BRITISH STANDARD

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Thickness measurement of coatings and characterization of surfaces with surface waves —

Part 1: Guide to the determination of elastic constants, density and thickness of films by laser induced surface acoustic waves

The European Standard EN 15042-1:2006 has the status of a British Standard

ICS 17.040.20

BSI
British Standards

National foreword

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The UK participation in its preparation was entrusted to Technical Committee STI/33, Electrodeposited and related coatings, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Summary of pages

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English Version

Thickness measurement of coatings and characterization of surfaces with surface waves - Part 1: Guide to the determination of elastic constants, density and thickness of films by laser induced surface acoustic waves

Mesure de l'épaisseur des revêtements et caractérisation des surfaces à l'aide d'ondes de surface - Partie 1 : Guide pour la détermination des constantes élastiques, de la masse volumique et de l'épaisseur des films à l'aide d'ondes acoustiques de surface générées par laser

Schichtdickenmessung und Charakterisierung von Oberflächen mittels Oberflächenwellen - Teil 1: Leitfaden zur Bestimmung von elastischen Konstanten, Dichte und Dicke von Schichten mittels laserinduzierten Ultraschall-Oberflächenwellen

This European Standard was approved by CEN on 2 March 2006.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 15042-1:2006) has been prepared by Technical Committee CEN/TC 262 “Metallic and other inorganic coatings”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2006, and conflicting national standards shall be withdrawn at the latest by October 2006.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This document gives guidance on methods of determining the elastic constants, density and thickness of thin films by laser-induced surface acoustic waves.

It defines terms and described procedures.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 11145:2001, *Optics and optical instruments — Laser and laser-related equipment — Vocabulary and symbols* (ISO 11145:2001)

International Vocabulary of Basic and General Terms in Metrology, 2nd Edition 1994, Beuth Verlag GmbH Berlin Wien Zürich

3 Terms and definitions

For the purposes of this document, the terms and definitions given in the International Dictionary of Metrology (VIM), EN ISO 11145:2001 and the following apply.

3.1
surface acoustic waves
ultrasonic wave propagating along the surface of the material

NOTE An important property of this wave is the penetration depth into the material, which depends on frequency.

3.2
phase velocity
velocity at which the phase of the wave propagates

3.3
group velocity
velocity at which the surface acoustic wave impulse induced by the laser propagates

3.4
dispersion
dependence of the phase velocity on the frequency of the wave

3.5
dispersion relation
ratio of angular frequency to the amount of the wave vector (wave number)

3.6
dispersion degree
difference between phase and group velocity

NOTE The dispersion degree is expressed as a percentage.

3.7
bandwidth
frequency range of the amplitude spectrum

3.8
measuring length
distance between the positions at which the dispersion curve is measured

3.9
thermo-elastic inducing
inducing a surface acoustic wave by locally rapid heating of the test material as the result of absorbing a pulsed laser radiation

4 Symbols and abbreviations

a	half length of the side of membrane for the membrane deflection technique;
c	phase velocity of the surface acoustic wave;
$c(E', E, \nu', \nu, \rho', \rho, d, f_k)$	theoretical values of the phase velocity (calculated for example according [2]);
$c(f_k)$	phase velocity of the measured dispersion curve;
C_1, C_2	constants (functions of the Poisson's ratio ν);
d	film thickness;
d_S	substrate thickness;
d_N	nitriding depth;
δ	indentation depth;
Δf	frequency shift;
Δd	uncertainty of the film thickness;
ΔE	uncertainty of Young's modulus of the film;
$\Delta \nu$	uncertainty of Poisson's ratio of the film;
$\Delta \rho$	uncertainty of the density of the film;
$\Delta E'$	uncertainty of Young's modulus of the substrate;
$\Delta \nu'$	uncertainty of Poisson's ratio of the substrate;
$\Delta \rho'$	uncertainty of the density of the substrate;
E^*	Young's modulus;
E	Young's modulus of the film;
E'	Young's modulus of the substrate;
E_o	Young's modulus of the indenter;
E_I	Young's modulus determined by indenter test;
E_{LA}	Young's modulus determined by the laser-acoustic method;
f_k	frequency values of the measured dispersion curve;
f	frequency;
f_0	resonance frequency of the resonance test method;
F	force;
h	deflection of membrane deflection technique;
h_p	plastic indentation depth of the indenter test;
k	magnitude of the wave vector;
λ_{light}	wavelength of the light of Brillouin-scattering technique;
p	pressure of the membrane deflection technique;
ν^*	Poisson's ratio;

ν	Poisson's ratio of the film;
ν'	Poisson's ratio of the substrate;
ν^{o}	Poisson's ratio of the indenter;
θ	scattering angle of the Brillouin-scattering method;
ρ^{*}	density;
ρ	density of the film;
ρ'	density of the substrate;
σ_{E}	residual stress;
ω	angular frequency;
T_{A}	annealing temperature;
U	voltage amplitude.

5 Description of the method

5.1 General principles

The elastic modulus (Young's modulus) of the film essentially determines the mechanical behaviour of the coated material, the development of residual stresses, the mechanical energy induced by externally loading the coated surface, influencing creation and growth of cracks in the film and, therefore, influencing essentially the failure behaviour of the coated material.

Especially for hard coatings, Young's modulus correlates with hardness that can be measured only with increasing error for reducing film thickness.

The structure of coatings can vary within a wide range, depending on the deposition process. This accompanies a Young's modulus of the film which varies considerably. The value tabulated for the bulk material therefore is only a very rough estimation for the material deposited as film. They are given for some selected materials in Annex A. Consequently, measuring the film modulus is a method for controlling the film quality and monitoring the technological process. For measuring Young's modulus of the film, several static and dynamic techniques are used, such as the membrane deflection test, indentation test, Brillouin-scattering, ultrasonic microscopy and resonance vibration test. An overview of the principles of these alternatives is given in Annex B.

These methods are characterised to require special sample preparation, to be time-consuming, or to fail for films of sub-micrometer and nano-meter thickness.

The laser-acoustic technique is a practicable method for reproducibly determining Young's modulus of films with thickness down to less than 10 nm without special sample preparation. The technique also enables the film thickness to be measured and provides access to the film density. The method can also be used to characterise layers with gradually varying properties perpendicular to the surface as created by transition hardening and nitriding steels or machining the surface of semiconductor materials. The applicability of the method can be limited by the ultrasonic attenuation of the test material.

5.2 Surface acoustic waves

5.2.1 Properties

The test method is based on measuring the dispersion of surface acoustic waves that have a vibration component perpendicular to the surface.

Surface acoustic waves propagate along the surface of the test sample. For isotropic media, their penetration depth is defined to be the distance to the surface where the wave amplitude is decreased to 1/e of the amplitude at the surface A (Figure 1). Approximately, the penetration depth can be equated with the

wavelength λ. The penetration depth of the surface acoustic wave reduces with increasing frequency, following the relation:

$$\lambda = \frac{c}{f} \tag{1}$$

where

c is the phase velocity, in m/s;

f is the frequency, in Hz.

The phase velocity depends on the elastic constants and the density of the material.

