Evaluation of anisotropic elastic properties by ultrasonic methods

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Abstrakt

Among the fundamental characteristic of solids are their elastic constants. As derivatives of the free energy, elastic constants are closely connected to thermodynamic properties of material. Extensive quantitative connections among thermodynamic properties can be made if the elastic constants are known as functions of temperature and pressure. Until recently, most often used methods for measurement of elastic constants have been ones based on ultrasonic pulse propagation. Another group is resonance method based on the measurement of natural frequencies of a sample and an inversion for its elastic parameters.

This contribution presents applications of both approaches in material science. The advantages and limitations are demonstrated on the preliminary experiments.

Keywords : pulse-echo measurement, resonant ultrasound spectroscopy, elastic properties of single crystals

1 Acoustic waves and elastic moduli

In an arbitrary direction of an anisotropic solid, sound may be propagated in a form of three elastic waves with different velocities. The wave polarization vectors (displacement motion directions) make an orthogonal triplet with general orientation with respect to the propagation axis. If the planar wave propagates in a principal material symmetry direction, Fig.1 a, then we can distinguish the longitudinal (L - the polarization is parallel along the propagation) and shear (transverse - T - the polarization is perpendicular to the propagation) waves, similarly as in the isotropic material.

The elastic-stiffness tensor of a cubic crystal has three independent elastic moduli C_{11}, C_{12}, and C_{44}, which may be determined from the velocities of waves propagating along the crystalographic direction [100]

$$c_L \equiv V_{[100][100]} = (C_{11}/\rho)^{1/2}, c_T \equiv V_{[100][001]} = V_{[100][010]} = (C_{44}/\rho)^{1/2}$$

and along the direction [110]

$$c_{qT} \equiv V_{[110][\bar{1}10]} = ((C_{11} - C_{12})/(2\rho))^{1/2} \quad \text{or} \quad c_{qL} \equiv V_{[110][110]} = ((C_{11} + C_{12} + 2C_{44})/\rho)^{1/2},$$

where the first index of the wave velocity symbol V means the wave propagation and the second index marks the wave polarization. The anisotropy magnitude of a cubic material is characterized by the anisotropy factor $A = 2C_{44}/(C_{12} - C_{12})$. The value $A = 1$ means an isotropic material and e.g. $A = 1.56, 3.21$ for single crystals Si, Cu (f.c.c.), resp.

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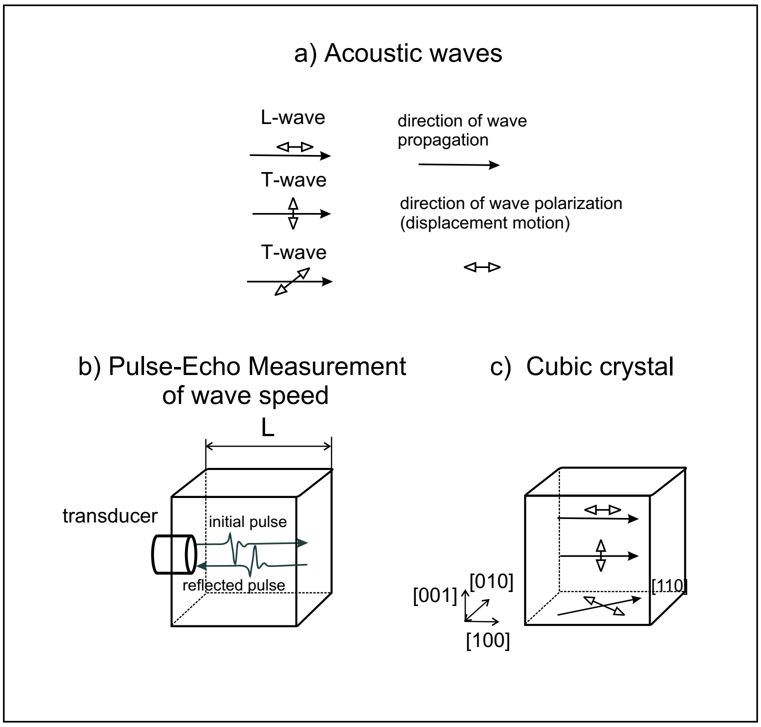


Obr. 1 Pulse - echo ultrasonic measurement.

