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1 Executive summary

Introduction

Quadrupole mass spectrometers (QMS) are used to measure partial pressures in vacuum. Industry uses QMS to control vacuum processes and to characterise materials built into vacuum chambers. Before this project there was no internationally agreed procedure to characterize QMS in order to use their measurement results in a quantitative manner traceable to the SI. This project established such procedures and published them by two Technical specifications at the ISO level.

The problem

Since no internationally accepted procedures existed on how to characterize QMS, the users of these instruments developed their own "localised" procedures as it happened in the semiconductor, pharmaceutical and coating industry as well as in particle accelerator facilities like CERN, DESY or Daresbury Laboratory. This caused the problem that results from the different fields of usage were not comparable and users had no acceptance guidelines when they wanted to accept the instrument from the manufacturer.

QMS are also used to characterize materials for vacuum applications in terms of outgassing rates. Also in this field, no internationally accepted procedures which offered traceability to the SI existed. This caused the problem that published results of outgassing rates were hardly comparable.

To change this situation, the ISO Technical Committee 112 "Vacuum Technology", the primary supporter of this project, requested that the necessary steps were taken to improve the traceability of partial pressure and outgassing rate measurements. This was possible, because the project EMRP IND12 'Vacuum metrology for production environments' had paved the way to write guidelines on how to characterise and calibrate QMS and how to perform traceable outgassing rate measurements.

The solution

This project has developed two Technical Specifications (TS) at the ISO level; the first Technical Specification was on how to characterise and calibrate quadrupole mass spectrometers (QMS), and the second was on how to perform traceable outgassing rate measurements. In an extension of the guidelines drafted in the former project EMRP IND12 now a much wider community was involved in writing the two TS, namely manufacturers of QMS, industrial users, scientific users and National Metrological Institutes. A workshop was held with these different communities to cover as many aspects as possible. The responsible working group WG2 of ISO TC 112 worked closely together with these groups to finally draft the TS.

Impact

ISO TC 112 as primary supporter initiated two new projects (in terms of ISO) to develop two TS, one for the characterisation and calibration of QMS, the second on procedures to measure and report outgassing rates. As project leader in the responsible ISO TC 112 working group WG2 the coordinator of this project was chosen. The WG2 and this project worked closely together and finally established the following two TS:

ISO TS 20175 Vacuum technology — Vacuum gauges— Characterization of quadrupole mass spectrometers for partial pressure measurement

ISO TS 20177 Vacuum technology — Vacuum gauges — Procedures to measure and report outgassing rates

ISO TS 20175 was published in April 2018, ISO TS 20177 in June 2018.

These two related Technical Specifications will enable the improvement of materials for the semiconductor industry and make the control of partial pressures in industrial processes more reliable. The Technical Specifications will also help manufacturers of QMS to improve their metrological performance.

The company ASML, which is focussed on semiconductor lithography systems by extreme ultraviolet radiation (EUV) at 13 nm, with their suppliers will incorporate the two TS in their procedures for evaluating quadrupole mass spectrometers and outgassing rates and propose the two TS to the SEMI (Semiconductor Equipment and Materials International) organisation for further consideration.

2 Need for the project

Key outputs from a previous EMRP project IND12 Vacuum (Vacuum metrology for production environments) were guidelines on how to characterise and calibrate QMS and how to perform traceable outgassing rate measurements. QMS are needed to perform outgassing rate measurements therefore they were a prerequisite for the latter and so the results were closely related.

The ISO Technical Committee 112 "Vacuum Technology" (ISO TC 112), the primary supporter of this project, has requested the implementation of the results from the project IND12 into Technical Specifications so that a wider community may use the results from the project and to ensure that the necessary steps to improve the traceability of partial pressure and outgassing rate measurements are taken.

The need for standardised procedures to characterise and calibrate QMS was highlighted in the results of an end user questionnaire conducted within IND12. The end users represented the field of high energy accelerators and fusion machines, suppliers of Extreme ultraviolet (EUV) components, of positioning devices of samples in vacuum chambers and research contract services from industry, (i.e. industries involved in production of semiconductors). In addition, IND12 demonstrated several metrological problems for QMS. It was concluded that particular care needs to be taken to overcome these problems in order to obtain valuable and reliable results for QMS and that some of these problems can only be solved by standardised procedures to characterise and calibrate QMS.

