# An alternative method for the analysis of Resonant Ultrasound Spectra of isotropic materials

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**An alternative method for the analysis of Resonant Ultrasound Spectra of isotropic materials**

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Abstract :

Resonant Ultrasound Spectroscopy is known as one of the most accurate techniques to determine materials' elastic properties. Its use has been limited until now since the extraction of elastic coefficients from resonance spectra requires some expertise. Recent efforts have been made to automatise the analysis and favour the widespread use of this method. Advanced mathematical tools have indeed been developed to produce robust exploitation of resonance spectra, mainly based on probabilistic approaches. Within this dynamics, we propose here a method that allows obtaining in a very efficient way the elastic coefficients of isotropic materials. The method is applicable to complete or incomplete resonance spectra. Our approach exploits the subtleties of resonance spectra in isotropic materials, the main one being that resonance frequencies depend in a complex manner on the Poisson's ratio $\upsilon$ but are proportional to the square root of Young's Modulus *E*. Starting from a set of resonance frequencies, from sample dimensions and density, our method is able to determine the real *E* and $\upsilon$ coefficients quickly and without making any hypothesis on their initial value. We also demonstrate that our approach is valid when the resonance spectrum presents some missing frequencies - which can occur in real cases - or when these frequencies are affected by

experimental error. Finally, we demonstrate in two real cases presented in previous articles that our approach is robust and rapid to determine accurate elastic coefficients.

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## I. Introduction

Elasticity, which correlates the matter displacements to the applied stress within a solid, appears as the first of the solid mechanical properties. It can be measured by many techniques such as compression tests [1-3], Impulse Excitation Technique [4-5], nano-indentation [6-8], Time-of-flight [9-11] or Resonant Ultrasound Spectroscopy (RUS) [12-17]. This last technique has many advantages over the other ones: a very high accuracy, no need for multiple measures for the whole elastic tensor determination, and no need for a big sample [12]. It has also been used in a wide range of thermodynamic conditions such as low temperature [17], high temperature [19-20], under electric or magnetic fields [21-23] or even in the case of samples showing high ultrasonic attenuation [24]. Despite these significant advantages, the use of RUS has been limited to a few groups all over the world: Los Alamos (USA) with 104 publications, Oak Ridge National Laboratories (USA) with 54 publications, M. Carpenter's group at Cambridge (U.K.) with 95 publications, Osaka University (Japan) with 51 publications, Ruhr University of Bochum (Germany) with 20 publications, M. Landa's group (Czech Republic) with 40 publications, to name some of the main actors in the field. It reflects the fact that a degree of expertise is needed to perform RUS measurements and analysis. The measurement by itself is indeed somewhat tricky, even if all the optimised experimental details were presented in previous articles [12-14, 25]. These articles reported on the preparation of a good sample for RUS (shape and size), on the requirement concerning electronics to acquire RUS spectra or on potential sources of errors like the pressure on the sample during acquisition. Commercial devices were also proposed, which help to minimise the source of errors [26, 27]. Considering this, the main difficulty during a RUS characterisation would lie in analysing acquired spectra. The least-square non-linear optimisation usually leads to the correct values of elastic coefficients once a correct association of modes is made and the potential absence

of modes is identified. The approach becomes hard to apply on materials with unknown elastic properties or complex elastic tensors, as in low-symmetry single crystals. This latter case represents a significant difficulty since as much as 21 elastic coefficients must potentially be retrieved if no knowledge is available about sample symmetry. Some methodologies were proposed either based on a step-by-step strategy coupled with complementary experiments [15, 28-29] or on user-independent advanced mathematical analyses, such as the Bayesian approach or the use of genetic algorithms [30-32]. Even if isotropic materials' elastic properties are by far easier to analyse, the limited use of the RUS technique, even in this case, underlines the expertise or time needed to analyse resonance spectra.

In the present work, we present an alternative approach applicable to isotropic materials only, leading to a fast and correct determination of the elastic coefficients. We expect our approach to make the RUS characterisation more accessible to scientists and engineers, following previous works aiming to popularise the RUS approach [17]. In the first part, we describe the hypothesis, approaches and mathematical tools that we used for the RUS analysis. We then apply our approach to 100 virtual isotropic rectangular parallelepiped samples chosen with random size, density and elastic coefficients. We demonstrate that it allows determining the elastic coefficients in the case of resonance spectra containing all the expected modes in a given frequency range and in the case of missing modes. We also show that we still obtain correct values for the elastic coefficients even if some statistical error is present in the spectrum resonance frequencies to be fitted. Finally, we apply our approach to two real examples that were presented in the literature as representative cases, and we show once more the efficiency of the approach.

## II. Calculations details

II.1. Theoretical approach

Our approach is adapted to the analysis of samples with some given characteristics:

- Samples are expected to be isotropic.
- Samples are supposed to be prepared accordingly to an assumed geometry [12-14]. In particular, care should be taken to prepare samples with a well-defined parallelepiped shape and suitable dimensions to avoid overlapping modes.
- Ultrasonic resonance spectra should be acquired with no loading on the sample.

