



# Determination of elastic constants via the impulse excitation method

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## 1. Introduction

Elastic constants are the most fundamental mechanical properties of linear elastic solid materials. They are defined by a linear constitutive equation called Hooke's law [1]. More specifically, the elastic behavior of isotropic linear elastic solids is completely described by only two independent elastic constants from the set of elastic moduli (Young's modulus, shear modulus and bulk modulus, all with dimensions [GPa]) and Poisson's ratio (a dimensionless ratio) [1]. Elastic constants can be determined by static methods (e.g. three- or four-point bending tests) or by dynamic methods (e.g. acoustic wave propagation or resonant frequency techniques) [2]. Also, the impulse excitation technique is a dynamic method, from which the resonant frequency can be obtained. Using dynamic methods adiabatic elastic constants are obtained, in contrast to static methods, by which isothermal elastic constants are obtained. Only for the shear modulus adiabatic and isothermal values are identical, while for Young's modulus and the bulk modulus the adiabatic values are always slightly higher (order of magnitude 1%) [3]. The elastic constants of heterogeneous materials are called effective elastic constants. They are dependent on the phase composition (volume fraction of the individual phases) and the microstructure, mainly the porosity (volume fraction of the pore space). When the phase composition is known, the elastic moduli, e.g. Young's modulus, of the dense material can be calculated rather accurately [4-8], while the porosity dependence can be estimated only very roughly (by rigorous upper bounds and tentative predictions in the form of model relations [4,9,10]). Even for these rough estimates, however, the (total) porosity must be known. Therefore, this laboratory work includes the determination of bulk density (and open porosity) via the Archimedes method and the calculation of the total porosity based on a knowledge of the true (theoretical) density of the solid phase (dense solid material).

## 2. Determination of bulk density and porosity

The bulk density  $\rho$  defined as the mass of a (possibly porous) solid body  $m_S$ , divided by its total volume  $V$ , which is the sum of the volume of the solid phase (matrix or skeleton)  $V_S$  and the volume of the pore space  $V_P$ , which consists of closed pores with volume  $V_{PC}$  and open pore space with volume  $V_{PO}$ :

$$\rho = \frac{m_S}{V} = \frac{m_S}{V_S + V_P} = \frac{m_S}{V_S + V_{PC} + V_{PO}}.$$

For bodies with simple geometric shapes (e.g. rectangular bars or plates, or cylindrical rods or disks) without major external defects the bulk density can in principle be determined just by weighing the dry sample and calculating the volume from the dimension measured by a slide caliper. However, the Archimedes method [11] has the advantage that it provides not only the bulk density, but also the apparent (i.e. open) porosity. For this purpose, in addition to the dry sample, also the water-saturated sample is weighed both in air and in water. The bulk density is then calculated as

$$\rho = \frac{m_S}{m_{WSA} - m_{WSW}} \cdot \rho_W ,$$

where  $m_{WSA}$  is the weight of the water-saturated porous body in air (obtained on an analytical balance after gently wiping off the surface with a wet cloth in order to remove excessive water),  $m_{WSW}$  is the weight of the water-saturated porous body in water (obtained by weighing the fully immersed sample using a hydrostatic balance) and  $\rho_W$  is the density of water (in principle, another liquid could be used as well, e.g. petroleum, but water is much more convenient and should be used wherever possible). According to the relevant international or national standards (e.g. ASTM standard B962-17), the body is usually boiled for 2 h in order to infiltrate the body (more precisely, its open pore space) with water. Boiling, or alternatively the application of a vacuum, efficiently supports the infiltration process, because the water can enter the narrow pores not only as a liquid, but also in the gaseous state as water vapor. The sample is usually left in water for 24 h, during which it is cooled down to room temperature.

The open porosity (also called apparent porosity) is defined as the ratio of the volume of open pores to the volume of the whole sample, i.e.

$$\Phi_{open} = \frac{V_{PO}}{V} .$$

Also, this quantity can be determined via the Archimedes method, using the relation

$$\Phi_{open} = \frac{m_{WSA} - m_S}{m_{WSA} - m_{WSW}} .$$

Note that the total porosity  $\Phi$ , defined as the ratio of the volume of closed and open pores together to the total porous body volume, cannot be determined by the Archimedes method alone. It can, however, be calculated via the relation

$$\Phi = \frac{V_P}{V} = 1 - \frac{\rho}{\rho_0} ,$$

when the bulk density has been measured and the true (or theoretical) density  $\rho_0$  of the solid phase (dense material) is known (either from the literature or an independent measurement via helium pycnometry). Note that  $\Phi = \Phi_{closed} + \Phi_{open}$  and that  $\rho_r = 1 - \Phi$  where  $\rho_r$  is the relative density (dimensionless or reduced density), defined as

$$\rho_r = \frac{\rho}{\rho_0} .$$

### 3. Young's modulus and its measurement via the impulse excitation method

Ceramic materials are known for their brittleness (i.e. very low values of fracture toughness), at least as long as the temperature is not too high (typically a few hundred °C). That means, they can sustain only small deformations, and fracture occurs as soon as a certain critical level of stress is exceeded (this critical stress level corresponds to the strength). Up to the fracture, most ceramic materials may be considered as linear elastic materials and behave according to Hooke's law, i.e.

$$\sigma = E \cdot \varepsilon ,$$

where  $\sigma$  is stress,  $E$  Young's modulus and  $\varepsilon$  strain. To describe elastic behavior of isotropic materials completely, one has to know at least two independent elastic constants.