For a homogeneous isotropic half-space, the following approximation is used

$$c = \frac{0{,}87 + 1{,}12\, v^*}{1 + v^*} \sqrt{\frac{E^*}{\rho^* \left(1 + v^*\right)}} \tag{2}$$

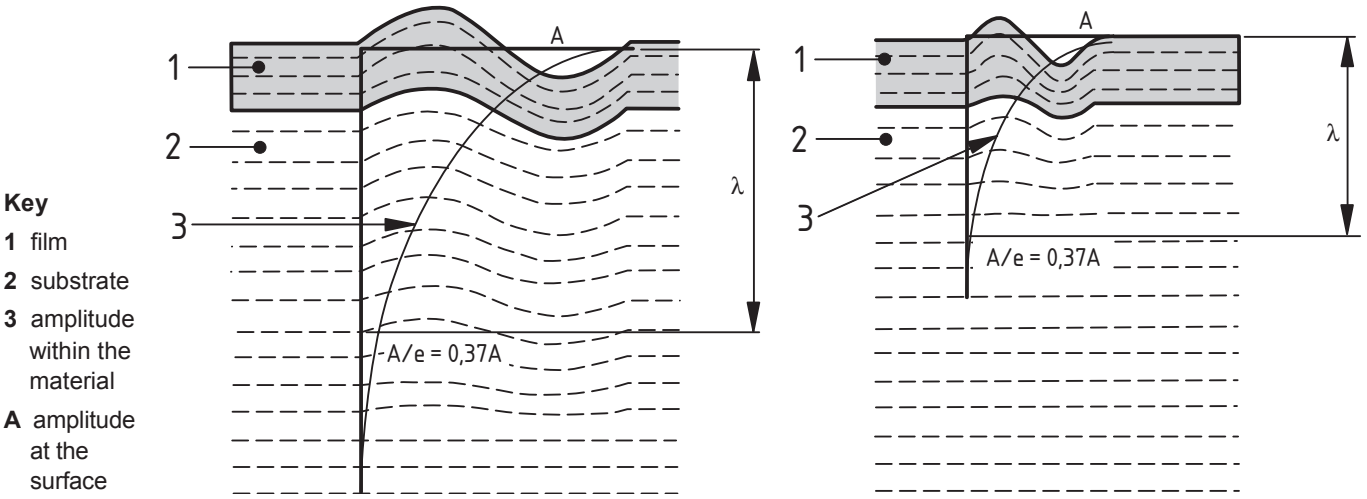
where

v^* is the Poisson's ratio;

E^* is the Young's modulus, in N/m^2;

ρ^* is the density, in kg/m^3.

Equation (2) does not apply to anisotropic materials which are more complex as described in [2].



1a) — Low frequency: long wavelength, high penetration depth, little effect of the film

1b) — High frequency: short wavelength, low penetration depth, large effect of the film

Figure 1 — Properties of the surface acoustic waves

5.2.2 Surface acoustic waves in coated materials

The surface wave velocity of a material varies by coating with a film with physical properties deviating from the substrate (see Figure 1).

It also depends on the elastic properties and the density of film and substrate material and the ratio of film thickness to wavelength. For a homogeneous isotropic film on homogeneous isotropic substrate, the following general relation applies:

$$c = \frac{\omega}{k} = c\left(E', v', \rho', E, v, \rho, d / \lambda\right) \tag{3}$$

where

c is the phase velocity, in m/s;

ω is the circular frequency, in Hz;

k is the magnitude of wave vector, in 1/m;

E' is the Young's modulus of the substrate, in N/m^2;

v' is the Poisson's ratio of the substrate;

ρ' is the density of the substrate, in kg/m^3;

E is the Young's modulus of the film, in N/m^2;

v is the Poisson's ratio of the film;

ρ is the density of the film, in kg/m^3;

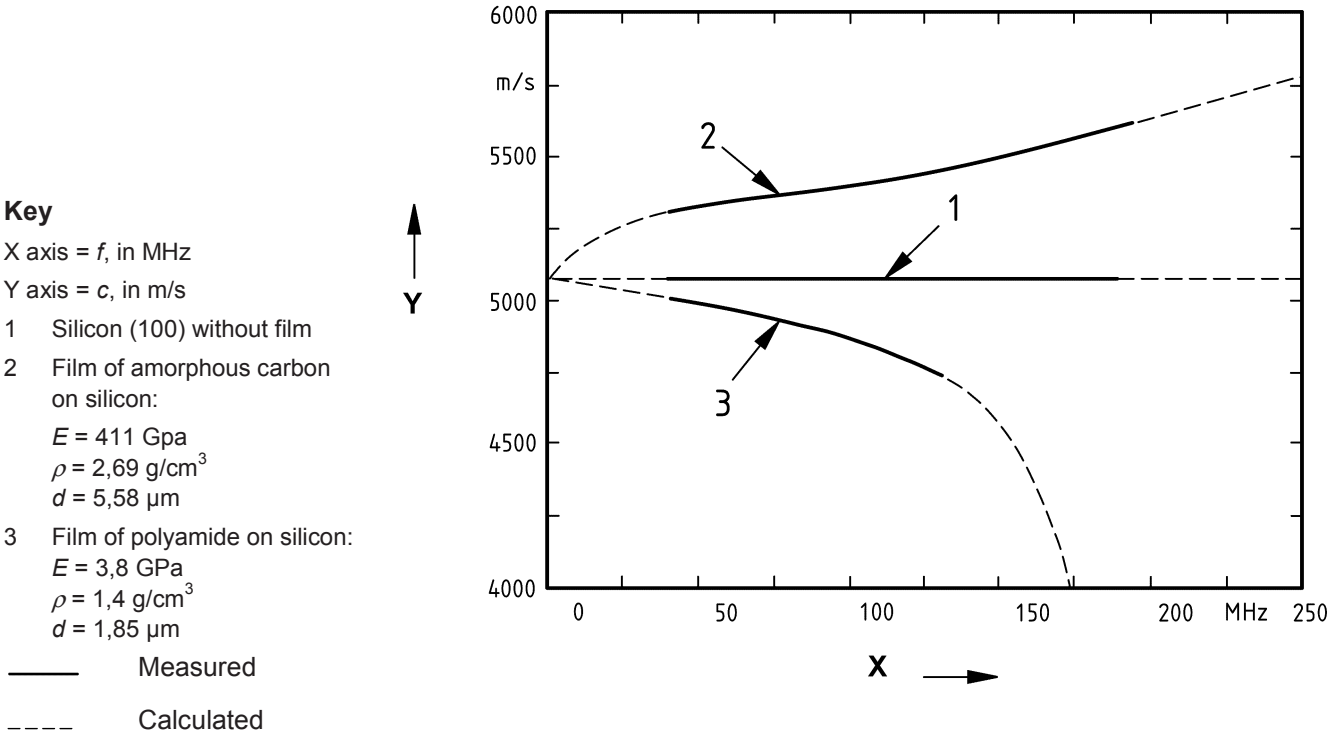
d is the thickness of the film in m;

λ is the wavelength, in m.

Equation (3) is the dispersion relation for the surface wave propagating in coated materials. The implicit form of this relation is deduced from the boundary conditions of stress and displacement components at the surface and the interface between film and substrate [2].

For anisotropic film and substrate materials, the elastic constants C_{ij} are used instead of Young's modulus and Poisson ratio.

The effect of the film on the wave propagation increases with increasing frequency of the wave due to its reducing penetration depth. This makes the wave velocity dependent on frequency. Figure 2 shows three characteristic cases.



