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ABSTRACT

An optical pressure standard, based on a multi-reflection interferometric technique, has been recently developed. This quantum-based standard realizes the pascal through the measurement of the refractive index of a gas by an unbalanced homodyne interferometer and it is currently capable of measuring gas pressure with a relative uncertainty of 10 ppm at 100 kPa. The performance of such optical-based standard have been preliminary evaluated by comparing it with two conventional primary pressure standards, namely a force balanced piston gauge and a pressure balance, in the range from 400 Pa to 120 kPa.

This work describes the performed study and discusses the results, which demonstrated the agreement between the optical pressure standard and the conventional standards in the considered range, within their related uncertainties ($k = 1$).

1. Introduction

Current realizations of the pascal are based on different techniques, depending on the pressure range. Below 100 Pa primary vacuum standards usually operate by means of static or dynamic expansion of pure gas [1–4]; above 100 Pa, the pascal is realized through piston gauges and mercury manometers [5–8], the latter progressively abandoned due to presence of mercury, not in line with World Health Organization resolutions [9].

In the SI system of units, the Pascal is defined as force per unit area. This “force per area” definition has been the basis of the most widespread realizations of the pascal, in particular by means of pressure balances, which represented, in the last decades, the more accurate and low-uncertainty primary standard available.

The SI redefinition through seven fundamental constants, including the Boltzmann constant, paved the way to realise the pascal through number density measurements via photonic methods, instead of “force per area” measurements, with the result of establishing a quantum-based traceability route to the pascal [10–25].

The current fixed value of the Boltzmann constant lays the foundation for the development of new photonic-based pressure standards, whose performance is primarily limited by the accuracies of quantum calculations of relevant gas parameters and temperature determination.

The paper reports the progress in the development of the so-named “UINT” optical pressure standard [17] based on a multi-reflection interferometer, i.e. the results of a comparison between this quantum-based standard and two conventional primary standards, as well as mentions the first steps taken at INRiM regarding the realization of a novel standard through Fabry-Pérot (FP) cavity based refractometry, in the framework of the European Partnership on Metrology (EPM) project 22IEM04 “MQB-Pascal”.

2. Methods and procedures

The UINT photonic pressure standard realizes the pascal by means of the measurement of the refractive index of a gas through an unbalanced homodyne interferometer with fixed arms. The measurement arm of the interferometer is formed by a multi reflection double mirror assembly, where a laser beam is reflected several times between two quasi-parallel mirrors A and B, to establish an unbalance length L between the two arms larger than 6 m in a compact set-up. The reference beam coming from the mirror M_R and the measurement beam are recombined by the beam splitter BS1 where interference occurs; the interference fringes are acquired by a CMOS camera and elaborated via a LabVIEW software. The system is also equipped with an additional four quadrant photodiode PD, which eventually can also be used.

The Fig. 1 shows the schematic of the system, including the homodyne UINT laser interferometer and the vacuum/pressure apparatus (see Fig. 2).

The optical components forming the interferometer are placed in a circular plate of 210 mm diameter, inserted inside an aluminium vacuum temperature-controlled chamber C, where the standard pressure p is generated. Both the double mirror assembly of dimension $(90 \times 60 \times 27)$ mm and the circular plate are made of ZERODUR® glass ceramic, to minimize deformations due to eventual temperature variations and gradients. The current operating temperature range of UINT system is between 20 °C and 30 °C, where the nominal mean coefficient of thermal expansion of ZERODUR® is equal to $(0 \pm 0.050 \times 10^{-6})/K$.

