Phase-sensitive eddy-current method of metallic coating thickness measurement.On question of calibration and verification of coating thickness gauges and metallic coating thickness standards.

Sergei S. Golubev Federal Agency on Technical Regulating and Metrology, Moscow, Russia +7 (499) 2360300 E-mail: golubev@gost.ru Vladimir A. Syasko, Nadezhda I. Smirnova Constanta ltd, St-Petersburg, Russia +7 (921) 9334343 E-mail: 9334343@gmail.com Kirill V. Gogolinskiy D.I. Mendeleev Institute for Metrology (VNIIM), St-Petersburg, Russia +7 (812) 2517601 E-mail: info@vniim.ru

Abstract

The international standardISO 21968:2005«Non-magnetic metallic coatings on metallic and nonmetallic basis materials -- Measurement of coating thickness -- Phase-sensitive eddy-current method» was approved in 2005. However, to ensure the required reliability of measurement results of metallic coatings by this method, it is necessary to develop and approve two more standards: «Non-magnetic metallic coatings on metallic and non-metallic basis materials -- Measurement of coating thickness -- Phase-sensitive eddy-current method. Part 2. Verification and calibration of the metallic coating thickness gauges» and «Non-magnetic metallic coatings on metallic and nonmetallic basis materials -- Measurement of coating thickness -- Phase-sensitive eddy-current method.Part 3. Calibration of metallic coating thickness standards». The following fundamental questions for the standards development are analysed with use of developed models of phasesensitive eddy-current transducers: the principles of metallic coating thickness standards completion and also influence of their basic electro-physical and geometrical parameters on measurement results. Based on this, suggestions on set, design and basic geometrical and electro-physical parameters of the metallic coating thickness standard, methods of their manufacture and calibration are given in the work. The calculation techniques of results uncertainty during measurements, the calibration of gauges, the calibration and the verification of metallic coating thickness standards and also structure of the standards (part 2 and part 3) and structure of the metrological chain for the definition and dissemination of metal coating thickness measurements are analysed.

Keywords: metallic coating, phase-sensitive, verification, metallic coating thickness standards

1. Introduction

International StandardISO 21968:2005«Non-magnetic metallic coatings on metallic and nonmetallic 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 on metallic and non-metallic basis materials, suchas:

- a) zinc, cadmium, copper, tin or chromium on steel;
- b) copper or silver on composite materials.

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 themagnetic 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 schemeof three-winding eddy current phase probefor measuring metallic coating thickness, using as a reference signal the voltage of the compensation winding is shown in Fig. 1.

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 $U_{a_q}(d)$ 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 R₁ operated microcontroller MC.

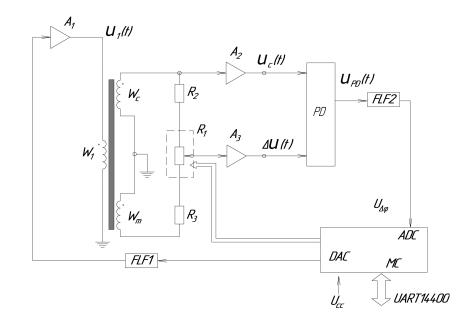


Figure 1. Block scheme of eddy current phase probe: A₁, A₃ – amplifiers, PD – phase detector, FLF₁ - FLF₂ - low frequency filters, W₁ – excitation coils, W_m and W_c – differentially connected measuring and compensating coils, MC – microcontroller, R₁ – an operated digital balancing potentiometer

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.

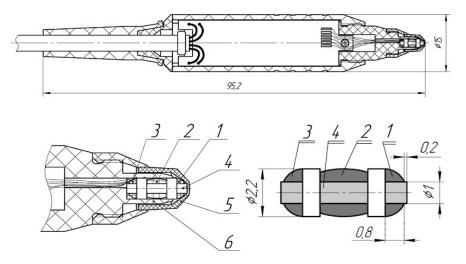


Figure 2. Consignment phase probe with a miniature sensor and removable protective cap: 1 measuring W_m , 2 - exciting W_1 and 3 - compensating W_c windings, 4 - ferrite core, 5 - cap 6 housing

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):

$$\max^{=0,8\,\delta_0}\tag{1}$$

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

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

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

where f is the probe operating frequency, Hz; σ is the electrical conductivity of the conductor, MSm/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 $\beta \approx 3...10$.