Key

X axis = *f*, in MHz

Y axis = *c*, in m/s

1 Silicon (100) without film

2 Film of amorphous carbon on silicon:
E = 411 Gpa
ρ = 2,69 g/cm^3
d = 5,58 µm

3 Film of polyamide on silicon:
E = 3,8 GPa
ρ = 1,4 g/cm^3
d = 1,85 µm

——— Measured

– – – – Calculated

Figure 2 — Two cases of dispersion of the surface acoustic wave in coated material compared to the case of non-coated material

The film properties in Figure 2 (Young's modulus, density, film thickness) were deduced from the measured curve by the inverse solution of the dispersion relation (3). The curves can be explained as follows.

The velocity is independent on the frequency for the non-coated silicon substrate.

The diamond-like carbon film on the silicon makes the dispersion curve to increase. The wave velocity is higher for the film than for the substrate.

The dispersion curve decreases with frequency for the silicon coated by a polyamide. The wave velocity is lower for the film than for the substrate.

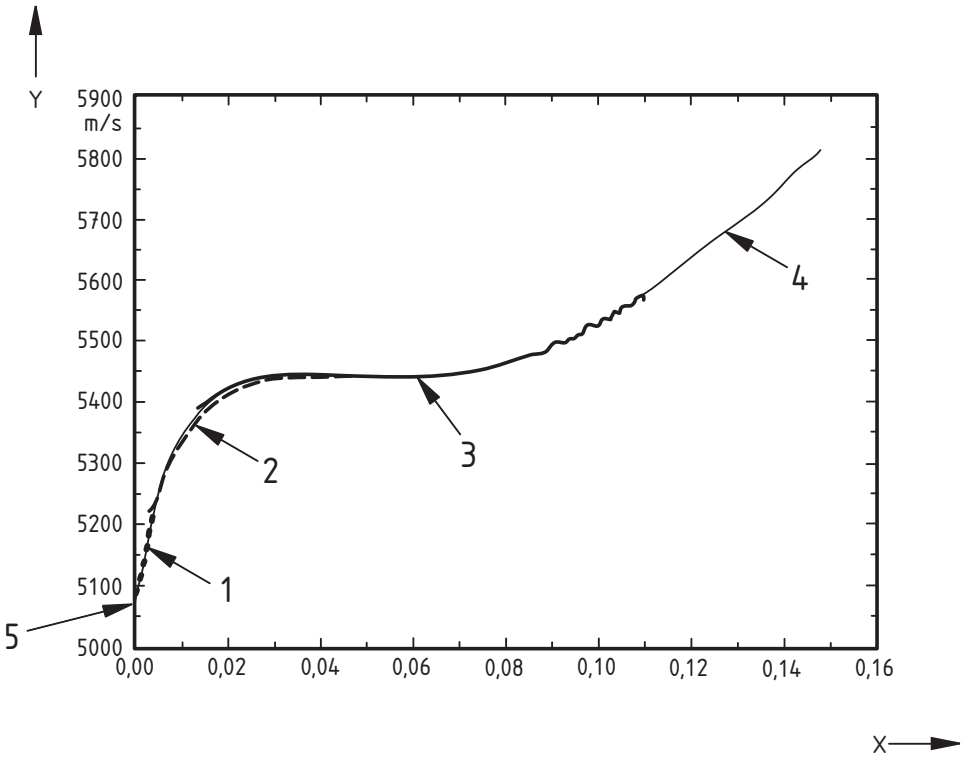
The shape of the dispersion curve characterises the film-substrate-compound. The intersection with the velocity axis at the frequency f = 0 defines the wave velocity of the substrate depending on the elastic parameters and the density of the substrate as given in relation (2) for isotropic materials.

The shape of the curve itself depends on the ratio of the elastic constants and the ratio of the density of film and substrate and on the film thickness as well.

The test method consists in measuring the dispersion curve and deducing the material parameters from the inverse solution of the dispersion relation.

For a given combination of film and substrate material, a generalised dispersion curve can be defined, depending on the film thickness normalised to the wavelength.

For the same material, all measuring points fit the same generalised curve independent of the film thickness. Figure 3 shows an example for the case of diamond-coated silicon.



Key

X axis = c, in m/s

Y axis = d/λ

1 d = 0,08 µm, measured

2 d = 0,95 µm, measured

3 d = 3,7 µm, measured

4 Theoretical dispersion curve calculated for d = 3,7 µm, E' = 1 029 GPa and ρ = 3,54 g/cm^3

5 Velocity of the surface acoustic waves for (100)-Silicon in [011]-direction

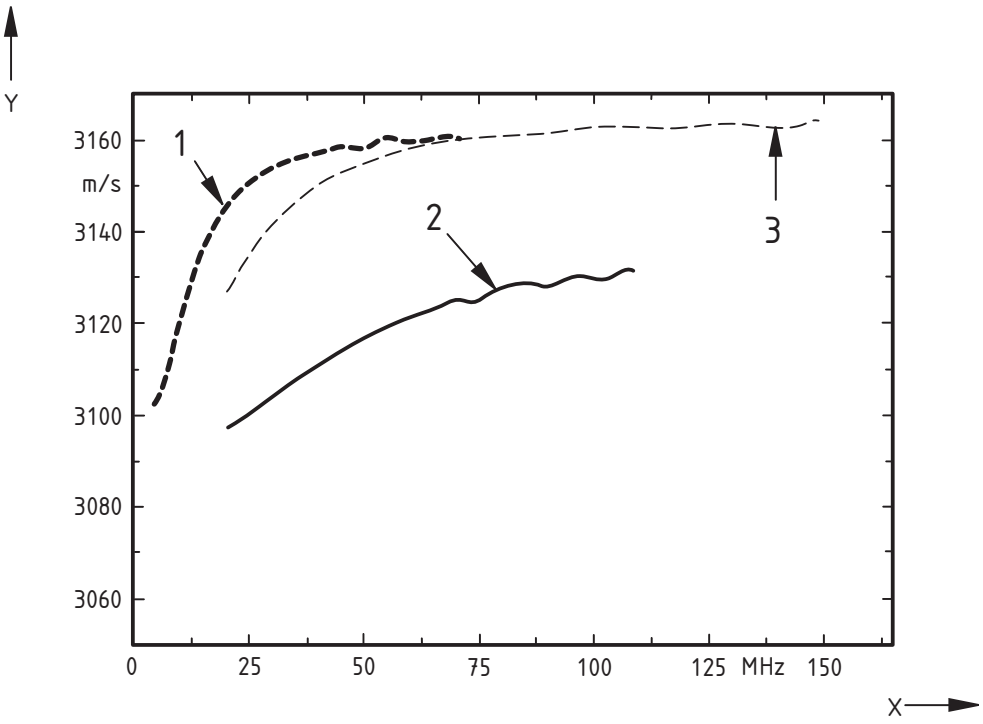
Figure 3 — Generalized dispersion curve for diamond films on silicon single crystals depending on the ratio d/λ

The curve shown in Figure 3 was measured for three samples with different film thickness. The measured segments of curve nearly exactly fit the theoretical curve. The restriction that the dispersion can only be measured with limited bandwidth, 200 MHz in this case, prevent one from measuring the complete dispersion for all film thickness. Therefore, the several measurements cover only a limited segment of the theoretical curve calculated for a wide range. Figure 3 reveals the measured curve to contain less information with reducing film thickness so that less film parameters can be obtained for thinner films.

5.2.3 Surface acoustic waves in non-homogeneously coated materials

The dispersion of surface acoustic waves can also be used to characterise surface modifications with gradually varying properties perpendicular to the surface instead of the step-like behaviour of the properties of coating. Figure 4 shows the example of a nitrided steel with three different nitriding depths. The surface treatment makes a diffusion layer with continually decreasing hardness into the material.