Outgassing rate measurements, which were, in the past, mainly performed by scientists in order to achieve very low pressures in vacuum systems, nowadays have great importance in industry; in particular, for EUV Lithography used in wafer illumination and for high energy accelerators used in cancer therapies. The results obtained within IND12 identified suitable traceable and validated methods for outgassing rate measurements for use by such industries, however in order to support comparability between outgassing rate measurement systems, using different methods, standardisation is required.

3 Objectives

The overall objective was to create impact from the results of JRP IND12 Vacuum via incorporating measurement techniques into formal ISO Technical Specifications. These results were explained to and coordinated with experts from other countries who were not involved in the JRP IND12. This was carried out within working group 2 (WG 2) "Vacuum Instrumentation" of ISO TC 112 "Vacuum Technology" which is the responsible group at the ISO level for technical drafts for this field.

This project addressed the following objectives:

1. To incorporate the results obtained for the characterisation and calibration of QMS as part of EMRP JRP IND12 into ISO/NP TS 20175 in order to support their wider dissemination and uptake.
2. To incorporate the results obtained for the procedures of outgassing rate measurements as part of EMRP JRP IND12 into ISO/NP TS 20177 in order to support their wider dissemination and uptake.

Having achieved these objectives the accuracy of partial pressure measurement in vacuum will increase in all fields and will make outgassing rate measurements more reliable.

4 Results

Objective 1 - To work with ISO TC 112 to develop a Technical Specification for the characterisation and calibration of QMS.

To fulfil the objective, two successive working drafts were developed, the first in October 2015, the second in May 2016. The workload between the participants was shared as follows during the whole project: PTB drafted the frame and content sections, worked out the sections on the parameters to be specified for different applications, the section on vacuum calibration systems by continuous expansion and comparison systems. IMT worked out the sections on in-situ vacuum calibration systems, the calibration procedure for mass resolution and on measurement uncertainties. A common effort of PTB and IMT were the introduction and the scope, the calibration procedures for minimum detectable partial pressure, minimum detectable concentration, dynamic range, sensitivity and interference effect ratio, linear response range, relative sensitivity factor, fragmentation pattern, outgassing and pumping of the instrument, PTB also made the communication with ISO TC 112 and organised the workshop and the TC 112 meetings. The first draft was discussed by the Working Group 2 (WG 2) members of "Vacuum Instrumentation" of ISO TC 112 in November 2015. The comments were considered in the succeeding working draft. This was published on the website of the project and uploaded on the Livelink website of ISO. Besides the Working Group 2, the wider community of manufacturers and users of QMS and researchers who use QMS were asked to comment by November 2016. The comments were discussed at a workshop from Jan 30 - Feb 1, 2017 and at the following WG 2 meeting on Feb 2-3, 2017. As a result of these discussions, a new draft was developed and put to vote within ISO TC 112 WG 2. The WG 2 decided that, after some minor changes, the draft should be forwarded to ISO TC 112 to be put to a vote. This vote took place from beginning of August to early November 2017.

As a result of the vote, the ISO DTS (Draft Technical Specifications) 20175 was approved without any further comment. On the TC 112 meeting on Nov 30, 2017, it was decided to submit the DTS for publication.

The headquarter of ISO in Geneva reviewed the DTS and proposed several additional editorial corrections, which were assessed in March 2018.

ISO/TS 20175 was published in April 2018 (https://www.iso.org/standard/67207.html)

ISO/TS 20175 " Vacuum technology — Vacuum gauges — Characterization of quadrupole mass spectrometers for partial pressure measurement" describes procedures to characterize quadrupole mass spectrometers (QMSs) with an ion source of electron impact ionization and which are designed for the measurement of atomic mass-to-charge ratios $m/z < 300$.

