EMRP Call 2012 – Open Excellence, Industry and SI Broader Scope

Selected Research Topic number: **SRT-s13**
Version: 1.0

Title: Outgassing rate control and pascal realisation in the extremely low-pressure range

Abstract

Vacuum techniques and technology in the ultrahigh vacuum range (UHV) and into the extreme high vacuum range (XHV) region have become crucial for many advanced scientific and industrial devices and technologies such as particle accelerators, storage rings, ion traps, the simulation of interstellar space, spectroscopy, deposition and epitaxy techniques, MEMS and other semiconductor device manufacturing.

The traceability to primary standards for pressure measurement and pressure related quantities in the UHV/XHV range is therefore important to underpin these fields; however the primary capability for the realisation of the pressure scale below $1x10^{-6}$ Pa is very limited in Europe and indeed worldwide and requires development. In addition the outgassing of the materials used is a principal obstacle to achieving this aim and conversely the outgassing rate of materials is one of the pressure related quantities which needs to be measured reliably.

Conformity with the Work Programme

This Call for JRPs conforms to the EMRP Outline 2008, section on “Grand Challenges” related to Industry & Fundamental Metrology on pages 12 and 37.

Keywords

UHV metrology, XHV metrology, XHV technology, pascal realisation, low outgassing rate materials, outgassing rate measurement, UHV/XHV transfer standard

Background to the Metrological Challenges

The primary capacity for the realisation of the pressure scale below $1x10^{-6}$ Pa is very limited in Europe and indeed worldwide. There is currently only one primary standard worldwide for which calibration and measurement capabilities (CMCs) below $1x10^{-7}$ Pa [1] are included in the BIPM key comparison database and within Europe this is also the only standard (operating from $1x10^{-2}$ Pa to $1x10^{-9}$ Pa) able to provide primary calibrations below $1x10^{-6}$ Pa.

This situation is undesirable and untenable in the long-term for a number of reasons. Being dependent on the one and only realisation of pressure unit over a certain range prevents any robust inter-laboratory comparison, and also precludes the possibility of identifying any unknown systematic effects. Similarly any estimation of the long-term stability of such a standard is exceedingly difficult, if not impossible. The consequence is that the declared uncertainties will tend to include ‘safety factors’. There are also obvious practical risks associated with the reliance on a single primary standard such as mechanical failure. There are some recent activities in the far East, in particular in Japan where a primary UHV calibration standard down to $1x10^{-9}$ Pa [2] has been constructed, and in China which recently announced construction of such a standard operating down to $1x10^{-10}$ Pa [3] (however, only with respect to the residual pressure).

The development of new primary standards operating in the UHV/XHV requires research, development and utilisation of advances in vacuum technology, including extremely low outgassing rate surfaces, stable and sensitive UHV/XHV pressure gauges (which do not unduly perturb the pressure equilibrium) pumping procedures (capable of reaching and maintaining very low stable pressures), and material preparation techniques. A number of individual technologies are well developed in Japan, namely the utilisation of the

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materials with low outgassing rates (research of barrier layers and coatings), XHV pumping techniques (tandems of turbomolecular pumps, bakeable cryopumps, cryopumps operating at 4 K and lower) and the new ionisation gauge designs.

Although research has been undertaken in the field of outgassing of materials, the main aim was to achieve extremely low outgassing rate with the available materials. The published results are often not fully comparable, due mainly to problems in describing the material, status, history and method of treatment. Even though the outgassing rates achieved would be near the required values, their stability and reproducibility together with the behaviour of materials in periodically vented real systems is not adequate. The reasons why some materials can be conditioned to the required state has been plausibly explained qualitatively, but this conditioning is not stable for majority of available materials (stainless steel, Fe- and Ni-based alloys) and many of the remaining materials (e.g. Al, Ti) are not suitable for metrological application for other reasons. It is therefore necessary to develop metrological procedures for the measurement of low outgassing rates with traceability to primary measurements of very low gas throughputs applicable for materials important for the design of metrological and industrial UHV and XHV devices

Scientific and Technological Objectives

Proposers should address the objectives stated below, which are based on the PRT submissions. Proposers may identify amendments to the objectives or choose to address a subset of them in order to maximise the overall impact, or address budgetary or scientific / technical constraints, but the reasons for this should be clearly stated in the JRP-Protocol.

