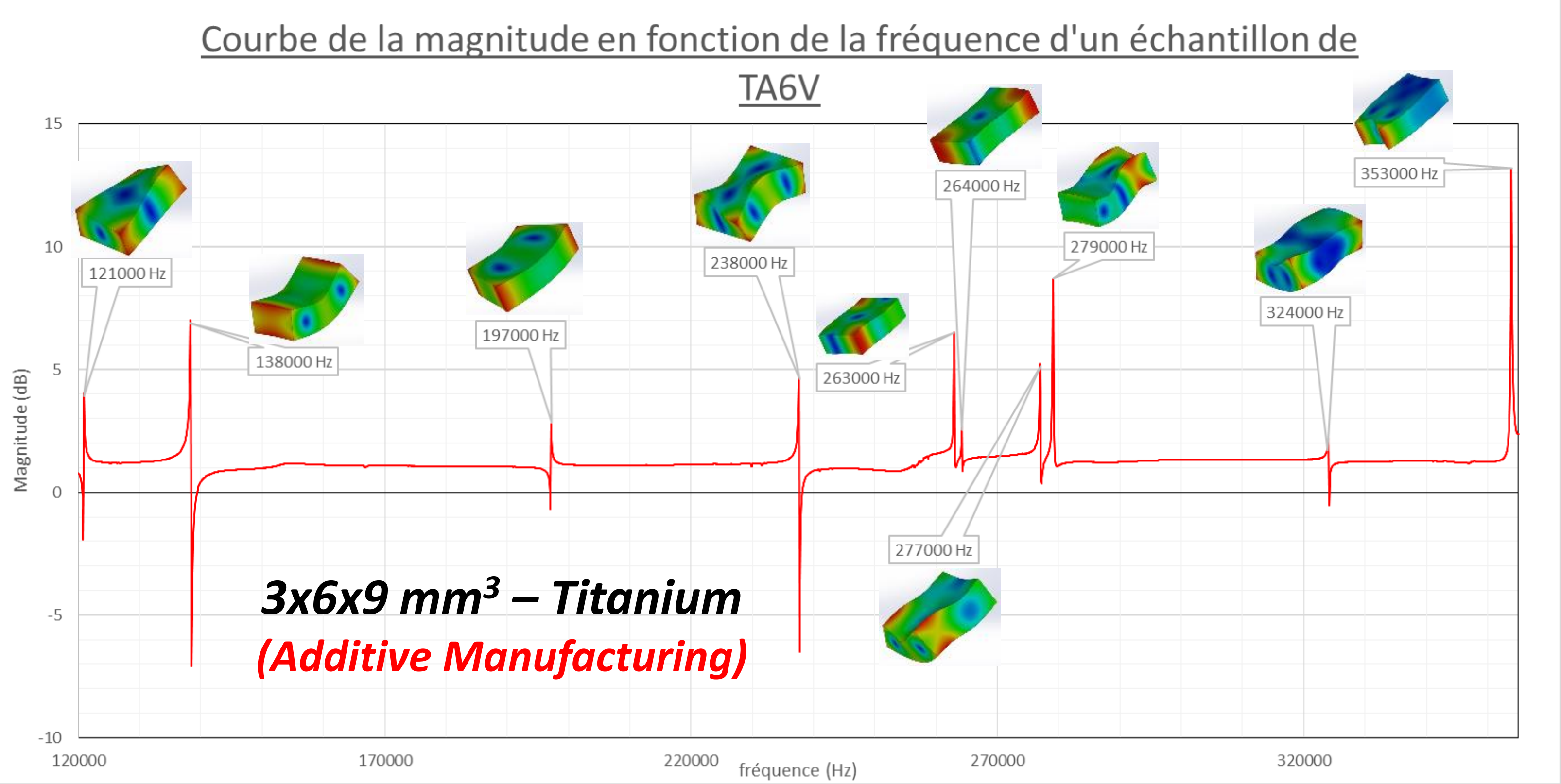
$$Obj = \sum_k \left(\frac{f_k^{exp} - f_k^{mod}(C_{ij})}{f_k^{exp}} \right)^2$$





spectrum for a copper sample and modal shapes

Mesoscopic Scale



3x6x9 mm^3 – Titanium

(Additive Manufacturing)

We measure « bulk » elasticity at the scale of the sample

(=> mesoscopic voxel of material)

