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Manufacturing and Metrological Certification of Samples of Metal Coatings Properties for Eddy-Current Thickness Gauges

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Abstract

In today's high-tech industries using large range of protective and functional metal coatings. The requirements for accuracy of measurement of the thickness *d* are constantly increasing. Manufacture and use of eddy-current thickness gauges of metal coatings implies the use of coating standards for calibration and verification in production, and also for setting before measurements. At the same time, for reliable measurements, it is necessary to ensure that the electromagnetic parameters of coating materials and bases are kept constant within the sets of coating thickness measures. However existing calibration schemes assume only control the geometric parameters of the coating standards.

To evaluate the influence of the electromagnetic parameters of the coating thickness measures on the *d* measurement results, a model of an eddy-current three-winding transformer primary measuring transducer with a ferrite core over a two-layer metal structure was developed. Based on the analysis of the influencing parameters: the electrical conductivity σ_c of the coating, the relative magnetic permeability μ_b, and the conductivity σ_b of the base, the requirements were formulated for the range of their variation, providing an error $|\Delta d| \leqq (0.02d + 1)$ µm.

Based on the calculations performed, a reference complex was developed to measure the electromagnetic parameters of the samples of the properties of metal coatings (coating thickness measures), allowing σ_c, μ_b and σ_b in the frequency range from 50 kHz to 200 MHz in the d = 5 - 300 µm range. Details considered complex structure, measurement algorithms and key metrological characteristics

1. Introduction

International Standard ISO 21968:2005 «Non-magnetic metallic coatings on metallic and non-metallic basis materials -- Measurement of coating thickness -- Phase-sensitive eddy-current method» describes a method of using phase-sensitive eddy-current instruments for non-destructive measurements of the thickness of non-magnetic metallic coatings, such as zinc, cadmium, copper, tin or chromium on steel.

The phase-sensitive method can be applied without thickness errors to smaller surface areas and to stronger surface curvatures than the amplitude-sensitive eddy-current method described in ISO 2360, and is less affected by the magnetic properties of the basis material. However, the phase-sensitive method is more affected by the electrical properties of the coating materials.

When measuring metallic coatings on metallic basis materials, the product of conductivity and permeability (σ, μ) of one of the materials should be at least a factor of 1.5 times the product of conductivity and permeability for the other material. Non-ferromagnetic materials have a relative permeability of 1.

2. Probes

The block scheme of three-winding eddy-current phase probe for measuring metallic coating thickness, using as a reference signal the voltage of the compensation winding is shown in Fig. 1.

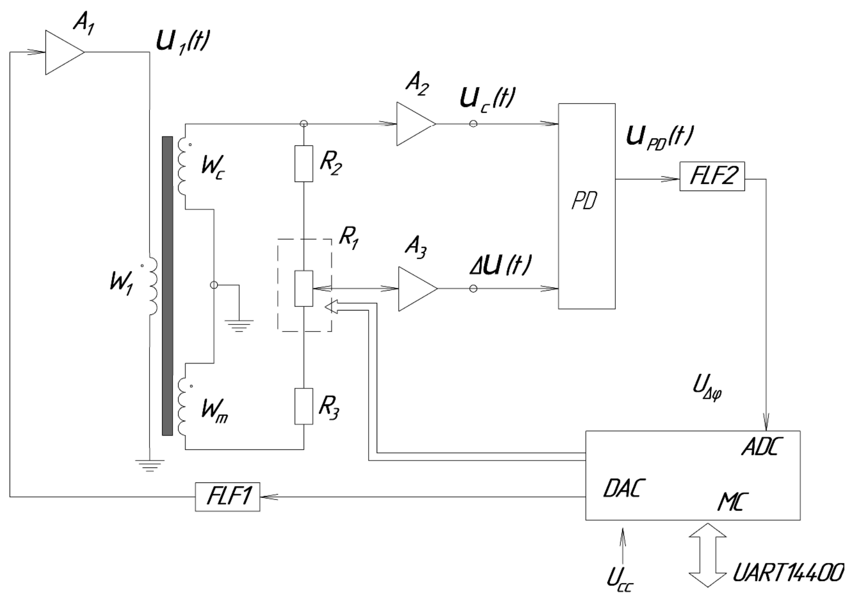

Figure 1. Block scheme of eddy current phase probe:
A_1, A_3 – amplifiers, PD – phase detector, FLF_1 - FLF_2 - low frequency filters, W_1 – excitation coils, W_m and W_c – differentially connected measuring and compensating coils, MC – microcontroller, R_1 – an operated digital balancing potentiometer