Quadrupole mass spectrometers (QMSs) are nowadays used not only in vacuum technology for leak detection and residual gas analysis but also in the process industry as an instrument to provide quantitative analysis in processes and to control processes such as physical and chemical vapour deposition, and etch processes. They are also used for quantitative outgassing rate measurements which are important to characterize vacuum components for critical applications like in the EUV lithography, semiconductor industry or medical instruments. TS 20175 provides standardized calibration procedures for QMSs for some important applications. These have been selected from the results of a survey of the international project EMRP (European Metrological Research Programme) IND12 which was conducted in 2013. This survey included manufacturers, distributors and users of quadrupole mass spectrometers.

It is well known from published investigations on the metrological characteristics of quadrupole mass spectrometers that their indications of partial pressures depend significantly on the settings of the instrument, the total pressure, and the composition of the gas mixture. For this reason, it is not possible to calibrate a quadrupole mass spectrometer for all possible kinds of use. The characterization procedures described in TS 20175 cover the applications of continuous leak monitoring of a vacuum system, leak rate measurement with tracer gas, residual gas analysis and outgassing rate measurements. The user can select that characterization procedure that best suits his or her needs. These characterization procedures can also be useful for other applications.

It is also well known that the stability of several parameters of quadrupole mass spectrometers, in particular sensitivity, are rather poor. Therefore, when a parameter has been calibrated, it needs frequent recalibration when accuracy is required. For practical reasons this can only be accomplished by in situ calibrations. To this end, TS 20175 not only describes how a quadrupole mass spectrometer can be calibrated by a calibration laboratory or a National Metrological Institute with direct traceability to the System International (SI), but also how calibrated parameters can be frequently checked and maintained in situ.

By their physical principle, quadrupole mass spectrometers need high vacuum within the instrument. By reducing dimensions or by special ion sources combined with differential pumping the operational range can be extended to higher pressures, up to atmospheric pressure. TS 20175, however, does not include quadrupole mass spectrometers with differential pumping technology.

TS 20175 does not describe how the initial adjustment of a quadrupole mass spectrometer by the manufacturer or by a service given order by the manufacturer should be made. The purpose of such an initial adjustment is mainly to provide a correct m/z scale, constant mass resolution or constant transmission, and is very specific to the instrument. Instead, it is assumed for TS 20175 that a manufacturer’s readjustment procedure exists which can be carried on-site by a user. This procedure is intended to ensure that the quadrupole mass spectrometer is in a well-defined condition for the characterization.

It is the intention of TS 20175 that the user gets the best possible metrological quality from his quadrupole mass spectrometer. It is not the intent of TS 20175 that all the parameters described be determined for each quadrupole mass spectrometer. However, it is intended that the value of a parameter addressed in TS 20175 be determined according to the procedure described in TS 20175 if it is given or measured (e.g. for an inspection test).

ISO TS 20175 addresses with different sections different communities:

- Section 5.2 "General characterization of the QMS" addresses the need of users of QMS to expect comparability of the important parameters of QMS like linear response range, sensitivity for pure nitrogen in the linear response range, minimum detectable partial pressure for helium and nitrogen, the dynamic range, minimum detectable concentration for helium in nitrogen and mass resolution.
- Section 5.3 "Leak rate measurement and leak rate monitoring (helium leak)" addresses the need of leak testing services and users who need to quantify leak rates of vacuum components, chambers and whole systems and even large facilities as accelerators, e.g. for their cooling systems of superconducting magnets.
- Section 5.4 " Leak rate monitoring (air leak)" addresses the need of those who need to continuously control a vacuum chamber at high vacuum, which may be a fusion reactor, an accelerator facility or a vacuum process chamber like glass coating machine.
- Section 5.5 " Leak rate monitoring (water leak) addresses of users which use water cooling for either generate energy as in a fusion reactor or have to dissipate energy as in a vacuum oven.
- Section 5.6 " Residual gas analysis" for manufacturers and users of vacuum chambers to agree on a common measurement standard of the seller and buyer.
- Section 5.7 " Outgassing rate measurement" for manufacturers and users of vacuum components to agree on a common measurement standard of the seller and buyer. This section is closely related to ISO TS 20177 and describes the necessary characterisation and traceability to perform quantitative outgassing rate measurements.

Further sections describe the vacuum systems and procedures to characterize the QMS for the different applications of the communities. A final section gives guidelines on the measurement uncertainties associated with the different parameters.

Objective 2 - To work with ISO TC 112 to develop a Technical Specification for procedures to measure and report outgassing rates.

