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Determination of the film thickness of coatings using an ultrasonic gage

Détermination de l'épaisseur du feuil de revêtement par mesurage ultrasons

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Foreword

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The committee responsible for this document is ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

Determination of the film thickness of coatings using an ultrasonic gage

1 Scope

This Technical Specification describes a method for determining the film thickness of coatings on metallic and non-metallic substrates using an ultrasonic gauge.

2 Normative references

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

ISO 4618, *Paints and varnishes — Terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and the following apply.

3.1
ultrasonic wave
acoustic wave having a frequency higher than the range of audibility of the human ear, generally taken as higher than 20 kHz

[SOURCE: EN 1330-4:2010, 3.1.1]

3.2
longitudinal wave
compressional wave
wave in which the particle motion in a material is in the same direction as the propagation of the wave

[SOURCE: EN 1330-4:2010, 2.3.1]

3.3
echo
ultrasonic pulse reflected to the probe

[SOURCE: EN 1330-4:2010, 5.5.2]

3.4
echo height
echo amplitude
height of an *echo* (3.3) indication on the screen

[SOURCE: EN 1330-4:2010, 5.5.5]

3.5
ultrasonic impulse
short-lived ultrasound signal

3.6
ultrasonic sensor
ultrasonic probe
device for sending and receiving *ultrasonic waves* (3.1), mostly based on piezoelectric materials

3.7
acoustic impedance
Z
product of sound velocity and density of a material

3.8
reflection coefficient
ratio of total reflected sound pressure to incident sound pressure at a reflecting surface

[SOURCE: EN 1330-4:2010, 3.4.11]

Note 1 to entry: For a wave the reflection coefficient R is calculated from the *acoustic impedances* (3.7) Z_1 and Z_2 of the bordering media, for which 1 is the medium of the incoming sound:

$$R = \frac{Z_2 - Z_1}{Z_2 + Z_1}$$

For a negative reflection coefficient the *phase* (3.9) of the reflected signal is changed by 180°.

3.9
phase
fraction of a complete wave cycle, expressed as an angle

[SOURCE: EN 1330-4:2010, 2.2.5]

3.10
interface
boundary between two media, in acoustic contact, having different *acoustic impedances* (3.7)

[SOURCE: EN 1330-4:2010, 3.4.1]

3.11
sound path travel time
time needed for the sound path travel distance

[SOURCE: EN 1330-4:2010, 5.6.3]

3.12
couplant
coupling film
medium interposed between the probe and the object under examination to enable the passage of *ultrasonic waves* (3.1)between them

[SOURCE: EN 1330-4:2010, 5.3.2]

3.13
A-scan presentation
display of the ultrasonic signal in which the X-axis represents the time and the Y-axis the amplitude

[SOURCE: EN 1330-4:2010, 5.5.16]

Note 1 to entry: Ultrasonic film thickness measuring devices, besides the numerical values of the obtained film thicknesses, normally display A-scans for checking the echo forms and echo sequences on a screen as well.

3.14
calibration
operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication

Note 1 to entry: A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

Note 2 to entry: Calibration should not be confused with *adjustment of a measuring system* (3.15), often mistakenly called "self-calibration", nor with verification of calibration.

Note 3 to entry: Often, the first step alone in the above definition is perceived as being calibration.

[SOURCE: ISO/IEC Guide 99:2007, 2.39]

3.15
adjustment of a measuring system
adjustment
set of operations carried out on a measuring system so that it provides prescribed indications corresponding to given values of a quantity to be measured

Note 1 to entry: Types of adjustment of a measuring system include zero adjustment of a measuring system, offset adjustment and span adjustment (sometimes called "gain adjustment").

Note 2 to entry: Adjustment of a measuring system should not be confused with *calibration* (3.14), which is a prerequisite for adjustment.

Note 3 to entry: After adjustment of a measuring system, the measuring system normally should be recalibrated.

