SLOVENSKI STANDARD
SIST EN 15042-2:2006
01-september-2006

Merjenje debeline nanosa prevlek in karakterizacija valovitih površin – 2. del: Vodilo za merjenje debeline prevlek s fototermično metodo

Thickness measurement of coatings and characterization of surfaces with surface waves - Part 2: Guide to the thickness measurement of coatings by photothermic method

Schichtdickenmessung und Charakterisierung von Oberflächen mittels Oberflächenwellen - Teil 2: Leitfaden zur photothermischen Schichtdickenmessung

Mesure de l'épaisseur des revetements et caractérisation des surfaces a l'aide d'ondes de surface - Partie 2 : Guide pour le mesurage photothermique de l'épaisseur des revetements

Ta slovenski standard je istoveten z: **EN 15042-2:2006**

ICS:
17.040.20

SIST EN 15042-2:2006 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15042-2

April 2006

ICS 17.040.20

English Version

Thickness measurement of coatings and characterization of surfaces with surface waves - Part 2: Guide to the thickness measurement of coatings by photothermic method

Mesure de l'épaisseur des revêtements et caractérisation des surfaces à l'aide d'ondes de surface - Partie 2 : Guide pour le mesurage photothermique de l'épaisseur des revêtements

Schichtdickenmessung und Charakterisierung von Oberflächen mittels Oberflächenwellen - Teil 2: Leitfaden zur photothermischen Schichtdickenmessung

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

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of 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.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 15042-2:2006: E

Contents

Page

Foreword

This document (EN 15042-2:2006) 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 describes methods for the measurement of the thickness of coatings by means of thermal waves generated by a radiation source.

The method can be used for coatings whose thermal properties (e.g. thermal conductivity) are different from those of the substrates in a range from a few microns to some hundred microns.

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.

ISO/DGuide 99998, *Guide to the expression of uncertainty in measurement (GUM) – Supplement 1: Numerical methods for the propagation of distributions*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

amplitude of the thermal wave
ΔT_0
maximum local temperature variation of the oscillating part for periodic-harmonic heating processes

NOTE See Equation 2.

3.2
penetration depth of thermal waves
depth at which the temperature variation below a modulated heated surface is still measurable.

NOTE In general, the penetration depth is of the order of magnitude of the thermal diffusion length

3.3
modulation frequency
f
frequency at which the intensity of the heating radiation varies periodically

3.4
phase (phase shift) of the thermal wave
$\Delta\ \Phi$
measure of the temporal delay of the temperature oscillation relative to the excitation for periodic-harmonic heating processes

NOTE See Equation 3.

3.5
photothermal efficiency
η
proportion of the incident radiation intensity that is converted into heat

NOTE In most technical applications it is approximately identical to the absorption.

3.6
thermal diffusion length
μ
characteristic length of the thermal diffusion with pulsed heating or periodically modulated heating, where the temperature amplitude has decreased to about 1/e or 37 %

NOTE 1 1/e, with natural number e = 2,71828.

NOTE 2 See Equation 4.

3.7
thermal diffusion time
τ
characteristic time that a thermal wave or a temperature pulse requires for penetrating a layer of finite thickness

NOTE See Equation 7.

3.8
thermal diffusivity
α
thermal parameter characterizing heat propagation in a body with time-dependent heating

NOTE See Equation 6.

3.9
thermal effusivity
e
thermal parameter determining the surface temperature of a body with time-dependent heating

NOTE See Equation 5.

3.10
thermal wave
spatiotemporally variable temperature field that is set up in a body (or medium) with time-dependent heating and is described by the heat conduction equation

NOTE 1 see Equation 1.

NOTE 2 The thermal wave is generated in one limiting case by a periodic-harmonic excitation, in the other limiting case by a pulsed excitation.

3.11
thermal reflection coefficient
R_{ls}
thermal parameter that is a degree of the reflection of the thermal wave at the boundary interface between two layers of different effusivity and thus describes the heat transfer across this boundary interface

NOTE See Equation 8.