After considering as a requirement that high-quality resonance spectra are obtained, our approach uses the fact that a suitable representation of the elastic tensor allows simplifying the problem deeply. Indeed, the elastic tensor for an isotropic material is represented as:

$$C_{ij} = \begin{pmatrix} C_{11} & C_{12} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{11} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{12} & C_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & C_{44} \end{pmatrix},$$

where $C_{44} = \frac{C_{11}-C_{12}}{2}$

It can also be represented as:

$$C_{ij} = \frac{E}{(1+\upsilon)(1-2\upsilon)} \begin{pmatrix} 1-\upsilon & \upsilon & \upsilon & 0 & 0 & 0 \\ \upsilon & 1-\upsilon & \upsilon & 0 & 0 & 0 \\ \upsilon & \upsilon & 1-\upsilon & 0 & 0 & 0 \\ 0 & 0 & 0 & \frac{1-\upsilon}{2} & 0 & 0 \\ 0 & 0 & 0 & 0 & \frac{1-\upsilon}{2} & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1-\upsilon}{2} \end{pmatrix},$$

Where *E* and $\upsilon$ are Young's Modulus and Poisson's ratio, respectively. From the second form, it is clear that the $C_{ij}$ elastic coefficients are a complex function of $\upsilon$ but are all proportional to Young's modulus *E*. This means that resonance frequencies are proportional to the square

root of *E*. Due to this form of the elastic tensor, isotropic materials with the same Poisson's ratios will present resonance spectra with the same resonance frequencies sequence. To illustrate this, we show in Figure 1, the calculated resonance spectra (standard procedure explained in the following) for three different Poisson's ratios and three different Young's moduli for an isotropic sample of a given size and density. From this comparison, it is evident that the value of *E* only affects the scale of the frequency axis, leaving the pattern of the spectrum the same, this pattern being controlled by $\nu$ (see vertical graphs in Figure 1).