2 Pulse-echo measurements of wave velocity

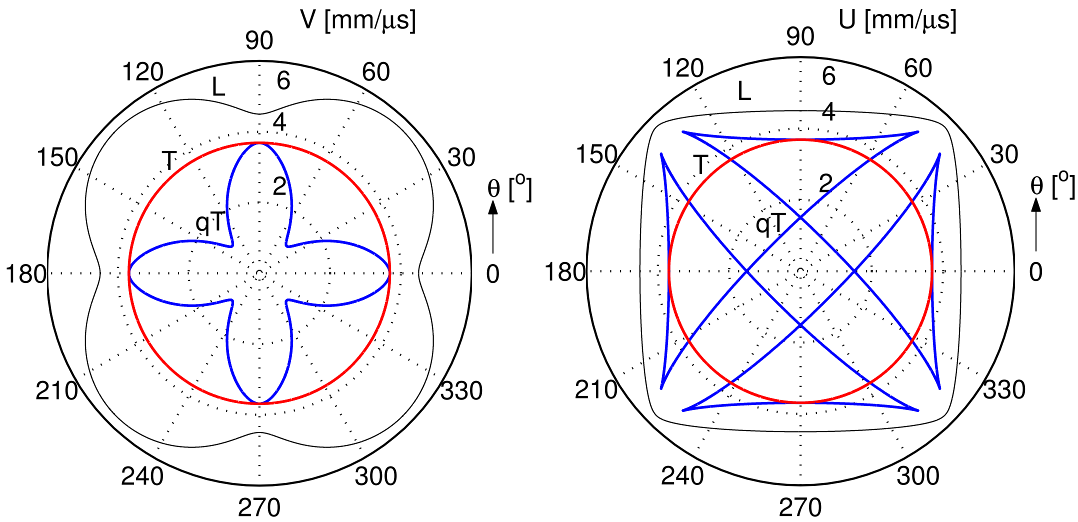
Conventional measurements of the elastic stiffness tensor C_{IJ} are made by measuring the velocity of acoustic waves along usually special directions in large specimens of the tested crystal, [2], [4]. In the example, Fig.1 b, shown here a short pulse of longitudinal wave is generated by a transducer on one crystal face; the pulse travels as a narrow beam through the crystal and reflects back from the opposite face to produce an echo that is picked up by the transducer. The measured time between the initiation of the pulse and the echo (called the pulse-echo time, τ) is equal to $2L/c_L$, where L is the distance between the two crystal faces and c_L is the longitudinal wave velocity. Thus the velocity can be calculated directly from τ. Further, if the density of the crystal, ρ, is known, the measurement also provides a direct determination of, C_{11}, one component of the elastic-stiffness tensor. By placing the transducer at various locations on the crystal and using both longitudinal and shear waves, all the components of the elastic-stiffness tensor can be determined, Fig.1 c.

3 Relative changes of wave velocity in prestressed single crystal

The material Cu-Al-Ni belong to Cu-based alloys which are studied for their shape memory effect. The single crystal Cu-Al-Ni was prepared in the Department of Metal Physics (Institute of Physics, AS CR). The cubic specimens with the edge length 10mm, were cut with orientation $[110][\bar{1}10][001]$.

The pulse-echo method (central frequency 10MHz) was used for wave velocities measurement in the stress-free crystal. The resulting values of velocities are : $c_L = 4.4992, c_{qL} = 5.7147, c_T = 3.6871, c_{qT} = 1.0859\mathrm{mm}/\mu\mathrm{s}$. and corresponding elastic constants : $C_{11} = 142.8, C_{12} = 126.2, C_{44} = 95.90\mathrm{GPa}$ are calculated with the mass density $\rho = 7.0546\mathrm{g/cm}^3$ (in the RT condition : $T_\circ = 24 - 25°\mathrm{C}$). The anisotropy factor of the Cu-alloy $A = 11.5$ indicates very strong anisotropy. It is also obvious from the polar diagrams of calculated phase and group velocity for crystalographic plane $\{001\}$, Fig.2, using the measured elastic constants.

The velocity changes with applied pressure are caused by both sample geometry changes and nonlinear elastic properties (in case the velocity changes are reversible). The nonlinear part is



normal surface (phase velocity) ray surface (group)

Obr. 2 Anisotropy of wave propagation in Cu-Al-Ni.

described by introducing of third order elastic constants (TOEC). These additional six quantities (for a cubic crystal) may be determined from the pressure derivative of relative change in wave velocity $|d(\Delta V/c)dp|_{p=0}$, where c is stress-free velocity, [1]. The nine combination of the direction of applied stress, the propagation vectors and the polarization vectors may be used for determination and verification TOEC values, Fig.3. The relative wave velocity dependencies on pressure, Fig.4, during loading and unloading are characteristics of a crystal stability before phase transformation processes.

