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Surface chemical analysis — Analysis of metal oxide films by glow discharge optical emission spectrometry

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National foreword

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2025-02

Surface chemical analysis — Analysis of metal oxide films by glow discharge optical emission spectrometry

Analyse chimique des surfaces — Analyse de films d'oxyde de métal par spectrométrie d'émission optique à décharge luminescente

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 8, *Glow discharge spectroscopy*.

This third edition cancels and replaces the second edition (ISO/TS 25138:2019), which has been technically revised.

The main changes are as follows:

— updated listed types of array detectors;

— anode size 2,5 mm has been added to those commonly offered in commercial instruments;

— verifying that test specimens and other samples form a good vacuum seal has been emphasized;

— revised optical system check to include instruments with array type detectors;

— text “setting of the high voltage for the detectors” replaced with “setting of the parameters for detector sensitivity” throughout this document; instructions to carry out these settings modified to accommodate array type detectors;

— instructions to optimize the crater shape has been changed from mandatory to optional;

— subclause 6.4 “Minimum performance requirements” has been revised.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Surface chemical analysis — Analysis of metal oxide films by glow discharge optical emission spectrometry

1 Scope

This document specifies a glow discharge optical emission spectrometric method for the determination of the thickness, mass per unit area and chemical composition of metal oxide films.

This method is applicable to oxide films 5 nm to 10 000 nm thick on metals. The metallic elements of the oxide can include one or more from Fe, Cr, Ni, Cu, Ti, Si, Mo, Zn, Mg, Mn, Zr and Al. Other elements that can be determined by the method are O, C, N, H, P and S.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 14707, *Surface chemical analysis — Glow discharge optical emission spectrometry (GD-OES) — Introduction to use*

ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The analytical method described in this document involves the following processes.

a) Preparation of the sample to be analysed, generally in the form of a flat plate or disc of dimensions appropriate to the instrument or analytical requirement (round or rectangular samples with a width of more than 5 mm, generally 20 mm to 100 mm, are suitable).

b) Cathodic sputtering of the surface metal oxide in a direct-current or radio-frequency glow discharge device.

c) Excitation of the analyte atoms in the plasma formed in the glow discharge device.

d) Spectrometric measurement of the intensities of characteristic spectral-emission lines of the analyte atoms as a function of sputtering time (qualitative depth profiling).

e) Conversion of the depth profile in units of intensity versus time to mass fraction versus depth by means of calibration functions (quantification). Calibration of the system is achieved by measurements on calibration samples of known chemical composition and measured sputtering rate.

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5 Apparatus

5.1 Glow discharge optical emission spectrometer

5.1.1 General

The required instrumentation includes an optical emission spectrometer system consisting of a Grimm type[4] or similar glow discharge source (direct-current or radio-frequency powered) and a simultaneous optical spectrometer as described in ISO 14707, capable of providing suitable spectral lines for the analyte elements. It is also common to combine this with a sequential spectrometer (monochromator), allowing the addition of an extra spectral channel to a depth profile measurement. An array-type detector, such as a charge coupled device (CCD), a complementary metal-oxide-semiconductor device (CMOS) or a charge injection device (CID) can also be used for simultaneous detection to cover a wide spectral range of the analytical lines.

The inner diameter of the hollow anode of the glow discharge source shall be in the range of 2 mm to 8 mm. A cooling device for thin samples, such as a metal block with circulating cooling liquid, is also recommended, but not strictly necessary for implementation of the method.

Since the principle of determination is based on continuous sputtering of the surface metal oxide, the spectrometer shall be equipped with a digital readout system for time-resolved measurement of the emission intensities. A system capable of a data acquisition speed of at least 100 measurements/second per spectral channel is recommended, but, for a large number of applications, speeds of >50 measurements/second per spectral channel are acceptable.

NOTE Pulsed mode of glow discharge source is available in some commercial instruments and can be beneficial in the analysis of heat-sensitive samples, for reduction of sputtering rate.