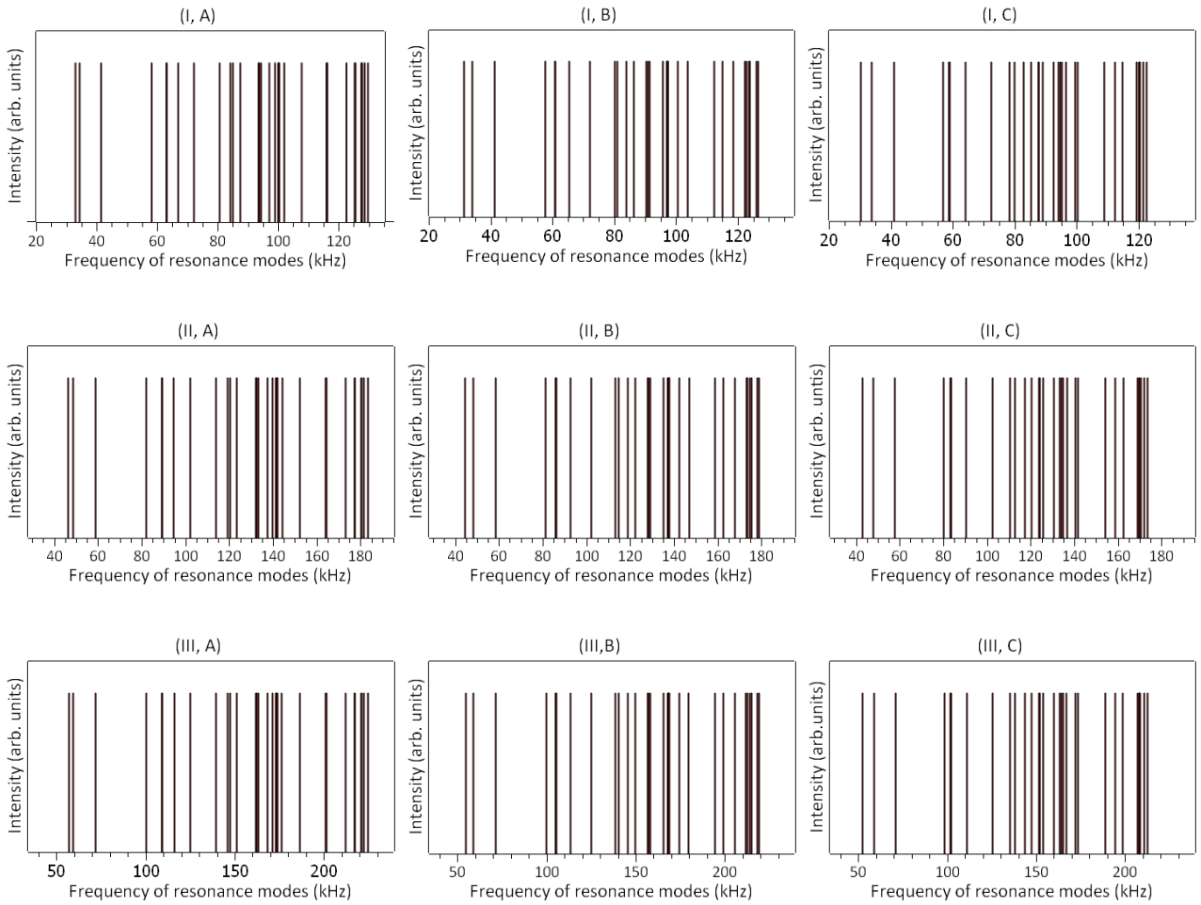



**Figure 1.** Resonance spectra of samples with size $L_1$=2 mm, $L_2$= 3 mm, $L_3$ = 5 mm, d = 2962 kg. $m^{-3}$ with Young's Modulus equal to (I) 100 (II) 200 (III) 300 GPa and Poisson's ratios of (A) 0.1 (B) 0.2 (C) 0.3

The problem of elastic coefficient extraction in isotropic materials can be drastically simplified through our approach. Once spectra are calculated for a given $E_0$ value and a range of $\nu$ Poisson's ratios, any spectrum corresponding to any ($E$, $\nu$) couple can be deduced from the ($E_0$, $\nu$) spectrum by multiplying all its frequencies by $(E/E_0)^{1/2}$. We can calculate spectra for a vast range of ($E$, $\nu$) efficiently and compare them to the original known spectrum. After this comparison, one can determine $E_{det}$ and $\nu_{det}$ the best optimised elastic coefficients. This is the basis of our optimisation scheme, represented in Figure 2.

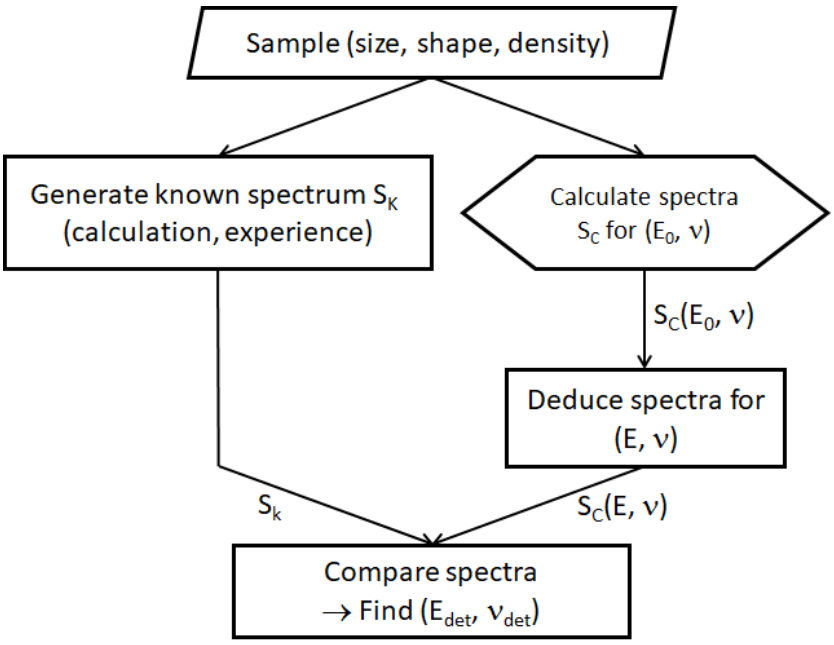



**Figure 2.** Explored scheme for the elastic coefficients extraction

II.2. Methodological details

The fundaments and procedure for the Resonant Ultrasound Spectroscopy forward model calculations have made the subject of many articles. We thus refer the reader to the previous articles mentioned in the introduction, particularly those of Migliori's group [12], which

constitute a complete and reliable source to better understand the ins and outs of RUS principle practice.

For the forward model, the evaluation of vibration modes of elastic objects with free boundaries can be simplified using the Rayleigh-Ritz method, allowing to calculate natural vibration frequencies. This approach supposes that the Lagrangian of a system is stationary with respect to small perturbations in the eigenfunctions. This requirement that Lagrangian is stationary reduces to finding the eigenvalues of the following equation:

$$\mathbf{K}\boldsymbol{a} = \omega^2 \boldsymbol{M}\boldsymbol{a} \qquad [1]$$

Where **K** is the stiffness matrix, **M** is the mass matrix, and **a** is a vector of displacements in three dimensions (see ref.12-17 and 28-31 for details). The interesting point is that the matrix **K** is proportional to the elastic constants $C_{ij,}$ *i.e.* proportional to Young's Modulus in the case of isotropic materials. By factorising *E* in the previous equation, one can easily demonstrate that the resonance frequencies of a given sample are directly proportional to Young's Modulus square root. This is the main property that we used here to fasten the calculations since the spectra for any ($E$, $\nu$) couple can be deduced from the spectrum ($E_0$, $\nu$) by simply multiplying all frequencies by $\sqrt{E/E_0}$.