If a suitable theory for the surface wave propagating in gradient layers is not available, the measuring method can be calibrated by samples with known hardening depth. This enables the hardening depth to be determined non-destructively for nitrided components.



Key
X axis = *f*, in MHz
Y axis = *c*, in m/s
---- 1 d_N = 105 µm
—— 2 d_N = 80 µm
– – – 3 d_N = 60 µm

Figure 4 — Dispersion curves for nitrided steel samples with different nitriding depth

The dispersion curve has a characteristic form for a special gradient of the microstructure, which can be used for controlling the technological process. These characteristic curves should be defined, belonging to the upper and lower limit for the tolerable quality.

5.3 Measuring technique

5.3.1 Principles

Obtaining reliable information on coatings or micro-structural gradients perpendicular to the surface requires measurement of the dispersion curve with a bandwidth as wide as possible. Therefore, a spectral measuring method is used.

Short laser pulses generate thermo-elastically wide-band surface acoustic wave impulses. Having passed the distance *x*, these impulses are received by a suitable detector, for example, a piezoelectric transducer or an interferometric technique.

Figure 5 presents two surface acoustic wave pulses received at two different distances x_1 and x_2 between the focus line of the laser beam and a piezoelectric detector. The different shape of the waveform at position x_2 compared to position x_1 reveals the dispersion of the surface acoustic wave. It contains the information of the film properties.

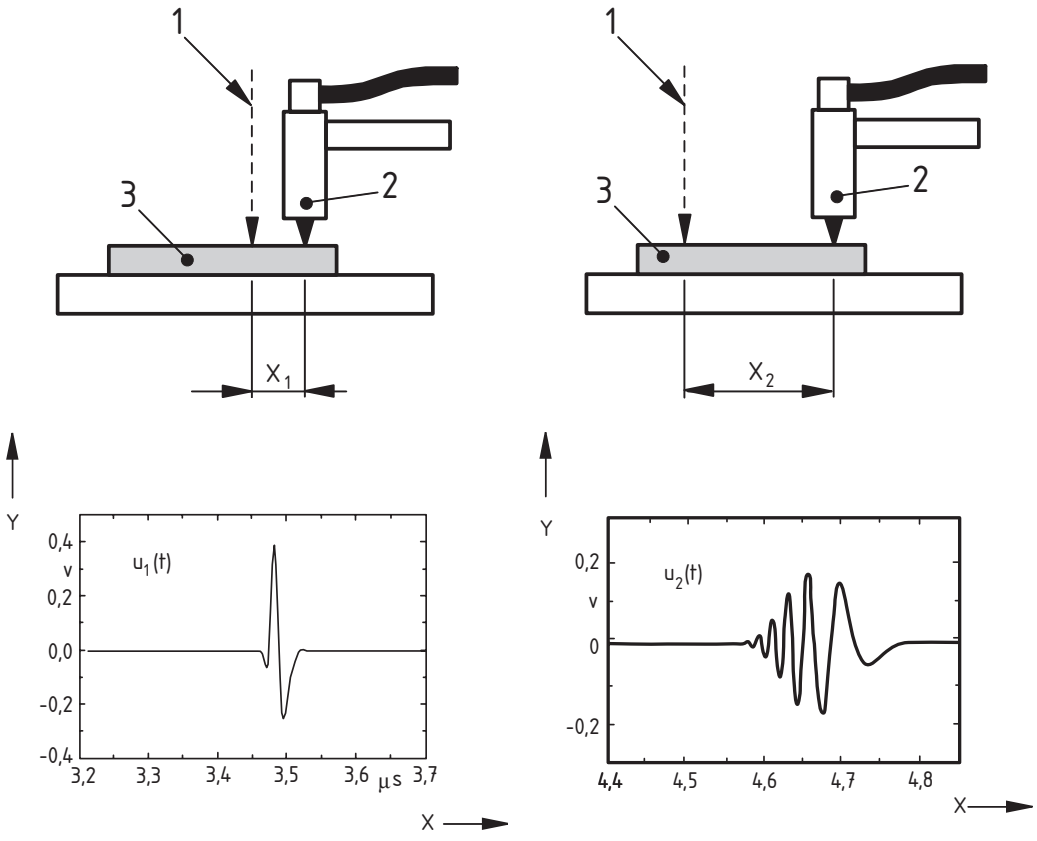
The detected impulses $u_j(t)$ (j = 1 and 2) are Fourier-transformed

$$U_j(f) = \int_{-\infty}^{\infty} u_j(t) \times \exp(i\,\omega\, t) \times d\,t \tag{4}$$

and the phase spectrum calculated as follows

$$\phi_j(f) = \arctan\left[\frac{\mathrm{Im}\,U_j(f)}{\mathrm{Re}\,U_j(f)}\right] + n2\pi \tag{5}$$

The ambiguity of the phase value n2π is determined from the dispersion degree following the procedure described in [3].



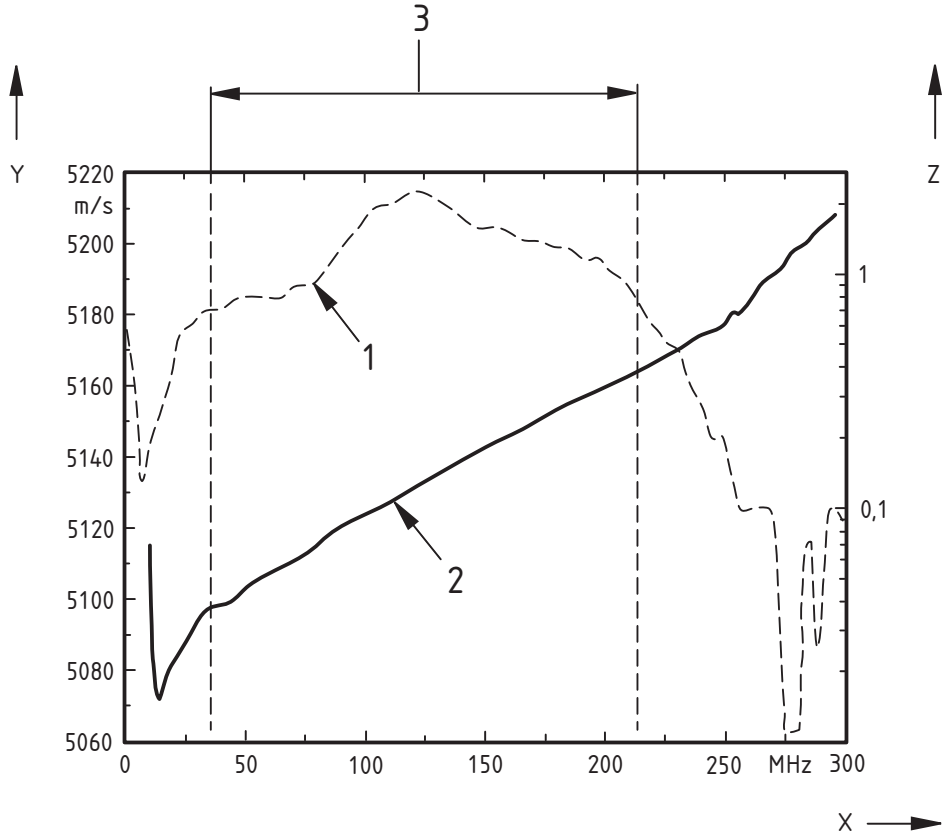
Key
X axis = t, in µs
Y axis = u, in V
1 laser beam
2 detector
3 sample
$u_1(t)$ signal, detected at distance x_1
$u_2(t)$ signal, detected at distance x_2

Figure 5 — Laser-acoustic signals for two different distances x1 and x2

The phase velocity is obtained from the relation

$$c\,(f) = \frac{(x_2 - x_1)\,\omega}{\Phi_2(f) - \Phi_1(f)} \tag{6}$$

The distance ($x_2 - x_1$) represents the measuring length. The final result is a spectrum of values of the phase velocity depending on frequency. Its frequency range is determined by the bandwidth of the surface acoustic wave impulse, illustrated in Figure 6.