The case of Bone

Bone ~ two-phase composite

{ solid anisotropic matrix (mineral/collagen) } + { soft material & fluids in pores }

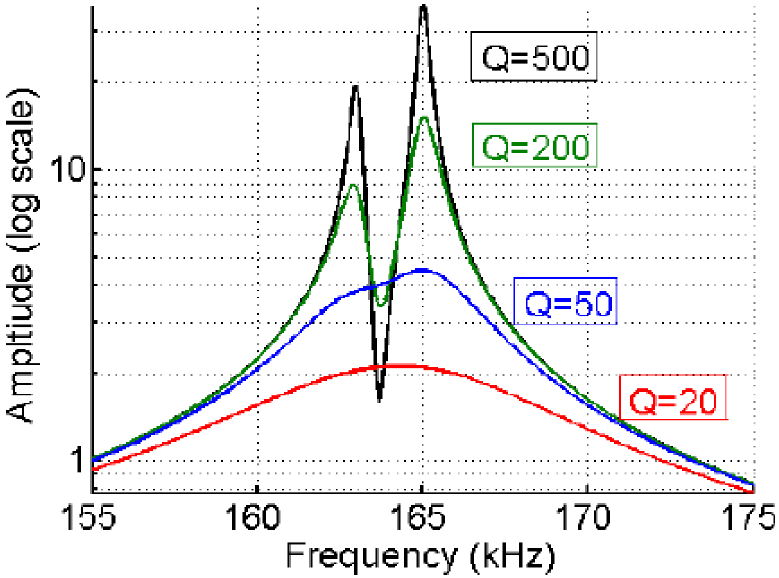
- Anisotopy (transverse isotropy or orthotropy)

$$\text{Stiffness tensor: } C = \begin{pmatrix} c_{11} & c_{12} & c_{13} & 0 & 0 & 0 \\ c_{12} & c_{22} & c_{23} & 0 & 0 & 0 \\ c_{13} & c_{23} & c_{33} & 0 & 0 & 0 \\ 0 & 0 & \cdot & c_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & c_{55} & 0 \\ 0 & 0 & 0 & 0 & 0 & c_{66} \end{pmatrix}$$

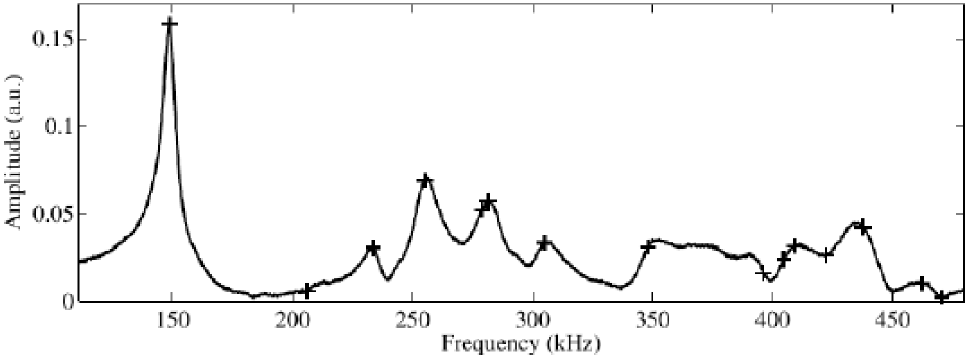
- viscoelastic : Q-factor ~ 30 (~ polycarbonate)

RUS applied to viscoelastic materials

- Unlike metals, bone is viscoelastic (low quality factor *Q*)!
- In a typical spectrum (bone), the peaks strongly overlap



