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TOWARDS AN IMPROVED HELIUM-BASED REFRATOMETER FOR PRESSURE MEASUREMENTS

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Abstract:

This paper describes a quantum realization of the pascal based a helium absolute refractometer at 532 nm. The short-term stability in pressure is ±2 mPa with a resolution in pressure of this new optical pressure standard is better than 1 mPa with temperature stability about 1 mK. A new design of the refractometer is presented to have better temperature stability and accurate temperature measurements to reach uncertainties in pressure better than current conventional methods.

Keywords: refractometry, Fabry-Perot, pressure measurement, quantum pascal

1. INTRODUCTION

For many decades and without any significant expected improvement, primary absolute pressure measurements in the range 1 Pa to 100 kPa are mainly based on liquid column manometers, gas-operated pressure balance or static expansions systems. The relative uncertainty in CMCs is about 10^{-6} at 100 kPa and about 10^{-4} at 1 Pa.

At LCM, over the past few decades, the best realizations of the pascal, see Figure 1, have been obtained using two different standards. First one in the range from 10 kPa to 500 kPa, an automatic absolute pressure balance APX50 equipped with 50 mm diameter piston-cylinder units is used. For the realization and the dissemination of the pressure scale, in the range 0.5 Pa to 10 kPa is achieved with a force-balanced piston gauge (FPG). For example, the extended relative uncertainty is 9×10^{-6} at 100 kPa and 6×10^{-4} at 1 kPa. Below 500 Pa, the relative uncertainty increases sharply.

There is a special need to reduce relative uncertainties below 100 kPa with, if possible, a single instrument in a wide range of pressures below atmospheric pressure. The Consultative Committee for Masses and Associated Quantities (CCM) and the Euramet Technical Committee for Masses and Associated Quantities (TC-M) have also identified the needs and limitations of conventional methods.

With the new international system of units (SI), the scientific community is exploring new avenues directly linked to fundamental laws and or constants of physics. Thus, for pascal, a standard could be based on optical or electrical measurements such as the refractive index or the permittivity of a gas, certain parameters of which can be calculated ab initio (with helium for example). In this context, it is necessary to continue the scientific and technological development of these methods to extend the measurement range and significantly the uncertainties of conventional methods.
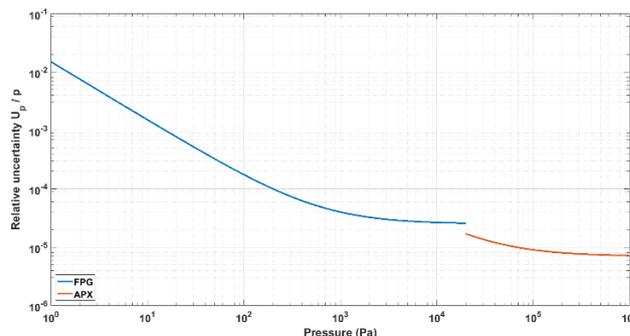

Figure 1: Current pressure relative uncertainties at LCM. In the range 10 kPa to 500 kPa, an automatic absolute pressure balance APX50 and in the range 0.5 Pa to 10 kPa a force-balanced piston gauge (FPG).

With the new international system of units (SI), many developments are now in progress for the realization of a "quantum pascal" by means of methods such as superconductive microwave resonators [1], Rayleigh scattering, multi-reflection interferometry [2], gas phase thermometry and absorption spectroscopy methods [3] as well as Fabry-Pérot (FP) cavity refractometers [4].

In the field of optical methods based on Fabry-Pérot, the National Institute of Standards and Technology (NIST) develop since 2004 an absolute refractometer based on a double Fabry-Pérot cavity in ULE using He-Ne lasers. NIST claims an extended

relative uncertainty $U_r(p) = 18 \times 10^{-6}$ at 100 kPa and 4×10^{-3} at 1 Pa [5], [6].

RISE (RISE Research Institutes of Sweden) and Umeå university in Sweden also develop from 2000's, this type of refractometer (double FP cavity in Zerodur®) using an original method of pressure modulation (GAMOR) to limit temperature effects on the cavity [7], [8]. NIM (National Institute of Metrology of China) in China, NMIJ (National Metrology Institute of Japan) in Japan [9] and PTB (Physikalisch-Technische Bundesanstalt) in Germany [10] start the development of such instruments.