5.1.2 Selection of spectral lines

For each analyte to be determined, there exist a number of spectral lines which can be used. Suitable lines shall be selected on the basis of several factors, including the spectral range of the spectrometer used, the analyte mass fraction range, the sensitivity of the spectral lines and any spectral interference from other elements present in the test samples. For applications where several of the analytes of interest are major elements in the samples, special attention shall be paid to the occurrence of self-absorption of certain highly sensitive spectral lines (so-called resonance lines). Self-absorption causes non-linear calibration curves at high analyte mass fraction levels, and strongly self-absorbed lines should therefore be avoided for the determination of major elements. Suggestions concerning suitable spectral lines are given in Table B.1. Spectral lines other than those listed may be used, so long as they have favourable characteristics.

5.1.3 Selection of glow discharge source type

5.1.3.1 Anode size

Most GD-OES instruments on the market are delivered with options to use various anode diameters, 2 mm, 4 mm and 8 mm being the most common. Some older instruments have one anode only, usually 8 mm, while the most commonly used anode in modern instruments is 4 mm. A larger anode requires larger samples and higher power during analysis; therefore, the sample is heated to a greater extent. On the other hand, a larger anode gives rise to a plasma of larger volume that emits more light, resulting in lower detection limits (i.e. higher analytical sensitivity). Furthermore, a larger anode helps to mask inhomogeneity within a surface metal oxide. This may or may not be an advantage, depending on the application. In a large number of applications, the 4 mm anode is a good compromise. However, in surface analysis applications, it is rather common to encounter problems of overheating of the samples due to e.g. surface layers of poor heat conductivity and/or very thin samples. In such cases, a smaller anode is preferable (typically 2 or 2,5 mm) even if there is some loss of analytical sensitivity.

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5.1.3.2 Type of power supply

The glow discharge source can be either a type powered by a direct-current (DC) power supply or a radio-frequency (RF) type. The most important difference is that the RF type can sputter both conductive and non-conductive samples; hence this is the only type that can be used for e.g. polymer coatings and insulating oxide layers. On the other hand, it is technically simpler to measure and control the electrical source parameters (voltage, current, power) of a DC type. Several commercially available GD-OES systems can be delivered with the option to switch between DC and RF operation, but RF-only systems are becoming increasingly common. In short, there are a very large number of applications where DC or RF sources can be used and several where only an RF source can be used.

5.1.3.3 Mode of operation

Both DC and RF sources can be operated in several different modes with respect to the control of the electrical parameters (current, voltage, power) and the pressure. There are several reasons for this:

— "historical" reasons (older instruments have simpler but functional power supplies, while the technology has evolved so newer models have more precise and easier-to-operate source control);

— different manufacturers have chosen different solutions for source control;

— there are some application-related issues where a particular mode of operation is to be preferred.

This document gives instructions for optimizing the source parameters based on several available modes of operation. The most important reason for this is to make these instructions comprehensive so as to include several types of instrument. In most applications, there is no major difference between these modes in terms of analytical performance, but there are other differences in terms of practicality and ease of operation. For instance, a system equipped with active pressure regulation will automatically be adjusted to the same electrical source parameters every time a particular analytical method is used. Without this technology, some manual adjustment of the pressure to achieve the desired electrical source parameters is normally required. Alternatively, a method to correct for impedance variations by means of empirically derived functions[5] can be used, provided it is implemented in the software of the GD-OES systems.

NOTE In this context, what is known as the emission yield forms the basis for calibration and quantification as described in this document.[6][7] The emission yield has been found to vary with the current, the voltage and, to a lesser extent, the pressure.[5][8][9] It is impossible in practice to maintain all three parameters constant for all test samples, due to variations in the electrical characteristics of different materials. In several instrument types, the electrical source parameters (the plasma impedance) can therefore be maintained constant by means of automatic systems that vary the pressure during analysis.

6 Adjusting the glow discharge spectrometer system settings

6.1 General

Follow the manufacturer's instructions or locally documented procedures for preparing the instrument for use.

RF sources differ from DC sources in the respect that for several instrument models, only the applied (forward) RF power can be measured, not the actual power developed in the glow discharge plasma. The applied RF power is normally in the range 10 W–100 W, but it must be noted that the RF power losses in connectors, cables, etc. vary considerably between different instrument models. Typical power losses are in the range 10 %–50 % of the applied power. Furthermore, the possibilities to measure the additional electrical parameters voltage and current in the plasma are generally restricted due to technical difficulties with RF systems, and several existing instrument models can only measure the applied RF power.