Peak overlapping for low *Q*



RUS applied to viscoelastic materials

- Resonance frequencies cannot simply be picked in the measured spectrum

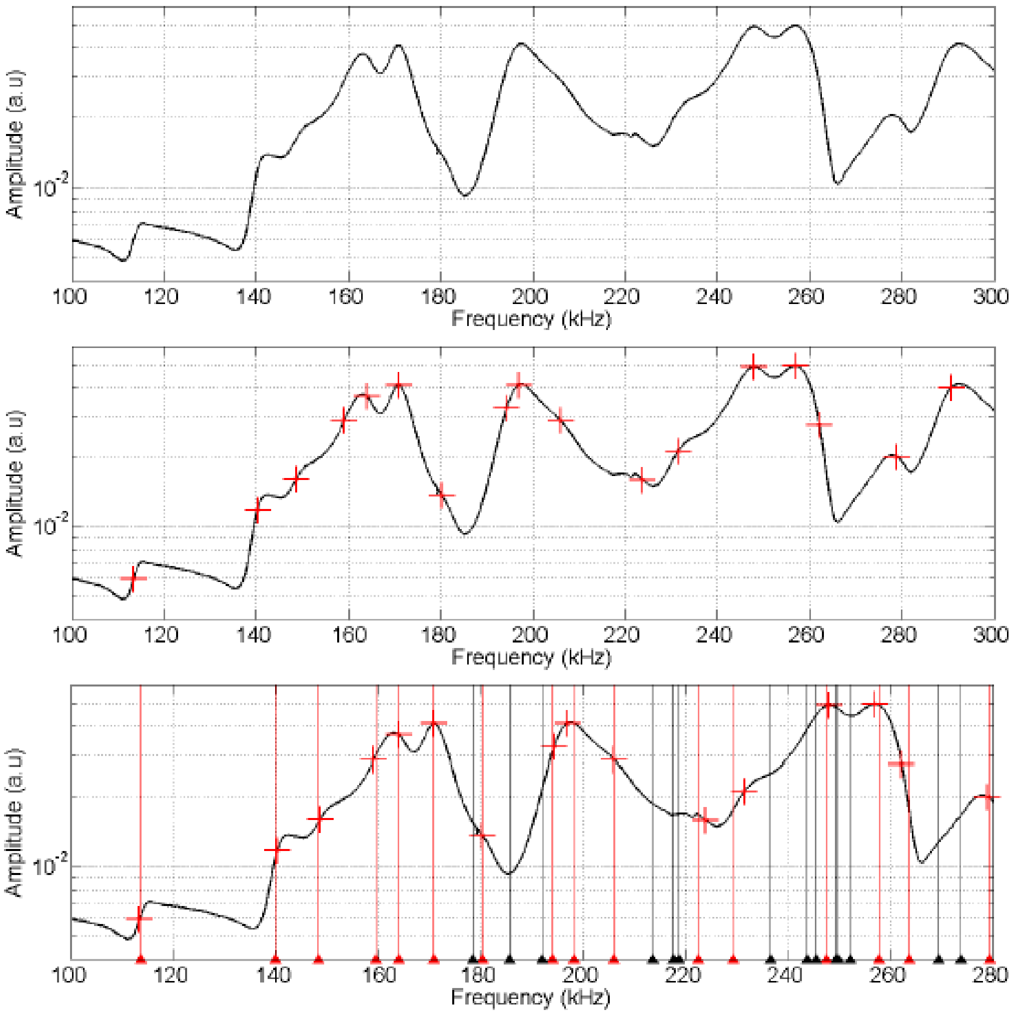
  The signal must be modeled as a sum of Lorentzian lineshapes

$$y(f)=\sum_{k=1}^{K} L(f;A_k,f_{0k},\phi_k,Q_k)$$

$$+\text{drift}+\text{noise}.$$

  K : unknown number of frequencies

- Problem solved with linear prediction filter (find K) & non-linear optimization [Lebedev 2002] [Bernard et al. J Acous Soc 2014] or Bayesian analysis [K Xu 2017 submitted]



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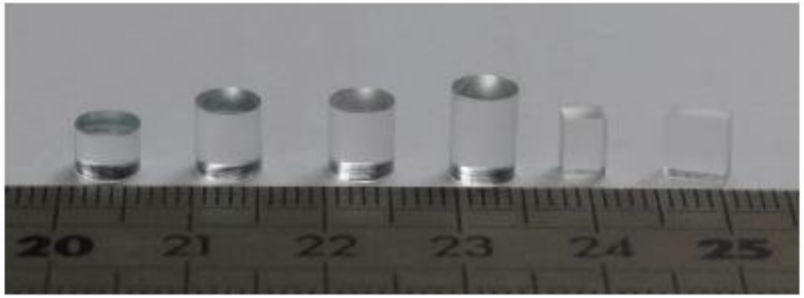
Precision - Accuracy

Two-types of errors need to be addressed

- Precision – reproducibility
  - **Random errors on measured frequencies**
  - **Random errors on** mass and **dimensions** measurements
- Accuracy (bias wrt a reference value)
  - Effect of measurement frequency
  - **Sample shape imperfections**
  - Orientation of sample's cut wrt material axes