[SOURCE: ISO/IEC Guide 99:2007, 3.11]

3.16
working standard
standard which is traceable to the national standard

[SOURCE: EN 60731:2007, 3.4.1.2]

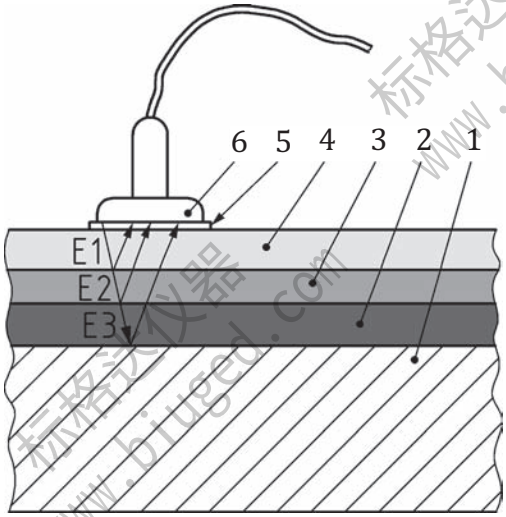
4 Principle

The method described in this Technical Specification determines single film thicknesses from the times of flight of an ultrasonic impulse that is partially reflected at the interfaces of the coating system. The strengths and weaknesses of the method are shown by measuring data for the different film-substrate combinations that are relevant in practice.

5 Physical principles of the measuring method and of the application

When measuring the film thickness using ultrasound, longitudinal waves are used because they can be easily generated and can be coupled into a work piece with almost every liquid. As shown in Figure 1, a sensor (6) consists of a piezoelectric disc, for sound generation and for reception, and of a "delay path". The ultrasonic impulse generated in the sensor first passes the delay path and then spreads through layers 1 to 3 down to the substrate (1) and beyond.

On each interface, a fraction of the impinging ultrasonic wave is reflected as a new ultrasonic impulse, while another fraction passes through. The first reflection occurs in the ultrasonic sensor when the ultrasonic impulse impinges on layer 1 (4).



Key

1 substrate
2 layer 3
3 layer 2
4 layer 1
5 couplant (liquid)
6 ultrasonic sensor (sender and receiver)
E echo 1, 2, 3

Figure 1 — Ultrasonic method

Ultrasonic impulses are recorded when they are received in the ultrasonic sensor. The distances of time between the ultrasonic impulses correspond to the sound path travel times T_i (i = 1, 2, 3) in the three individual layers. The amplitude or echo height of the ultrasonic impulse reflected on each interface depends on the respective reflection factors. If the sound velocity in each single layer is known, the respective film thickness can be calculated by means of the times of flight. For each layer, Formula (1) applies:

$$v = \frac{t_\mathrm{d}}{T / 2} \tag{1}$$

where

v is the sound velocity;

t_d is the dry film thickness;

T is the sound path travel time in the layer (back and forth).

In order to be able to resolve echoes with short intervals of sound path travel times with the naked eye (e.g. 20 ns in a 20 µm thick coating), the ultrasonic impulses shall be at least just as short. For this, the ultrasonic frequencies shall be respectively high (at least the reciprocal of half of the time of flight) or the A-scan shall be generated from lower frequencies by means of digital signal processing. For an example of an A-scan, see Figure 2.

When layers are too thin, the echoes of the individual layers merge into each other. In this case, an optic control of the evaluation in the A-scan presentation is no longer possible.

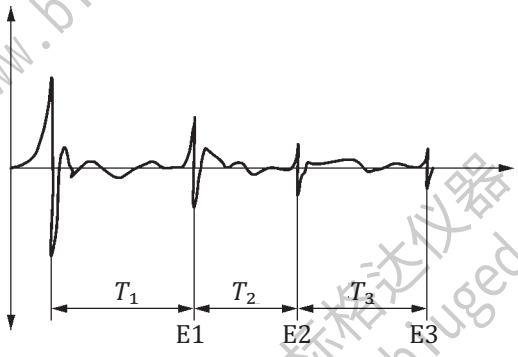
In cases where the A-scan presentation shows positive and negative half-waves, it shall be taken into account that for a negative reflection factor ($Z_2 < Z_1$) the phase of the ultrasonic impulse changes by 180°. If this is ignored, a relative time delay of a half wavelength can occur.