4 Symbols and abbreviation

Symbol	Unit	Description	See Equation
$\Delta T(x,t)$	K	amplitude of the temperature oscillation of the thermal wave	1
$\Delta T_0(x)$	K	amplitude of the temperature oscillation of the thermal wave at the surface (x = 0)	2
$\Delta\,\Phi$	rad	phase of the temperature oscillation of the thermal wave	3
μ	m	thermal diffusion length	4
e	$Ws^{1/2}/(m^2K)$	thermal effusivity	5
α	m^2/s	thermal diffusivity	6
τ	s	thermal diffusion time	7
F_0	W/m^2	heat flow/excitation power density	7
η		photothermal efficiency	
k	W/(m·K)	thermal conductivity	12
ρ	Kg/m^3	mass density	13
c	J/(kg·K)	specific heat capacity	13
f	s^{-1}	modulation frequency	
i_0	W/m^2	incident radiant power density	2
x	m	location below the boundary interface	
t	s	time	

5 Foundations of photothermal materials testing

5.1 Physical foundations

5.1.1 Thermal waves

The concept "thermal wave(s)" describes a spatially and temporally variable temperature field that is generated in a body by time-dependent heating. Besides the concept thermal wave the term "temperature wave" is also used in technical literature. The excitation of the spatiotemporally variable temperature field mathematically described by a diffusion equation - the heat conduction equation - can occur in the one limiting case periodic-harmonically and in the other limiting case pulsed.

The physical foundations [1], [5], [6], [7] can be derived both for the harmonic excitation and for the pulsed excitation, and are related by a Fourier transformation. This clause considers primarily the harmonic excitation; the derivation for the pulsed excitation can be found in [8].

An example of thermal waves observable in nature is the temperature distribution in the ground. This distribution is dependent on the time of day and year, with the daily variation in temperature reaching a penetration depth of nearly 30 cm and the variation in the course of the year penetrating up to several meters [9].

The example of thermal waves [10] excited by the harmonic-periodic and large-area irradiation of homogeneous, semi-infinite bodies absorbing only at the surface, can be used to describe the most important properties of thermal waves and to identify the physical variables and parameters that are measurable by means of thermal waves during materials testing.

$$\Delta T(x,t) = \Delta T_0(x) \cdot \cos\left(2\pi ft + \Delta\phi(x)\right) \tag{1}$$

NOTE Terms are defined in Clause 4.

$$\Delta T_0(x) = \frac{\eta\, i_0}{e\sqrt{2\pi f}} \exp(-x/\mu) \tag{2}$$

$$\Delta\phi(x) = -\frac{x}{\mu} - \frac{\pi}{4} \tag{3}$$

$$\mu = \sqrt{\frac{\alpha}{\eta\, f}} \tag{4}$$

The amplitude of the thermal wave (Equation 1) decreases exponentially with the depth, if the heated surface is taken to be x = 0. The measurable penetration depth has the order of magnitude of the thermal diffusion length μ. Conditioned by the frequency-dependency of the thermal diffusion length μ (Equation 4), the penetration depth can be adjusted by precisely varying the modulation frequency f of the heating. The amplitude of the thermal wave $\Delta T_0(x)$ (Equation 2) and the phase shift $\Delta\phi(x)$ (Equation 3) depend on the following thermal properties:

The thermal effusivity (thermal penetration coefficient), e, is given by the equation:

$$e = \sqrt{k\, \rho\, c} \tag{5}$$

and the thermal diffusivity, α, is given by the equation:

$$\alpha = \frac{k}{(\rho\, c)} \tag{6}$$

Accordingly, frequency-dependent measurements of the amplitude and phase of the thermal wave provide depth-resolved information on these combined thermal parameters. In Equations (5) and (6), k is the thermal conductivity, ρ the mass density and c the specific heat capacity.

The amplitude of the thermal wave measurable at the surface is proportional to the photothermal efficiency η, which specifies the proportion of the incident radiant power converted into heat.

With layered systems the amplitude and the phase shift of the temperature oscillation are determined on the one hand by the ratio of the thermal effusivity of layer and substrate $e_{\text{layer}}/e_{\text{substrate}}$, and on the other hand by the thermal diffusion time for the layer:

$$\tau_{layer} = \frac{l_{layer}^{\;2}}{\alpha_{layer}} \tag{7}$$

where

l_{layer} is the geometrical thickness of the layer;

α_{layer} is the thermal diffusivity of the layer.

Given a known value of the thermal diffusivity of the layer and a sufficiently large thermal contrast, describable according to [11] by the thermal reflection coefficient:

$$R_{ls} = \frac{e_{layer} - e_{substrate}}{e_{layer} + e_{substrate}} \tag{8}$$

contactless and non-destructive layer thickness determination is possible by means of thermal waves (see Clause 6).

5.1.2 Thermal Properties

The significance of the thermal effusivity and the thermal diffusivity can be made especially clear by means of special time-dependent heating (step function).