The impulse excitation method is a dynamic and non-destructive method of measurement [12]. Its principle consists in the excitation of vibrations in a sample by impact with a small projectile or flexible hammer. These vibrations are recorded by a microphone and the corresponding signal (a damped oscillation) is transformed by fast Fourier transform (FFT) into a frequency spectrum, from which the elastic constants can be calculated, based on the (fundamental modes of the sample) resonant frequencies. For long bars or rods there is only measured one resonant frequency (usually from a flexural or longitudinal vibration), while for thin disks there are two resonant frequencies (an axisymmetric flexural one and a non-axisymmetric so-called anti-flexural one, which is sometimes called torsional). In the former case the Poisson's ratio is essentially irrelevant, so that for sufficiently long samples Young's modulus can be calculated without knowledge of the Poisson's ratio, whereas in the latter case two elastic constants, usually the Poisson's ratio and Young's modulus, are measured at the same time. The measurement equipment used in this laboratory work is the "Resonant Frequency and Damping Analyzer" RFDA 23 (IMCE N. V., Belgium). The measurement itself can be performed in a way to obtain flexural or longitudinal vibration. Proper attachment with respect to the nodal planes (0.224 of the specimen length from either side) is desired (but not necessarily essential) for obtaining the resonant frequency of long bars or rods (note, however, that for obtaining the damping the precise fixation in the node planes is absolutely essential [5]). The cross-wire fixation used for thin disks necessarily suppresses the axisymmetric flexural vibration, which makes these measurements more tricky. The excitation is both cases performed with a special flexible hammer or automatic projectile equipment, such than the sample is hit essentially by a free-flying bullet or projectile.

Relations for the calculation of Young's modulus are different for different sample geometries and types of vibration. In particular, the relations for calculating Young's modulus of a rod and bar, respectively, from flexural vibrations are

$$E = 1.6067 \left( \frac{l^3}{d^4} \right) (m \cdot f_f^2) T_1 ,$$

$$E = 0.9465 \left( \frac{l^3}{t^3} \right) \left( \frac{m \cdot f_f^2}{b} \right) T_2 ,$$

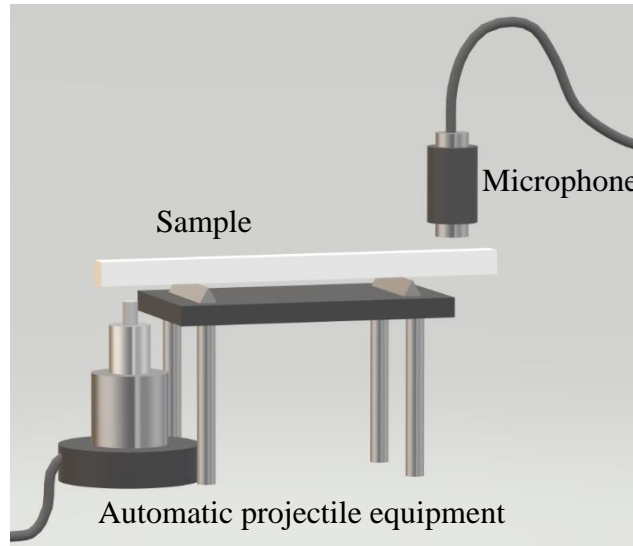
where  $l$  is length,  $m$  mass,  $d$  diameter,  $t$  thickness,  $b$  width and  $f_f$  is the fundamental flexural frequency.  $T_1$  and  $T_2$  are correction factors which is a (weak) function of diameter, mass (i.e. the aspect ratio) and Poisson's ratio. For sufficiently long sample this correction factor is very close to unity, i.e. can be set to 1. In this case the accurate value of the Poisson's ratio is not required to determine an accurate value of Young's modulus.

In the case of thin disks, the resonant frequency ratio of flexural and anti-flexural vibrations is used to determine first the Poisson's ratio (from a numerical solution that is given in the ASTM standard E1876-15 in tabular form). Then, based on the Poisson's ratio, Young's modulus is calculated using the relations given in the ASTM standard both for the flexural and the anti-flexural vibrations, and finally the arithmetic average of the two Young's modulus values, which should be very close to each other, is calculated as the final result. Then, if the material is isotropic all other elastic constants can be calculated, in particular the shear modulus and bulk modulus using the relations

$$G = \frac{E}{2(1 + \nu)},$$

$$K = \frac{E}{3(1 - 2\nu)},$$

where  $\nu$  is Poisson's ratio and  $G$  and  $K$  are shear and bulk modulus, respectively.