The precondition for ultrasonic impulses with signal amplitudes sufficiently high for evaluation forming at the interface of two layers is

— a sufficiently high reflection coefficient or respectively different acoustic impedances Z, and

— a clearly defined intersection between the materials.

Otherwise, the reflections can become too low for detection. This can also occur with curved work pieces where, due to geometry, not all sound fractions simultaneously re-impinge from the interface on the sensor.



Key

T sound path travel times for layers 1, 2, 3

E echo for layers 1, 2, 3

Figure 2 — Example of an A-scan presentation

6 Apparatus and materials

6.1 Ultrasonic film thickness measuring device

A device with an ultrasonic sensor for sending and receiving ultrasonic impulses and an evaluation unit for determining the film thickness over the time of flight (see Figure 1).

NOTE The ultrasonic sensors used for measuring the film thickness generate ultrasonic signals (longitudinal waves), which spread perpendicularly to the surface of the work piece and the coating. In the schematic diagram in Figure 1, a slant representation of the sound propagation was selected only for illustrating the sound generation.

6.2 Couplant

An acoustic contact between probe and test specimen with sufficient coupling shall be enabled. Commonly, a liquid (e.g. water or oil) or a gel couplant is applied.

6.3 Calibration standards

For checking the function of a measuring device, a working standard shall be used.

For checking the measuring method and for adjusting the device prior to use, a working measurement standard shall be used; one which largely corresponds to the respective test object to be measured with regard to film thickness, coating system, substrate and thicknesses of the layers.

7 Calibration, adjustment and checking of the measuring device

7.1 Calibration

Calibrate the device in accordance with the manufacturer's information.

NOTE Primarily, the time of flight of a signal is measured with the measuring devices. This time of flight can be checked, if necessary, with a calibration standard with defined thickness and known sound velocity.

7.2 Adjustment

Adjust the device in accordance with the manufacturer's information.

When adjusting the entering of sound velocities, it is recommended to use test specimens that are based on the minimum and maximum film thicknesses that would be expected for the determination of these sound velocities. Using the ultrasonic film thickness measuring device, the time of flight of the ultrasonic impulse and the sound velocities, in accordance with Formula (1), are determined in the same measuring area in which the film thickness was or will be determined with an alternative method.

The determination of the time of flight is carried out repeatedly on each test specimen and the mean is taken for each test specimen.

When selecting the test specimens to use for the determination of the sound velocity, it should be considered that the thinner the test specimens and the lower the sound path travel times, the more imprecise the determination of the sound velocity from time of flight and thickness.

When calculating, it shall be observed that the data are entered in the units used by the manufacturer of the device and, after a calculation in SI units, that these have been converted.

7.3 Checking the adjustment

Prior to recording the measurements, after turning the device on, and for alterations of the coating system or substrate of the test object, the adjustment of the measuring device shall be checked by means of a working standard.

All the device settings should always be done in accordance with the manufacturer's information.

After the adjustment, device settings that influence the ultrasonic signals shall not be changed. When exchanging the probe or changing the device settings another adjustment shall be carried out.

8 Procedure of measurement

Operate the device in accordance with the manufacturer's information.

Apply some couplant to the coating and measure its film thickness. Lay the sensor perpendicularly onto the coating and press so that the film of couplant becomes as thin as possible. Keep the probe calmly in the measuring position until a stable measuring value is displayed.

9 Temperature influence during the measurement

Most of the probes are intended to be used between –20 °C and +60 °C. However, it is recommended to carry out ultrasonic film thickness measurements, preferably in the range of common ambient temperatures, in order to keep the test object, couplant and probe at an equilibrium temperature during measuring. Temperature gradients in the delay path of the probe or in the test object influence the measuring results due to uncontrollable changes and fluctuations of the sound velocity.