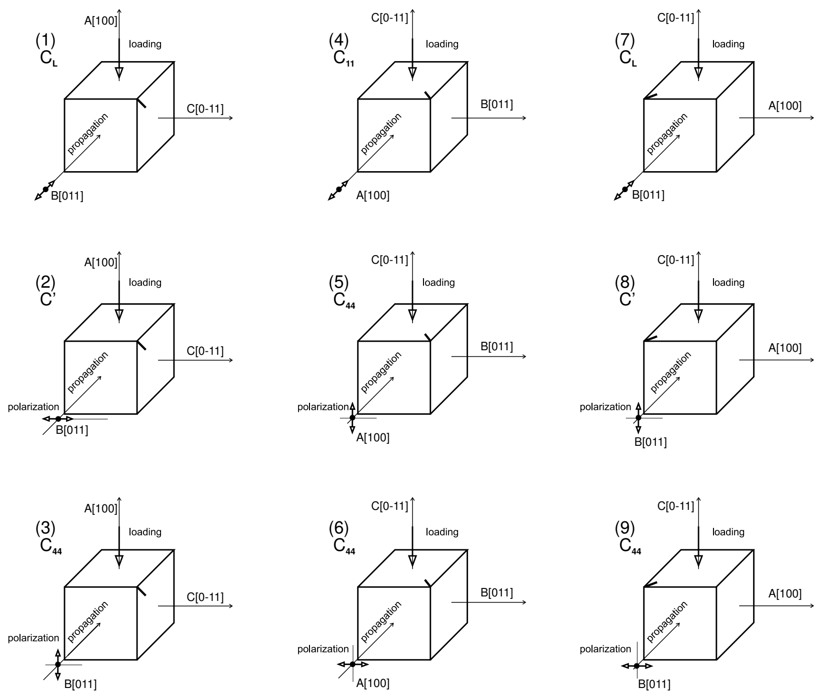
4 Resonant ultrasound spectroscopy

More recent determinations of elastic constants have used a technique resonant ultrasound spectroscopy (RUS), Fig.5, in which one measures the natural frequencies of elastic vibration for a number of a sample's normal modes and analyses these, along with the shape and mass of the sample. In the data processing, one must first solve the problem of calculating the natural frequencies (the forward problem), and apply a nonlinear inversion procedure to find the demand elastic constants from the measured natural frequencies (the inverse problem). The Rayleigh-Ritz method is usually used for solving the forward problem, [3]. Adopting of finite element method seems to be very suitable for this purpose. The inverse problem is solved by hybrid Newton and steepest descents method of nonlinear multidimensional data modelling (Levenberg-Marquart).

The RUS analysis requires that the measurement determine the natural frequencies of a sample with stress-free boundary conditions. Resonance oscillations are excited by one transducer. The second transducer receives the amplitude and the phase of the sample response. To obtain the desired accuracy, one must minimalize sample loading by the transducers. It was proposed to a rectangular sample which was supported by the transducers at its opposite corners. The displacements have a maximum in the corners.

The RUS is promising for measurement small specimens (of the order 1mm) of single crystals, namely for evaluation of elastic properties temperature dependence. We have been testing RUS technique on glass (isotropic), Si and Cu (cubic) specimens with perspective to elastic properties measure of each individual phase of studied shape memory alloys.

Glass prismatic specimens of typical dimensions $(2.3\times2.9\times3.8)\mathrm{mm}^3$ were used for error estimation of resonance measurement, Fig.6 a. The elastic constants $C_{11} = 82.0407$, $C_{12} = 23.5666$ ($C_{44} = 29.237$)GPa were pre-determined by pulse-echo method and mass density $\rho =$