Along the years, several significant advances were made to facilitate the calculation of the forward model [33-35], and RUS calculation codes were subsequently made available to the scientific community, for instance, from the Los Alamos National Laboratory [36], and the Physical Acoustic Lab (Auckland, New-Zealand) [37]. We used the "RUS.m" Matlab code developed by Matt Fig [38], adapted to our specific purpose. This code is essentially a forward model which allows obtaining the resonance frequencies of a given sample with known

dimensions, density and elastic coefficients. The virtual samples (see below) spectra were obtained by assuming a n = 20 polynomial order for the forward model; the calculated spectra used during elastic coefficient extraction were obtained considering a n = 12 polynomial order.

Based on the scheme presented above, a more detailed calculation procedure is the following:

- We first consider a known spectrum $S_K$ associated with a real or virtual sample with a known size and density. This spectrum, typically containing 30-40 frequencies, can be obtained from experiments or from calculations and is the one from which we want to extract the elastic coefficients.
- We used the forward model to calculate the spectrum for the same sample (size, density, shape) fixing $E_0$ = 100 GPa and $\nu$ varying from 0.1 to 0.4. In our case, we calculated 601 spectra ($S_C$) with thus a 0.0005 step in $\nu$.
- We obtained the theoretical spectra for all $E$ in a given range by multiplying the frequencies obtained in the previous step by $\sqrt{\frac{E}{E_0}}$. In our case, we used a 0.1 GPa step in $E$.
- We calculate a distance measure between the known spectrum and all the calculated spectra (601 x n). In our case, we simply associated the frequencies of the known spectrum to the closest frequencies in the calculated spectra and sum all the absolutes values of differences in frequencies to obtain a distance measure:

$$distance(S_T, S_C) = \sum_{\forall f_i \in S_K} min_{\forall f_j \in S_C} |f_i - f_j| \quad [2]$$

As a result, we obtain a couple ($E_{det}$, $\nu_{det}$) presenting the smaller distance between the known and the calculated spectra. The proposed distance measure which compares sets of different sizes is similar to the Hausdorff distance [39].

II.3. Virtual and real samples

To validate our approach, we input the dimensions and density of the "virtual sample," chosen of a rectangular parallelepiped (RP) geometry. It is a virtual sample for the purpose of the proof of concept worked out here (random dimensions are used), equivalent to the investigated sample in a real experiment, where the mass and dimensions would be measured directly. We considered 100 virtual samples having different sizes and elastic properties. These virtual samples are rectangular parallelepipeds with dimensions $L_1$, $L_2$ and $L_3$ in the range 0.5 – 5.5 cm (the absolute size has actually not much importance on the final result), their density between 2000 and 7000 kg/m$^3$, and their elastic coefficients $E$ and $\nu$ in the range 100 – 400 GPa and 0.1 – 0.4 respectively. We represent, in Table 1, ten of those samples. We also report in Table 1 two real samples taken as good examples for applying an original RUS analysis procedure from previous articles [29-31].

| **Sample** [a,b] | **Samples dimensions and density** | | | | **elastic coefficients (known)** | | **elastic coefficients (determined)** | |
|---|---|---|---|---|---|---|---|---|
| | **L1 (cm)** | **L2 (cm)** | **L3 (cm)** | **Density (kg/m$^3$)** | **$E_K$ (GPa)** | **$\nu_K$** | **$E_{det}$ (GPa)** | **$\nu_{det}$** |
| 1 | 4.573 | 5.029 | 1.135 | 2487 | 374.01 | 0.289 | 373.8 | 0.289 |
| 2 | 1.892 | 3.234 | 5.287 | 6852 | 389.46 | 0.147 | 389.36 | 0.147 |
| 3 | 5.289 | 2.927 | 4.501 | 6578 | 142.56 | 0.226 | 142.55 | 0.226 |
| 4 | 4.461 | 5.297 | 3.779 | 6670 | 110.71 | 0.355 | 110.73 | 0.354 |
| 5 | 3.894 | 4.289 | 4.215 | 2856 | 217.66 | 0.297 | 217.64 | 0.296 |
| 6 | 4.030 | 0.659 | 1.884 | 6117 | 113.85 | 0.129 | 113.85 | 0.130 |
| 7 | 3.974 | 2.085 | 5.251 | 3907 | 110.33 | 0.232 | 110.32 | 0.231 |
| 8 | 4.327 | 4.475 | 4.434 | 5231 | 246.92 | 0.233 | 246.88 | 0.233 |
| 9 | 4.047 | 4.273 | 1.880 | 2813 | 303.91 | 0.296 | 303.89 | 0.296 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 10 | 1.095 | 2.992 | 5.299 | 3119 | 202.12 | 0.275 | 202.12 | 0.276 |
| 11 | 1.192 | 1.093 | 0.986 | 2788 | 71.51 | 0.3381 | 71.5 | 0.3390 |
| 12 | 0.775 | 0.905 | 1.319 | 4402 | 118.6 | 0.3209 | 118.3 | 0.3192 |

[a] *1-10 : virtual samples*

[b] *11, 12 : real samples taken from [29-31]*

**Table 1.** Virtual (n° 1-10) and real (n° 11 and 12) samples characteristics including the dimensions of the rectangular parallelepiped samples (*L1*, *L2*, *L3*), the density and elastic isotropic coefficients $E_K$ and $\nu_K$. Results of the elastic coefficients extraction procedure.