Beside the determination of relevant gas parameters, in particular the molar polarizability, the realization of an accurate photonic-based pressure standard implies an assessment of temperature with an uncertainty at millikelvin level: to satisfy this requirement, a double-stage temperature control was implemented, as reported in Ref. [17], where the most important steps concerning the realization of the UINT

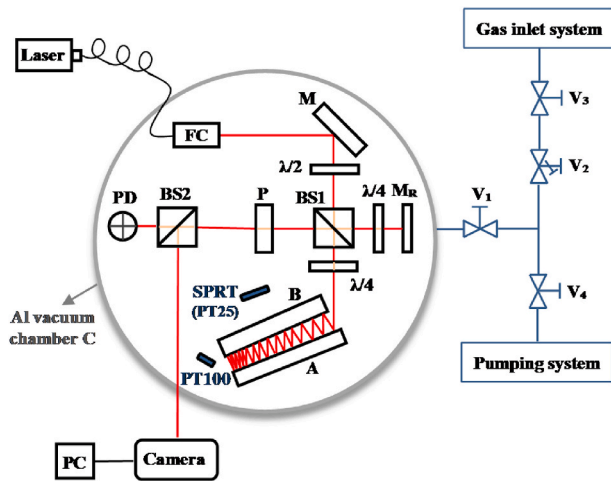


Fig. 1. Schematic of the UINT optical pressure standard; C: aluminium vacuum chamber containing the interferometer; FC: collimator; Mr: reference mirror; BS1 and BS2: beam splitters; P: polarizer; PD: photodiode; V₁, V₃, V₄: valves; V₂: variable leak valve.

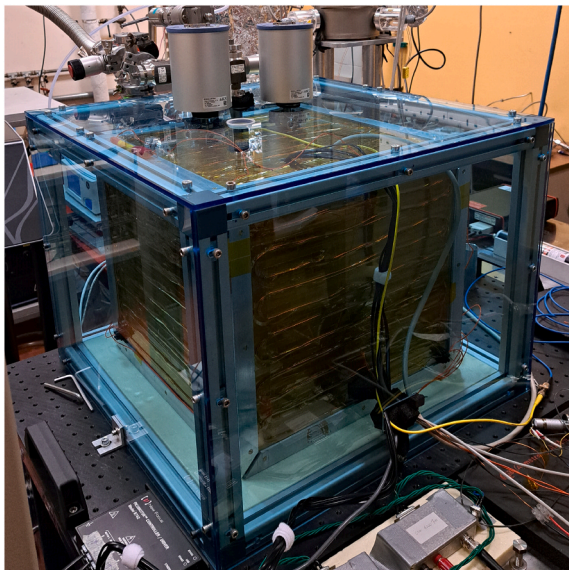


Fig. 2. UINT photonic pressure standard.

standard are also covered.

The UINT interferometer is used as a refractometer determining the refractive index n of the gas at generated pressure p , through the formula:

$$n = n_{vac} + \frac{\varphi\lambda}{L} \quad (1)$$

where n_{vac} is the refractive index in vacuum, at residual pressure p_{res} , φ is the number of interference fringes occurred between p_{res} and p , λ is the laser wavelength and L is the unbalance of the interferometer under vacuum, i.e. the optical path difference between reference and measurement arm of the interferometer, considering the forward and backward optical path.

Once determined the refractive index n , the molar density ρ can be determined through the Lorentz-Lorenz equation:

$$\frac{n^2 - 1}{n^2 + 2} = \rho(A_R + B_R\rho + C_R\rho^2 + \dots) \quad (2)$$

in which A_R is the molar polarizability, B_R and C_R are the second and third order refractivity virial coefficients.

At last, the standard pressure can be deduced from a virial expansion in the molar density, which takes into account the real gas behaviour:

$$p = \rho RT(1 + B\rho + C\rho^2 + \dots) \quad (3)$$

where R is the molar gas constant, B and C are respectively the second and third order density virial coefficients. It is worth underlining that R is the product of Boltzmann's constant k_B and the Avogadro number N_A , defined in the new SI as constants with exact values and no uncertainties.

The details of the uncertainty evaluation of the UINT standard are reported in Ref. [17], fulfilling the main goal of having the ability to measure pressure with a relative uncertainty of 10 ppm at 100 kPa.

A further and fundamental step towards a complete characterization of UINT system is the final evaluation of its performance by comparing it with two different INRiM primary standards, namely a force balanced piston gauge for pressures up to 10 kPa, and a pressure balance for pressures above 10 kPa up to 120 kPa.

