Publishable Summary for 16NRM05 Ion gauge
Towards a documentary standard for an ionisation vacuum gauge

Overview

The ionisation gauge is the only vacuum gauge type for high and ultrahigh vacuum. The pertinent standardisation committee for vacuum technology ISO TC 112 had indicated that important applications need better accuracy, reproducibility and the sensitivity for many gas species, properties which all current types of ionisation gauges lack. This project designed a new type of ionisation vacuum gauge that is accurate (total relative uncertainty: 1 %), robust and long-term stable, with known relative gas sensitivity factors, and provides the relevant parameters for an ISO standard gauge. The gauge can be made by any experienced manufacturer and provides the opportunity for improved traceability within industries that employ high vacuum processes.

Need

High and ultrahigh vacuum is an indispensable tool for science and industry. Fields of application for science include high-energy accelerators, plasma and fusion science, surface science, and thin film studies, which have a great impact on industry, e.g. optics, optoelectronics, and solar cells. Additional areas of application for industry include the semiconductor industry, the coating industry, and extreme ultraviolet (EUV) lithography, in which the Dutch company ASML, in cooperation with Zeiss, is the only instrument manufacturer worldwide.

The ionisation gauge is the only vacuum gauge type for high and ultrahigh vacuum but is lacking in robustness, as well as long-term and transport stability.

The pertinent technical committees ISO/TC 112 "Vacuum Technology" and the DIN NA 060-07 “Vacuum Technology” section made clear that the reliability and usefulness of ionisation gauges can be greatly improved by standardisation and encouraged research towards a standardised ionisation gauge. Their requirements in detail:

- For pumping speed measurements, ISO 21360-1 requires a standard uncertainty of 3 % of pressure measurement with an ionisation gauge. This is possible for nitrogen, but at present not for any other gas. A standardised ionisation gauge could provide this accuracy for many kinds of gases. Also, the measurement of compression ratios according to ISO 5302 and ISO 21360-1 requires an ionisation gauge with well-known relative gas sensitivity factors which are not available at present.
- Support is needed for the implementation of the two Technical Specifications ISO TS 20175 and ISO TS 20177 by means of a standardised ionisation gauge. This was one of the major needs identified in the EMRP Joint Research Project IND12 and its follow-up Support for Impact project 14SIP01, which will benefit from the standardisation developed in this project.
- Calibration laboratories for vacuum gauges in the HV and UHV ranges do not have reliable reference standards below 1 mPa. An ionisation gauge that is stable over a long-term (relative uncertainty of 1 % over 1 year) will provide this in order to apply ISO 3567 and ISO 27893.

This project has successfully produced and validated a gauge fulfilling these requirements.

Objectives

The overall objective was to determine and specify all relevant parameters to enable an ISO standard for an ionisation gauge so that this gauge is accurate, robust and long-term stable. Such a standard also strengthens the metrological and technological basis of the ISO TS 20175 and 20177 which require a reliable ionisation gauge.

The specific objectives were:

1. To provide a substantial contribution to the resolution 2015-09 of ISO TC 112. This meant in detail to determine and specify all relevant parameters that are needed to elaborate an ISO standard of an accurate ionisation gauge (total relative standard uncertainty according to GUM [2]: 1 %) in the measurement range from 10^{-6} Pa to 10^{-2} Pa.

2. To make a substantial contribution to the implementation of the two Technical Specifications ISO TS 20175 and 20177 by providing new data material for a stable ionisation gauge and by providing 10 relative gas sensitivity factors of this ionisation vacuum gauge. This is needed for the calibration of quadrupole mass spectrometers and outgassing rate measurement systems as outlined in the aforementioned two Technical Specifications.
3. To work closely with ionisation gauge manufacturers in order to consider their experiences and to make sure that the standard for the ionisation gauge will result in an instrument that is easy to use and economical to produce.
4. To work closely with ISO TC 112 and national standards developing organisations, as well as the future users of the standard, to ensure that the output of the project will cover their need for a reliable ionisation gauge. This includes close communication with their respective working groups in order to consider their input and to make the output of the project easily available to them. That will make the results applicable to a Standard at the earliest possible opportunity.

Progress beyond the state of the art

All manufacturers of ionisation gauges develop different products even if belonging to the same type of gauge. They differ in the selection of materials, potentials and, most important, geometry. For this reason, they also significantly differ in their relative sensitivity factors. In addition, all available types are lacking long-term and transport stability, the instability being presently about 5 % over one year. Important reasons are that the electrodes are not rigid enough, the spatial emission from the cathode is not stable and the materials show a too high and unstable secondary electron yield.