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, basismetal thickness, edge effects, surface curvature *R*, surface roughness *Rz*, lift-off effect *h*, probe pressure, probe tilt α , temperature *t*.

As is known, this method is weakly sensitive to deviations of the *R*, *Rz*, *h*, α and *t*. The mainfactors, affecting measurement accuracy: σ_b , μ_b and 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.

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. 3) is used as the primary informative parameter $\Delta \phi$ of introduced voltage \ddot{U}_{BH} .

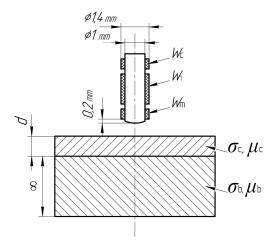


Figure 3. Model of the eddy current sensor of three-winding probe on the two-layer calibration standard.

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, zin cand copper, thee lectrical conductivity over lapping range of 7 to 58 MSm/m.

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

$$\Delta d(\sigma_b) = k(\Delta \sigma_b / \sigma_b) d \quad [\mu_b = \text{const}, \ \sigma_c = \text{const}]$$
(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 MSm/m) with the variation μ_b within 10% the brought measurement error Δd will be equal to

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

where k changes in the range from 0.86 to 1,01.

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

$$\Delta d(\sigma_{\rm c}) = k(\Delta \sigma_{\rm c}/\sigma_{\rm c})d \quad [\mu_{\rm b} = \text{const}, \sigma_{\rm b} = \text{const}] \tag{6}$$

where *s* 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

$$\Delta d \le \pm (0.02d + 1) \,\mu\mathrm{m} \tag{7}$$

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 spauges for calibration and verification measures to the thickness of the maximum permissible error d must not exceed the value

$$\Delta d \le \pm (0.02d + 1) \,\mu\mathrm{m} \tag{8}$$

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

$$|\Delta d| \le |\Delta d(\sigma_{\rm b})| + |\Delta d(\mu_{\rm b})| + |\Delta d(\sigma_{\rm c})| \tag{9}$$

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

$$|\Delta d_{\rm m}(\sigma_{\rm b})| = |\Delta d_{\rm m}(\mu_{\rm b})| = |\Delta d_{\rm m}(\sigma_{\rm c})| = 0.33 |\Delta d|. \tag{10}$$

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*

$$\begin{aligned} |\Delta d_{\rm m}(\sigma_{\rm b})| &\leq 0.33(0.02d+1) = 0.0066d + 0.33 \ \mu{\rm m}; \\ |\Delta d_{\rm m}(\sigma_{\rm c})| &\leq 0.33(0.02d+1) = 0.0066d + 0.33 \ \mu{\rm m}; \end{aligned} \tag{11} \\ |\Delta d_{\rm m}(\mu_{\rm b})| &\leq 0.33(0.02d+1) = 0.0066d + 0.33 \ \mu{\rm m}. \end{aligned}$$

Fig. 4 - 6 shows the dependence of the allowable deviation $\sigma_b(d)$, $\mu_b(d)$ and $\sigma_c(d)$ to Equation (8).

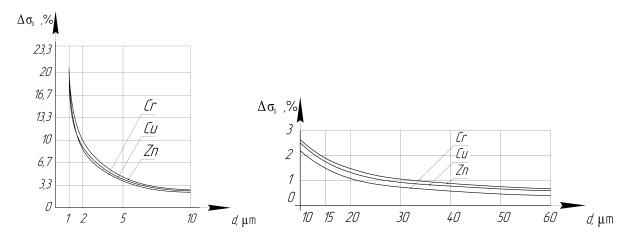


Figure 4. The maximum permissible deviation bases conductivity $\sigma_b(d)$ of calibration standards.