To fulfil the objective, two successive working drafts were developed, the first in October 2015, the second in May 2016. The workload between the participants was shared as follows during the whole project: PTB drafted the frame and content sections, worked out the sections on the systems and measurement procedures applying the throughput method, as well as the section on measurement uncertainty with these systems. IMT worked out the sections on the systems and measurement procedures applying the accumulation method, as well as the section on measurement uncertainty with these systems. A common effort of PTB and IMT were the introduction and the scope, the general requirements of the measurement systems, the section on the measurement report and the informative annex, PTB also made the communication with ISO TC 112 and organised the workshop and the TC 112 meetings. The first draft was discussed by the Working Group 2 (WG 2) members of "Vacuum Instrumentation" of ISO TC 112 in November 2015. The comments were considered in the succeeding working draft. This was published on the website of the project and uploaded on the Livelink website of ISO. Besides the Working Group 2, the wider community of manufacturers and users of QMS and researchers who measure outgassing rates were asked to comment by November 2016. The comments were discussed at a workshop from Jan 30 - Feb 1, 2017 and at the following WG 2 meeting on Feb 2-3, 2017. As a result of these discussions, a new draft was developed and put to vote within ISO TC 112 WG 2. The WG 2 decided that, after some minor changes, this drafts should be forwarded to ISO TC 112 to be put to a vote. This vote took place from beginning of August to early November 2017.

As a result of the vote, the two ISO DTS (Draft Technical Specifications) 20177 was approved with just a few comments. On the TC 112 meeting on Nov 30, 2017, it was decided to submit the DTS for publication with some editorial changes discussed at the meeting.

The headquarter of ISO in Geneva reviewed the DTS and proposed several additional editorial corrections, which were assessed in April 2018.

ISO/TS 20177 was published in June 2018 (https://www.iso.org/standard/67208.html).

ISO/TS 20177 "Vacuum technology — Vacuum gauges — Procedures to measure and report outgassing rates" describes procedures to measure outgassing rates from components designed for vacuum chambers and of vacuum chambers as a whole. The outgassing rates are expected to be lower than 10−5 Pa m3 s−1

(10^{-2} Pa L s^{-1}) at 23 °C and that the devices are suitable for high or ultra-high vacuum applications. The molecular mass of the outgassing species or vapour is below 300 u.

For many practical applications it is sufficient to determine the total outgassing rate. If a measuring instrument, which sensitivity is gas species dependent, is used, this total outgassing rate should be given in nitrogen equivalent. In cases, however, where the total outgassing rate is too high, there is a clear need to identify the disturbing gas species and measure its outgassing rate in order to improve the sample material. This Technical Specification covers both cases.

Some outgassing molecules may adsorb on a surface with a residence time that is much longer than the total time of measurement. Such molecules cannot be detected by the detecting instrument when there is no direct line of sight. This is considered as a surface effect and surface analytical investigations will be more useful than general outgassing rate measurements considered here. Also, molecules that are released from the surface by irradiation of UV light or X-rays, are out of the scope of the Technical Specification.

TS 20177 was written to standardize the measurement of outgassing rates in such a way that values obtained at different laboratories and by different methods will be comparable. To this end, for any of the described methods it is necessary to provide traceability to the SI for the most important parameters of each method and according to the metrological level.

Outgassing rate measurements by mass loss which were mainly developed for testing of spacecraft and satellite materials are not gas specific. For acceptable measurement times, mass loss measurements require significantly higher outgassing rates (> 10-5 Pa m3 s-1) than typical for high and ultrahigh vacuum components. Also it is not possible to measure the sample in situ due to the weight of the vacuum chamber, since the balances are not vacuum compatible. For these reasons, mass loss measurements were not considered in TS 20177.

TS 20177 offers different options as measurement systems. The reason is that, at the present stage, there are no scientific reasons to prefer one method against the other, as long as traceability to the SI is ensured in some way. In the past, lack of traceability was a great deficit of systems and procedures. All measurement systems listed in the TS ensure some kind of traceability to the SI. The higher the metrological level, the more direct is the traceability, and the lower are the uncertainties of the traceable quantities which are significant. In the future, when the TS is established and comparisons between different system have been carried out, it may turn out that some systems should be preferred against others, because the kind of traceability is insufficient or data are less reliable for other reasons. This will lead to new recommendations.