This project improved this situation by providing a design of an ionisation gauge that exceeds present performance and can be standardised. The new gauge improved the relative standard uncertainty due to long-term and transport instability from about 5% to below 1 % for nitrogen gas and makes it unnecessary to calibrate relative gas sensitivity factors for each individual gauge and gas species, because the spread of the sensitivity factors was reduced from about 10 % to 1 %-3 % depending on gas species. This gauge can be produced by any experienced manufacturer so that reliably known sensitivity factors can be provided together with the gauge. Transport and long-term stability ensure that the gauge can be used by calibration laboratories applying ISO 3567 and by users applying ISO TS 20175 and ISO TS 20177.

Results

Objective 1. Relevant parameters for ISO standard on ion gauge

A literature review of ionisation vacuum gauges with hot cathodes was performed. 260 relevant papers dating from the 1950s to the present were identified, reviewed and conclusions drawn. A report collated the most important results. In addition, a review paper of 18 pages and 174 references was published in the journal VACUUM. This was the broadest review paper on ionisation vacuum gauges published so far. These reports identified the most relevant parameters of Bayard-Alpert type and of some other types of ionisation gauges which are important to make the gauge more stable and applicable to standardisation. Electron and ion trajectories were simulated for the preferred design of the ionisation gauge. Before this, different software options were tested by benchmarking of a commercial gauge. It turned out that 2 of the 4 software packages had severe problems or could not deliver the desired parameter to be compared to the real gauge. For this reason, the simulation of the proposed design was made with the remaining software packages OPERA and SIMION. Later, a third software package COMSOL with an additional module was also used to simulate the gauge characteristics. The design was optimised in terms of electrode positions and dimensions. During the first period of the simulations it was found that some modification of the design was needed to make it more robust to changes of the electrode position, in particular of the position of the cathode as the electron source. A statistical evaluation was carried out in order to find the tolerances of the electrode positions for manufacturing. The literature review and the simulations were supported accordingly by a study of possible suitable materials for the electrodes and first simulations of sputtering of the materials. Typical temperatures of the electrodes in a hot cathode ionisation gauge were determined to inform the material investigations.

The surface investigations were studied in an experimental ion gauge simulator built for this project. The research focused on the changes of ion induced secondary electron yield (IISEY) due to the exposure to an ionisation gauge environment. The IISEY measurements were performed on five different materials, with three ion species. The investigations revealed that there is a strong coverage of hydrocarbons on all materials. This contamination is caused by the hot cathode in the ion gauge. The contamination effect with an yttrium oxide coated iridium cathode is somewhat lower than with a tungsten cathode, but still significant. Most interestingly, all work functions tend to reach the same value, independent of the substrate. The consortium concluded that the presently best way to stabilise the ion induced secondary electron yield is to condition the gauge by an operation in an argon environment before a measurement. This conditioning produces the hydrocarbon layer on the collector with the more stable ion induced secondary electron yield.

TRIDYN simulations of low energy ion sputtering of different materials were performed, reaching satisfactory agreement with the experimental results.

This objective has been successfully completed.

Objective 2. New data material for a stable ionisation gauge

The consortium agreed on two commercial gauges to be tested as a benchmark for the laboratory and model gauge and the quantities to be investigated were fixed and measured. These results were compared to the data obtained with the new gauge developed by the project.

The NMIs adapted their existing calibration systems to the gases and measurement uncertainties needed to test the laboratory gauges being developed in the project, and to measure the 10 relative gas sensitivity factors with the new gauge.

In November 2017, the consortium agreed on the gauge design to be pursued. It is a design that cannot be found on the market at present. This was somewhat risky, since experiences with existing models could not be adopted. The consortium, however, was of the mind that the technical reasons causing instabilities in ionisation gauges could not be overcome by modifying existing designs. One of the industrial partners developed the technical drawings of the laboratory gauges and produced ten laboratory gauges to be tested by the consortium. The tests of the laboratory gauges showed that the design is very successful. Issues were of technical nature only. For this reason, only technical improvements concerning robustness for transport stability and better electrical insulation between electrodes were proposed for the model gauges, maintaining the principal design. With these changes new or modified technical drawings of the model gauges were developed and 23 model gauges delivered by the two different consortium gauge manufacturers.

The tests of the model gauges in the different NMIs produced outstanding results and justified the risk that the consortium took with its decision. The new design exceeds the performance of the existing designs of ionisation vacuum gauges by far in terms of predictability of sensitivity, linearity, repeatability, reproducibility, robustness, and transport stability. The relative uncertainty from these effects is below 1 % of measured vacuum pressure values. On top, the measured sensitivity for nitrogen agrees with the one expected from the simulation with the three software packages. There were no significant differences between the layouts of the two manufacturers, which is a necessary prerequisite that the gauge type can be standardised. The electron transmission through the ionization region is close to 100% so that the electron path length is well-defined. Relative gas sensitivity factors were measured for 12 gas species.