Key

X axis = f, in MHz
Y axis = c, in m/s
Z axis = u, in V
1 Amplitude spectrum
2 Phase velocity
3 Frequency range of the measurement

Figure 6 — Deducing the frequency range of the measured dispersion curve from the amplitude spectrum of the impulse

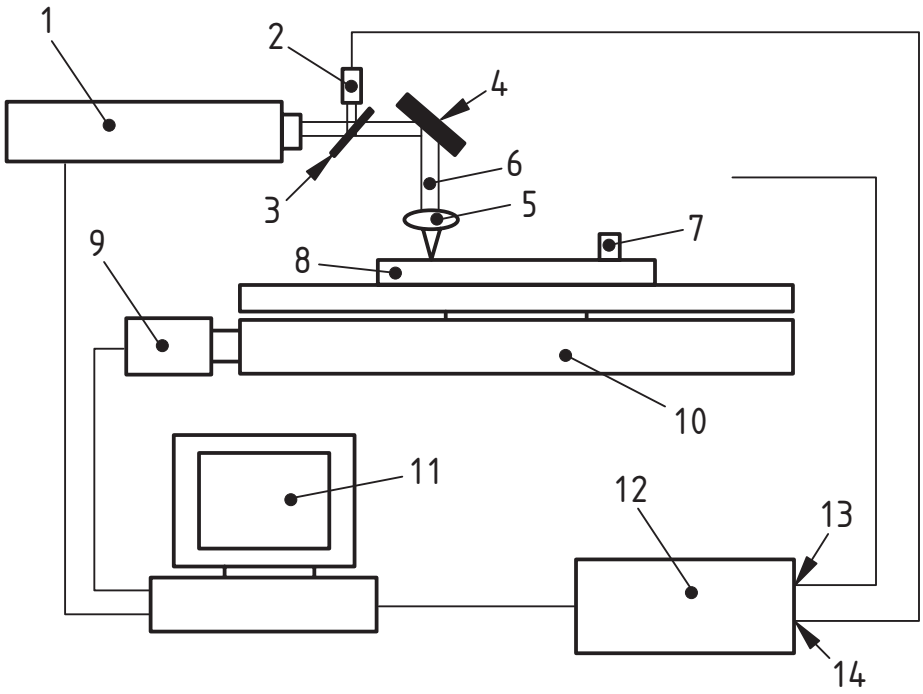
The measurement provides reliable values for the phase velocity only in the frequency range of a high enough amplitude $\left|U_{\mathrm{j}}(f)\right| = \sqrt{\left\{\mathrm{Re}\left[U_{\mathrm{j}}(f)\right]\right\}^2 + \left\{\mathrm{Im}\left[U_{\mathrm{j}}(f)\right]\right\}^2}$. This frequency range is defined by the 3 dB-bandwidth of the amplitude spectrum.

Taking into account at least two impulses received at different distances between the source of the ultrasound (position of the focused laser beam) and the detector enables a difference measuring method to be performed. In this way, the effect of the measuring device is eliminated.

5.3.2 Example of realising a measuring equipment

An example of a measuring equipment is shown in Figure 7. A nitrogen pulse laser (wavelength: 337 nm, pulse duration: 0,5 ns, pulse energy: 0,4 mJ) generates thermo-elastically surface acoustic wave impulses. The laser beam is focused by a cylindrical lens on the sample surface. The surface acoustic wave impulses are detected by a piezoelectric transducer [4].

Sample and detector are fixed together on a translation stage that moves perpendicular to the laser beam to position the detector to different distances to the laser focus. The signals are recorded with a digitising oscilloscope. A computer does the controlling and the mathematical processing. The accuracy of positioning and signal recording and the stability of the laser focus determine the uncertainty of measurement.



Key
1. Pulse laser
2. Photo diode
3. Beam splitter
4. Mirror
5. Cylindrical lens
6. Laser
7. Piezoelectric detector beam
8. Sample
9. Motor drive
10. Translations stage
11. Computer
12. Oscilloscope
13. Signal input
14. Trigger input

Figure 7 — Schematic representation of the measuring equipment

5.3.3 Test sample

Check the reproducibility using to the following conditions.

— Shape of the sample: plane surface, standard roughness R_A < 1 µm;

— Typical dimension of the area of measurement: 5 mm × 5 mm;

— Measuring smaller areas increases the measuring uncertainty.

— Minimum thickness of the substrate:

a) for silicon single crystal ≥ 0,3 mm;

b) for polycrystalline substrate ≥ 2 mm.

— Measuring the given minimum film thickness is done with following minimum bandwidth:

— Minimum thickness:

c) 5 nm: bandwidth at least 200 MHz;

d) 10 nm: bandwidth at least 150 MHz;

e) 100 nm: bandwidth at least 50 MHz;

f) 1 000 nm: bandwidth at least 10 MHz.

5.3.4 Performing the measurement

Carry out the measurement as follows.

a) Put the sample on the sample table with the measuring area in the plane defined by detector and laser focus.

b) Ensure that the laser focus and detector are so far away from the sample edge that interferences of the measuring signals with echoes from the sample boundaries do not occur.

c) Ensure that the measuring area is free of contamination.

d) Avoid material ablation at the surface caused by the effect of the laser, reducing the laser power below the ablation threshold by use of suitable filters.

5.3.5 Reproducibility

Determine the reproducibility using the following conditions:

a) recording the laser-acoustic signal with a sample rate ≥ 1 GSa/s (stability of the oscillator (clock of the time basis): ≤ 10^{-7});

b) resolution of the amplitude record: ≥ 8 bit;

c) positioning error of the laser spot on the sample surface in reference to the detector: $\Delta x \leq 1$ µm;

d) measuring distance: $x_2 - x_1 \geq 5$mm;

e) signal-to-noise ratio (SNR) of the laser-acoustic signal: ≥ 50;

f) temperature: 10 °C to 35 °C.

For repetitive measurements of the dispersions curve, the data points with frequencies ≥ 20 MHz scatter with a standard deviation of $\sigma_{n-1} \leq 1$ m/s.

5.3.6 Checking the measuring equipment

Check the measuring equipment using the following conditions:

a) performing the measurement according to the procedure in 5.3.4;

b) silicon single crystal of (100) cut as reference sample, non-coated, average roughness depth Rz < 10 nm, dispersion of surface acoustic wave not higher than 0.5 m/s within the bandwidth from 50 MHz to 150 MHz;

c) measuring direction [110];

d) at least 10 measurements;

e) calculating the mean value for the frequencies 60 MHz, 100 MHz, and 140 MHz;

The mean values shall be in the range of 5 078 m/s ± 2 m/s and the standard deviation shall be $\sigma_{n-1} \leq 1$ m/s.