## III. Results

### III.1. Results for virtual test-samples

In addition to the results shown in Table 1, we show in Figure 3 the results of our analysis for samples having perfect RUS spectra *i.e.* calculated with the forward model using a high polynomial order (n = 20), thus having all the resonance modes present and determined with great precision. Figure 3 plots the Poisson's ratio and Young's Modulus as deduced from our procedure and compared to those of 200 virtual samples. This figure shows a perfect agreement between the determined values and the known ones. As a whole, the calculated and known values differ by less than 0.1% for *E* and less than 0.5% for $\nu$, a precision here only limited by the precision we used for both *E* and $\nu$ variables. Concretely we used steps of 0.1 GPa for *E* screening, and 0.0005 for $\nu$ screening.

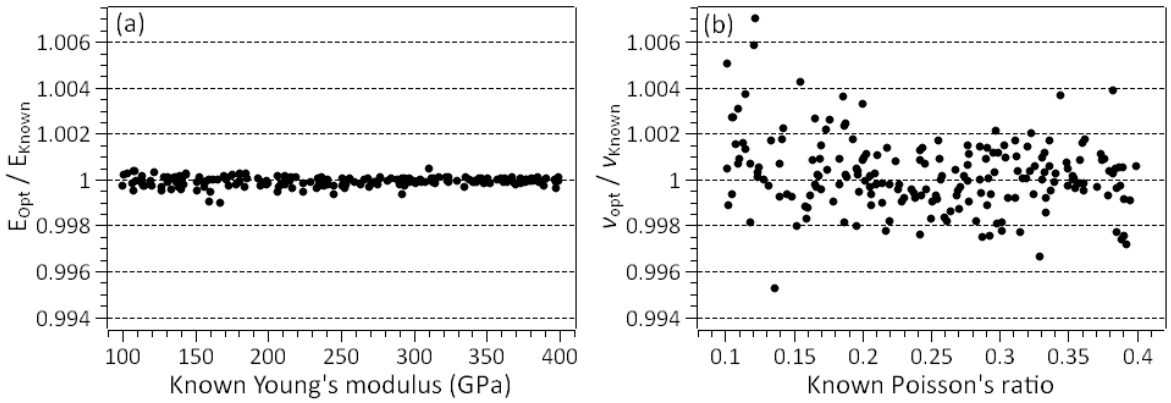



**Figure 3.** Ratio between determined and known elastic coefficients for virtual samples: (a) Young's Modulus and (b) Poisson's ratio.

III.2. Results for virtual test-samples with altered spectra

*III.2.1. Influence of missing modes on elastic coefficients determination*

The results presented above confirm the method's potential interest since it leads to the determination of correct elastic coefficients without any initial guess value and required action. We consider in this part RUS spectra in which some resonance modes are missing. We have artificially removed a given percentage of modes (from 0% to 70%) from the original virtual spectra. The removed frequencies may include the first frequency, which is always considered as important experimentally due to its pure shear character.

The analysis of missing modes' influence was performed in an exhaustive way using 20 different combinations for each percentage of missing modes explored for 50 virtual samples, representing more than seven thousand cases. In the following graph, we present the ratio between the determined values and the known elastic properties as a function of the percentage of missing peaks. Even for a very high percentage of missing modes, our approach gives determined values of *E* close to the known value, the error being always less than 0.5% and 99% of the time less than 0.2%. The error on $\nu$ determination is more important reaching

around 2% for some samples and a high percentage of missing modes. For 30% of missing modes, the percentage of solutions determined for which the error on $\nu$ remains within 0.5% is greater than 95%. These results are particularly interesting since the approach allows determining the elastic coefficients even with incomplete spectra, without the need for assuming it or without the need to evaluate the number of missing modes.

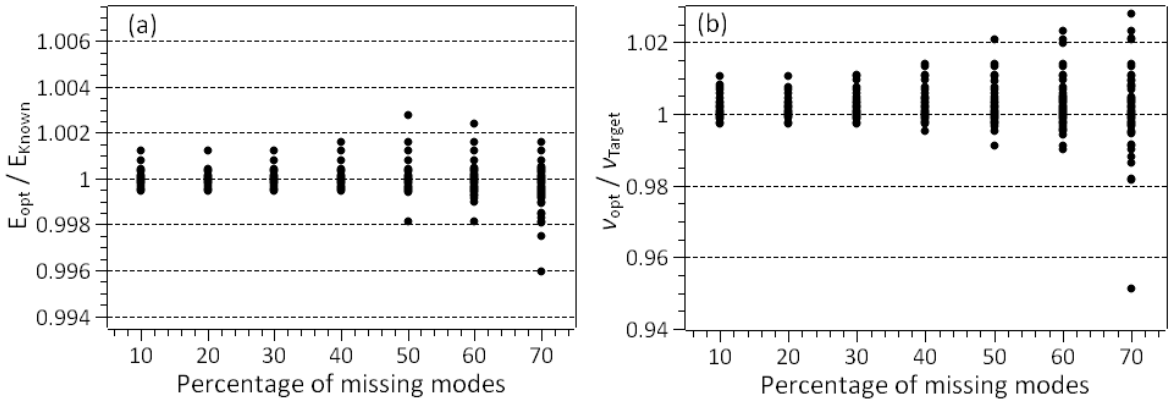



**Figure 4.** Ratio between determined and known elastic coefficients for virtual samples as a function of the percentage of missing peaks (artificially removed) for (a) Young's Modulus and (b) Poisson's ratio

