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Incorporating Crystallographic Orientation in the Development of Resonant Ultrasound Spectroscopy

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Abstract. Resonant ultrasound spectroscopy (RUS) measures the mechanical resonance of solids and uses the resonance frequen-cies to extract a complete set of elastic constants of the solid material. One of the advantages of the RUS method is its applicability to small single crystals. In the past two decades, the RUS technique has gained more acceptance as a nondestructive method to measure elastic properties. The inherent assumptions in the conventional RUS algorithm include free boundary condition on the specimen faces and the faces of the specimens are normal/parallel to the principal crystallographic axes. This assumption is fulfilled through a time consuming procedure that typically involves multiple iterations of sample cutting and inspection using an x-ray Laue method. Such an intensive method is not suitable for many samples in engineering applications. To estimate the elastic constants of such samples, a modified RUS algorithm has been developed to incorporate the sample crystallographic orientation expressed in terms of Euler angles. This modified RUS algorithm has been applied to estimate the elastic constants of cubic and hexagonal crystal structure samples with known orientation. The obtained values are comparable to literature values. With the incorporation of crystal orientation into the RUS algorithm, the elastic constants of samples with random crystal orientation were obtained.

INTRODUCTION

Resonant ultrasound spectroscopy (RUS) [1, 2, 3, 4] is a proven, non-destructive method used to determine a complete set of elastic constants in millimeter-scale solid samples with known canonical geometries such as rectangular parallelepiped and crystallographic orientation. While traditional methods based on the velocity of sound propagating through a sample require several measurements along different crystallographic directions to obtain a full elastic stiffness matrix, whereas RUS is able to measure the complete set of elastic constants of a solid in one frequency sweep using a spectrum of its mechanical resonances. The RUS measurement technique assumes that the specimen faces are normal to principal crystallographic axes, as shown in Figure 1. In practice, this is not always possible. A solution to this limitation is to perform time consuming specimen reorientation during sample preparation to ensure that the faces are normal to principal crystallographic axes. However, in materials that cannot be grown in large sizes, it is difficult to cut and polish so that its faces are aligned normal to the principal crystallographic axes.

To overcome this limitation, this paper presents a method that incorporates crystallographic orientation information into the RUS method of determining elastic constants. The method is demonstrated by applying it to single crystals of cubic and hexagonal crystal structures.

Resonant Ultrasound Spectroscopy

Resonant ultrasound spectroscopy technique is a two stage process involving both experimental measurements and computation as shown in Figure 1. The experiment consists of measuring the normal mode frequencies of a vibrating solid. An iterative computational technique is then utilized to extract the elastic constants. This physics based inversion process makes use of a forward model of the experiment which predicts the resonance frequencies for

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a given set of elastic constants. The given set of elastic constants are then adjusted iteratively to find the best fit, in a multidimensional non-linear least squares fitting between calculated and measured frequencies. The forward model typically utilizes a numerical algorithm based on minimum energy configuration of the body known as the Rayleigh-Ritz (RR) energy minimization technique, where a number of different basis functions have been applied [5, 6, 7, 8]. Visscher *et al.* [9] proposed a simple computational scheme that uses a basis function in the form of $x^p y^q z^r$ has been applied to specimens with well defined geometries.

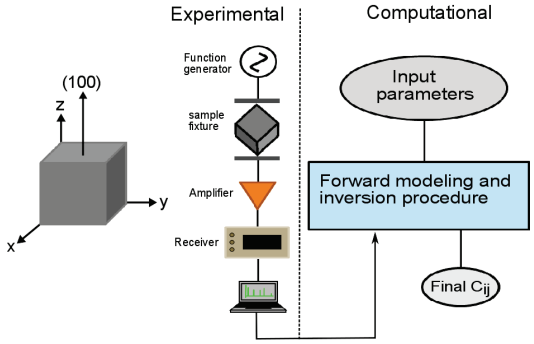

FIGURE 1. The components of RUS experimental measurements including the measurements of natural frequencies and the inversion procedure. The assumption that sample faces are normal to the principal crystallographic axes is illustrated here.