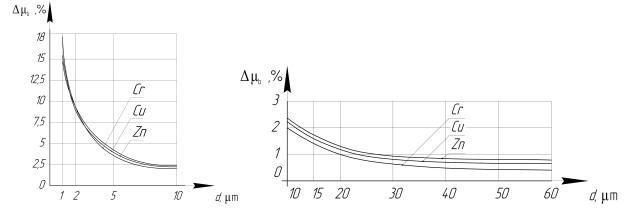


Figure 5. The maximum permissible deviation bases permeability $\mu_b(d)$ of calibration standards

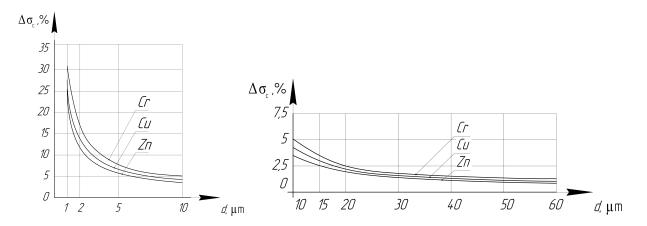


Figure 6. The maximum permissible deviation coating conductivity $\sigma_c(d)$ of calibration standards.

4. Metrological chain and structure of the second and third volumes of the standards

Fig. 7 shows four-level structure of the metrological chain to define and disseminate metallic coating thickness measurement scales. These are used to "produce" reference calibration standardsat user level. Naturally, direct calibration and the verification should be at the highest possible accuracy.

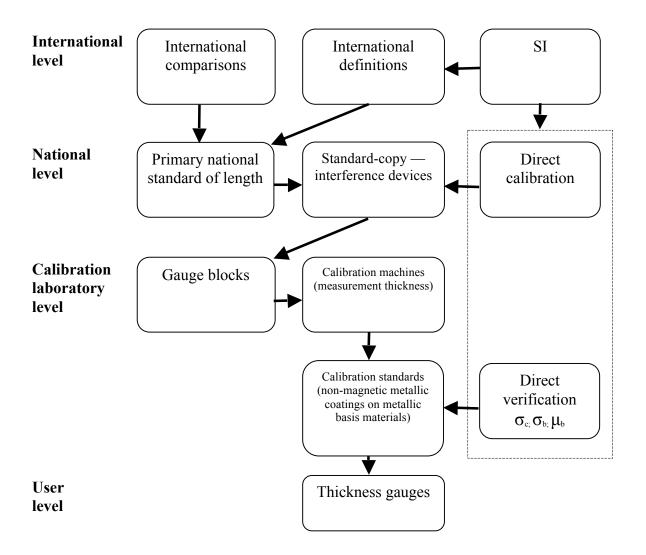


Figure 7. Four-level structure of the metrological chain to define and disseminate metallic coating thickness measurement scales.

The structure of the second volume of the standard should include the following sections:

- 1 Scope
- 2 Normative references
- 3 Generalcon ditions
- 4 Indirect verification

- Genera lProcedure
- Variation coefficient
- Error of Thickness gauges
- Measurement uncertainty
- 5 Intervals between verifications
- 6 Verification report / calibration certificate

Annex A (informative) Measurement uncertainty of calibration results

The structure of the third volume of the standard should include the following sections:

- 1 Scope
- 2 Normative references
- 3 Manufacture of reference calibration standards
- 4 Calibration
- General
- Traceability
- 5 Calibration procedure
- 6 Number of test measurement
- 7 Uniformity of measurement
- 8 Marking
- 9 Validity

Annex A(informative) Measurement uncertainty of reference calibration standards

Annex C (informative) Examples of reference calibration standards

5. Conclusion

The modeling of probe allowed to formulate the requirements for electromagnetic characteristics of the calibration standard.

The proposed four-level structure of the metrological chain and provides traceability standards, the possibility of international comparisons and improve the accuracy of the results of calibration and verification.