This objective has been successfully completed.

Objective 3. Cooperation with gauge manufacturers

The consortium incorporated two-gauge manufacturers as partners to contribute with their experiences and to make sure that the standard for the ionisation gauge will result in an instrument that is easy to use and economical to produce. One of the manufacturers delivered the design of the new type of ionisation gauge according to the results gained from the literature review and simulations and produced ten laboratory gauges to be tested by the consortium. After the tests of the laboratory gauges, some improvements were decided for the design of the model gauges. These were implemented by both manufacturers. Both manufacturers produced at least ten model gauges of the same electrode design, but with different technical layouts and using their own production lines. The test showed agreement between the different gauge layouts produced by these manufacturers. This ensures that, when the gauge type has been standardised, there will be no gauge characteristic dependence introduced by different manufacturer production lines.

This objective has been successfully completed.

Impact

Key Highlights of Dissemination Activities:

- Consortium partners presented the project and its research results to the ISO TC 112 Working Group 2 on Vacuum Instrumentation (the advisory group of the project) in November 2017, May 2019, and November 2020
- The consortium gave two presentations and presented one poster at the 15th European Vacuum Conference in June 2018
- The consortium gave four presentations and presented a poster at the 21st International Vacuum Congress in July 2019
- The consortium published four articles in open access peer reviewed journals, three of them with authorship from at least two partners, a fifth article has been submitted.

Impact on industrial and other user communities

As a result of this project, vacuum gauge manufacturers now have the option of producing in-house a reference ionisation gauge that due to its inherent sensitivity enables significant reductions in calibration costs and the time penalties that this introduces. In addition, the ability to change the gauge's cathode, or to replace it in a production process without the need of recalibration or readjustment offers substantial benefits to users in the semiconductor, coating and aerospace industries.

For pumping speed measurements, ISO 21360-1 requires an accuracy of 3 % of pressure measurement with an ionisation gauge. By introducing the project designed ionisation vacuum gauges, high-vacuum pump manufacturers will be able to fulfil this requirement not only for nitrogen, but also for other gas species – an important attribute for many of their industrial and academic customers.

The exchangeability of a standardised gauge, one of its most important features, will have a significant impact in the vacuum market by reducing maintenance costs. The company ASML, the only producer of EUV lithography equipment, has set-up a working group with its suppliers and PTB to establish guidelines for traceable outgassing rate measurements based on ISO TC 20175 and 20177. The role of a standardised ionisation gauge has been emphasised in this group.

Impact on the metrological and scientific communities

Achieving ultrahigh vacuum is important to European researchers at for example the high energy and fusion physics facilities of CERN, DESY, ESRF, and ITER. They rely on the ability to make reliable measurements of many different gas species. This is another area expected to benefit from the use of the project developed reference/standardised vacuum gauge. Examples of uses are determinations of nuclear/atomic collision cross sections, ionisation probabilities, nuclear/atomic absorption cross sections and gas exposure measurements in surface adsorption experiments.

The impact in the metrology area is twofold: (i) The function of ionisation gauges as reference and transfer standards for calibration services has been improved and widened to relevant gases other than nitrogen. National Metrology Institutes now have a reliable transfer gauge for high and ultrahigh vacuum to compare their primary standards. (ii) The use of ionisation gauges to calibrate quadrupole mass spectrometers in situ is much more accurate with a standardised ionisation gauge as relative gas sensitivity factors are reliably known. Such an in-situ-calibration is frequently needed for outgassing rate measurements.

The consortium held a web meeting with the CCM WG PV. The group appreciated the success of this development and qualified the new ionisation vacuum gauge as good candidate for a transfer standard in future CCM KCs in the high vacuum range. NMIJ as pilot laboratory for the next comparison in this range indicated interest to test it as transfer standard and asked for a copy.

Impact on relevant standards

As a result of this project it will be possible to develop an ISO standard (Technical Specification) for an ionisation vacuum gauge as outlined in the business plan of ISO TC 112. The consortium held a web meeting with the ISO TC 112 WG 2. The group appreciated the success of this project in specifying the requirements for a standard ionisation gauge and recommended starting the ballot process within ISO TC 112 for a new project to cast our draft 'working' technical specification into a standard Technical Specification. The ballot of ISO/NP TS 6737 was started in January 2021. With such a standardised ionisation gauge, ISO TS 20175 and 20177 will be able to be effectively implemented. ISO 3567, 5302 and 21360-4 and other newly developed standards for high vacuum pumps in the 21360 series will also greatly benefit from a standardised gauge.