There is no difference between DC and RF concerning the possibilities to measure the pressure. However, there are large pressures differentials in a Grimm type source, and pressure readings obtained depend on the location of the pressure gauge. Some instrument models have a pressure gauge attached to measure the actual pressure in the plasma, while others have a pressure gauge located on a "low pressure" side of the source closer to the pump. Therefore, the pressure readings can, for several instruments, just be used

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to adjust the source parameters of that particular instrument, not as a measure of the actual operating pressure in the plasma.

For test specimens, calibration samples and validation samples, it is important to ensure before analysis that once located in the glow discharge source there are no vacuum leaks. Special attention must be paid on the sealing between the sample and glow discharge source.

For an optical system with photomultiplier detectors in fixed spectral positions, the most important preparation step is to check that the entrance slit to the spectrometer is correctly adjusted, following the procedure given by the instrument manufacturer. This ensures that the emission intensities are measured on the peaks of the spectral lines for optimum signal-to-background ratio. For further information, see ISO 14707. For an optical system with CCD detectors, the corresponding control is to check that the wavelength calibration is correct, following the procedure given by the instrument manufacturer.

The most important step in developing a method for a particular application is to optimize the parameters of the glow discharge source. The source parameters shall be chosen to achieve three aims:

a) adequate sputtering of the test sample, to reduce the analysis time without overheating the sample;

b) good crater shape, for good depth resolution;

c) constant excitation conditions in calibration and analysis, for optimum accuracy.

Trade-offs are often necessary among the three specified aims. More detailed instructions on how to adjust the source parameters are given in the following subclauses.

The settings of the parameters for detector sensitivity depend on the source parameters, but the procedure is the same for all modes of operation of the source. This procedure is therefore only described for the first mode of operation.

Similarly, the steps to adjust and optimize the source settings in terms of signal stability and sputter crater shape are also similar in principle for all modes of operation. Therefore, these procedures are only described in detail for the first mode of operation.

6.2 Setting the parameters of a DC source

6.2.1 Constant applied current and voltage

The two control parameters are the applied current and the applied voltage. Set the power supply for the glow discharge source to constant-current/constant-voltage operation (current set by the power supply, voltage adjusted by pressure/gas flow regulation). Then, set the current and voltage to the typical values recommended by the manufacturer. Alternatively, set the power supply to constant voltage/constant current operation (voltage set by the power supply, current adjusted by pressure/gas flow regulation). If no recommended values are available, set the voltage to 700 V and the current to a value in the range 5 mA to 10 mA for a 2 mm or 2,5 mm anode, 15 mA to 30 mA for a 4 mm anode or 40 mA to 100 mA for a 7 mm or 8 mm anode. If no previous knowledge of the optimum current is available, it is recommended to start with a value somewhere in the middle of the recommended range.

NOTE For the purposes of this document, there is no difference between the two alternative modes of operation described above.

6.2.1.1 Setting the parameters for detector sensitivity

Select test samples with surface layers of all types to be determined. For all test samples, run the source while observing the output signals from the detectors for the analyte atoms. Parameters for detector sensitivity in such a way that sufficient sensitivity is ensured at the lowest analyte mass fraction without

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saturation of the detector system at the highest analyte mass fraction. For array type detectors (CCD and CID), adjust the integration time in the same way as the high voltage for PMT.

NOTE Some instruments allow to work in dynamic mode, where all measured intensities are converted into the intensity, which would be measured at the lowest sensitivity (shortest integration time or lowest PMT voltage). In this case, you can calibrate and measure the samples at optimal sensitivity. For this dynamic operation mode to function, it is necessary to determine the ratio of the sensitivities as a function of integration time/PMT voltage beforehand.

6.2.1.2 Adjusting the source parameters

For each type of test sample, carry out a full depth profile measurement, sputtering it in the glow discharge for a sufficiently long time to remove the metal oxide completely and continue well into the base material. By observing the emission intensities as a function of sputtering time (often referred to as the qualitative depth profile), verify that the selected source settings give stable emission signals throughout the depth profile and into the substrate. If this is found not to be the case, reduce one of the control parameters by a small amount and sputter through the metal oxide again. If the stability is still unsatisfactory, reduce the other control parameter by a small amount and repeat the measurements. If found necessary, repeat this procedure for a number of control parameter combinations until stable emission conditions are obtained.

NOTE Unstable emission signals can indicate thermal instability in the sample surface layers; sample cooling is beneficial in this regard.