6 Determination of the elastic constants, density thickness of the film

6.1 Determination of Young's modulus of the film

6.1.1 Procedure

Young's modulus of the film is calculated by the inverse solution of the dispersion relation (3). A theoretically calculated curve (for examples as described in [2]) is fitted to the measured curve by varying Young's modulus of the film and one material parameter of the substrate (for example Young's modulus).

The method of the least-square-error is used for the process of fitting.

$$\sum_{\mathrm{k}} \left[c\left(f_{\mathrm{k}}\right) - c\left(E', E, \nu', \nu, \rho', \rho, d, f_{\mathrm{k}}\right) \right]^2 \rightarrow \min \tag{7}$$

NOTE 1 For anisotropic substrate materials, the elastic constants C_{ij} are used instead E and ν. The elastic deformation of cubic materials such as silicon is described by C_{11}, C_{12} and C_{44}.

NOTE 2 The fit procedure yields a parameter of the substrate (for example Young's modulus), too.

NOTE 3 A known empirical correlation of Young's modulus with density can be used as additional condition for the fit process to fit both Young's modulus and density of the film. Figure 8 shows such a correlation found for diamond-like carbon films. Rutherford backscattering (RBS), x-ray reflectometry (XRR), and electron-energy-loss spectroscopy (EELS) were used to determine the density of the films. The error of the results for the density depends on the statistic uncertainty of this correlation. In the presented case, the uncertainty of the density is $\Delta\rho/\rho$ = ± 0,07 with a probability of 95 %.

Input:

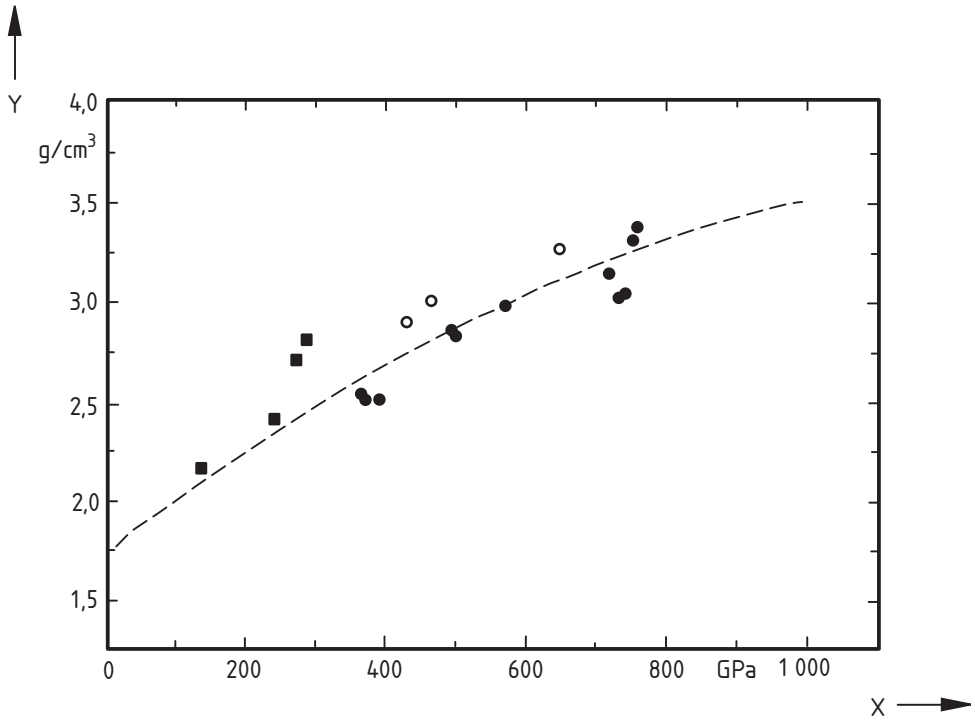
a) The Poisson ratio and the density of the substrate are put into the fit procedure.

b) The density of the film is put into the fit procedure.

c) The Poisson ratio of the film material is put into the fit procedure. If the Poisson's ratio is not known, a value typical for this class of material is put in. Its error has an effect of less then 1/20 on the final result.

The fit procedure needs start values for the material parameters involved. For some selected materials, appropriate values Young's modulus, Poisson's ratio and density are given in the table of Annex A.

NOTE 4 Annex A contains a list of elastic constants and density for some materials. These data can be used as start values for the fit procedure.



Key
X axis = ρ, in g/cm^3
Y axis = E, in GPa
Methods to measure the density:
∘ RBS
• XRR
▪ EEL

Figure 8 — Correlation of Young's modulus and density for diamond-like carbon films [6]

6.1.2 Accuracy

Keeping to the conditions

a) measuring error of the dispersion curve $\Delta c/c \leq \pm$ 0,01;

b) bandwidth B > 30 MHz;

c) number of measuring points in the dispersion curve N > 50.

and a mathematical processing following relation (7) with a theoretical curve calculated as described in [2] yield a standard deviation of s < 2,5 % for the repetitive measurement of the Young's modulus of the film that is only the results of statistic error of the dispersion curve.

The effect of the other material parameters on the result of the film modulus can usually be evaluated only numerically. Neglecting non-linear dependencies, this error can also be estimated using relation (8), assuming Young's modulus of the substrate E' to have been obtained from the measurement, too.

For determining the total uncertainty, see [19].

$$\left|\Delta E / E\right| \leq \left|\Delta d / d\right| + \left|\Delta v/(20v)\right| + \left|\Delta\rho / \rho\right| + \left|\Delta v'/(20v')\right| + \left|\Delta\rho' / \rho'\right| \quad (8)$$

6.2 Determination of the film thickness

6.2.1 Procedure

The thickness of the film is calculated by the inverse solution of the dispersion relation (3). A theoretically calculated curve (for examples as described in [2]) is fitted to the measured curve by varying the film thickness d and one material parameter of the substrate (for example Young's modulus E').

The method of the least-square-error is used for the fitting process (relation (7).

Input:

a) The elastic constants and the density of the substrate are put into the fit procedure.

b) One of these parameters can also be obtained from the fit procedure.

c) The elastic constants and the density of the film are put into the fit procedure.

Determining the film thickness requires the elastic parameters and the density to put into the procedure. For some selected materials, appropriate values of Young's modulus, Poisson's ratio and density are given in the table of Annex A.

6.2.2 Accuracy

Determine the accuracy using the following conditions:

a) measuring error of the dispersion curve $\Delta c/c \leq \pm$ 0,01;

b) bandwidth B > 30 MHz;

c) number of measuring points in the dispersion curve N > 50.

Mathematical processing using equation (7) with a theoretical curve calculated as described in [2] yields a standard deviation of s < 2,5 % for repetitive measurements of the thickness of the film that is only the results of the statistic error of the dispersion curve.

The effect of the error of the other material parameters on the result can usually be tested only numerically. Neglecting non-linear dependencies, this error can also be estimated as described in relation (9), assuming Young's modulus of the substrate E' to have been obtained from the measurement, too.

For determining the total uncertainty, see [19].

$$\left|\Delta d / d\right| \leq \left|\Delta E / E\right| + \left|\Delta \nu /(20\nu)\right| + \left|\Delta \rho / \rho\right| + \left|\Delta \nu' /(20\nu')\right| + \left|\Delta \rho' / \rho'\right| \quad (9)$$

6.3 Multi-parameter fit

6.3.1 Procedure

A wide enough bandwidth enables more film parameters to be measured. Additionally, this option depends on the film thickness and the difference of the properties of film and substrate.

