ISO TC201 SC8 N348

ISO /DTS 15338 (Ed.3): xxxx(E)

ISO /TC 201/SC 8

Secretariat: JISC

Date: 2024-11-25

Surface chemical analysis — Glow discharge mass spectrometry — Operating procedures

DTS stage

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

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Foreword

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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 ~~documents~~ 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 15338:2020), which has been ~~minor~~ technically revised.

The main changes ~~compared to the previous edition~~ are as follows:

— ~~Minor editorial changes to and~~ additional technical information have been added to the principle, apparatus and routine operations~~.~~

~~A list of all parts in the ISO/TS15338 series can be found on the ISO website.~~

— minor editorial changes.

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Surface chemical analysis — Glow discharge mass spectrometry — Operating procedures

1 Scope

This document givesspecifies procedures for the operation and use of glow discharge mass spectrometry (GD-MS). There are several GD-MS systems from different manufacturers in use and this document describes the differences in their operating procedures when appropriate.

NOTE This document is intended to be read in conjunction with the instrument manufacturers' manuals and recommendations.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Principle

In a glow discharge source, a potential difference is applied between the cathode (the sample to be analysed) and the anode, and a plasma is supported by the introduction of an inert gas, normally argon, but other inert gases can be used. This potential difference can be either direct current (DC) or radio frequency (RF), the advantage of RF being that electrically insulating materials can be analysed directly. The impacts of inert gas ions and fast neutrals formed within the plasma on the surface of the sample result in the production of neutrals by sputtering from surface.

These neutrals diffuse into the plasma where they are subsequently ionised within the equipotential area of the plasma and can then be extracted to a mass spectrometer for analysis. Both magnetic sector and time of flight spectrometers are available.

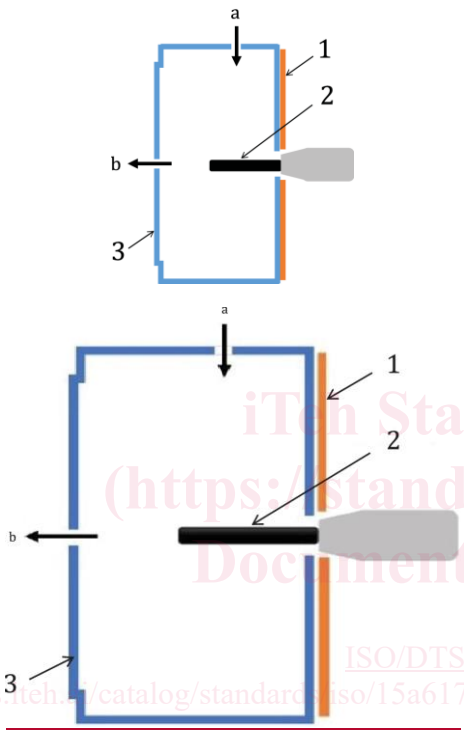
4 Apparatus

4.1 5.1 Ion source

There are two fundamental types of ion source used for the GD-MS, a low flow or "static" source, and a fast flow source. Both types can accept pin samples or samples with a flat surface. A typical pin would be 20 mm long with a diameter of 3 mm, and a typical flat sample would be 20 mm to 40 mm diameter. It must be big enough to cover the hole in the chosen anode plate and provide a good gas seal. More details of these dimensions can be found later.

In the low flow source, the plasma cell is effectively a sealed unit held within a high vacuum chamber, with a small exit slit or hole to allow the ions to exit the cell and enter the mass spectrometer. The cell body is at

anode potential, the acceleration potential of the mass spectrometer, and the sample is held at cathode potential, typically 1 kV below anode potential. In this type of source, the argon flow is typically one sccm (standard atmosphere cubic centimetres per minute) or less, and the gas used, normally argon, should be of very high purity, six nines five or better. The power of the plasma is relatively low, typically 2 W or 3 W; the potential difference is typically 1 kV and the current 2 mA or 3 mA.



Key

1 insulator
2 sample (cathode)
3 anode (GD cell)
[a] Gas inlet (0,3 sccm to 0,6 sccm~~)~~.
[b] To mass spectrometer.