Obr. 3 Configurations for acoustoelastic measurement in the cubic crystal

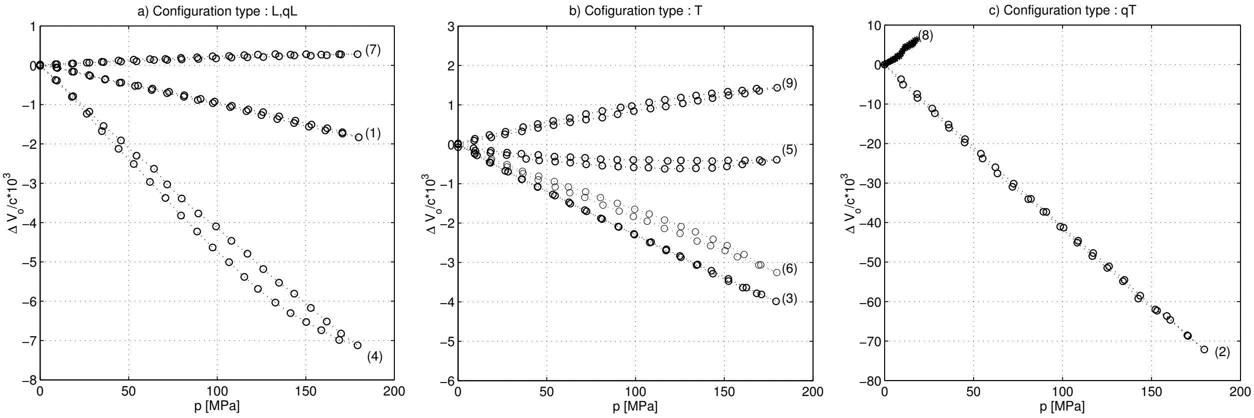
$2.4599\mathrm{g/cm^3}$) was measured by Archymedes technique. Eigenfrequencies were calculated using FEM system PMD (Package for Machine Design). The 20-nodes solid element was adopted and the mesh was suggested as $10 \times 10 \times 10$ elements. The first 14 nontrivial values of the natural frequencies f_i in the frequency range from 0.3 to 1.0MHz were taken into account. The sufficient numerical accuracy were shown on deviations from double-dense meshing. The deviation of the experimental data ($f\mathrm{x_i}$) from numeric values (f_i) are depended on the magnitude of the measured resonance. The averaged values $(f\mathrm{x}_i - f_i)/f_i$ from four measured specimens also include influence of geometrical and dimensional variations, Fig.6 b. The averaged root-mean-square deviation (this way is defined a functional for minimalization) $\sqrt{(f\mathrm{x}_i - f_i)^2/f_i^2}$ is 1.3%. This is reasonable value for the termination of an inverse procedure iteration.

5 Concluding remarks

This paper shows possibilities of pulse-echo and RUS methods to measure elastic constants of single crystals. Based on the experience with both techniques, we can make following conclusions :

Pulse-echo methods

- Advantages
  - The resulting elastic properties are obtained immediately from the measurement by a simple way (direct measurement)
  - A shape and dimensions of specimen edges are not important
  - The measurement of prestressed specimen is not problem (acoustoelasticity measurement)
  - Measurement accuracy is possible improve by means of advance digital signal processing
- Disadvantages
  - Minimal sample dimension (about 10mm)



Obr. 4 Relative changes in the natural wave velocities during applied unidirectional compression; a) L-wave: (4); $c = c_L = 4.4992\mathrm{mm}/\mu\mathrm{s}$, qL-wave: (1), (7); $c = c_{qL} = 5.7147\mathrm{mm}/\mu\mathrm{s}$, b) T-wave: $c = c_T = 3.6871\mathrm{mm}/\mu s$, c) qT-wave: $c = c_{qT} = 1.0859\mathrm{mm}/\mu s$.

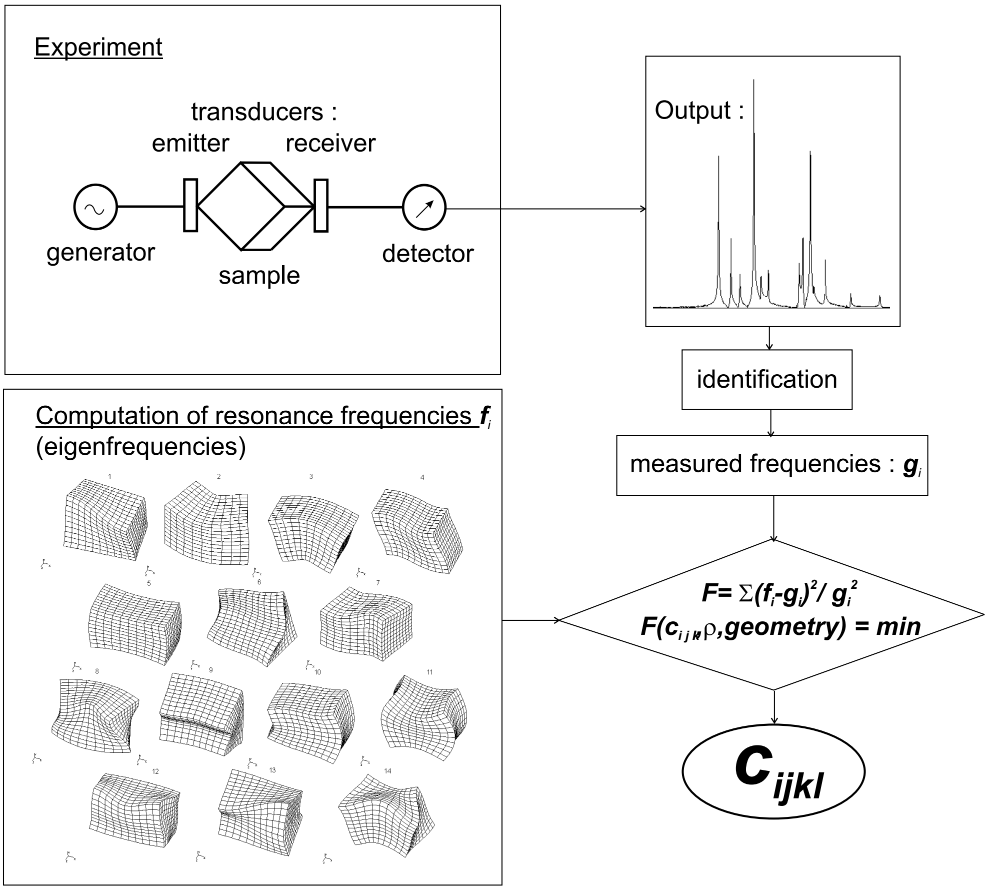
- The sufficient amount of planparallel cuts with respect to principal material axes (usually more than one type of specimen) is needed
- The broadband measurement is typically in the range of 10MHz (a wave diffraction from a single aperture in anisotropic solids may be important)
- Influence, stability and reproducibility of acoustic coupling layer between the transducer and the specimen