Assuming, the velocity of film and substrate differs by more than 50 %, two film parameters (E and d, E and ρ, ρ and d) can be calculated if the signals have a minimum bandwidth satisfying the requirement 0,005 < d/λ < 0,2, and three film parameters (E, ρ and d) can be calculated if the signals have a minimum bandwidth satisfying the requirement 0.005 < d/λ < 0,4.

To judge the effect of the bandwidth, transferring the dispersion curve into the normalised form depending on the ratio of film thickness to wavelength d/λ is preferred, as shown for diamond films on the silicon in Figure 3.

The bandwidth is limited to higher frequency by two effects. The material attenuates the ultrasonic waves with increasing frequency. For anomalous dispersion (ascending dispersion curve), a wave of Rayleigh mode does not exist above the velocity of the transversal wave of the substrate.

The fit procedure needs start values for the material parameters involved. For some selected materials, appropriate values Young's modulus, Poisson's ratio and density are given in the Table of Annex A.

6.3.2 Accuracy of the film parameters

Keeping to the conditions

a) measuring error of the dispersion curve $\Delta c/c \leq \pm 0{,}01$;

b) normalised bandwidth for fitting two parameter $0{,}005 < d/\lambda < 0{,}2$;

c) number of measuring points in the dispersion curve $N > 50$.

and a mathematical processing following relation (7) with a theoretical curve calculated as described in [2] yield a standard deviation of $s < 10$ % for repetitive measurements of Young's modulus and density or thickness of the film that is only the result of statistic error of the dispersion curve.

d) normalised bandwidth for fitting three parameter $0{,}005 < d/\lambda < 0{,}4$.

For calculating the Poisson ratio additionally to Young's modulus of the film, a considerably higher uncertainty cannot be excluded [7].

7 Test report

Report the determination of the elastic constants, density and thickness of the surface coating. As a minimum the report shall contain the following:

a) all information necessary for identification of the sample tested;

b) a reference to this document (EN 15042-1: 2006);

c) details of the method used including:

   i) measuring accuracy of the dispersion curve;

   ii) number of measuring points;

   iii) bandwidth;

   iv) reference to the model used for the inverse solution of the dispersion relation;

   v) references of the input parameters;

   vi) list of the material parameters used for the calculation inclusive the measuring uncertainty (in the case of literature data: references);

d) results of the test, including:

   vii) at least five individual determinations and their mean;

viii) data evaluating the reproducibility of the measurement;

ix) numeric data of the measuring uncertainty of the test result, or estimation in accordance with relation (8) or (9);

x) other data usually presented in the test reports.

e) name of the person carrying out the tests;

f) any deviations from the procedure specified;

g) any unusual features (anomalies) observed during the test;

h) date of the test.

Annex A
(informative)

Material data

The values for the film parameters are deduced from fitting a theoretical curve to the dispersion curve measured. Additionally, the elastic modulus of the substrate is yielded. The fit procedure needs start values that should be as close as possible to the values expected to make sure the mathematical algorithm finds the proper solution.

Tables A.1 and A.2 summarize values for Young's modulus, Poisson's ratio and density from the literature for some materials frequently applied as coating and substrate material.

Table A.1 — Elastic parameters and density of some materials with isotropic or cubic material behaviour

Material	Young's Modulus E^*, GPa	Poisson's ratio, ν^*	Density ρ^*, g/cm^3	Literature
Fe	211	0,27	7,874	[15]
Ti	120,2	0,361	4,54	
Cr	279	0,21	7,19	
Ni	199,5	0,312	8,902	
Cu	129,8	0,343	8,96	
Zn	104,5	0,249	7,133	
Al	70,6	0,345	2,698	
Zr	98	0,38	6,506	
Mo	324,8	0,293	10,22	
W	411	0,28	19,3	
Mg	44,7	0,291	1,738	
Ge	79,9	0,32	5,323	
Sn	49,9	0,357	5,75(a) / 7,31(b)	
Au	78,5	0,42	19,32	
SiO_2 (fused silicon)	70	0,17	2,2	[16]
TiC	439,4	0,187 – 0,189	4,92 – 4,938	[17]
TiN	79,1 – 250,34	k, A,	5,43	
WC	696	k, A,	15,7	[17]
BN (cub.)	(IIa) 85,9 (IIc) 33,86	k, A,	3,49	[18]
SiC (cub./CVD)	427,59	0,183 – 0,192	3,21	
Si_3N_4 (reaction sin tered)	80 – 200	0,22 – 0,25	2,3 – 2,8	[19]
Si_3N_4 (sintered)	260 – 310	0,23 – 0,28	3,2 – 3,3	
Si_3N_4 (hot pressed)	290 – 325	0,26 – 0,32	3,16 – 3,35	

Table A.2 — Cubic materials					
Material	**C_{11}, GPa**	**C_{12}, GPa**	**C_{44}, GPa**	**Density ρ^*, g/cm^3**	**Literature**
Si	165,3	63,9	79,6	2,33	
GaAs	12,2	5,5	6,0	5,32[a]	[19]
InAs	8,3	4,5	4,0	k. A.	[23]
[a] [20]					

Annex B
(informative)

Other methods for determining Young's modulus of film materials

B.1 Membrane deflection technique

Before testing, the substrate material is removed within a special sample area to make the film a freestanding membrane. The sample is put in a chamber and a vacuum is applied, resulting in a deflection of the membrane. The deflection is measured very accurately, for example by a laser-interferometer [9]. The pressure in the chamber is varied and Young's modulus of the membrane is deduced from the load-deflection curve:

$$p = \frac{C_1 \sigma_E d}{a^2} h + \frac{C_2 E d}{a^4} h^3 \qquad \text{(B.1)}$$

Laser-acoustic test and membrane-deflection test were compared for films of polysilicon on (100)-substrate. Film thickness was 460 nm. The samples were tempered. The results of both methods are presented in Figure B.1. The results of both methods do not deviate more than 5 %. This is within the range of error estimated for both methods.

