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Surface chemical analysis — Glow-discharge mass spectrometry (GD-MS) — Introduction to use

Analyse chimique des surfaces — Spectromètrie de masse à décharge luminescente (GD-MS) — Introduction à l'utilisation

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Contents

Foreword

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ISO 15338 was prepared by Technical Committee ISO/TC 201, *Surface Chemical analysis*, Subcommittee SC 8, *Glow discharge spectroscopy*.

Surface chemical analysis — Glow-discharge mass spectrometry (GD-MS) — Introduction to use

1 Scope

This Standard is a guide to the operation and recommendations for the use of glow discharge mass spectrometry (GD-MS).

Note: This Standard should be read in conjunction with the instrument manufacturer's manuals and recommendations.

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 18115:2001/PDAM 1, *Surface chemical analysis — Vocabulary — Supplement*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 5725-6:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values.*

ISO/DIS 18115, *Surface chemical analysis — Vocabulary*

1 Safety

3.1 General

The following precautions should be taken to ensure the safety of operators and their environment, during operation of glow discharge mass spectrometry:

(a) Use of high voltage power supply and connection of the instrument.

(b) Use and storage of compressed gas cylinders.

(c) Handling of cryogenic materials.

3.2 Use of high voltage power supply and connection of the instrument

Electrical connection should comply with the regulations in force. Particular care should be taken to ensure that connection of the instrument to ground/earth is correct, and the efficiency of the ground/earth connection should be checked.

3.3 Use and storage of compressed gas cylinders

The compressed gas cylinders should be regularly tested by the appropriate authorities. Preferably, cylinders should not be stored or used inside the laboratory. Rather, they should be located outside the laboratory in a place that is well ventilated, away from direct heat, and accessible to service and safety personnel. The cylinders should be provided with suitable pressure reducing valves. If more than one cylinder is to be used or stored in close proximity, it is advisable to indicate in some way which cylinder or cylinders are currently in use.

3.4 Handling of cryogenic materials

The installation of vessels of cryogenic materials shall be located so as to minimize the risk to personnel. Area where cryogenic liquids are stored and used shall be ventilated to prevent the accumulation of gas or vapour which could evaporate from the liquid. It is good practice to keep areas where cryogenic liquids are used very clean. All transfer operation shall be in accordance with statutory requirement. When a cryogenic liquids is being transferred from one vessel to another, precautions shall be taken to minimize any spills and splashing. The requirement of the relevant regulatory authority shall also be met.

4 Principle

In a glow discharge source electrical power is supplied between the sample (cathode) and the anode by a power supply typically operated in direct current (dc) at 0.5 to 2 kV and 1 to 30 mA. Argon (or other inert gas such as neon, krypton or helium) is introduced into the discharge cell. The pressure inside the discharge cell is typically a few hundred Pascals (Pa). The potential difference between the cathode and the anode is applied and a glow discharge (plasma) is established. Sample material (single atoms and/or clusters) which are sputtered by ions and neutrals diffuse into the plasma.

Ions formed in the glow discharge are extracted from the cell and pass into a mass analyzer. The mass analyzer is used to transmit ions of given mass to charge ratio to the detector(s). The ions reaching the detector(s) are measured directly as ion current or counted by a counting system. Information is stored in a computer system. Elemental mass fractions are typically calculated by the instrument software using the ion currents of isotopes, by normalizing the signal to the signal of a matrix element and subsequently comparing the normalized signals with those arising from the corresponding elements in calibration samples.

5 Terms and definitions

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

5.1
Abundance sensitivity
ratio of the maximum ion current recorded at a mass m to the ion current arising from the same species recorded at an adjacent mass ($m \pm 1$).

5.2
Accuracy of measurement
the closeness of the agreement between a test result and the accepted reference value.

5.3
Anode
more positively charged electrode in a glow discharge device.

5.4
Cathode
more negatively charged electrode in a glow discharge device.

5.5
Chemical specie
atom, molecule, ion or functional group.

5.6
Detection limit (DL)
smallest amount of an element or compound that can be measured under specified analytical conditions.

5.7
Elemental intensity
the amount of ion current recorded for a particular element.

5.8
Flat cell
a sample cell used for the analysis of flat samples.

5.9
Glow discharge, abnormal
glow discharge operated in a current/voltage regime for which an increase in current is accompanied by an increase in voltage.

5.10
Glow discharge, normal
glow discharge operated in a current/voltage regime for which an increase in current is accompanied by little or no detectible change in voltage.

5.11
Ion beam ratio (IBR)
the signal intensity of the analyte ion divided by the intensity of the matrix ion(s), both corrected for isotopic abundance.