Longer-term economic, social and environmental impacts

As a wider impact, more accurate pumping speed values for a greater number of gases will provide designers of vacuum plants with reliable data for sizing pumps and gas flows. With more accurate and more reliable data, safety margins can be reduced, and gas consumption minimised. This will save resources, improve work security for explosive and poisonous gases in the semiconductor and coating industry and reduce environmental pollution.

The vacuum gauge manufacturers will benefit from a standardised ionisation gauge design as less effort will be required for the calibration of the manufactured gauge with no need to calibrate sensitivity for gases other than nitrogen.

When the project developed standardised ionisation gauges are used in plants, an exchange of the gauge will be possible without readjusting process parameters in the semiconductor and coating industry, because the sensitivities for all process gas species will be known with high accuracy. The exchangeability of the standardised gauge may have a great impact on the vacuum market where time consuming vacuum process readjustments lead to unscheduled production stoppages and lost productivity.

The ionisation gauge developed by the project will improve the control of vacuum processes in the semiconductor and coating industry by the more accurate pressure measurements at the different places in a plant. At present, due to the sensitivity scatter of ionisation gauges gas flow measurements under high vacuum conditions are unreliable. This better control will lead to higher cost efficiency, greater safety, and improved environmental protection due to a better waste management of vacuum processes. It will improve quality assurance procedures of European vacuum equipment manufacturers helping increase customer confidence in European products.

The European vacuum industry is traditionally at the forefront worldwide, especially in the excellence of its mechanical engineering. There are several companies in Europe that manufacture ionisation gauges. The knowledge gained by the project will give them an improved competitive edge compared with Asian and American companies.

The improvement of the measurement possibilities brought about by this project will enable European manufacturers of process tools, vacuum pumps, and vacuum and partial pressure gauges to improve their products.

List of publications

1. R. Silva, N. Bundaleski, A.L. Fonseca, O.M.N.D. Teodoro, *3D-Simulation of a Bayard Alpert ionisation gauge using SIMION program*, Vacuum **164** (2019) 300-307 https://doi.org/10.1016/j.vacuum.2019.03.039
2. I.G.C. Figueiredo, *Investigation and characterization of materials towards building ionization vacuum gauges* (Master's thesis), Nova University of Lisbon (2018) http://hdl.handle.net/10362/52578
3. R.A.S. Silva, *Desenvolvimento de um manómetro de ionização de elevada estabilidade* (Master's thesis), Nova University of Lisbon (2018) http://hdl.handle.net/10362/59610
4. Jousten K, Boineau F, Bundaleski N, Illgen C, Setina J, Teodoro OMND, Vicar M, Wüest M, *A review on hot cathode ionisation gauges with focus on a suitable design for measurement accuracy and stability*, Vacuum 179 (2020) 109545, https://doi.org/10.1016/j.vacuum.2020.109545.
5. Jenninger B, Anderson J, Bernien M, Bundaleski N, Dimitrova H, Granovskij M, Illgen C, Setina J, Jousten K, Kucharski P, Reinhardt C, Scuderi F, Silva RAS, Stöltzel A, Teodoro OMND, Trzpil-Jurgielewicz B, Wüest M, *Development of a design for an ionisation vacuum gauge suitable as a reference standard*, Vacuum 183 (2021) 109884, https://doi.org/10.1016/j.vacuum.2020.109884.
6. I. Figueiredo, N. Bundaleski, O.M.N.D. Teodoro, K. Jousten, C. Illgen, *Influence of ion induced secondary electron emission on the stability of ionisation vacuum gauges*, Vacuum 184 (2021),109907, https://doi.org/10.1016/j.vacuum.2020.109907.

This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

Project start date and duration:	01 June 2017, 42 months	
Coordinator: Karl Jousten, PTB Tel: +49 30 3481 7262 E-mail: Karl.jousten@ptb.de Project website address: http://www.ptb.de/empir/16nrm05-home.html		
Chief Stakeholder Organisation: ISO TC 112	Chief Stakeholder Contact: Juergen Eisenreich	
Internal Funded Partners: 1. PTB, Germany 2. CMI, Czech Republic 3. IMT, Slovenia 4. LNE, France 5. RISE, Sweden	External Funded Partners: 6. CERN, Europe 7. FCT-UNL, Portugal 8. VACOM, Germany	Unfunded Partners: 9. INFICON LI, Liechtenstein
RMG: -		