**Fig. 1:** Arrangement of rectangular sample during measurement of flexural vibrations.

#### 4. Porosity dependence of elastic constants

The effective properties of heterogeneous and porous materials are in the vast majority affected by the microstructure. The microstructure of material is a result of its technological processing starting from raw materials.

In particular, mechanical properties, including elastic moduli, are significantly influenced by porosity. However, they can be predicted only to a certain degree.

For all porous materials (with arbitrary microstructure, isotropic or anisotropic, random or periodic etc.) Young's modulus  $E$  is bounded from above by the upper Paul bound (also called Voigt bound)

$$E \leq E_0(1 - \Phi),$$

where  $E$  is the effective Young's modulus of the porous material,  $E_0$  is Young's modulus of the solid phase (dense, i.e. pore-free material) and  $\Phi$  the porosity.

It is useful to work with relative values of Young's modulus  $E_r$  instead of absolute values, especially when materials of different composition are compared

$$E_r = \frac{E}{E_0},$$

so the previous inequality can be written as

$$E_r \leq 1 - \Phi,$$

If the microstructure of the porous material is isotropic, the value of Young's modulus is limited by the upper Hashin-Shtrikman bound

$$E_r \leq \frac{1 - \Phi}{1 + \Phi}.$$

These upper bounds provide maximum limit values which cannot be exceeded. That means, values that exceed these bounds are wrong, i.e. incorrectly measured. This is sometimes a useful guideline, but is of course far from any prediction, since the corresponding lower bounds do not exist for porous materials (in contrast to composites). The advantage of these bounds is that they are model-independent, universal laws of nature. Other model-independent relations of this type do not exist.

More concrete, but only tentative, predictions can be made by relations than require model assumptions (model relations), mainly concerning the shape of pores (if existent). When other more concrete information is lacking, the most reasonable model choice is the assumption of spherical pore shape. The exact solution for a single spherical pore in an infinitely extended solid is

$$E_r = 1 - 2\Phi.$$

This is an exact solution, but for an idealized (i.e. unrealistic) situation (one single pore). However, the lower the porosity (volume fraction of pores), the closer is this situation realized. Therefore, from the viewpoint of real materials, this is called a linear approximation. According to this linear approximation the value of Young's modulus becomes zero at and above 50% porosity, and it can be expected to provide a good prediction only for sufficiently low porosities. It is clear that for realistically high porosities, nonlinear relations are needed. In rational engineering science all of these nonlinear relations must fulfil two requirements: First, they must reduce to the aforementioned exact single-inclusion solution, i.e. the linear approximation, for small porosities. And second, they must never violate (i.e. exceed) the aforementioned upper bounds, not even at arbitrarily high porosity. There are only a few non-linear relations that fulfill these requirements. For spherical (and isometric) pores these are e.g. the power law relation

$$E_r = (1 - \Phi)^2,$$

next the Pabst-Gregorová exponential relation [9,10]

$$E_r = \exp\left(\frac{-2\Phi}{1 - \Phi}\right),$$

and the so-called sigmoidal average (of the Hashin-Shtrikman bounds) [13]

$$E_r = \frac{(1 - \Phi)^2}{1 + \Phi}.$$

Up to this point all relations are parameter-free predictions. Sometimes, however, it is useful to fit the measured values by a curve. In principle, there is an infinite number of possible fit curves. The simplest one is the Pabst-Gregorová fit relation [9]

$$E_r = (1 - \Phi) \left( 1 - \frac{\Phi}{\Phi_{crit.}} \right),$$

where  $\Phi_{crit.}$  is fitting parameter which represents a critical porosity, i.e. the value of porosity at and above which Young's modulus becomes zero.

### Tasks:

1. Determine the bulk density and open porosity (apparent porosity) of pre-boiled samples based on Archimedes method, and calculate the total porosity from the bulk density and the true (theoretical) density.
2. Measure the value of Young's modulus of samples by impulse excitation method at least three times. Check the reproducibility. The final value will be their average.
3. Compare the measured values graphically with the Voigt and upper Hashin-Shtrikman bounds and the predictive models (linear approximation, power law, exponential relation and sigmoidal average of the Hashin-Shtrikman bounds). Comment the differences.
4. Determine the value of the critical porosity by curve fitting with the help of Solver Microsoft Excel add-in. What does this value mean?
5. For disk-shaped samples determine all elastic constants (Young's, shear and bulk modulus and Poisson's ratio).
6. Aforementioned tasks will be included in the laboratory report (.doc file) which will be submitted via email within one week. Laboratory report includes short principle of measurement, experimental procedure (step by step), measured and calculated values with all equations and their discussion and conclusion.

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