5.12
Intensity, peak
measure of signal intensity for a constituent spectral peak.

5.13
Intensity, signal
strength of a measured signal at a spectrometer detector or after some defined processing.

5.14
Interference signal
signal measured at the position of mass of interest due to another, undesired, species.

5.15
Mass to charge ratio
mass of an ion divided by the number of electrons added to or removed from it to form an ion.

5.16
Pin cell
sample cell used for the analysis of wire and rod samples.

5.17
Plasma
gas consisting of ions, electrons, and neutral particles.

5.18
Preburn
period during which preburning occurs, i.e. period when plasma is on before analysis.

5.19
Presputtering period
process of sputtering, prior to signal registration, employed to allow steady state sputtering to be established and analytical signals to stabilise.

5.20
Precision of measurements
the closeness of the agreement between independent test results obtained under stipulated conditions, normally reported as a standard deviation.

5.21
Reference material
material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of measured methods, or for assigning value to materials.

5.22
Reference material, certified
reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified values is accompanied by an uncertainty at a stated level of confidence.

5.23
Resolution of spectrometer
contribution of the spectrometer to the measured full width at half maximum (FWHM) or at 10% height of maximum intensities of spectral peaks above their local backgrounds.

5.24
Resolving power of a spectrometer
ratio of the mass to the resolution of the spectrometer at that mass.

5.25
Pin, rod and wire sample
a sample with cylindrical or square cross section of nominal length typically 20 mm and not normally exceeding 10 mm across.

5.26
Secondary cathode
electrically conductive mask, containing an aperture, used to enable sputtering of an electrically nonconductive sample surface in a direct glow discharge device.

5.27
Sensitivity factor, relative (RSF)
coefficient for an element with which the measured intensity of a mass peak for that element, divided by the measured intensity of a mass peak of a matrix element, is multiplied to yield the mass fraction of that element in the sample divided by the mass fraction of the matrix element.

NOTE RSF values for some other techniques are sometimes calculated as the inverse of the definition used here for GD-MS.

5.28
Transmission
a ratio of the number of ions reaching the detector relative to the number of ions entering the mass analyzer.

6 Materials (Reagents)

Water- deionized water, 18 M ohm or better.

Argon gas (Ar) - Of purity at least 99.9995% argon (or other gases of high purity)

Liquid nitrogen (LN2) - For cryogenic cooling of discharge cell.

Compressed air - To operate pneumatic valves.

7 Apparatus

7.1 General

A glow discharge mass spectrometer typically consists of the following parts:

(a) Ion source

(b) Mass analyzer

(c) Detector system

(d) Data acquisition and control

7.2 Ion source

7.2.1 General

A glow discharge ion source consists of a glow discharge cell and a power supply. The ion source also may contain a series of focusing plates, external to the cell, whose function is to extract ions from the cell and focus these ions into the mass spectrometer.

Typically the body of the discharge cell is connected to the anode output of the power supply. The sample serves in the glow discharge cell as a cathode and is connected to the cathode output of the power supply. The discharge cells have been designed to accommodate samples in the geometries recommended in 8.3 and examples of the discharge cells are illustrated with the appropriate sample holders in Figures 1a and 1b.

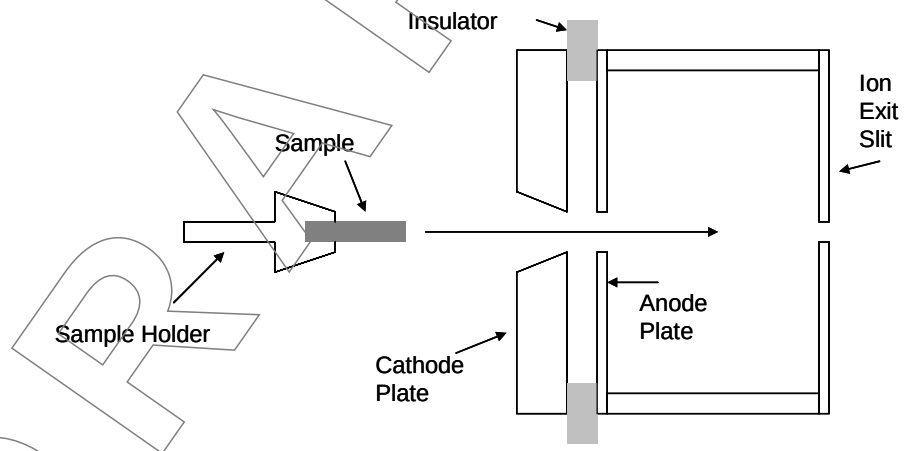

Figure 1a — Example of a cell used for the analysis of pin samples