Validation of RUS on Polymethyl Metacrylate (PMMA)

- 4 cylindrical samples ($\phi = 5.15$ mm, height: 3.7 to 7.2 mm)
- 2 rectangular parallelepiped samples ($2.5 \times 3.5 \times 5$ mm and $2.5 \times 5 \times 5$ mm)

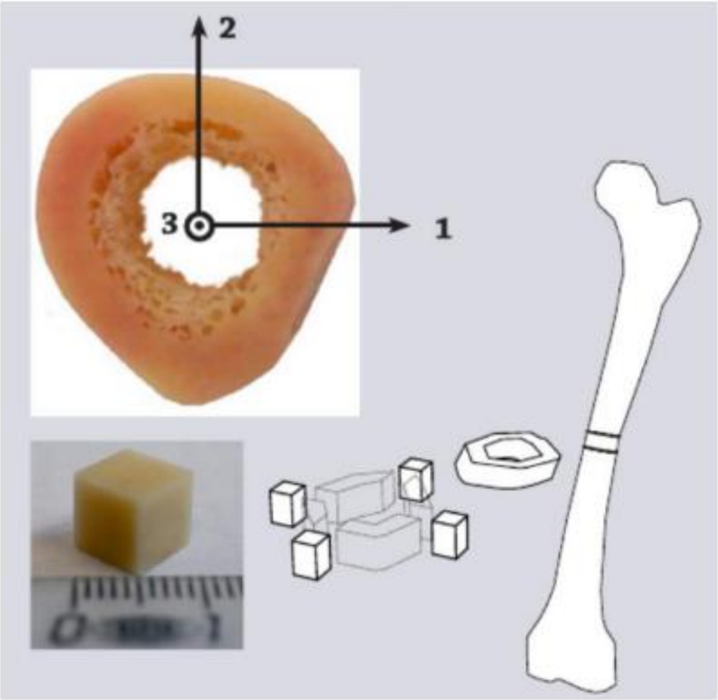


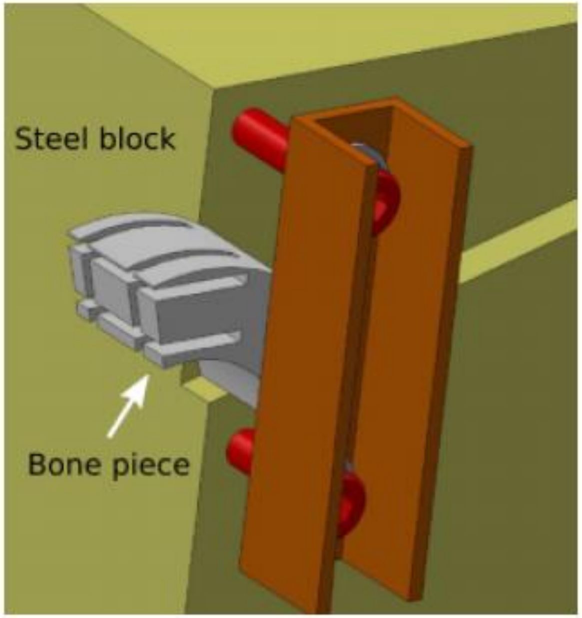
- 13 to 24 resonant frequencies measured for each sample
- Precision (std. dev.) over 6 samples: **1% (C_{11}) and 0.5% (C_{44})**
- Agreement within ± 2.5 % of tabulated values

[Bernard et al. J Acous Soc 2014]

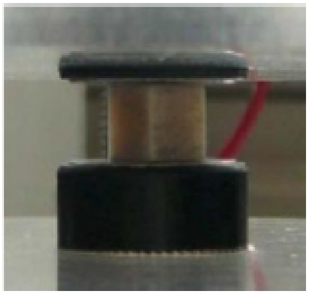
Cortical bone samples

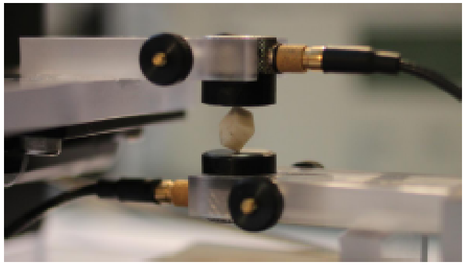
- 53 specimens harvested from 26 left human femurs
- Rectangular Parallelepiped-shape (nominal size: 3x4x5 mm^3)
- Transverse isotropy assumed, i.e. $C_{11} = C_{22}$, $C_{13} = C_{23}$, $C_{44} = C_{55}$, $C_{12} = C_{11} - 2C_{66}$