Incorporation of Orientation Parameters in the Forward Problem

The mathematical formulation of the forward problem of computing the natural resonances of a vibrating solid has been described in detail in the literature [3, 9]. This formulation does not include specifically the crystallographic orientation of the single crystal. In this section we provide a description of the forward problem of resonance computation with the incorporation of crystallographic orientation. At resonance, an elasic solid oscillates between extremes of maximum strain and zero velocity (maximum potential energy and zero kinetic energy) and vice versa. The potential and kinetic energy can be expressed in terms of a field $\vec{\psi}(\vec{x})$ which is the displacement of a point in the solid as a function of its equilibrium position $\vec{x}$. The potential energy density of the solid with elastic constants c_{ijkl} is $\frac{1}{2}c_{ijkl}\frac{\partial\psi_i}{\partial x_j}\frac{\partial\psi_k}{\partial x_l}$ And the kinetic energy density for a normal mode of frequency ω and mass density ρ is $\frac{1}{2}\rho\omega^2\psi_i\psi_i$ where $i, j, k, l = (1, 2, 3)$ represent Cartesian coordinates of the sample.
The Lagrangian becomes

$$\mathcal{L} \approx \int\int\int \left(\frac{1}{2}\rho\omega^2\psi_i\psi_i - \frac{1}{2}c_{ijkl}\frac{\partial\psi_i}{\partial x_j}\frac{\partial\psi_k}{\partial x_l}\right) dV \tag{1}$$

The displacement field can be approximated by an expansion of some suitable set of basis functions:

$$\psi_i(x, y, z) \approx \sum_\lambda a_{i\lambda}\Phi_\lambda(x, y, z) \tag{2}$$

where $a_{i\lambda}$ are the expansion coefficients and $\Phi_\lambda(x, y, z)$ are the basis functions.
The assumption of free boundaries for the vibrating solid makes it possible such that the displacements are solutions for which the Lagrangian is minimized. The minimum is determined by equating derivatives of the Lagrangian with respect to expansion coefficients, $\vec{a}$ to zero. The minimization can be cast in eigenvalue form as:

$$\omega^2\vec{E}\vec{a} = \vec{\Gamma}\vec{a} \tag{3}$$

where $\vec{E}$ and $\vec{\Gamma}$ are matrices resulting from the kinetic energy and potential energy terms respectively. The solutions to this eigenvalue equation give the oscillation frequencies (eigenvalues) of the solid and its physical displacement (eigenvectors).

In the treatment of specimen where the faces are not normal to the principal crystallographic axes, it is required that the elastic tensor be transformed under rotation [2, 10]. So, there is need to determine the orientation of the faces of a rectangular parallelepiped specimen and how far off they are from the principal crystallographic axes in the form of Euler angles. The Euler angles are used to define a transformation matrix that will be applied to the elastic tensor

in order to calculate a new set of transformed elastic tensor, $c_{i'j'k'l'}$. The transformed elastic tensor will then replace c_{ijkl} in equation (1), that is

$$c_{ijkl} \longrightarrow c_{i'j'k'l'} = \sum_{i,j,k,l} R_{i'i} R_{j'j} R_{k'k} R_{l'l} c_{ijkl} \tag{4}$$

The elements of the rotational matrix R depend on the notation used to determine the Euler angles. In this project, the Bunge notation was used. By definition, the crystallographic axes (X-Y-Z) are rotated with respect to the sample axes by an angle α about Z, then by an angle β about the new X axis, and finally by an angle γ about the final Z axis. In this notation, R takes the form

$$R = \begin{pmatrix} cos\alpha\ cos\gamma - sin\alpha\ cos\beta\ sin\gamma & -cos\alpha\ sin\gamma - sin\alpha\ cos\beta\ cos\gamma & sin\alpha\ sin\beta \\ sin\alpha\ cos\gamma + cos\alpha\ cos\beta\ sin\gamma & -sin\alpha\ sin\gamma + cos\alpha\ cos\beta\ cos\gamma & -cos\alpha\ sin\beta \\ sin\beta\ sin\gamma & sin\beta\ cos\gamma & cos\beta \end{pmatrix} \tag{5}$$

The Inverse Problem

The inverse problem is the determination of some or all of the parameters from the measured frequencies. Migliori and Maynard [11] presented an approach for the inverse problem of the conventional RUS technique.The elastic constants are usually the parameters of interest, but sample dimensions or crystal orientation with respect to sample axes can be inferred. A very efficient technique for determining the parameters of interest was developed by Migliori *et al* [12]. This approach involves estimating how good a particular forward model is (in terms of predicting the experimental data) by defining a function which measures the closeness of fit between the calculated and measured frequencies using the Levenberg-Marquardt nonlinear least squares fitting scheme.