6.2.1.3 Optimizing the crater shape (optional)

If a suitable profilometer device is available, adopt the following procedure. Sputter a sample with a metal oxide typical of the test samples to be analysed to a depth of about 10 µm to 20 µm, but still inside the metal oxide. This is only possible for applications where surface metal oxide layers of such thickness are available. If no such sample is available, use a steel or brass sample. Measure the crater shape by means of the profilometer device. Repeat this procedure a few times using slightly different values of one of the control parameters. Select the conditions that give an optimally flat-bottomed crater. These conditions are then used during calibration and analysis, provided that the stability of the emission conditions obtained in step 6.2.1.2 is not compromised. In some cases, there is a certain trade-off between these two requirements.

6.2.2 Constant applied current and pressure

The two control parameters are the applied current and the pressure. Set the power supply for the glow discharge source to constant-current operation. Then set the current to a typical value recommended by the manufacturer. If no recommended values are available, set the current to a value in the range 5 mA to 10 mA for a 2 mm or 2,5 mm anode, 15 mA to 30 mA for a 4 mm anode or 40 mA to 100 mA for a 7 mm or 8 mm anode. If no previous knowledge of the optimum current is available, it is recommended to start with a value somewhere in the middle of the recommended range. Sputter a typical coated test sample, and adjust the pressure until a voltage of approximately 700 V is attained in the metal oxide.

Set the parameters for detector sensitivity as described in 6.2.1.1.

Adjust the discharge parameters as described in 6.2.1.2, adjusting first the current and, if necessary, the pressure.

Optimize the crater shape as described in 6.2.1.3 by adjusting the pressure. These conditions are then used during calibration and analysis (optional).

Before sputtering a new sample type, make a test run in order to ensure that the voltage has not changed by more than 5 % from the previously selected value. If this is the case, readjust the pressure until the correct value is attained.

6.2.3 Constant voltage and pressure

The two control parameters are the applied voltage and pressure. Set the power supply for the glow discharge source to constant voltage operation. First set the voltage to a typical value recommended by the manufacturer. If no recommended values are available, set the voltage to 700 V. Sputter a typical coated

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test sample, and adjust the pressure until a current in the approximate range 5 mA to 10 mA for a 2 mm or 2,5 mm anode, 15 mA to 30 mA for a 4 mm anode, 40 mA to 100 mA for a 7 mm or 8 mm anode is attained in the surface layers. If no previous knowledge about the optimum current is available, it is recommended to start with a value somewhere in the middle of the recommended range.

Set the parameters for detector sensitivity as described in 6.2.1.1.

Adjust the source parameters as described in 6.2.1.2, adjusting first the voltage and, if necessary, the pressure.

Optimise the crater shape as described in 6.2.1.3, by adjusting the pressure. These conditions are then used during calibration and analysis (optional).

Before sputtering a new sample type, make a test run in order to ensure that the current is not altered more than 5 % from the previously selected value. If this is the case, readjust the pressure until the correct value is attained.

For analysis of very thin layers < 100 nm, it is recommended to test and evaluate all three modes described above. There may be a small difference in the very short start-up of the discharge, affecting the analytical results to some extent. In the constant voltage – constant current mode, the pressure is increased until ignition occurs. The pressure must then be decreased to reach the selected current. In the constant current – constant voltage mode the voltage increases until ignition occurs, if the correct start pressure is set the voltage rapidly reaches the set value with minimal pressure regulation. It is therefore important to adjust the start pressure before ignition to a value such that the voltage and current values in the thin layer come very close to those used for calibration of the instrument. A few trials on a sample with the correct type of thin layer can be necessary. The purpose is to minimize changes in the discharge parameters during the short time the nanolayer is sputtered. Detailed instructions are given in 6.4.4.

6.3 Setting the discharge parameters of an RF source

6.3.1 General

The most common operating modes of RF sources are the following: constant applied power and constant pressure; constant applied voltage and constant pressure; or constant effective power and applied RF voltage (the RF voltage is defined here as the RMS voltage at the coupling electrode without DC bias). In addition, the mode constant applied power and DC bias voltage is sometimes used, but less common. All RF operational modes are allowed in this document, provided they meet the three aims listed in 6.1. In the following, separate instructions are provided on how to set the parameters for the different operational modes.