According to [12], the thermal effusivity e (Equation 5) is a measure of the time-dependent heating of a surface:

$$\Delta T\,(x=0,t) = \frac{2\,F_0}{e\sqrt{\pi}}\sqrt{t} \tag{9}$$

where F_0 is the constant heat flow absorbed at the surface and $\Delta T(x = 0,t)$ represents the heating of the surface at time t after the start of heating. The thermal effusivity determines the contact temperature between bodies and layers having different thermal properties. An example is the contact temperature:

$$T_{contact} = \frac{\left(e_1 \cdot T_1 + e_2 \cdot T_2\right)}{\left(e_1 + e_2\right)} \tag{10}$$

occurring at the boundary interface between two semi-infinite bodies having different thermal effusivity e_1 and e_2 and different initial temperatures T_1 and T_2, after these bodies have been brought into contact with one another.

The thermal diffusivity α (Equation 6) is a measure of the propagation of the temperature through a homogenous body:

$$\Delta T\,(x,t) = \frac{F_0}{e\sqrt{\pi}} \int_0 \exp\left(\frac{-x^2}{4\,\alpha\,t'}\right) / \sqrt{t'}\,\mathrm{d}t' = \Delta T\,(x=0,t)\sqrt{\pi}\,\mathrm{ierfc}\left(\frac{x}{\sqrt{4\,\alpha\,t}}\right) \tag{11}$$

Given measurements of the thermal effusivity and thermal diffusivity by means of thermal waves, the heat conductivity and the heat capacity per unit volume can be determined using Equations (5) and (6):

$$k = \sqrt{\alpha} \cdot e \tag{12}$$

$$(\rho\,c) = \frac{e}{\sqrt{\alpha}} \tag{13}$$

Here it shall be kept in mind that with Equations (12) and (13) effective parameters shall be determined for the actual test object that include the influence of porosity, surface roughness and anisotropy on the heat transfer [13].

5.1.3 Thermal depth profiling

The thermal diffusion length μ (Equation 4) is a measure of the penetration depth of the thermal wave. Since the thermal diffusion length and hence the penetration depth can be varied via the modulation frequency of the heating, a depth-resolved measurement of thermal properties is possible. The resolution limits basically depend on the thermal contrast of the individual layers, on the detection procedure used and on the technical quality of the detectors.

5.1.4 Measurable variables and possibilities of measurement

In principle, thermal waves can be used to measure any physical variable that affects the heat transfer and temperature distribution in a body, i.e. the spatial distribution of the thermal effusivity and of the thermal diffusivity or the layer thickness in layered systems with different thermal properties.

Accordingly, it is possible to measure directly or infer other characteristic data, such as the hardness of metallic materials, porosity and moisture in solid bodies, if these variables affect heat transfer properties. In these cases, however, the correlation of, for example, the porosity, moisture [13], [14] or hardness [15] with the effective thermal properties shall be determined through calibration.

Optical variables, such as the photothermal efficiency η and the absorption coefficient β for electromagnetic radiation, which affect the intensity and depth profile of the heat sources can also be determined.

In addition, the processing and modification of technical surfaces (e.g. by plasma etching, ion implantation, heat treatment, machining, friction wear, etc.) can be determined by means of thermal waves, if these surfaces have a measurable reactive effect on the optical or thermal properties. Using photothermal methods of measurement facilitates on the one hand quantitative determination of the features of the test object, such as coating thickness, thermal diffusivity, thermal effusivity, absorption coefficient, adhesion of layers or cracks, etc. On the other hand, monitoring of the production process, such as uniform coating thickness or implantation dose is possible.

For the surface study of test objects, thermal imaging methods are used; here, thermal waves are used pointwise to form a surface raster, or in the case of large-area modulated heating, to make time-dependent recordings with an IR camera.

5.2 Structure of a photothermal measuring system

A photothermal measuring instrument comprises components to carry out the following functions: excitation, detection and measured value processing. As a rule, the measurement data are compared with calibration data, so that quantitative statements on deviations in the production process or on deviations from the desired functional properties can be made. The only requirement on the test object (besides optical accessibility) is the supply of energy (through the absorption of radiation, for example) and its conversion into a detectable form (by increasing the thermal radiation to the level of IR detection, for example). The detectable signals (amplitude and phase) then provide information on material properties and/or their modifications in layers near the surface.

Figure 1 shows one possible way of integrating a photothermal measuring system in a production line or in the production process. Feedback coupling serves to control the production process and ensure the functional properties of a component. As a non-destructive and contactless procedure, thermal waves are suitable for testing materials and components. They can be used as an on-line measuring procedure for production monitoring and quality testing.