Resonant Ultrasound Spectroscopy

- Advantages
  - Small samples are suitable (up to under 1mm)
  - All independent elastic moduli are determined from one measurement on one specimen
  - The dependence of elastic moduli on an external condition (temperature, humidity, etc.) is measured by a simple way
  - This technique is not only usable to measure of elastic constants, but other application in nondestructive testing and utilization of non-linear acoustics are investigated
- Disadvantages
  - The method is inversive (higher demands to analysis and computation)
  - Elastic constant measurements expect high quality of resonances (low material damping)
  - The sample geometry and dimensional accuracy have a great influence on results
  - The preliminary vibration analysis is necessary to avoid multiply eigenvalues
  - Some resonant modes may be underexcitated ("hide" modes)
  - The analysis expects stress-free boundary condition. The elasticity stress dependence measurement is a problem.

Acknowledgements

The authors wish to express their thanks to Ing. Jan Zídek for support of experiments, Mr.Vladimír Novák (Inst. of Physics AS CR) for the specimens preparation and Ing. Radek Kolman for the numerical computation by PMD. This work has been supported by Czech Grant Agency under the Post-doc. project No. 106/00/D106 and project No. 106/01/0396.



Obr. 5 Resonant ultrasound spectroscopy

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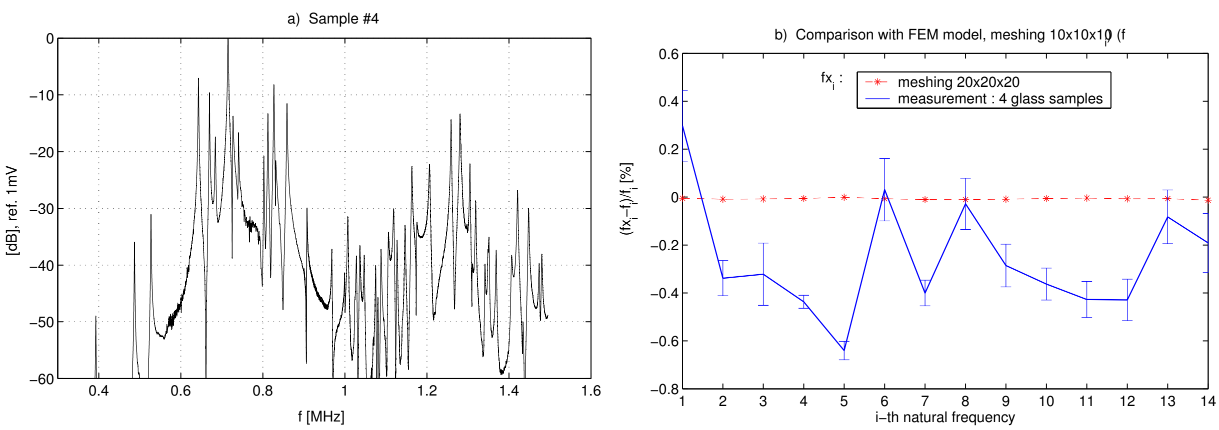

Obr. 6 a) Typical amplitude-frequency response; b) Comparison of measured and computed frequencies.