The criteria which system is best to use are:

- Need to measure time dependence
- Need to identify outgassing species
- Outgassing of vapour (e.g. water vapour) in a significant magnitude
- Expected outgassing rate
- Needed accuracy or uncertainty of measurement
- Effort, budget and experience

Associated measurement procedures and uncertainties are then described for the different measurement systems in the following sections.

Industry and research often need a complete characterisation of the vacuum process environment, which includes (in addition to the total pressure) the gas composition i.e. which species are present in the vacuum and their partial pressures. Therefore the "cleanliness" of a vacuum is vital. Although oil–free vacuum pumps and other components have improved cleanliness considerably, components can still be a source of unwanted gasses that can be released or "outgassed" into the vacuum. Outgassing rates are therefore a vital quality assurance figure in vacuum technology. However, before this project there was no internationally standardised way to measure such rates and to establish their traceability to the SI system of units.

The cooperation between IMT and PTB was important for the success of this project. The PTB experience with writing standards was complemented by the metrological experience of IMT from the lead of the work package on partial pressure measurement in the former project IND12. From the point of measurement experience, PTB had the metrological experience with the throughput method while IMT had broad experience with accumulation systems. In addition, the consolidation of the knowledge about the pertinent community of the two partners increased the impact of the project.

5 Impact

After the preparation of drafts of the two Technical Specifications, the project involved the Working Group 2 "Vacuum Instrumentation" (WG 2) of ISO TC 112 to discuss the drafts and develop them further with the project partners. In a meeting of ISO TC 112 in November 2015 the business plan and schedule was harmonized with the schedule of this project. After this step, the project opened the discussion of the standards to a wider community including manufacturers and users in industry, science and metrology. This wider community was addressed via the website, the social network LinkedIn and by email contacts and invited to a workshop from Jan 30 - Feb 1, 2017. On this workshop there were intense discussions and the results were discussed at the following WG 2 meeting on Feb 2-3, 2017. After agreeing a new draft with this community, the primary supporter, ISO TC 112, used the draft for a ballot which ended in an approval of the draft. From Nov 29 to Dec 1, 2017 another ISO TC 112 meeting was held and it was decided to submit ISO DTS 20175 and 20177 to the ISO headquarter for publication.

ISO TS 20175 was published in April 2018, ISO TS 20177 in June 2018.

The Technical Specification for the characterisation and calibration of QMS will allow end users to compare the performance of QMS and use them with the best possible accuracy. A more accurate characterisation of QMS will also support their development.

The Technical Specification for outgassing rate measurements will make these measurements more reliable and comparable and therefore costly "local" solutions (used in industry to provide agreement between different suppliers of vacuum components) should become obsolete.

The two Technical Specifications which were developed in the project are important in particular for end users from the field of high energy accelerators, suppliers of EUV components and positioning devices and from the coating and semiconductor industries. The new Technical Specifications enable the users and manufacturers to reliably quantify outgassing rates. Users and suppliers can agree on established methods how to characterize QMS and measure outgassing rates which will avoid judicial conflicts.

The company ASML, which is focussed on semiconductor lithography systems by extreme ultraviolet radiation (EUV) at 13 nm, with their suppliers will incorporate the two TS in their procedures for evaluating quadrupole mass spectrometers and outgassing rates and propose the two TS to the SEMI (Semiconductor Equipment and Materials International) organisation for further consideration.

This last initiative from industry shows in which direction the results of this project will have further impact: On the basis of the Technical Specifications users and suppliers of vacuum equipment will agree on certain standards of how "clean" components for vacuum have to be in terms of outgassing rates. The community can concentrate on material development and characterisation instead of arguing about measurement methods. This standardisation will save energy and costs for the companies and due to the more efficient material development help to producible sustainable and protect the environment. The still ongoing miniaturization of all kinds of sensors, communication, semiconductor and biomedical equipment will greatly profit from the two Technical Specifications, because EUV lithography systems are made possible with clear specifications of outgassing rates.

6 Website address and contact details

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