In all materials the sound velocity depends more or less on the temperature.

NOTE For polymers, the changes are typically in the range of 0,1 % to 0,3 % per °C for a negative temperature coefficient.

In order to minimize errors due to temperature changes, constant temperature conditions should be observed. Adjusting and subsequent measuring shall be carried out at the same temperature. In cases where there are prolonged breaks between the measurements and changes of the ambient temperature the adjustment shall be checked.

10 Precision

10.1 General

For further information on the determination of precision see Annex B.

10.2 Repeatability limit

The repeatability limit *r* is the value below which the absolute difference between two test results (each the mean of three valid determinations) can be expected to lie when this method is used under repeatability conditions. In this case, the test results are obtained on identical material by one operator in one laboratory within a short interval of time using the described test method. The repeatability limit *r* in accordance with this specification, calculated with a probability of 95 %, corresponds to the values given in Tables 1 and 2.

Table 1 — Repeatability limit (*r*) for individual test specimens

Test specimen	Film thickness µm	Repeatability limit (*r*) µm
Spruce veneered	100	4
Spruce sanded	100	6
Beech veneered	100	6
Beech sanded	100	6
Aluminium	22	3
Aluminium	44	4
Carbon-fibre composite	22	3
Carbon-fibre composite	44	3
PP (polypropylene)	22	4
PP (polypropylene)	44	4
SMC (sheet moulding compound)	22	5
SMC (sheet moulding compound)	44	9
Steel	22	3
Steel	44	4

Table 2 — Repeatability limit (*r*) for product groups

Test specimen	Film thickness µm	Repeatability limit (*r*) µm
Wood test specimens	100	5
Metal (aluminium and steel)	22	3
Metal (aluminium and steel)	44	4

10.3 Reproducibility limit

The reproducibility limit *R* is the value below which the absolute difference between two single test results (each the mean of two valid determinations) can be expected to lie when this method is used under reproducibility conditions. In this case, the test results are obtained on identical material by operators on succeeding days during shift operation using the described test method. The reproducibility limit *R* in accordance with this specification, calculated with a probability of 95 %, corresponds to the values given in Tables 3 and 4.

Table 3 — Reproducibility limit (*R*) for individual test specimens

Test specimen	Film thickness µm	Reproducibility limit (*R*) µm
Spruce veneered	100	13
Spruce sanded	100	20
Beech veneered	100	13
Beech sanded	100	11
Aluminium	22	5
Aluminium	44	7
Carbon-fibre composite	22	4
Carbon-fibre composite	44	8
PP (polypropylene)	22	5
PP (polypropylene)	44	12
SMC (sheet moulding compound)	22	10
SMC (sheet moulding compound)	44	14
Steel	22	6
Steel	44	13

Table 4 — Reproducibility limit (*R*) for product groups

Test specimen	Film thickness µm	Reproducibility limit (*R*) µm
Wood test specimens	100	17
Metal (aluminium and steel)	22	6
Metal (aluminium and steel)	44	11

11 Test report

The test report shall include at least the following information:

a) all details necessary to identify the tested product;

b) a reference to this Technical Specification, i.e. ISO/TS 19397;

c) the result of the measurement;

d) any deviation from the specified test method;

e) any unusual observation (deviation) during testing;

f) the date of the test.

Annex A
(informative)

Qualification of the personnel

An operator carrying out ultrasonic thickness measurements in accordance with this Technical Specification should be introduced to the physical principles and the metrological processes of this method by highly qualified personnel and, prior to testing, should have had metrological experience guided by that personnel.

For coating systems that are metrologically untested, information on the product and the materials to be tested shall be available for the operator. For the reliable setting of the devices, knowledge of the physical principles of ultrasound and a detailed understanding of the metrological processes are required. Due to metrological similarity, knowledge of the most commonly used method of ultrasonic thickness measurement is helpful (see the Bibliography).

