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Merjenje debeline nanosa prevlek in karakterizacija valovitih povrģin ï 1. del: Vodilo za ugotavljanje proģnosti, specifiļ ne teģe in debeline prevlek z laserjem, ki povzroļ a valovito akustiļ no povrģino

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

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

Mesure de l'épaisseur des revetements et caractérisation des surfaces a 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 a l'aide d'ondes acoustiques de surface générées par laser

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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

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

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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of an identical text or by endorsement, at the latest by October 2006, and conflicting national standards shall be withdrawn at the latest by October 2006.

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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

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

where

ν^* 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].

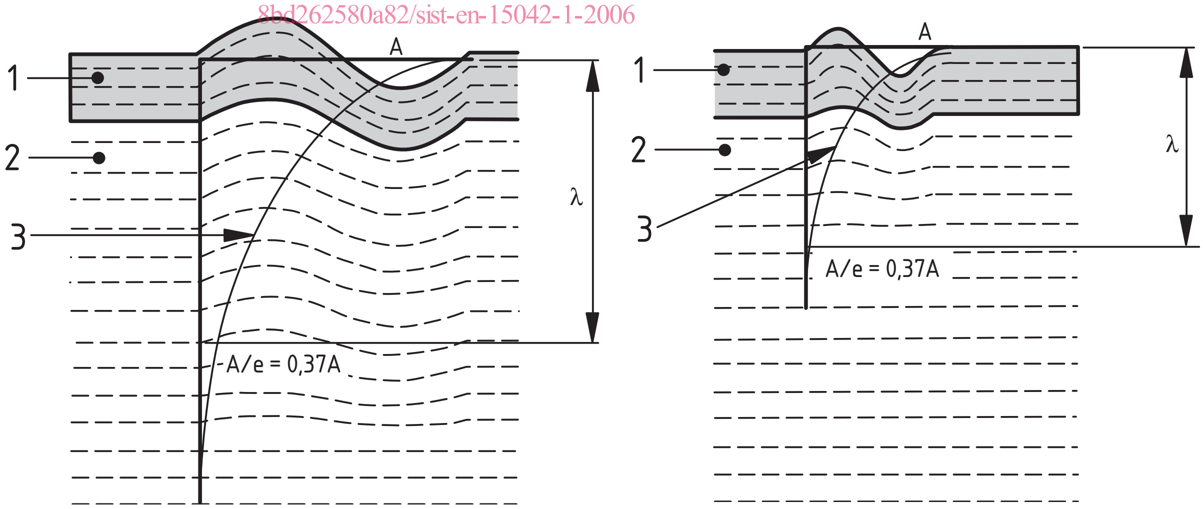
Key

1 film

2 substrate

3 amplitude within the material

A amplitude at the surface



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:

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

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;

ν' 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;

ν 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

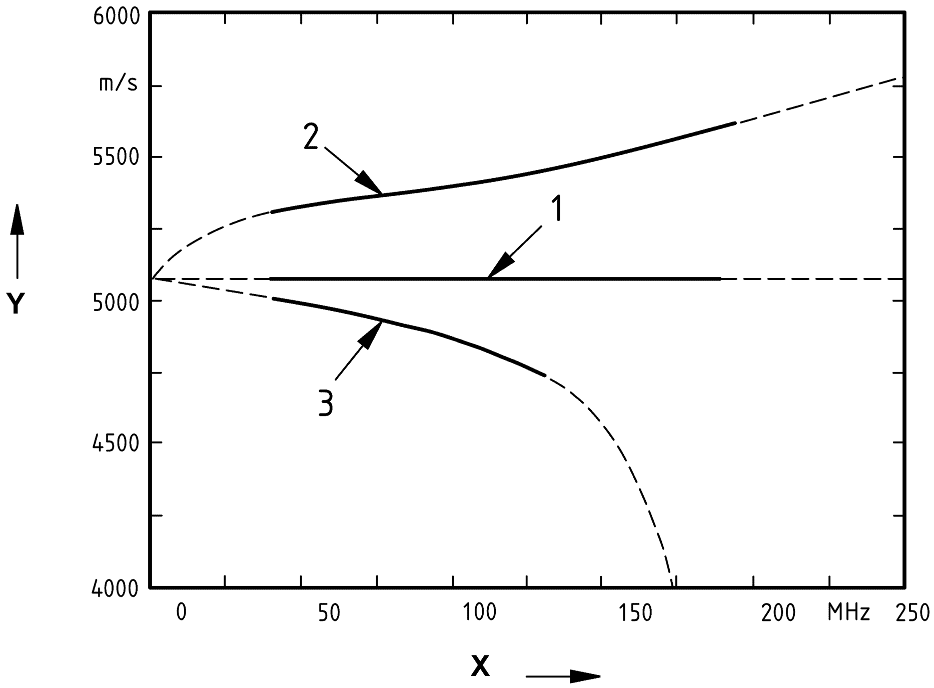


Figure 2 — Two cases of dispersion of the surface acoustic wave in coated material compared to the case of non-coated material

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.