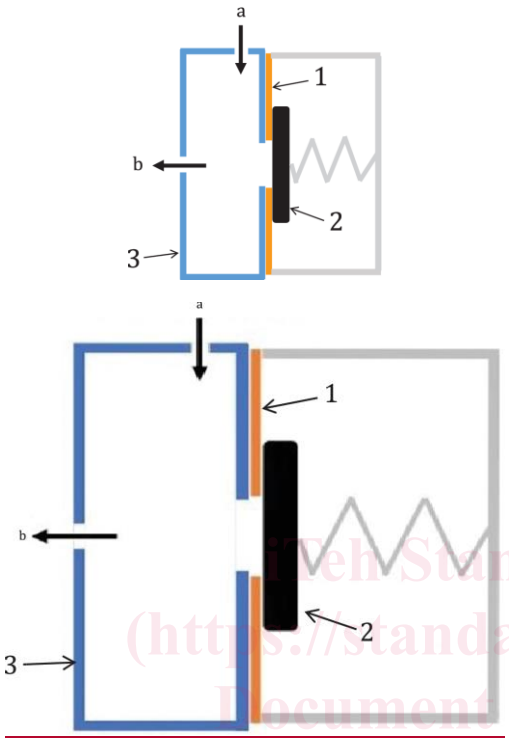
Figure 1 — Low flow source pin geometry

A schematic diagram of the low flow source in pin geometry is shown in ~~Figure 1.~~Figure 1. The gas is introduced into the cell through a metal pipe which forms a metal to metal seal with the cell body. On some systems an alternative of a PEEK tube with a ferrule seal to the cell body is used. If a metal pipe is used, then an insulating material must be included in the gas line as the cell is at anode potential. This is normally a piece of quartz with a very small diameter hole through which the gas passes. ~~–~~The pin sample is held in a chuck which sits at cathode potential and the cell body is at anode potential, so the two are separated by an insulating disc. The chuck is actually located against a metal (tantalum) plate which also sits at cathode potential (not shown in the schematic diagram). The whole assembly forms a good gas seal while maintaining good electrical insulation. The only escape for the gas and any ions formed in the plasma is through a small slit or hole at the back of the cell, and this creates a pressure differential between the cell and the surrounding source vacuum

chamber. It is normal to measure the pressure outside the cell in a low flow source rather than in the cell itself, the presence of a plasma making the measurement difficult. In this geometry, the potential difference between the anode and cathode "drops" in a small sheath approximately ~~1mm~~1 mm around the sample, thus leaving the main gas volume in the cell at the same potential. So any ions formed in the "plasma cloud" will not be electrically attracted back to the cathode.

It is standard practice in the low flow source to cool the plasma to near liquid nitrogen temperatures. This has been shown to reduce significantly the formation of molecular species associated with the matrix and plasma support gas combined as dimers or trimers, or with gas backgrounds such as hydrogen, nitrogen and oxygen. Cooling the sample in this way also allows for the measurement of low melting point materials such as gallium and indium, materials that would melt under normal plasma conditions.

Heat transfer between the components of the plasma cell needs to be considered. The whole cell assembly is floated up to the accelerating potential, so the anode will typically be around 6 kV to 8 kV while the sample (cathode) is at approximately 1 kV lower during operation. The design of the heat exchanger, or cooling assembly, means that it will be sitting at ground potential, and so it is connected to the cell insulating disc which is of a larger diameter than the cell body (not shown in ~~Figure 1).~~Figure 1). Thus, it is necessary for the insulating disc to have a good coefficient of heat transfer at the same time as being electrically insulating; the material boron nitride is ideal for this and is used in most systems. It is important to consider heat transfer through all junctions, particularly from the cell body and cathode plate through the insulator to the heat exchanger. ~~And in~~In order to make the sample cold, it is important to use a sample holder, or chuck, that is of a similar diameter to the sample itself. It is possible to maximise the heat transfer if attention is paid to detail, for example, a piece of Indium foil can be cut and shaped to fit between the heat exchanger and the insulator, providing that care is taken to ensure that there can be no electrical leakage. If a pin sample of square cross section is analysed, then the contact with the round hole in the holder gives very poor heat transfer, limiting the cooling of the sample. This can be improved by warming the chuck and filling with pure Gallium before inserting the sample.



Key

1 insulator
2 sample (cathode)
3 anode (GD cell)
a Gas inlet (0,2 sccm to 0,4 sccm~~)~~.
b To mass spectrometer.

Figure 2 — Low flow source flat geometry

~~Figure 2~~Figure 2 shows a schematic of the low flow source in flat geometry. The sample, which forms the cathode in the plasma, is pressed against an insulator which in turn is pressed against the cell body which is at anode potential. Again, a good gas seal is formed and the only escape route for the gas and the sputtered particles is through the ion exit slit or hole. The area of the sample exposed to the plasma can be varied by the choice of the insulator used. If a larger area of sample is exposed then a larger ion beam will be produced, but it is possible to use smaller insulators to allow smaller samples to make a gas seal~~. Or,~~ or to reduce the area exposed to analysis. Commercial systems will generally have insulators available to allow areas from 2 mm to 20 mm diameter to be exposed, but 10 mm to 15 mm is the normal. The most important need is that the sample surface shall be flat in order to make a good gas seal with the insulating disc.

The gap between the sample (cathode) and the anode has to be small (less than a critical distance, typically 1 mm) to avoid creation of a discharge in the gap. It is often a problem that sputtered material can be deposited on the inner diameter of the insulator, creating a short circuit between anode and cathode. This can be avoided by the use of two insulators with different diameter holes.