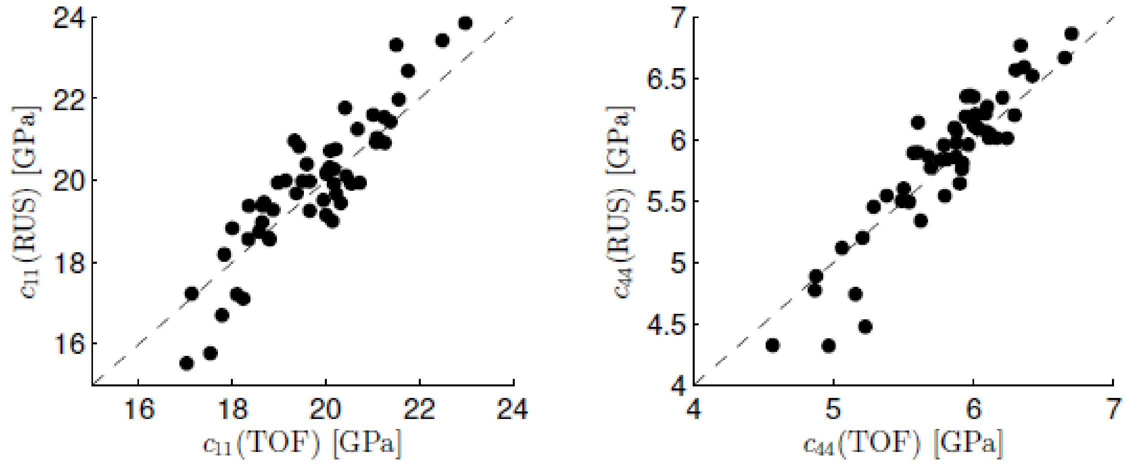




Comparison with Time-of-flight method







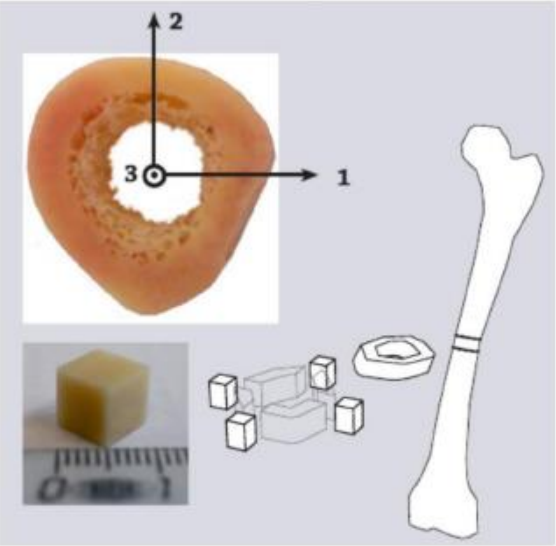
$C_{11}; R^2 = 0.825$; $C_{44}; R^2 = 0.896$