Coil W_1 gets sinusoidal voltage $u_1(t)$ with frequency f. Voltage on measuring coil W_m is equal to the sum of the voltages $\mathring{U}_0$ arising in absence of a controllable product, and brought (differential) voltage $\mathring{U}_i$, arising due the influence of a product: $\mathring{U}= \mathring{U}_0+ \mathring{U}_i$. Strengthened differential (brought) voltage $\Delta u(t)$ arrives on phase detector PD. Voltage $u_c(t)$ from an exit of compensating coil W_c serves as a basic signal for the phase detector. Constant voltage $U_{\Delta\varphi}(d)$ on an exit of the filter of low frequency is proportional to phase difference $\Delta\varphi$ between voltage $u_c(t)$ and differential (brought) voltage $\Delta u(t)$. Balancing of coils of the transducer is made with use of the digital potentiometer R_1 operated microcontroller MC.

The probe is made in a small cylindrical holder, attached to the electronic control unitcable, which is fed by the supply voltage and organized communication channel UART14400 (Fig. 2) for communication with the block processing and presentation of results.

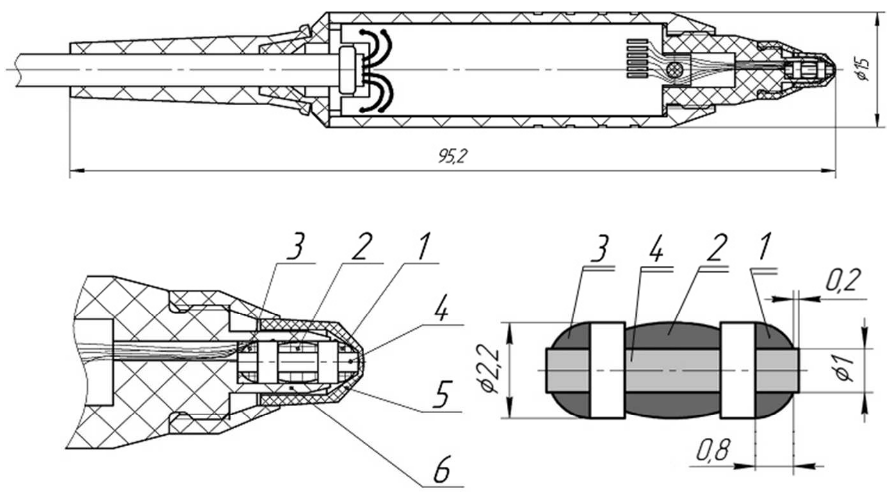

Figure 2. Consignment phase probe with a miniature sensor and removable protective cap: 1 - measuring W_m, 2 - exciting W_1 and 3 - compensating W_c windings, 4 - ferrite core, 5 - cap 6 – housing

3. Theory. Factors, affecting measurement accuracy

Since this thickness range depends on both the applied frequency of the probe system and the electrical properties of the coating, the maximum thickness should be determined experimentally, unless otherwise specified by the manufacturer.

However, in the absence of any other information, the maximum measurable coating thickness, d_{max}, can be estimated using Equation (1):

$$d_{max}= 0.8\ \delta_0 \qquad (1)$$

where δ_0 is the standard penetration depth of the coating material.

For non-magnetic metallic coatings the standard penetration depth, δ_0, is a useful value for some important rough estimations. It may be calculated, in mm, using Equation (2):

$$\delta_0 = 503/\mathrm{sqrt}(f\sigma) \tag{2}$$

where f is the probe operating frequency, Hz; σ is the electrical conductivity of the conductor, MS/m.

For the analysis of characteristics of the probe it is convenient to use the generalized parameter

$$\beta = R\sqrt{2\pi f \sigma_i \mu_0} \tag{3}$$

where R – equivalent radius of excitation coil; f – frequency of excitation current; σ_i – integrated electrical conductivity of material; μ_0 – a magnetic constant.