French metrology is currently developing a quantum realization of the pascal based on a helium absolute refractometer at 532 nm [11]: the optical path travelled by a laser beam in a single Fabry-Perot cavity is measured either with helium gas or under vacuum. The ratio between these two optical paths, with appropriate corrections, gives the refractivity of the helium. By comparison with an *ab-initio* determination of helium refractive index and an accurate measurement of the gas temperature, we determine the absolute gas pressure inside the refractometer using Lorentz-Lorenz equation. Other gases can be used, after determining their atomic parameters by comparison with helium.

This paper describes shortly the current experimental set-up and the latest results on the performance of the refractometer for pressure measurements in the range 100 Pa to 100 kPa. We also present an improved version to insure the best stability in temperature, a limited temperature gradient and accurate temperature measurements with an uncertainty lower than 1 mK.

2. EXPERIMENTAL SET-UP

2.1. General view

The experimental set-up was formerly used as an absolute refractometer of air and helium [12] for the determination of refractive index with a standard uncertainty of 10^{-8}. It is mainly composed of a large vacuum chamber that hosts the Fabry-Perot cavity and two frequency doubled Nd:YAG lasers at 532 nm: one is servo-controlled on transition iodine and second one is servo-controlled on the FP cavity. A frequency beat is performed between the two lasers to measure the frequency of second laser.

The Fabry-Perot cavity in Figure 2 is a plano-concave optical resonator which spacer is made of Zerodur® of 100 mm-long and 25 mm-diameter. It is placed inside a copper and dural cylinders with a temperature regulated system using thermofoils. The assembly is then placed in a 10L vacuum enclosure. Gas temperature is measured with 4 wires 100 Ω RTD sensors along the FPM cavity.
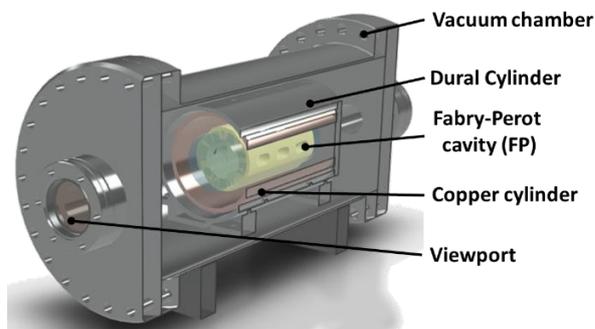

Figure 2: Cross-section of the vacuum chamber with a Fabry-Perot cavity. Fabry-Perot resonator composed of two silica mirrors and a spacer made in Zerodur®.

2.2. Methods to pressure determination

Pressure P can be determined using two methods (Figure 3). In a first approach, refractometer can be used in absolute way: starting from vacuum as reference and measuring frequency variation ν of the servo-controlled laser on the cavity between vacuum and pressure to measure. Frequency variation allows to determine refractive index n variation of the gas and therefore pressure variation if the thermodynamic temperature T is accurately measured.
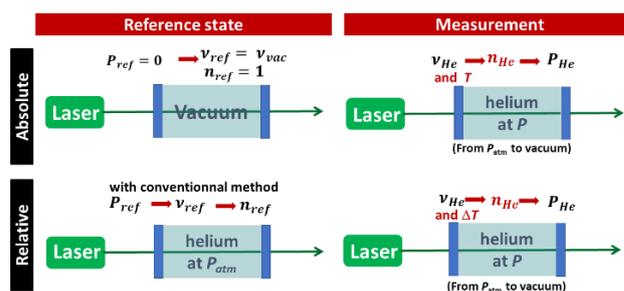

Figure 3: Schematic of the two methods (absolute and relative) to determine a pressure using a refractometer. P is a pressure, n is the refractive index et ν the frequency. ref index indicates the reference state of the parameters and He index indicates the measurement state.

Second way consist to use the refractometer in relative mode. In this case, reference state is a gas near the atmospheric pressure. This pressure is measured with a conventional method (uncertainty better than 10^{-5}). Then, frequency variation of the laser is measured between this reference pressure and pressures up to 100 Pa. It is not necessary to measure thermodynamic temperature of the gas but just only the temperature difference. In this case, the traceability of the pressure is given by the pressure balance for the absolute value and by atomic gas parameters for pressure variation.

