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 has 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 will provide the relevant parameters for an ISO standard of an ionisation gauge so that this gauge is accurate (total relative uncertainty: 1 %), robust and long-term stable, with known relative gas sensitivity factors, and can be built by any experienced manufacturer.

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 of the DIN NA 060-07 “Vacuum Technology” section have made clear that the reliability and usefulness of ionisation gauges can be greatly improved by standardisation and have encouraged research towards a standardised ionisation gauge. The need 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.

Objectives

The overall objective is 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. This standard will also strengthen the metrological and technological basis of the ISO TS 20175 and 20177 which require a reliable ionisation gauge.

The specific objectives are:

1. To provide a substantial contribution to the resolution 2015-09 of ISO TC 112. This means in detail to determine and specify all relevant parameters that are needed to elaborate an ISO standard of an ionisation gauge (total uncertainty: 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 to consider their input and to make the output of the project easily available to them in order to 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 will improve this situation by providing the design of an ionisation gauge that exceeds present performance and can be standardised. The new gauge shall improve the relative standard uncertainty due to long-term and transport instability from about 5% to below 1 % for nitrogen gas and make it unnecessary to calibrate relative gas sensitivity factors for each individual gauge and gas species, because the spread of the sensitivity factors will be reduced from about 10 % to 2 %-3 % depending on gas species. This gauge will then be able to be produced by any experienced manufacturer so that reliably known sensitivity factors can be provided together with the gauge. The aimed-for transport and long-term stability will 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

1. Relevant parameters for ISO standard on ion gauge

A literature review of ionisation 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. This report 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. The design was optimised in terms of electrode positions and dimensions. During this 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 in the experimental ion gauge simulator focused on the measurements of ion induced secondary electron yield (IISEY) on five materials. 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 yttriumoxide 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 highest stability in terms of IISEY changes was achieved with graphite coated substrates.

This is an important finding for the development of stable ion gauges in the project. It was also found that baking reduces the amount of hydrocarbon layer and, as a consequence, increases the unwanted IISEY.

TRIDYN simulations of low energy ion sputtering were performed, reaching satisfactory agreement with the experiments. This software package, supported by the deterministic sampling method, was also used to evaluate the coating lifetime of the cathode under low energy ion bombardment.

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 will be 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. The industrial partners developed the technical drawings of the laboratory gauges and produced ten gauges to be tested by the consortium.

The tests of the laboratory gauge are ongoing in the different NMIs. The results obtained so far are very encouraging. The measured sensitivity agrees with the one expected from the simulation and the electron transmission through the ionization region is close to 100% so that the electron path length is well-defined, and it can be expected that the relative sensitivities will exactly relate as the ionization probabilities for different gases. 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.

3. Cooperation with gauge manufacturers

The consortium incorporated two gauge manufacturers as partners to include 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. Either of the two manufacturers will provide ten model gauges of the same electrode design, but with different technical layouts and production lines.

Impact

Key Highlights of Dissemination Activities:

- Consortium partners presented the project and its expected research results to the ISO TC 112 Working Group 2 on Vacuum Instrumentation in November 2017
- The consortium gave two presentations and presented a poster at the 15th European Vacuum Conference in June 2018
- The consortium partners informed the advisory group ISO TC 112 Working Group 2 on Vacuum Instrumentation about the progress of the project in May 2019
- The consortium gave four presentations and presented a poster at the 21st International Vacuum Congress in July 2019

Impact on relevant standards

By carrying out this research it will be possible to develop an ISO standard for an ionisation gauge which is in the business plan [1] of ISO TC 112. With such a standardised ionisation gauge, ISO TS 20175 and 20177, which were recently (2018) published, will be able to be effectively implemented. ISO 3567, 5302 and 21360-4 will also greatly benefit.

Impact on industrial and other user communities

For pumping speed measurements, ISO 21360-1 requires an accuracy of 3 % of pressure measurement with an ionisation gauge. The results of this project will enable pump manufacturers to fulfil this requirement not only for nitrogen, but also other gas species important for buyers of high vacuum pumps.

The project will have a significant impact on the semiconductor and coating industry, EUV lithography, high energy and fusion physics research facilities in Europe (CERN, DESY, ESRF, ITER) and the aerospace industry by ensuring traceability and comparability of outgassing measurement results. The exchangeability of a standardised gauge will lead to great impact in the vacuum market by reduced costs for maintenance. The company ASML, the key player in EUV lithography, 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

The impact in the scientific area will be that there is a reliable measurement of many different gas species in the high and ultrahigh vacuum. Examples of use are determinations of collision cross sections, ionisation probabilities, absorption transitions and gas exposure measurements in surface adsorption experiments.

The impact in the metrology area will be twofold: (i) The function of ionisation gauges as reference and transfer standards for calibration services will be greatly improved and widened to relevant gases other than nitrogen. National Metrology Institutes will 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 will be much more accurate with a standardised ionisation gauge, because the relative gas sensitivity factors will be reliably known.

Longer-term economic, social and environmental impacts

The project will improve the control of vacuum processes. This better control will lead to higher cost efficiency, more safety, and greater environmental protection due to a better waste management of vacuum processes. It will improve quality assurance procedures of European vacuum equipment manufacturers.

The European vacuum industry is traditionally at the forefront worldwide, especially by the capability of excellent mechanical engineering. There are several companies in Europe that manufacture ionisation gauges. The knowledge gained by the project will give them a winning margin compared with Asian and American companies.

As a wider impact, more accurate pump speed values for a greater number of gases will provide designers of vacuum plants with reliable numbers for sizing pumps and gas flows. This will save resources, improve work security for explosive and poisonous gases 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 standardised ionisation gauges are used in plants, an exchange of the gauge will be possible without readjusting process parameters, 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.

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.

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

Project start date and duration:	01 June 2017, 36 months	
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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
RMGs:		