For the considered task optimal ß ≈ 3…10.

Further, the standard provides an overview of factors, affecting measurement accuracy: electrical properties σ_b and μ_b of the basis materials, electrical properties σ_c of the coating materials, basis-metal thickness, edge effects, surface curvature R, surface roughness Rz, lift-off effect h, probe pressure, temperature t.

As is known, this method is weakly sensitive to deviations of the R, Rz, h, d and t. The main factors, affecting measurement accuracy: σ_b, μ_b and σ_c.

Fig. 3 shows functional connection insert voltage $\mathring{U}_i$ from measurement value d and influence parameters σ_b, μ_b , σ_c.

In this regard, in the description of the calibration process on the plant and verification of the devices and probes indicated that: «the electrical conductivity and magnetic permeability of both coating and basis materials should be identical to the corresponding properties of the parts to be measured. As calibration standards are subject to wear and deterioration with time and use, they should be recalibrated and/or replaced periodically at time intervals established locally or after consultation with the manufacturer”. But does not indicate the magnitude of σ_b, μ_b and σ_c deviation between the samples, providing the required accuracy for the verification.

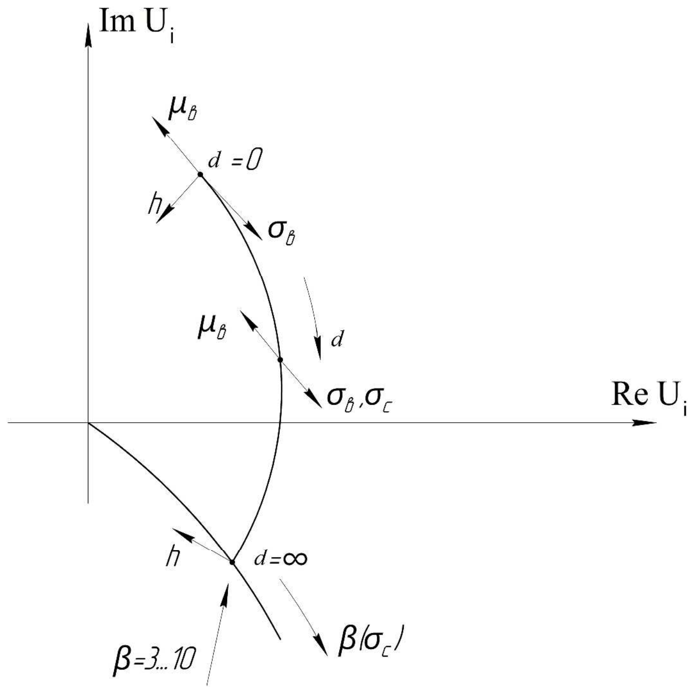

Figure 3. Functional connection insert voltage $\mathring{U}_i$ from measurement value *d* and influence parameters σ_b, μ_b , σ_c

4. Modeling

To determine the component of the expanded uncertainty introduced by the influence of variations of values σ_b, μ_b and σ_c was developed calculation model of a three-winding eddy-current probe with a ferrite core on the sample coverage (Fig. 4) is used as the primary informative parameter $\Delta\varphi$ of introduced voltage $\mathring{U}_i$.

There were calculated $\Delta\varphi(d, \sigma_b, \mu_b, \sigma_c)$ for f = 65 kHz, 200 kHz and 1.8 MHz. As a base material considered Steel 1020, which has enough homogeneous structure and provides the required surface finish. The coating materials analyzed: chromium, zinc and copper, thee electrical conductivity over lapping range of 7 to 58 MS/m.

The analysis of the dependencies demonstrated that for the considered coatings (σ_c from 7 to 60 MS/m) with the variation σ_b within 10% the brought measurement error Δd will be equal to

$$\Delta d(\sigma_b)= k(\Delta\sigma_b/\sigma_b)d \quad [\mu_b=\text{const}, \sigma_c= \text{const}] \qquad (4)$$

where k changes in the range from 1.49 to 1.62.