The medium used in the comparison was nitrogen gas with 99.9999 % purity.

The measurements were performed at the following 7 values of nominal target pressure p_{nom} of nitrogen: 0.4 kPa, 1 kPa, 3 kPa, 10 kPa, 30 kPa, 100 kPa, 120 kPa.

3. Results and discussion

The measurements in the range between 400 Pa and 10 kPa have been carried out comparing the UINT system with INRiM force balanced piston gauge (FPG), a digital non-rotating piston gauge FPG8601 manufactured by DH-Instruments. The heart of FPG is formed by a piston-cylinder unit having an effective area of about 10 cm² made of tungsten carbide. The piston is not rotating and it is maintained in the centred position by a constant lubricating gas flow through the annular gap. In this type of primary standard, the pressure is defined by means of the force measured using a high precision load cell and the effective area of the piston cylinder assembly,

The standard uncertainty of the INRiM FPG is given by:

$$(0.01 + 1.5 \times 10^{-5}p) Pa \quad (4)$$

The measurements in the range above 10 kPa have been performed comparing the UINT system with INRiM pressure balance DHI-FLUKE PG7601 with piston-cylinder assembly 7100-10.

This pressure balance is based on a simple type piston-cylinder assembly (free deformation) in tungsten carbide and can be used both for relative and absolute pressure measurements. It is equipped with an automated mass handling technology that enables a completely automated operation over the full pressure range. It arranges two different piston-cylinder assemblies:

- DHI -PG7000 -L in the range 15 kPa ÷ 380 kPa (used for this comparison);
- DHI -PG7000 -H in the range 140 kPa ÷ 7.5 MPa.

The standard uncertainty of pressure generated with the piston-cylinder assembly DHI -PG7000 -L used in this work is:

$$(0.04 + 1.0 \times 10^{-5}p) Pa \quad (5)$$

The relative standard uncertainty of the pressure p_{st} generated by the conventional primary standards, as well as by UINT system is summarized in the Table 1.

Three measurement cycles have been carried out both for the comparison UINT-FPG and for the comparison between UINT and the pressure balance PG7601.

Table 1

Relative standard uncertainty (ppm) of the pressure p_{st} generated by INRiM conventional pressure standards and by UINT optical pressure standard.

p_{nom}	$u(p_{st})/p_{st}$	$u(p_{UINT})/p_{UINT}$
Pa	ppm	ppm
400	40	130
1000	25	53
3000	18	20
10000	16	10
30000	11	10
100000	10	10
120000	10	10

The results are presented in the Fig. 3.

The results show the agreement of the UINT system and the conventional pressure standards FPG (400 Pa - 10 kPa) and the pressure balance DHI-FLUKE PG7601 (30 kPa–120 kPa) within their related uncertainty ($k = 1$). Beside the aforementioned agreement, it should be noted that, at lower pressure, the value of the standard pressure generated by the UINT pressure standard is systematically lower than the value associated to the conventional pressure standards: such effect is more evident for pressure below 1 kPa, where the UINT system appears to be less performing and the relative standard uncertainty is higher than 50 ppm.

The research line dedicated to the quantum-based pressure standards at INRiM is continuing, through the realization, within the EPM project 22IEM04 “MQB-Pascal”, of a novel FP-based refractometry pressure standard with main future goal of designing the FP system so it can also be used as a transfer standard and perform a direct measurement comparison between UINT system and the new FP standard.

A detailed treatment of the FP cavity based refractometry to assess pressure is reported in Ref. [26].

In the last years, two typical paths, in terms of used materials, have been explored to realized FP refractometers: cavities bored in low thermal expansion glass, as Zerodur and ULE [12,20,21] or invar-based systems, which have been recently developed, in conjunction with the so-named GAMOR technique [15,24], resulting an alternative and, in some aspects, advantageous approach compared to experimental setup based on the use of low thermal expansion glass.