In this project, the average magnitude of percentage difference, $\%D$ between the measured frequencies, f_i^{meas} and the calculated frequencies, f_i^{calc} is used to determine the optimum fit and the best estimation of the elastic constants.

$$\%D = \frac{1}{N} \sum_{i}^{N} \frac{|f_i^{meas} - f_i^{calc}|}{f_i^{meas}} \tag{6}$$

Experiments

In this project, the complete set of elastic constants of two materials with different crystal structures were determined. Rectangular parallelepiped specimens of a cubic crystal, Nickel Aluminum (NiAl) and an hexagonal crystal material, α-phase Ti-7Al were prepared with all the surfaces polished to 0.05 micron surface finish. Subsequently, electron backscatter diffraction (EBSD) measurement were collected on the samples to determine the orientation in terms of Euler angles, α, β and γ. The dimensions, densities and Euler angles of the samples are given in Table 1.

TABLE 1. Properties of NiAl and α-phase Ti-7Al single crystal samples. There is an uncertainty of ±0.002 mm with the dimensions measurement and $\pm 1^o$ in the angle measurements.

Properties	Nickel Aluminum (NiAl)	α-phase Ti-7Al
Length (mm)	3.05	4.99
Width (mm)	3.01	8.02
Height (mm)	4.54	6.00
Density (g/cm^3)	5.84	4.35
α (deg)	43.2	195
β (deg)	91.8	74.9
γ (deg)	179	102

Subsequently, resonance frequencies measurements were collected for both samples, using the experimental setup of Figure 1. Ten independent measurements were made on the samples. After each measurement, the samples were removed and then remounted between the transducers to take another measurement. Lists of resonance spectrum was extracted from the result of these measurements and used with parameters in Table 1 in the fitting scheme (described in the inverse problem) to calculate the complete set of elastic constants.

Results

The final set of fitted elastic constants of NiAl and Ti-7Al specimens are shown in Tables 2 and 3. In each of these fittings, the Euler angles and dimensions are fixed as the measured values of Table 1.

TABLE 2. Elastic constants of NiAl single crystal sample.

	c_{11} (GPa)	c_{12} (GPa)	c_{44} (GPa)	Avg |% diff | (%)
Without orientation	310.5	213.5	125.9	2.56
With orientation	203.2	136.1	114.9	0.15
Pulse echo [13]	204.6	135.4	116.8	

TABLE 3. Elastic constants of Ti-7Al single crystal sample.

	c_{11} (GPa)	c_{33} (GPa)	c_{44} (GPa)	c_{12} (GPa)	c_{13} (GPa)	Avg |% diff | (%)
Without orientation	163.0	175.1	48.1	80.8	62.7	0.89
With orientation	164.0	172.8	44.7	76.8	56.3	0.68

In both cases when orientation information are explicitly included in the fitting, the elastic moduli are much closer to literature values and the average absolute percentage difference between measured and calculated resonance frequencies are much lower. Another effect that crystallographic orientation inclusion has on the fitted moduli is that when Euler angles are very different from 90^o or multiples of 90^o, the conventional orientation assumption results in large errors in the fitted elastic moduli. There is also a much greater disparity in the average percentage difference between measured and calculated frequencies. The fitted elastic moduli of NiAl in Table 2 illustrate this effect. On the other hand, as the Euler angles are closer to 90^o and its multiples, the fitted elastic moduli are similar to that obtained by assuming the convention (sample surfaces are normal to the principal crystallographic axes). This is clearly shown in the results of Ti-7Al single crystal, Table 3.

SUMMARY

In the development of resonant ultrasound spectroscopy technique for the determination of elastic constants of materials, the following assumptions were made; the sample is under stress free boundary conditions, the sample has a canonical geometry, and the surfaces of the sample are normal to the principal crystallographic axes. In this article, the crystallographic orientation information (Euler angles) has been incorporated as parameters in the RUS technique so that it can be applied in the measurements of elastic constants of specimen with arbitrary crystallographic orientation.

The modified RUS routine that included orientation information in the form of Euler angles as parameters has been successfully employed to determine elastic moduli of a cubic crystal material, Nickel Aluminum (NiAl) and an hexagonal material, α-phase Ti-7Al.

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