*II.2.2. Influence of noise in the spectrum*

In this part, we introduced some errors artificially on frequency measures in the original known resonance spectra. These errors are set to follow a normal distribution and are supposed to reflect potential sources of error coming from a poor sampling of frequencies during RUS spectrum acquisition. It is not expected to mimic bad acquisition conditions like those due to pressure on the sample, non-homogeneous dimensions, etc... which would induce non-random frequency shifts.

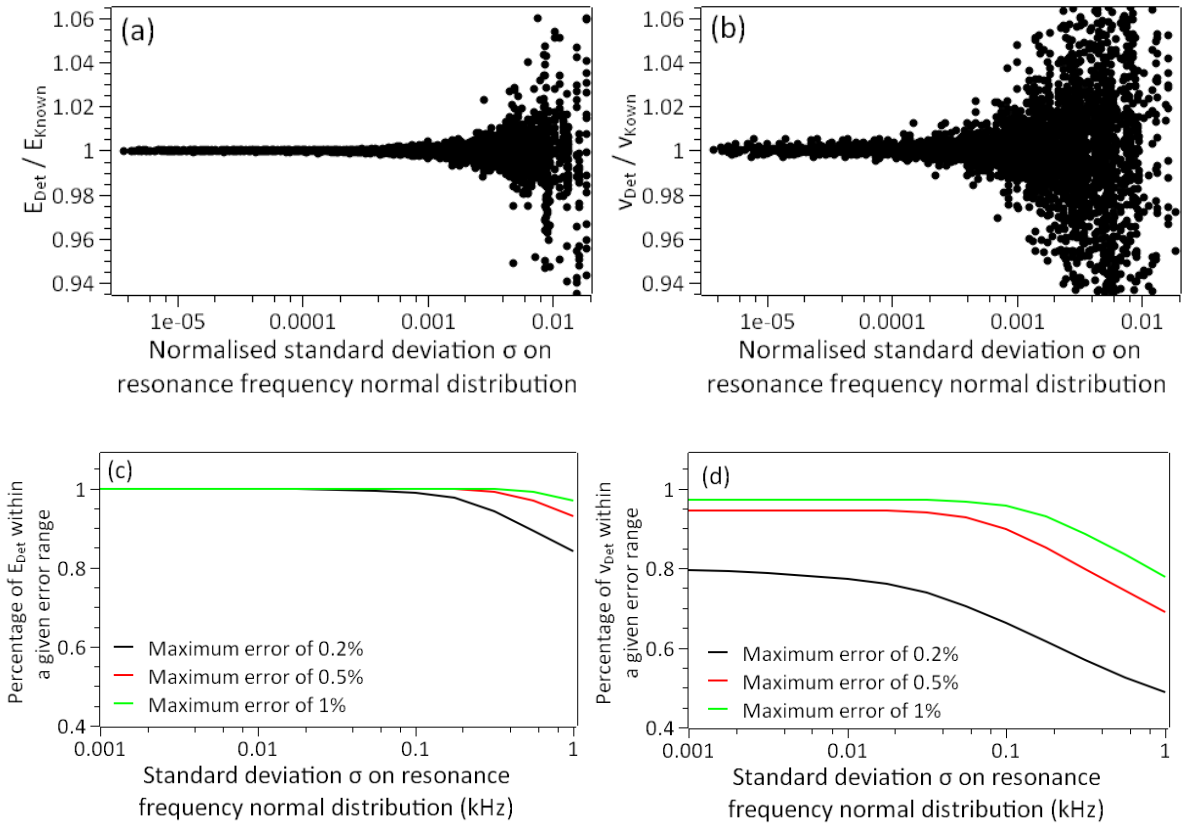



**Figure 5.** Ratio between determined and known values of elastic coefficients for virtual samples as a function of the normalised width of the normal distribution for (a) Young's Modulus and (b) Poisson's ratio. Proportion of determined values within a given range of error (0.2%, 0.5%, 1%) for (c) Young's Modulus and (d) Poisson's ratio.

The results of the effect of random frequency shift on elastic coefficients extraction are shown in Figure 5. The standard deviation is normalised by the 15$^{th}$ frequency of the spectrum to take into account the size and density of the considered virtual sample. Our method appears robust in dealing with potential sources of error and can accept a broad standard deviation on frequency measurements, still leading to correct elastic coefficients.