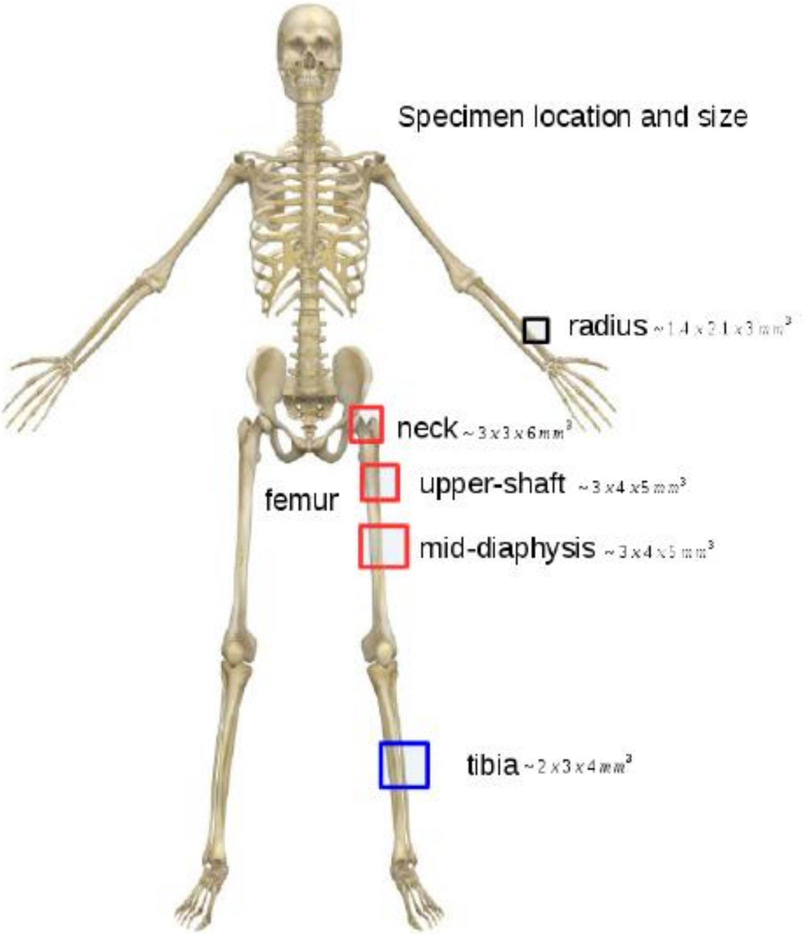
No significant difference between stiffness assessed from RUS and Time-of-flight (first arrival) [Peralta et al. Ultrasonics 2017]

More samples

Specimens of cuboid shape, sized 1~5 mm

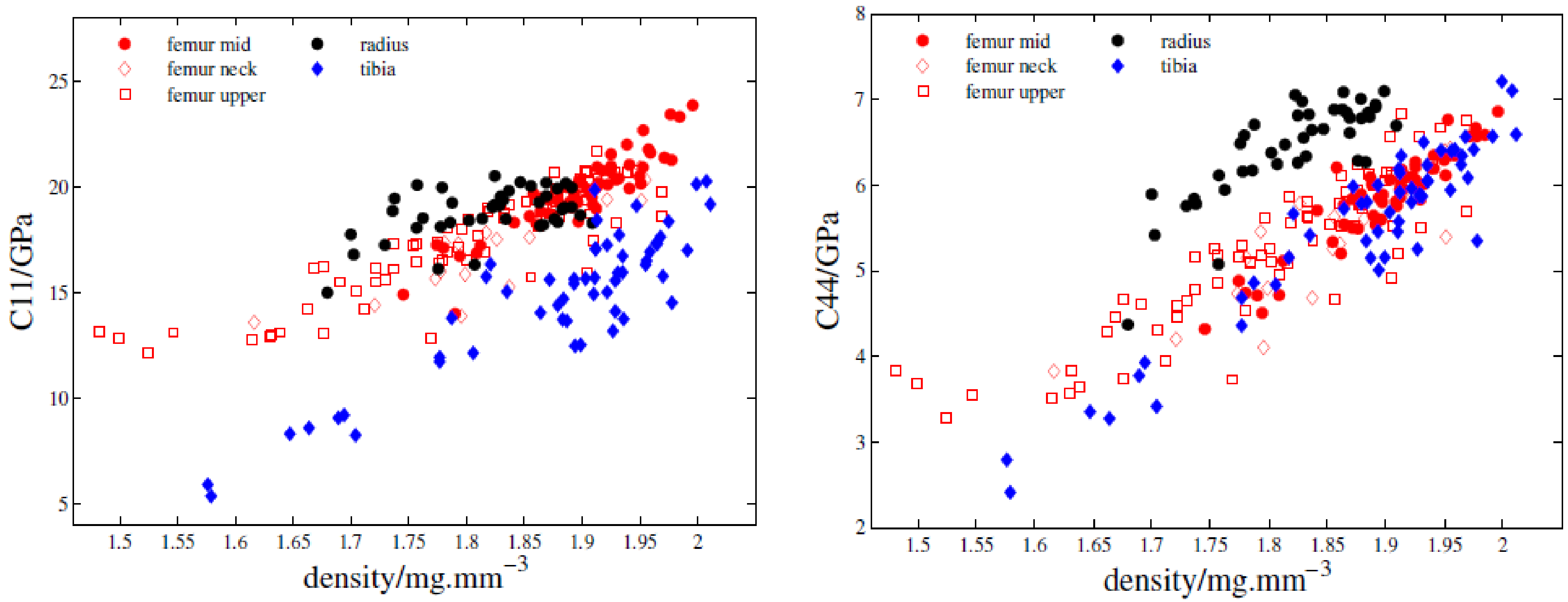
- Femurs : neck (19 donors, 19 specimens), upper-shaft (19 donors, 73 specimens), mid-diaphysis (30 donors, 55 specimens)
- Radius (one-third proximal (20 donors, 42 specimens)
- Tibia (20 donors, 55 specimens)