The analysis of the dependencies demonstrated that for the considered coatings (σ_c from 7 to 60 MS/m) with the variation μ_b within 10 % the brought measurement error Δd will be equal to

$$\Delta d(\mu_b) = k(\Delta\mu_b/\mu_b)d \quad [\sigma_c=\text{const}, \sigma_b=\text{const}] \tag{5}$$

where k changes in the range from 0.86 to 1.01.

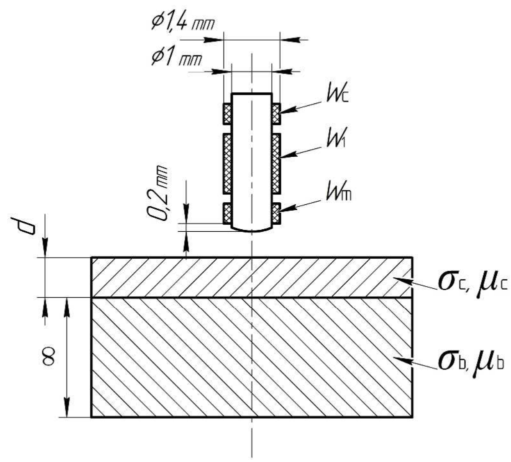

Figure 4. Model of the eddy current sensor of three-winding probe on the two-layer calibration standard

The analysis of the dependencies demonstrated that for the considered bases (σ_b from 7 to 9 MS/m) with the variation σ_c within 10 % the brought measurement error Δd will be equal to

$$\Delta d(\sigma_c) = k(\Delta\sigma_c/\sigma_c)d \quad [\mu_b=\text{const}, \sigma_b=\text{const}] \tag{6}$$

where k changes in the range from 1.56 to 1.69.

For the most of the technological processes of galvanic coatings application the measurement error Δd during the verification with use of coating thickness reference standards shall not exceed

$$|\Delta d| \leq (0.02d + 1)\ \mu\text{m} \tag{7}$$

As specified above the main stray parameters that determine quality of coating application are σ_b, μ_b, σ_c.

Given requirements for the purpose (measuring galvanic coating thickness), for the majority of modern thickness gauges for calibration and verification measures to the thickness of the maximum permissible error d must not exceed the value

$$|\Delta d| \leq (0.02d + 1)\ \mu\text{m} \tag{8}$$

As mentioned above, the main nuisance parameters influencing the error Δd are σ_b, μ_b, σ_c.

$$|\Delta d| = |\Delta d(\sigma_b)| + |\Delta d(\mu_b)| + |\Delta d(\sigma_c)| \tag{9}$$

Let us, as a condition that the maximum insertion components of measurement error

$$|\Delta d_m(\sigma_b)| = |\Delta d_m(\mu_b)| = |\Delta d_m(\sigma_c)| = 0.33|\Delta d|. \tag{10}$$

On this basis, taking into equations (4) - (6) we can calculate the allowable deviation σ_b, μ_b and σ_c for measuring ranges d

$$|\Delta d_m(\sigma_b)| \leq 0.33(0.02d + 1) = 0.0066d + 0.33\ \mu m \tag{11}$$
$$|\Delta d_m(\sigma_c)| \leq 0.33(0.02d + 1) = 0.0066d + 0.33\ \mu m \tag{12}$$
$$|\Delta d_m(\mu_b)| \leq 0.33(0.02d + 1) = 0.0066d + 0.33\ \mu m. \tag{13}$$

Fig. 5 - 7 shows the dependence of the allowable deviation $\sigma_b(d)$, $\mu_b(d)$ and $\sigma_c(d)$ to Equation (8).