III.3. Study of real samples

In this section, we discuss the case of two real samples, which were presented in previous articles as good examples to illustrate the difficulty to identify normal modes and obtain the resulting elastic constants in isotropic solids.

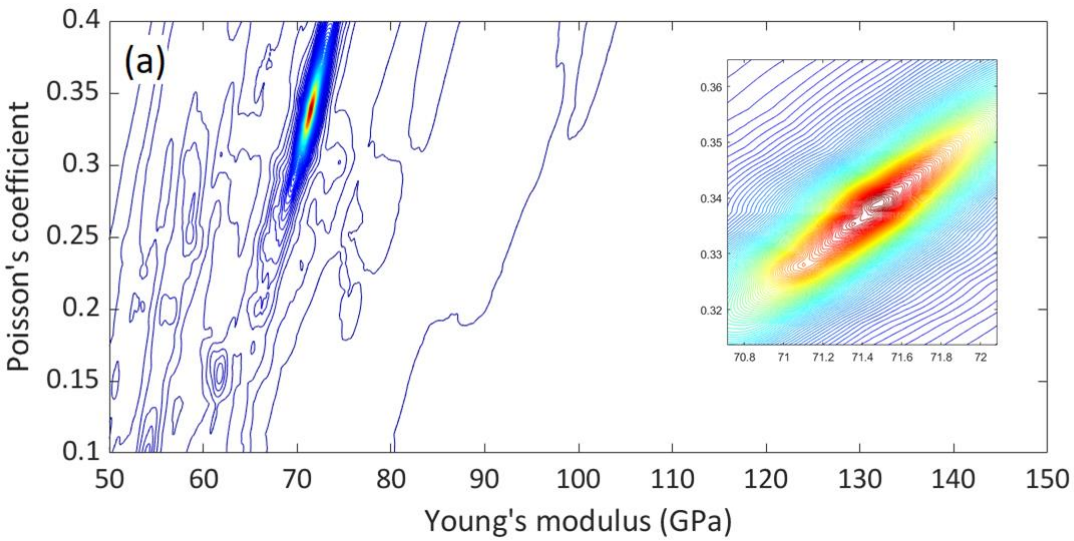
*Real sample 1*

The first one numbered 11 in Table 1 corresponds to a polycrystalline Al alloy, presented first by Ogi *et al.* [29]. This sample was used to demonstrate the interest of using laser-assisted RUS to identify the symmetry of resonance modes and thus facilitate modes association. The same sample was further used by S. Bernard *et al.* [30] to demonstrate the Bayesian-based approach's efficiency for the determination of elastic coefficients. In the original article, the authors mentioned: "*We tried to deduce the $C_{ij}$ using the usual RUS approach with the initial set of $C_{ij}$ used to compute the displacements … pairing the closest resonance frequencies from the measurements and calculations. The inverse calculation failed to converge, indicating that the usual RUS method is sensitive to the initial guesses* ". These authors then succeeded in refining elastic constants only after identifying vibration modes from displacement measurements through an interferometry method, taking benefit from the fact that "the displacement distribution patterns are insensitive to the elastic constants ".

*Real sample 2*

The second example, n°12 in Table 1, is a polycrystalline Ti-6Al-4V alloy with fine grains. This sample was used to demonstrate the Bayesian inference efficiency to determine elastic coefficients and prove the isotropic character of the sample [31]. This sample did not make the object of elastic coefficients determination from the standard procedure, by which we can only compare with the results obtained from the Bayesian approach.

As reported in Table 1, we obtained good determined values for both $E$ and $\nu$ for these two real examples. Through our optimisation scheme, $E_{det}$ and $E_{Known}$ (resp. $\nu_{det}$ and $\nu_{Known}$) differ by ~0.2% (resp. ~0.3%) for sample 1 and by ~0.1% (resp. ~0.5%) for sample 2. We did not have to make any assumption on the first mode, to presuppose any elastic coefficients, or to propose a specific mode association to make the analysis. Besides, our approach allows representing the solution as a map, which may help apprehend better the solution's uniqueness. In Figure 6, we represent the inverse of the distance (Equation 2) between calculated and known spectra. This map shows that the proposed solution is the only one giving acceptable values, the rest of the ($E$, $\nu$) variable space presenting a poor agreement with target data.