The design of the first INRiM FP cavity-based pressure standard has been finalized, according to the aforementioned idea of having a transportable system capable of acting also as a transfer standard. The Fig. 4 shows the first recent realization of such FP cavity.

The FP refractometer, with total length of about 120 mm, is essentially composed by a spacer, two closing flanges and two fused-silica high-refractivity mirrors, each of which is placed between the spacer and a closing flange. The spacer is derived by an invar rod of 40 mm diameter, in the centre of which is dug the cavity whose diameter is equal to 6 mm; between each mirror and the spacer, as well as between each mirror and correspondent closing flange, a custom copper gasket is interposed, ensuring the tightness of the whole system, acting itself as a vacuum/pressure chamber.

The laser mirrors are coated to optimally work at the wavelength of 1550 nm, providing a resonator with high finesse (≈ 10000).

First measurements are in progress, aimed to evaluate the vacuum tightness of the system: the preliminary tests are encouraging, showing typical gas leaks less than 10^{-6} Pa/s.

4. Conclusions

Recently, a novel photonic pressure standard, named UINT, based on multi-reflection interferometry, has been developed, demonstrating the ability of measuring gas pressure with a relative uncertainty of 10 ppm at 100 kPa.

The new UINT pressure standard has been compared with two

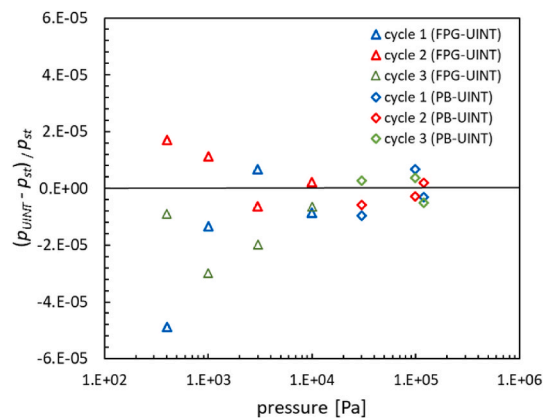


Fig. 3. Results of comparison between UINT photonic pressure standard and INRiM primary conventional pressure standards, namely FPG and pressure balance (PB) DHI-FLUKE PG7601.

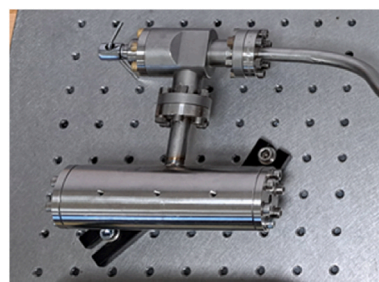


Fig. 4. INRiM invar Fabry Péro-based cavity for pressure measurement.

conventional pressure standards, i.e. a force balanced piston gauge and a pressure balance, in the range from 400 Pa to 120 kPa, to preliminary evaluate the agreement of such novel quantum-based standard with typical traditional standards, based on “force per area” definition of the pascal. The results demonstrated the agreement of the UINT standard and the conventional pressure standards within their related uncertainty ($k = 1$).

After this encouraging result, a novel pressure standard based on a Fabry Péro refractometer is under development at INRiM for the double main purpose of realizing an accurate transfer standard of the pascal and perform a direct comparison between this system and the UINT standard.

From a purely theoretical point of view, both quantum-based methods, based on UINT interferometer and FP cavity, rely essentially on the accurate measurement of the refractive index and the temperature of a gas, using the Lorentz-Lorenz equation and an equation of state. Nevertheless, they operate at different wavelength, through totally different experimental setup, determining the refractive index starting from the measurement of different quantities, i.e. the interference fringes with the UINT system, or the frequency in case of the FP cavity-based device. In this context it will certainly be interesting and instructive to carry out measurement comparisons between these two quantum-based standards, not only to test their mutual agreement within their uncertainty, but also to provide additional important information regarding the experimental determinations of relevant gas parameters, primarily the molar polarizability of gases at different wavelength.

In addition, in the medium-long term, the quantum-based primary standards could be miniaturised, providing faster and calibration-free pressure measurements for industry at a fraction of the present cost.

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