Specimen location and size

Density – Elasticity relationships

A contribution to bone biomechanics : first multi-site caracterization of the entire stiffness tensor



- Important elasticity relative variations (> 100%) in the physiological density range
- A trend of relatively higher elasticity in radius and lower elasticity in tibia

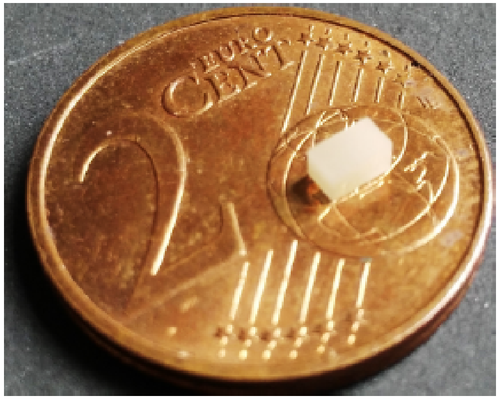
Summary

RUS is a practical method to measure large series of small (~ 1 mm^3) anisotropic (orthotropy) samples with low Q-factors ($Q \sim 25$)

The **precision and accuracy of the determined elastic constants depend on**:

- Quality of the spectrum (sample-transducer **contact conditions**)
- **Accuracy of peak picking** (can be automated but risk of false peaks / missing peaks for viscoelastic materials)
- **Ambiguity in inverse problem** (non unique solution) when frequencies are missing → key role of regularization through a priori on material (stiffness tensor) and anisotropy (class, orientation).

Future work → measure more resonances



Acknowledgements

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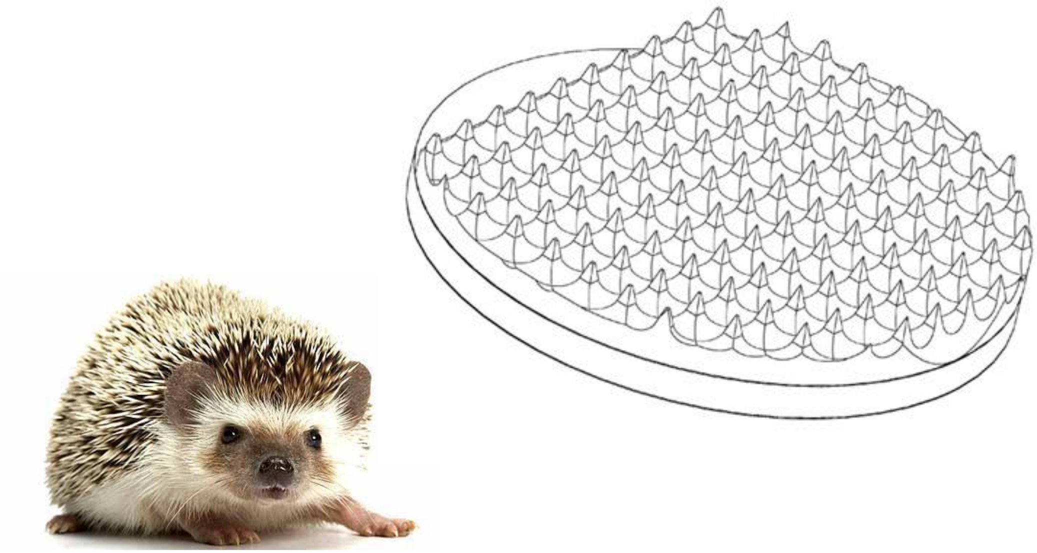
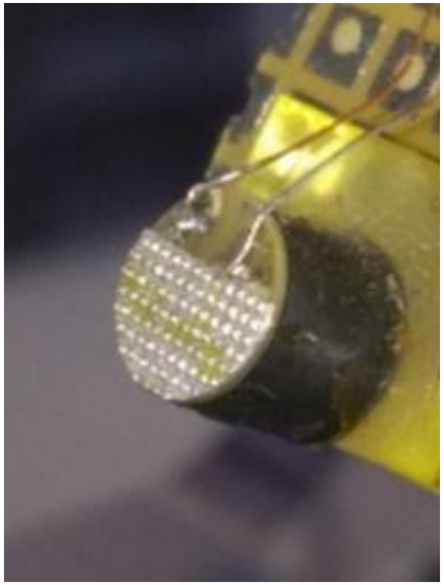
Next steps : Hedgehog Transducer

