

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

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National foreword

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Thickness measurement of coatings and characterization of
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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.

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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of an identical text or by endorsement, at the latest by October 2006, and conflicting national standards shall be withdrawn at the latest by October 2006.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_0	Young's modulus of the indenter;
E_1	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^0	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:

$$\lambda = \frac{c}{f} \quad (1)$$

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

$$c = \frac{0,87 + 1,12 \nu^*}{1 + \nu^*} \sqrt{\frac{E^*}{\rho^* (1 + \nu^*)}} \quad (2)$$

where

ν^* is the Poisson's ratio;

E^* is the Young's modulus, in N/m²;

ρ^* is the density, in kg/m³.

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

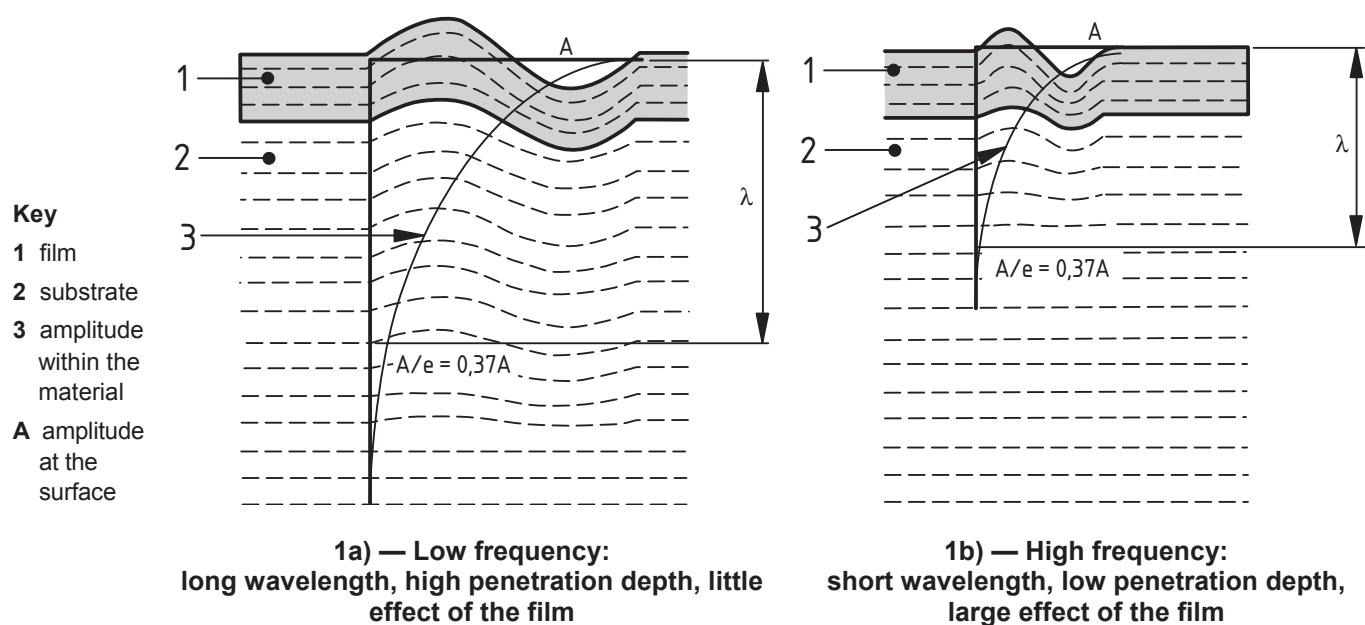


Figure 1 — Properties of the surface acoustic waves