3. MAIN RESULTS

To achieve the final objectives in terms of pressure uncertainties (5×10^{-4} at 1 Pa, 10^{-5} à 1 kPa), ageing and mechanical deformation of the FP cavity

are two parameters which limit the uncertainty in relative mode [11]. In the case of absolute use, the thermodynamic temperature of the gas is also a limiting factor. We need to measure the temperature helium gas with an uncertainty lower than 1 mK and to maintain inside the refractometer a pressure with a stability lower than 0,01 Pa over several tens of seconds.

3.1. Ageing, xxx and uncertainties

The uncertainty of the ageing of the cavity is 1×10^{-11}/day . This value leads to a pressure variation of 3 mPa for N_2 and 20 mPa for He. It is necessary to improve the uncertainty on this parameter by a factor greater than three for pressure below 100 Pa by using a pressure balance.

For the thermal expansion coefficient, a relative uncertainty about 4% is enough if the temperature of the cavity can be reproduced at 10 mK. These two conditions are already met today with a relative uncertainty of 0,15 mPa for N_2 and 1,2 mPa for He.

The uncertainty of 5×10^{-15} obtained for the pressure-induced deformation for the absolute mode (vacuum as a reference) do not ensure the required uncertainty. In the case of the relative mode measurements, the uncertainty is sufficient. However, recent changes to the cavity mirrors have significantly improved the uncertainty on the measurement of the free spatial range (about 200 Hz in vacuum) and should improve this uncertainty component.

3.2. Temperature stability and measurement of the temperature

To stabilize the temperature in the FP cavity, we therefore set up a temperature regulation system at ±1 mK (completely autonomous system based on Arduino). For that, to the Dural cylinder, we are glued two thermofoils in the opposite side. The copper cylinder allows to create a heat shield. Finally, the entire enclosure was confined in an expanded polystyrene box. The temperature is measured inside the cavity by 4 Pt100 RTD probes (4-wire measurement) distributed over the entire cavity.

The temperature stability inside the cavity is about 2 mK but the temperature gradient along the 100 mm of the cavity is around 5 mK. Long-term stability is also not guaranteed due to the excessive volume of the vacuum enclosure.

With the Pt100 probes, it is not possible to measure thermodynamic temperature better than 10 mK. For an absolute pressure measurement with a relative uncertainty of 10^{-5} an uncertainty of less than 3 mK is required (whatever the pressure to be measured). It is therefore necessary to improve the uncertainty on the value of the gas temperature by temperature probes and its location. If the refractometer is used in relative mode, the only constraint is that the temperature between the

reference state and the measurement state is the same to within 3 mK.

3.3. Pressure stability

In previous works [11], we were limited by the stability of the pressure due to the use of the commercial pressure regulator. The pressure resolution was then about 0.2 Pa at the atmospheric pressure. To reduce this pressure stability, we have installed and tested a DHI PG7607 pressure balance associated with a 20 cm² piston-cylinder whose pressure expanded uncertainty is $0.20\,\text{Pa} + 8.0 \times 10^{-6}p$ This has led to reach the target pressure stability in Figure 4 and then allows us to determine the pressure by means of the refractometer with a stability of ±2 mPa over 20 min.
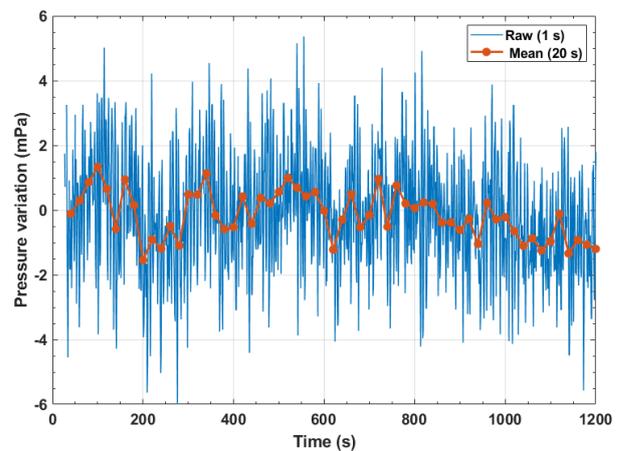

Figure 4: Pressure variation measured by the refractometer over 1200 s.