Pat. FR 3 057 667

Funding : SATT LUTECH

This new kind of transducer is based on the use of an array of tiny spikes at the surface of a piezoelectric disc.

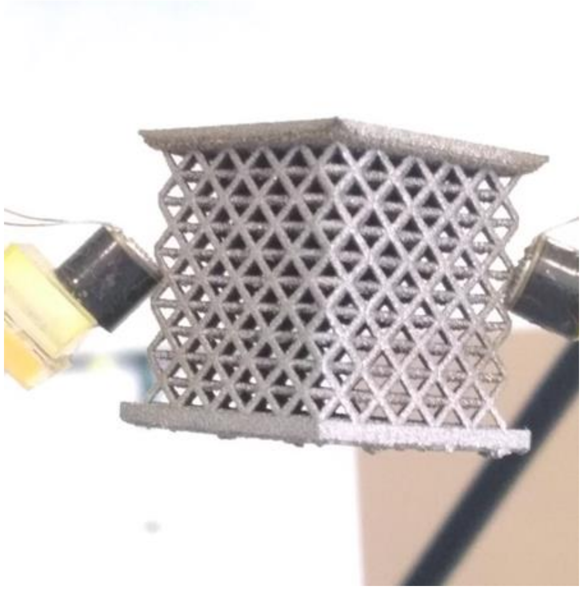


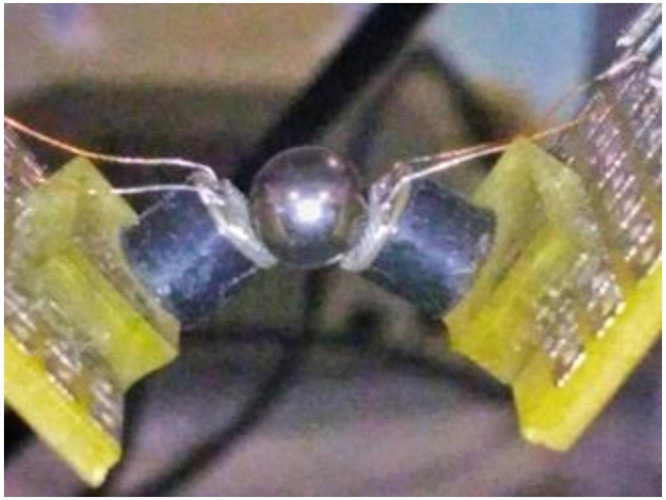


Hedgehog Transducer

Thanks to spikes, sample positionning is isostatic :

- only three points are in contact (2 + 1)
- Boundery conditions of sample are closer to « free-free » conditions
- Sample is easily positionned in stable conditions
- Even smallest samples can be analysed (for modes < 1 MHz)





Standards

Referenced documents « ***related*** » to RUS :

- ASTM C747-16 : Standard Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance
- ASTM C1198-09 : Standard Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Sonic Resonance
- ASTM C1259-15 : Standard Test Method for Dynamic Young’s Modulus, Shear Modulus, and Poisson’s Ratio for Advanced Ceramics by Impulse Excitation of Vibration
- ASTM E1875-13 : Standard Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio by Sonic Resonance
- ASTM E1876 : Standard Test Method for Dynamic Young’s Modulus, Shear Modulus, and Poisson’s Ratio by Impulse Excitation of Vibration
- ASTM E2001-98 : Standard Guide for Resonant Ultrasound Spectroscopy for Defect Detection in Both Metallic and Non-metallic Parts
- BS ISO 17561-2016 = ISO 17561-2002 : Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for elastic moduli of monolithic ceramics at room temperature by sonic resonance
- NF EN 23312-1993 : Matériaux métalliques frittés et métaux-durs - Détermination du module de Young