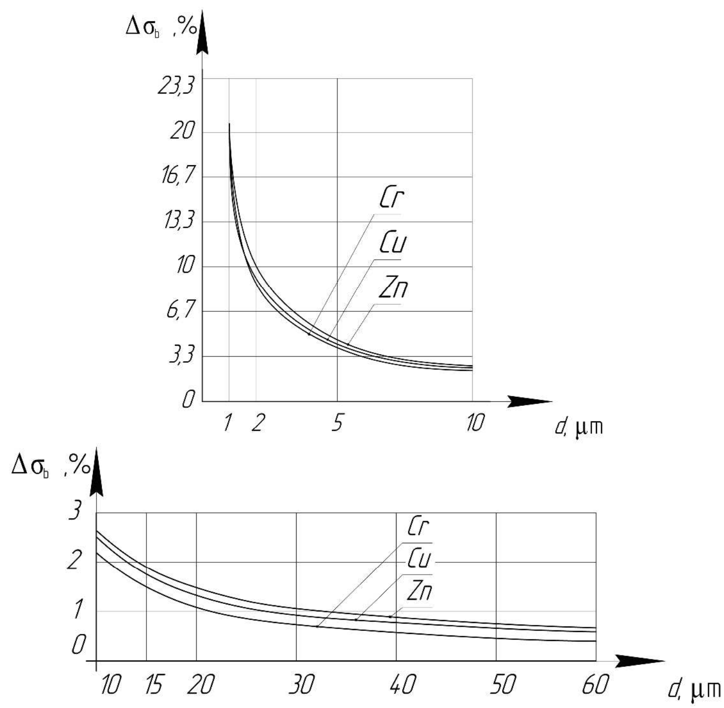

Figure 5. The maximum permissible deviation bases conductivity $\sigma_b(d)$ of calibration standards

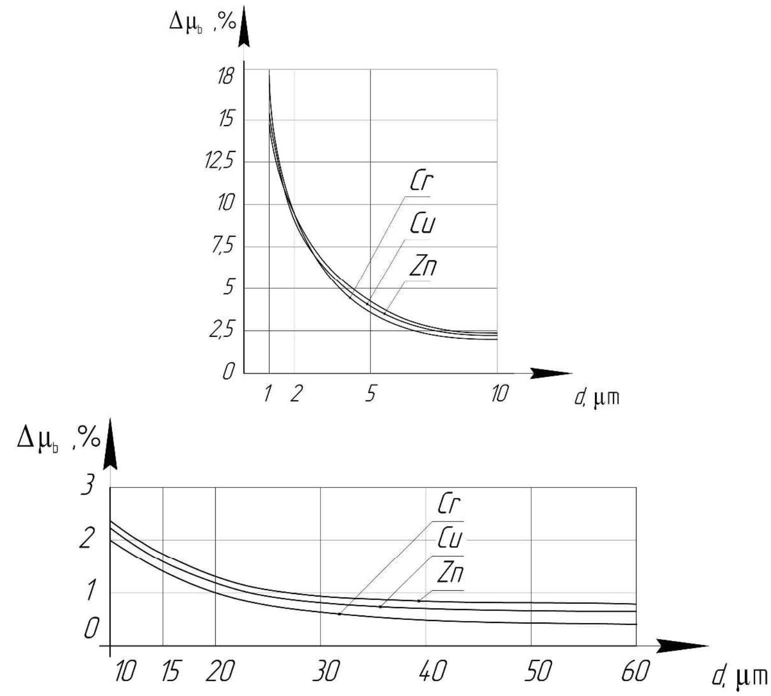

Figure 6. The maximum permissible deviation bases permeability $\mu_b(d)$ of calibration standards