Annex B
(informative)

Determination of precision

B.1 General information on the round robin test

A round robin test was carried out to determine the precision of film thickness measurements using ultrasonic film thickness measuring devices. Five companies participated in the round robin test.

B.2 Test specimens

For the round robin test, an aqueous standard dispersion was applied to ten different substrates (see Table B.1). The dispersion was applied repeatedly to some of the substrates in order to achieve higher film thicknesses.

Five test specimens of each type of substrate were prepared for each value of film thickness.

The film thickness of the wood test specimens was 100 µm.

For the other types of substrate, test specimens with film thicknesses of 22 µm and 44 µm were prepared.

For the basic calibration, separate calibration panels with identical parameters and cross sections for split-beam microscopy were prepared.

Further cross-sectional samples for the split-beam microscopy were prepared for the direct comparison of the film thickness measurement microscopy/ultrasound.

Table B.1 — Substrates used in the round robin test with the respective film thicknesses

Substrate	Nominal film thickness µm
Spruce veneered	100
Spruce sanded	100
Beech veneered	100
Beech sanded	100
Aluminium	22
Aluminium	44
Root wood	44
Carbon-fibre composite	22
Carbon-fibre composite	44
PP (polypropylene)	22
PP (polypropylene)	44
SMC (sheet moulding compound)	22
SMC (sheet moulding compound)	44
Steel	22
Steel	44

B.3 Ultrasonic film thickness measuring devices

The round robin test was carried out using five different ultrasonic film thickness measuring devices operating in the frequency range > 200 MHz. Devices with frequencies below 200 MHz are not suitable for all substrates.

Prior to measuring, all of the devices were calibrated in accordance with the manufacturers' information.

B.4 Repeat determination

On each test specimen the determination was carried out in triplicate.

The three measuring points were marked on each test specimen.

B.5 Evaluation

B.5.1 General

The evaluation of the measuring results was carried out in accordance with ISO 5725-2 and ISO/TR 22971.

The test specimen root wood was determined as an outlier and therefore was ignored in the precision calculation for ultrasound.

The repeatability limit and the reproducibility limit were calculated separately for each type of substrate and for each film thickness (see Table 1 and Table 3).

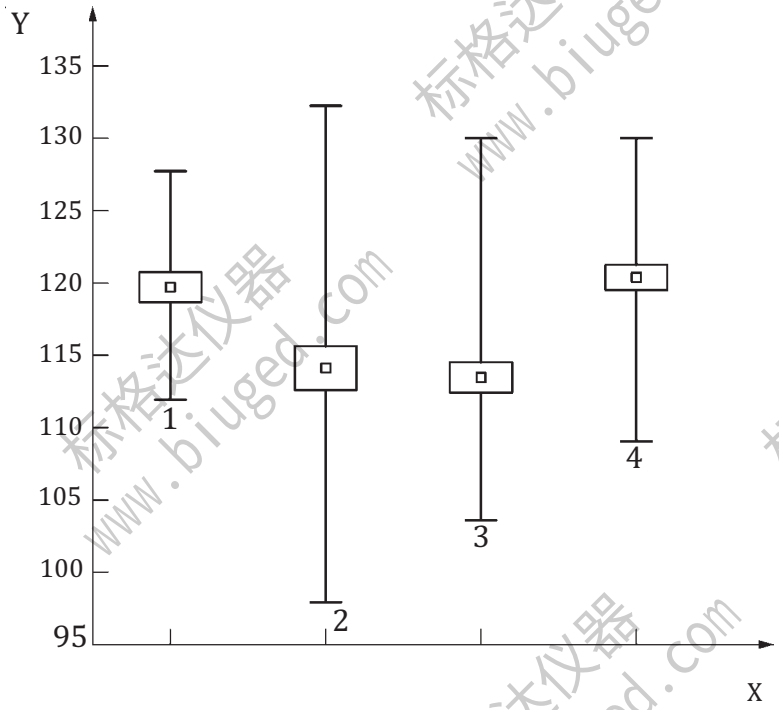
Since the repeatability limits and reproducibility limits are similar for the wood types and also for aluminium and steel, groups of test specimens were formed for simplification and their respective repeatability limits and reproducibility limits were calculated.