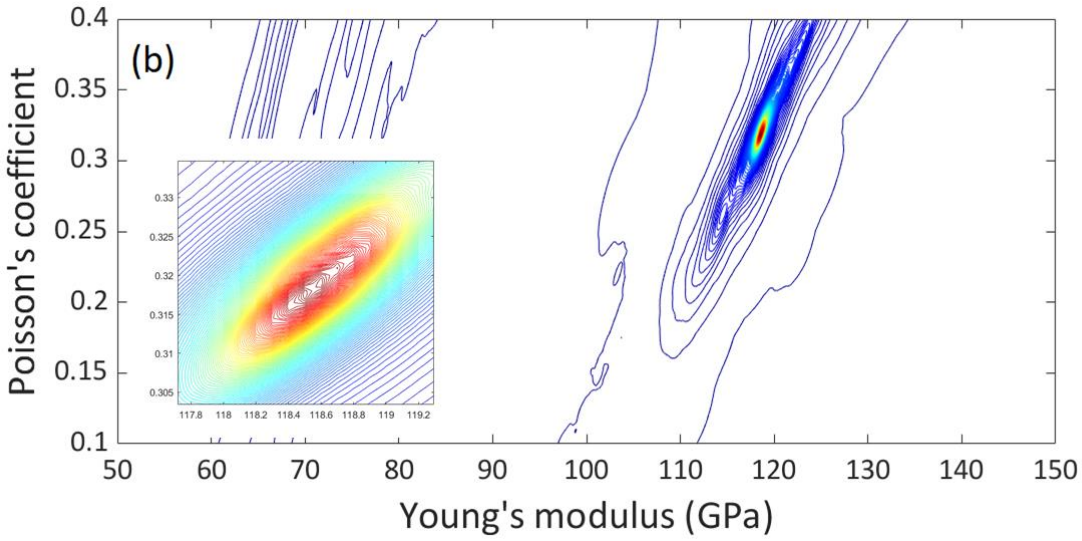



**Figure 6.** Map of the inverse of the distance between calculated and target spectra for a wide (E, ν) explore space: (a) sample n°11 (b) sample n°12.

A zoom on this map, close to the determined elastic coefficients region, provides a natural path toward error estimation. Following an approach similar to the one proposed by Migliori *et al.* [12], A 2D-ellipsoid in the parameter space surrounding the minimum of distance between calculated and known spectra can be determined corresponding to a 2% increase in the distance measure. This ellipsoid may provide a realistic error estimate for both *E* and *v* parameters. Following this approach, the error estimation for $E_{det}$ for samples 11 and 12 is smaller than 0.1% in both cases, and the error estimation for $\nu_{det}$ for samples 11 and 12 is 0.3% and 0.6%, respectively.

## IV. Discussion

The approach presented here exploits the elastic tensor's unique properties in isotropic materials, particularly the fact that we can factorise Young's Modulus in the expression of $C_{ij}$,

making the calculation of resonance modes for a wide range of $E$ and $\nu$ coefficients more straightforward. The approach does not pretend to provide new conceptual tools in the field of Resonant Ultrasound Spectroscopy. It simply proposes some method to efficiently obtain elastic coefficients without any expertise needed, any identification of the peaks to specific resonance modes and any assumption on $E$ and $\nu$ values. It is nevertheless brute-force in that it explores the whole variable space to find the determined solution.

In practice, the extraction of elastic coefficients following our scheme, *i.e.* scanning a wide range of $E$ and $\nu$, can last for some tens of seconds. For instance, an optimisation for $E$ ranging from 100 to 400 GPa with a 0.1 GPa resolution and with $\nu$ from 0.1 to 0.4 (with a 0.0005 step), would typically last 90 s on a personal computer. We have to mention here that all the results presented here were obtained with a code that was not specially optimised for speed. It could perform faster if the writing of elastic matrices during resonance mode calculation and interpolation of frequencies were optimised.

Our approach is also interesting in that it allows obtaining a solution map, which further gives a visual representation of the solution. This representation could help reveal potential concurrent solutions in case of unreasonable determined values ($E_{det}$, $\nu_{det}$) or even be used as proof of isotropicity. Our approach for elastic coefficient determination is only adapted to the RUS analysis of isotropic samples. Contrary to some previous approaches, which suggest obtaining guess values for $C_{44}$ by considering the shear character of the very first mode, the approach proposed here makes no hypothesis on elastic coefficients. Besides, the fact that we can manage till 30% of missing modes without significant degradation of the results could be interesting when dealing with incomplete spectra. Thus our approach, even if conceptually very simple, is potentially very useful for non-expert users or for *in situ* characterisations, during which it is often difficult to control the absence of resonance modes. Indeed, while

several experiments can easily be performed at room temperature to obtain a complete set of experimental resonance frequencies, such an approach is hard to apply on samples studied *in situ,* for instance, as a function of temperature or of a magnetic field. Obviously, our approach aims not to enter into competition with more advanced or general methodologies based on probabilistic approaches, which are especially powerful in determining elastic coefficients of samples with lower symmetry, *i.e.* for polycrystalline samples with texture or for single crystals.

## V. Conclusion

This work proposes an alternative approach to analyse the Resonant Ultrasound Spectra of elastically isotropic samples to determine their two independent elastic coefficients. The procedure is straightforward, robust and efficient and can be implemented easily. Elastic coefficients can be obtained in a few tens of seconds with high accuracy without prior knowledge of the sample's elastic behaviour. It is also reliable when some resonance modes are absent in the original spectrum, which drastically simplifies the measure for the interpretation of *in situ* RUS measurements.

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Data availability statement

The raw/processed data required to reproduce these findings cannot be shared at this time due to technical or time limitations.

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