We can note that this excellent result is only possible in the short term because, it is not possible to guarantee stability in pressure and temperature more than 20 min.

4. IMPROVED DESIGN OF THE REFRACTOMETER

To resolve the main issues of the current refractometer, we have designed a new set-up still using a helium absolute refractometer at 532 nm. This design has the advantage to be more compact (20 times smaller in volume) to avoid temperature gradient and temperature drift. Figure 5 presents a project design of the improved set-up: a 50 mm-squared single Fabry-Perot resonator (two silica mirrors and a spacer made in Zerodur®) and an enclosure in copper working under vacuum and gas.

We also will use encapsulated high quality Pt25 probe used at gallium point to have an uncertainty lower than 1 mK.

In the longer term, a FP cavity made of a more stable material than Zerodur® can be used. Several publications report a ceramic, NEXCERA™ (Krosaki Harima), which has an aging almost 5 times lower than Zerodur® [13].
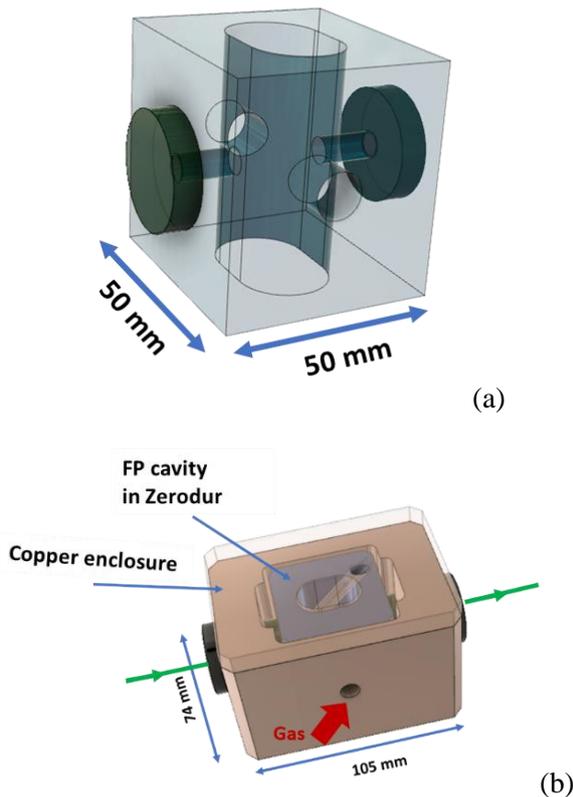
(a)

(b)

Figure 5: Project design of our new quantum pascal realization set-up. (a) 50 mm single Fabry-Perot (FP) resonator composed of two silica mirror and a spacer made in Zerodur®. (b). Cross section of the copper enclosure including the FP resonator.

Simulations of the pressure-induced deformation have been performed on with new design of the Fabry-Pérot cavity using ANSYS Workbench Rxx academic version. We use the same methodology presented in [14]. The pressure-normalized deformation value is $\frac{\frac{\Delta L}{L}}{P} = -6.5810 \times 10^{-12}$ Pa^{-1}. The simulated value for the current FP cavity presented in this paper is $\frac{\frac{\Delta L}{L}}{P} = -6.3899 \times 10^{-12}$ Pa^{-1}.

5. SUMMARY

This paper describes a quantum realization of the pascal based a helium absolute refractometer at 532 nm. The short-term stability in pressure is ±2 mPa with a resolution in pressure of this new optical pressure standard is better than 1 mPa with temperature stability about 1 mK. Pressure can be determined using an absolute method or a relative method. Aging and pressure-induced deformation of the FP are the main contributions of the uncertainty in the relative mode. In the case of absolute mode, the thermodynamic temperature of the gas is also a limiting factor. For that, a new design of the refractometer is presented to have better temperature stability and accurate temperature measurements to reach uncertainties in pressure better than current conventional methods. Improvements (performance, uncertainties, etc.) are mainly linked to the objectives of the "QuantumPascal" EMPIR project.

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