B.5.2 Repeatability limit

Figures B.1, B.2 and B.3 show the diagrams of the respective groups of test specimens:

— wood test specimens with a film thickness of 100 µm (Figure B.1);

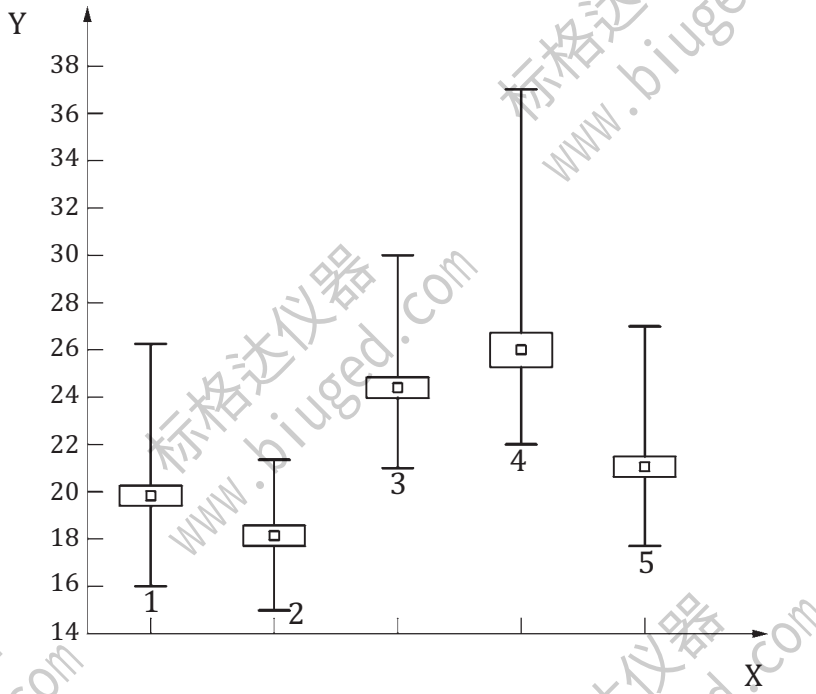
— aluminium, carbon-fibre composite, PP, SMC and steel test specimens with a film thickness of 22 µm (Figure B.2) and 44 µm (Figure B.3).



Key

Y	film thickness, in micrometres
X	substrate
1	spruce, veneered
2	spruce, sanded
3	beech veneered
4	beech, sanded
☐	mean value
▭	mean value ± 0,95 confidence interval
⊥	min.-max

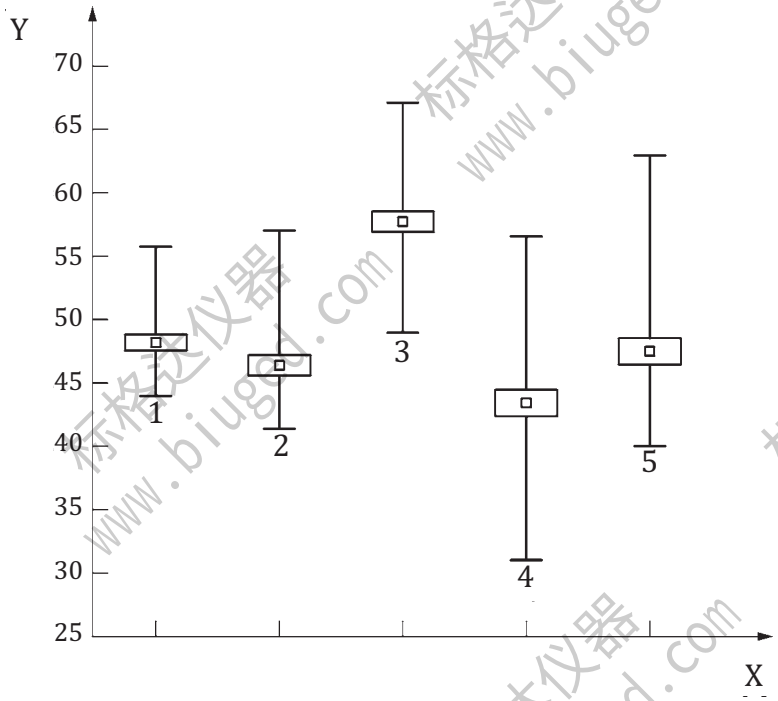
Figure B.1 — Diagram of the repeatability limit of the wood test specimens with a film thickness of 100 µm