6.3.2 Constant applied voltage and pressure

The two control parameters are the applied power and the pressure. First set the applied power and adjust the source pressure to the values suggested by the manufacturer. If recommended values are not available, set the applied power and pressure to somewhere in the middle of the ranges commonly used for depth profiling of metal samples. Measure the penetration rate (i.e. depth per unit time) on an iron or steel sample, adjusting the power to give a penetration rate of about 2 µm/min to 3 µm/min.

Set the parameters for detector sensitivity as described in 6.2.1.1.

Adjust the discharge parameters as described in 6.2.1.2, adjusting first the applied power and, if necessary, the pressure.

Optimize the crater shape as described in 6.2.1.3 by adjusting the pressure (optional).

Re-measure the penetration rate on the iron or steel sample and adjust the applied power, if necessary, to return to about 2 µm/min to 3 µm/min. Repeat the cycle of power and pressure adjustment until no significant change is noted in the penetration rate or crater shape. Note the power and pressure used in units provided for the instrument type. These conditions are then used during calibration and analysis.

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6.3.3 Constant applied power and DC bias voltage

The two control parameters are the applied power and the DC bias voltage. First, set the applied power and adjust the source pressure to attain a DC bias typical of the values suggested by the manufacturer. If recommended values are not available, set the applied power and DC bias voltage to somewhere in the middle of the range commonly used for depth profiling of metal samples. On instruments equipped with active pressure control, this can be achieved automatically. Measure the penetration rate (i.e. depth per unit time) on an iron or steel sample, adjusting the power to give a penetration rate of about 2 µm/min to 3 µm/min.

Set the parameters for detector sensitivity as described in 6.2.1.1.

Adjust the discharge parameters as described in 6.2.1.2, adjusting first the applied power and, if necessary, the DC bias voltage.

Optimize the crater shape as described in 6.2.1.3 by adjusting the DC bias voltage (optional).

Remeasure the penetration rate on the iron or steel sample and adjust the applied power, if necessary, to return to about 2 µm/min to 3 µm/min. Repeat the cycle of power and DC bias voltage adjustment until no significant change is noted in the penetration rate or in the crater shape. If this is not the case, readjust the DC bias voltage until the correct value is attained. Note the power and DC bias voltage used in units provided for the instrument. These conditions are then used during calibration and analysis.

NOTE This mode can only be used if conductive samples are measured, since the DC bias vanishes at non-conductive samples.

6.3.4 Constant effective power and RF voltage

The two control parameters are the effective power and the RF voltage. Constant effective power is defined here as the applied power minus the reflected power and the “blind power” measured with the sample in place but without plasma (vacuum conditions). The RF voltage is defined here as the RMS voltage at the coupling electrode without DC bias.

Set the power supply for the glow discharge source to constant effective power/constant RF voltage operation. First set the power to a typical value recommended by the manufacturer. If no recommended values are available, set the RF voltage to 700 V and the power to a value in the range 10 W to 15 W for a 4 mm anode, 5 W to 10 W for a 2 mm anode to give an example. If no previous knowledge of the optimum power is available, it is recommended to start with a value somewhere in the middle of the recommended range.

Set the parameters for detector sensitivity as described in 6.2.1.1.

Adjust the discharge parameters as described in 6.2.1.2, adjusting first the effective power and, if necessary, the RF voltage.

Optimize the crater shape as described in 6.2.1.3 by adjusting the RF voltage. Select the conditions that give an optimally flat-bottomed crater. These conditions are then used during calibration and analysis (optional).

NOTE In order to determine the RF RMS voltage, it is essential to include the offset caused by the DC bias voltage, which is not possible to measure at the coupling electrode when non-conductors are sputtered. However, when only relatively thin (<100 µm) non-conductive layers on a conductive specimen are analysed, the offset can be estimated and the amplitude of the RF voltage multiplied by 1,22 can be used as a good estimate of the RMS voltage[10]. For thick non-conductors more complex calculations must be carried out.[11] However, analysis of thick non-conductors is outside the scope of this document.

6.4 Minimum performance requirements

6.4.1 General

It is desirable for the instrument to conform to the performance specifications given in 6.4.2 and 6.4.3 below, for applications to very thin layers < 100 nm also to 6.4.4.

NOTE Setting up for analysis commonly requires an iterative approach to the adjustment of the various instrumental parameters described in this document.

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