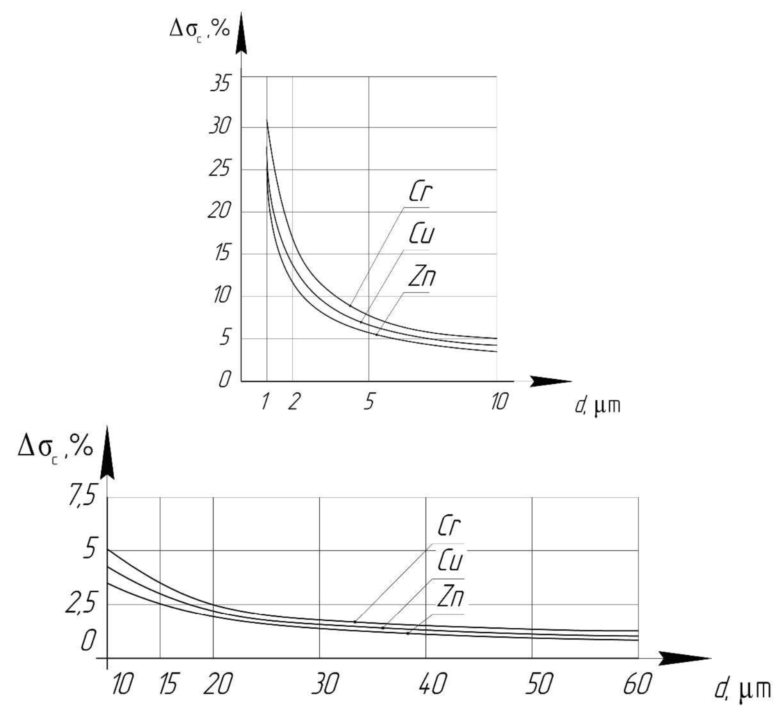

Figure 7. The maximum permissible deviation coating conductivity $\sigma_c(d)$ of calibration standards

5. Metrological chain

Fig. 8 shows four-level structure of the metrological chain to define and disseminate metallic coating thickness measurement scales. These are used to “produce” reference calibration standards at user level. Naturally, direct calibration and the verification should be at the highest possible accuracy.