The JRP shall focus on the development of a primary standard for realisation of the pascal in the UHV and XHV region and the development of techniques for the traceable measurement of low out-gassing rates of materials. A parallel objective is the strengthening of European science and research together with European industry in the field of vacuum and XHV applications currently in development.

The specific objectives are

1. To design, develop and characterise a primary vacuum standard covering the complete UHV pressure range and into the XHV, i.e. down to the pressure 10^{-10} Pa. The residual pressure in a calibration chamber should be below $1x10^{-10}$ Pa and the smallest incremental calibration pressure generated by the system should be of the order of 10^{-11} Pa. A theoretical study should be undertaken to identify a suitable operating principle feasible with the available technique.
2. To develop techniques for the measurement of low out-gassing rates traceable to primary measurements of very low gas throughputs, and which are applicable for materials important in the design of metrological and industrial UHV and XHV devices. The methods should be based on the most contemporary technology and enable the classification of the behaviour of materials in situations typical of metrological applications. The advances in high vacuum, UHV and XHV techniques and technologies should be employed and tested in these metrological applications.
3. To transfer knowledge of the advanced vacuum techniques gained during the development of the primary standard and the outgassing measurement techniques to the metrology community and wider stakeholder group.

Proposers should include target uncertainties based on documented industrial needs.

These objectives will require large-scale approaches that are beyond the capabilities of single National Metrology Institutes and Designated Institutes. To enhance the impact of the research work, the involvement of the larger community of metrology R&D resources outside Europe is recommended. A strong industry involvement is expected in order to align the project with their needs and guarantee an efficient knowledge transfer into industry.

Proposers should establish the current state of the art, and explain how their proposed project goes beyond this. In particular proposers should clearly explain how their proposed project goes beyond and is complementary to the currently funded EMRP JRP IND12 ‘Vacuum metrology for production environments’.

The total eligible cost of any proposal received for this SRT is expected to be around the 2.7 M€ guideline for proposals in this call. The available budget for integral Research Excellence Grants is 42 months of effort.

Potential Impact

Proposals must demonstrate adequate and appropriate participation/links to the “end user” community. This may be through the inclusion of unfunded JRP partners or collaborators, or by including links to industrial/policy advisory committees, standards committees or other bodies. Evidence of support from the “end user” community (eg letters of support) is encouraged.

You should detail how your JRP results are going to:

- feed into the development of urgent documentary standards through appropriate standards bodies
- transfer knowledge to the semiconductor manufacturers, MEMs device manufacturers, spectroscopists, users of deposition and epitaxy techniques, particle physics researchers, space researchers, and wider metrology community.

You should detail other impacts of your proposed JRP as detailed in the document “Guide 4: Writing a Joint Research Project”

You should also detail how your approach to realising the objectives will further the aim of the EMRP to develop a coherent approach at the European level in the field of metrology and includes the best available contributions from across the metrology community. Specifically the opportunities for:

- improvement of the efficiency of use of available resources to better meet metrological needs and to assure the traceability of national standards
- the metrology capacity of Member States and countries associated with the Seventh Framework Programme whose metrology programmes are at an early stage of development to be increased
- outside researchers & research organisations other than NMIs and DIs to be involved in the work

Time-scale

The project should be of up to 3 years duration.

Additional information

The references were provided by PRT submitters; proposers should therefore establish the relevance of any references.

[1] JOUSTEN, K., MENZER, H., WANDREY, D., NIEPRASCHK, R.: New, fully automated, primary standard for generating vacuum pressures between 10^{-10} Pa and $3x10^{-2}$ Pa with respect to residual pressure. Metrologia 36 (1999), 493-497.

[2] YOSHIDA, H., ARAI, K., HIRATA, M., AKIMICHI, H.: Calibration of ultra-high vacuum gauge from 10^{-9} Pa to 10^{-5} Pa by the two-stage flow-dividing system. In: AVS 57th International Symposium and Exhibition. October 17-22, 2010, Albuquerque, USA.

[3] DETIAN, L., MEIRU, G., YONGYUN, C., YAN, F., DIXIN, Z.: Vacuum-calibration apparatus with pressure down to 10^{-10} Pa. J. Vac. Sci. Technol. A 28 (2010), 1099-1104.