Key

Y	film thickness, in micrometres
X	substrate
1	aluminium
2	carbon-fibre composite
3	PP (polypropylene)
4	SMC (sheet moulding compound)
5	steel
□	mean value
▭	mean value ± 0,95 confidence interval
I	min.-max

Figure B.2 — Diagram of the repeatability limit of the other test specimens with a film thickness of 22 µm



Key

Y	film thickness, in micrometres
X	substrate
1	aluminium
2	carbon-fibre composite
3	PP (polypropylene)
4	SMC (sheet moulding compound)
5	steel
☐	mean value
▭	mean value ± 0,95 confidence interval
⊥	min.-max

Figure B.3 — Diagram of the repeatability limit of the other test specimens with a film thickness of 44 µm

The calculation of the repeatability limit (r) of the individual test specimens (see Table 1) is based on three measuring values for each substrate and film thickness.

The repeatability limit (r) for the product groups wood and metal (see Table 2) almost corresponds to the individual repeatability limit values.

B.5.3 Reproducibility limit

The calculated reproducibility limit (R) of the individual test specimens (see Table 3) is based on all measured film thickness values.

The reproducibility limit (R) of the test specimen groups wood and metal (see Table 4) show larger deviations compared to the results of the individual test specimens.

B.5.4 Influencing factors

The calculated repeatability limits and reproducibility limits also include other different influencing factors besides the measuring device variation, for example:

— inhomogeneities of the substrate, such as wood grain or variations of the density of the substrate;

— influence due to application, such as uneven application or orange peel;

— poor reflection of the ultrasound at the surface of the substrate.

In sum, the highest percentage of the measurement uncertainty was related to the application.

B.6 Error propagation

There are ultrasound measuring devices that have to be calibrated using measuring results from microscopy. One of those devices was also used in the round robin test.

For calibration, several calibration plates were prepared.

Formula (B.1) applied:

$$\frac{t_c}{T_c} = \frac{t_p}{T_p} \tag{B.1}$$

where

t_c is the film thickness of the coating of the calibration panel, determined microscopically;

T_c is the ultrasonic path travel time of the coating of the calibration panel;

t_p is the calculated film thickness of the coating of the test panel.

T_p is the ultrasonic path travel time of the coating of the test panel;

Derivations for the error propagation:

$$\frac{\delta t_p}{\delta t_c} = \frac{T_p}{T_c} \quad ; \quad \frac{\delta t_p}{\delta T_c} = -\frac{t_c \cdot T_p}{T_c \cdot T_c} \quad ; \quad \frac{\delta t_p}{\delta T_p} = \frac{t_c}{T_c}$$

From this, t_c results for the error propagation for the calculated film thickness.

For the error propagation of Δt_p based on t_c and the ultrasonic path travel times T_c and T_p results:

$$\Delta t_p = \frac{\delta t_p}{\delta t_c} \cdot \Delta t_c + \frac{\delta t_p}{\delta T_c} \cdot \Delta T_c + \frac{\delta t_p}{\delta T_p} \cdot \Delta T_p = \frac{T_p}{T_c} \cdot \Delta d_c + \frac{t_c \cdot T_p}{T_c \cdot T_c} \cdot \Delta T_c + \frac{d_c}{u_c} \cdot \Delta T_p$$

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