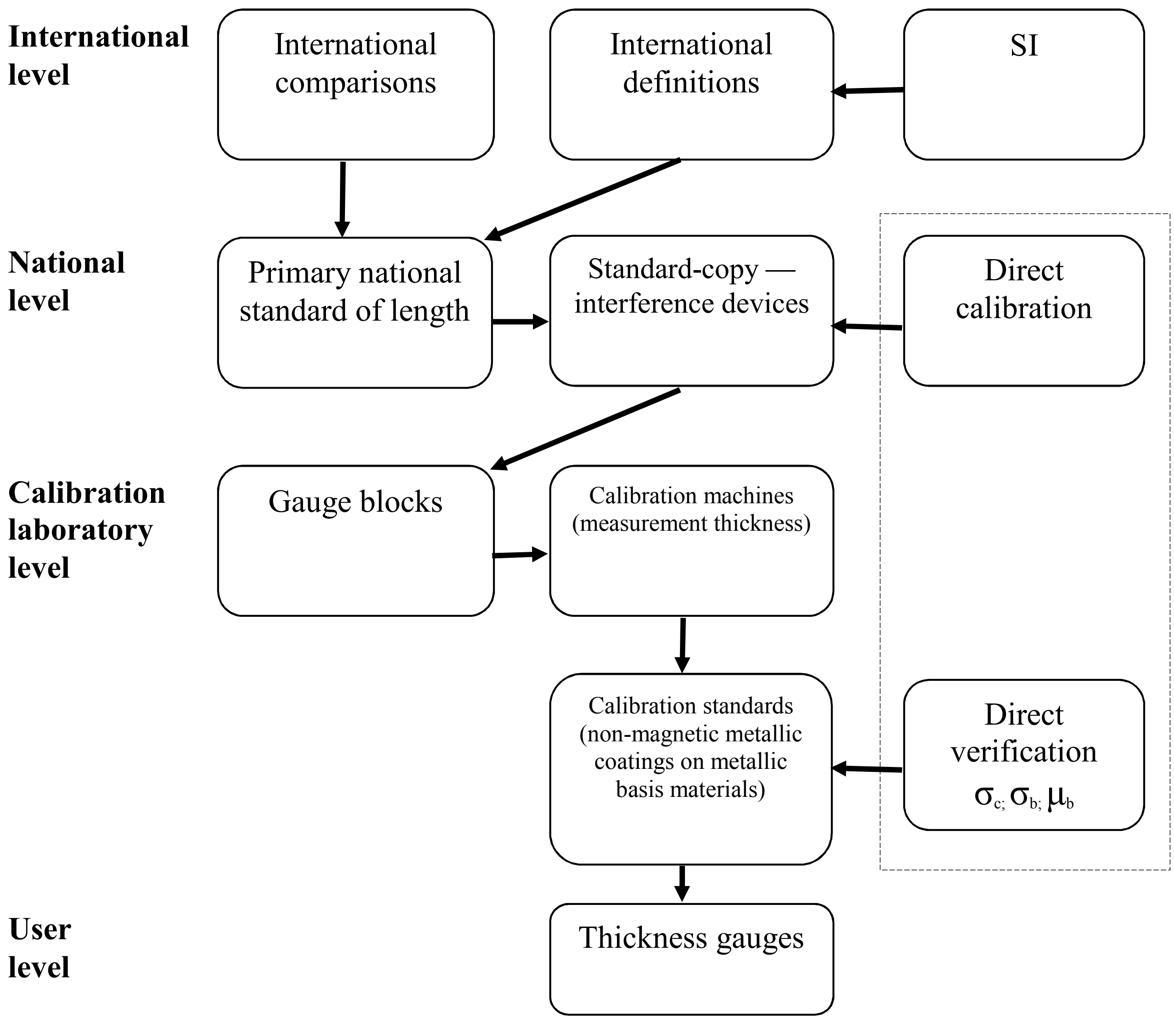

Figure 8. Four-level structure of the metrological chain to define and disseminate metallic coating thickness measurement scales

6. Design calibration standards and measurement of their parameters

Fig. 9 shows general view of presupposing realization problems control d, σ_c, σ_b, μ_b calibration standards.

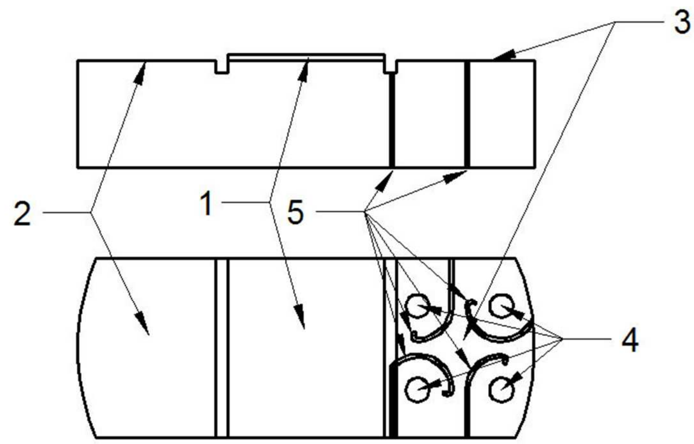

Figure 9. General view of calibration standard: 1 – area with metallic coating; 2 – area without coating; 3 – area measuring the conductivity of the base metal; 4 – contacts for connecting the cables of the power supply, ammeter and voltmeter; 5 – figured cuts

The calibration standard has the three areas: 1 – area with metallic coating, 2 – area without coating. The areas 1 and 2 are used for eddy-current probes of thickness gauges installation at use of the calibration standard on purpose And then μ_b is measured in the area 2 during transfer the quantity value μ_b. Also the areas 1 and 2 are designed for coating thickness d measurement with the use of profilometer. The area 3 is used for σ_b measurement. The contacts 4 for connecting the cables of the power supply, ammeter and voltmeter and also figured cuts 5 forming the optimal shape of the leak area DC in the measurement of σ_b by the method of Van der Pau are located in this area.

It is necessary to complete the calibration machines for the measurement, calculate the operating modes and to develop a measurement procedure for metallic coating thickness control.

When completing the calibration machine, magnetic permeability blocks and conductivity blocks are made and measured.

The preparatory part includes: a graduation laid transformer eddy-current probe with a U-shaped core, designed to measure μ_b on the magnetic permeability blocks and a graduation high frequency laid transformer eddy-current probe, designed to measure σ_c on the conductivity gauge blocks.

The standard parameters measurement procedure consists of the following steps:
- d measurement by using profilometer ($d \leq 400$ µm);
- σ_b measurement by van der Pau method using voltmeter, current source, ammeter and micrometer with digital counting device;
- μ_b measurement using transformer eddy-current probe with a U-shaped connected to the impedance analyzer;
- σ_c measurement using a high-frequency eddy-current probe connected to the impedance analyzer;

- processing of the all measurement results using the application software (based on MS Excel) of personal computer.

7. Conclusions

The modeling of probe allowed to formulate the requirements for electromagnetic characteristics of the calibration standard. The proposed design of the standard and the measurement procedure its parameters provide possibility of international comparisons and improve the accuracy of the results of calibration and verification of eddy-current thickness gauges of metal coatings.