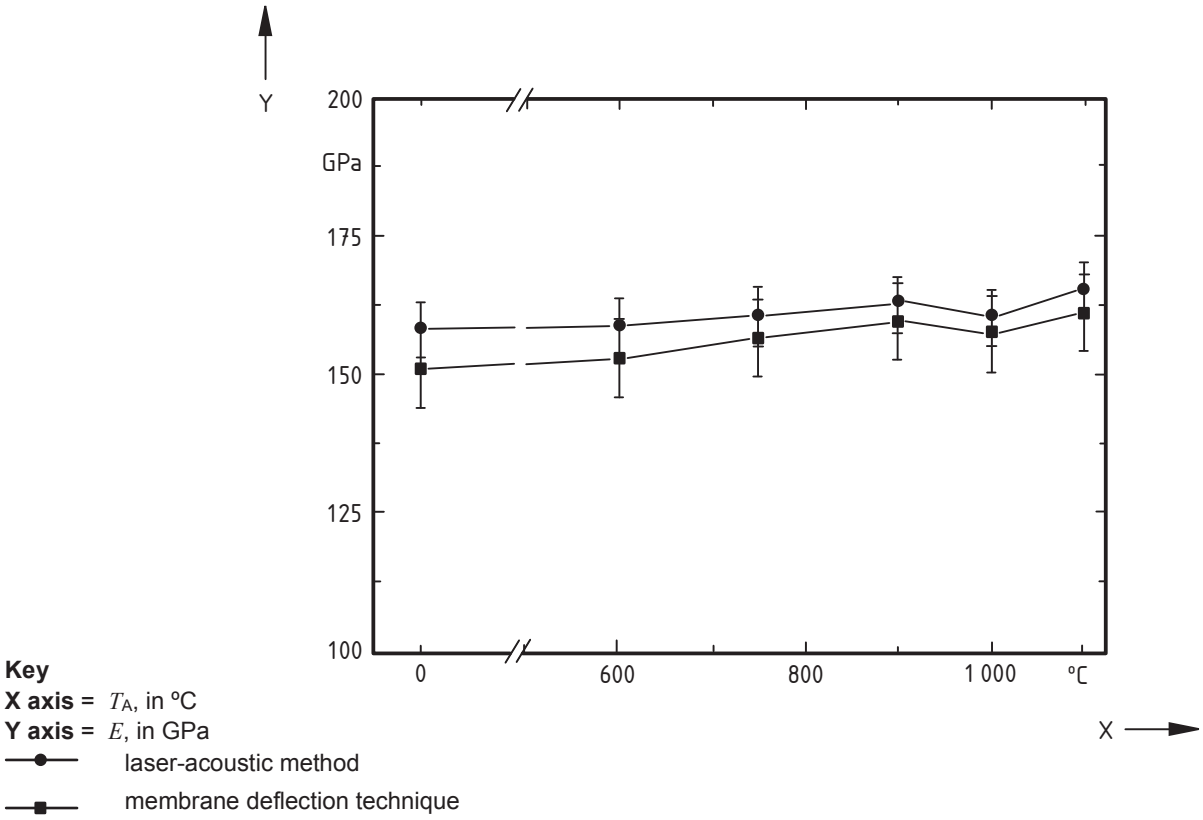


Figure B.1 — Comparing the results obtained for Young's modulus by the laser-acoustic and membrane-deflection methods, performed for films of polycrystalline silicon (film thickness: 460 nm) [8]

B.2 Indentation technique

Young's modulus can be deduced from the un-loading curve of the recorded indentation test (see ISO 14577-1 [1]). An indenter of defined geometry (usually diamond pyramids with the geometry proposed by Vickers, Knoop, or Berkovich) is pressed into the surface with a given time-dependent loading regime $F(t)$ and is un-loaded afterwards. Measuring simultaneously with the load the indentation depth δ yields the force-indentation curve $F(\delta)$, containing information of both plastic and elastic deformation [9]. Young's modulus is obtained from the slope of the un-loading curve.

It is

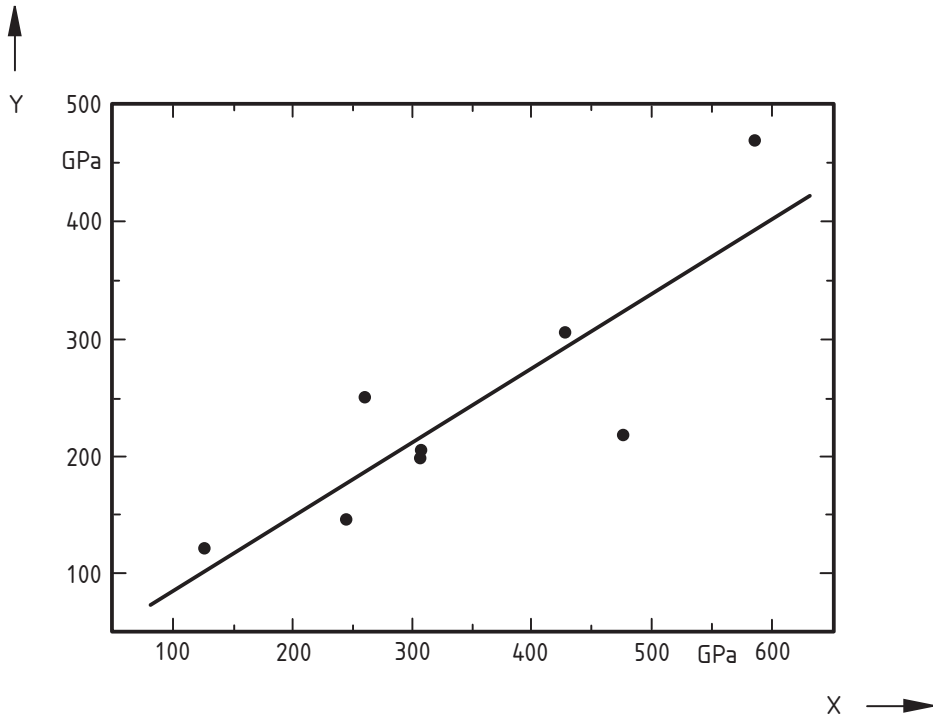
$$E_{\mathrm{r}} = \frac{1}{2h_{\mathrm{p}}}\left(\frac{\pi}{24{,}5}\right)^{0{,}5}\left.\frac{dF}{d\delta}\right|_{h_{\mathrm{p}}} \tag{B.2}$$

and

$$\frac{1}{E_{\mathrm{r}}} = \frac{1-\nu^2}{E} + \frac{1-\nu_{\mathrm{o}}^2}{E_{\mathrm{o}}} \tag{B.3}$$

The elastic-plastic deformation should not reach into the substrate. The relations applied are only valid for a plastic indentation depth of less then one tenth of the film thickness.

Figure B.2 presents comparing measurements of Young's modulus obtained by laser-acoustic and indentation method [13]. Film thickness was in the range from 1 to 2 µm.



Key
X axis = T_{LA}, GPa
Y axis = E_{I}, in GPa

Figure B.2 — Comparing results of measuring Young's modulus of diamond-like carbon films with the laser-acoustic and indentation method [13]

B.3 Resonance method

For the resonance test, the sample is induced to vibrate with its resonance frequency [10]. Young's modulus is obtained from the resonance frequency. For a bending beam coated on one side, it is

$$E = E'\,(2\Delta\, f / f_0\; d_s / d + \rho / \rho')/3 \tag{B.4}$$

Δf is the frequency shift of the resonance frequency caused by the film. d_s and d denote the thickness of substrate and film, ρ and ρ' denotes the density of substrate and film.

The sensitivity of the method to the effect of the film reduces with reducing ratio of d/d_s.

B.4 Brillouin-scattering

The Brillouin-scattering uses very high frequencies in the range of 10 GHz. It is based on the scattering of monochromatic light at lattice vibrations. This process can be treated as exchange of impulses between photons and phonons. It varies the energy (frequency) and the propagation direction of the photon. The velocity of the phonons can be deduced from [11].

$$c = \Delta F \lambda_{\text{light}} / 2 \sin \vartheta \tag{B.5}$$

Due to the low penetration depth of the inducing radiation and the short wavelength of the sound waves, the method can measure the sound velocity directly in the film also for very thin films. Using relation (2) enables Young's modulus of the film to be determined if the film density is known.

B.5 Ultrasonic microscopy

Using special piezoelectric transducers (ultrasonic lenses) of frequencies up to about 1 GHz, the ultrasonic microscope focuses an ultrasonic wave field to the sample surface. The interference of the wave reflected directly at the surface with the leaky surface acoustic wave creates a signal amplitude varying with the distance between the acoustic lens and the surface [12]. Mathematically analysing the signal yields the propagation velocity of the surface acoustic wave. The measurement is performed for a defined frequency. Creating a dispersion curve of many data points is time consuming and requires the corresponding number of lenses.

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