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PRIMARY VACUUM STANDARD CMI FOR UHV PRESSURE RANGE 1×10^{-6} TO 1×10^{-10} Pa

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Abstract: A primary vacuum standard for UHV pressure range is under development in Czech Metrology Institute (CMI). In order to cover the UHV range down to the limit 1×10^{-10} Pa a method of calibration in non-equilibrium gas (so called dynamic extension method) was employed. The apparatus is divided into three parts: the dynamic extension stage, the orifice flow standard and the flowmeter. The dynamic extension stage is designed as an XHV apparatus manufactured from a low outgassing rate material – beryllium copper alloy. A special 4 K bakeable refrigerator cryopump is used as the main pump.

Keywords: Primary vacuum standard, vacuum gauge calibration in UHV range.

1. INTRODUCTION

In spite of long term performed attempts to develop an absolute gauge for ultra high vacuum (UHV) pressure range the primary standards for this range are conceived almost entirely as vacuum apparatuses. A defined gas pressure is adjusted in by means of certain defined thermodynamic process [1]. Nonetheless, also this way of primary vacuum standard design is difficult for the range of high and very high vacuum (i.e. very low pressure range) because in addition to the intentionally invoked processes performed with the gas admitted controlled way some undesirable processes run in vacuum apparatuses with a gas spontaneously appearing in the chambers. These undesirable processes cause uncertainty of the adjusted value of pressure. In order to keep this way originating uncertainty acceptably low the amount of the undesirable gas spontaneously emerging in the apparatus has to be negligible compared to the amount of gas used for calibration.

Another requirement is that the gas pressure in the calibration chamber has to be well defined, thus, the gas has to be in thermodynamic equilibrium with Maxwellian molecule velocity distribution. This equilibrium can be at small amount of gas in the vacuum chamber easily disturbed.

The possibility to increase the amount of gas intentionally admitted into the apparatus (working gas) in order to make the amount of gas released in vessel spontaneously (undesirable gas) negligible is used in orifice flow standards some decades already. The working gas is continuously admitted and simultaneously pumped out. In order to disturb the thermodynamic equilibrium only negligibly the admitting of gas and pumping speed are strongly limited. The pumping aperture area must not exceed a small fraction of vessel inscribed sphere surface, thus, also even smaller fraction of the vessel real inner surface. Because the rate of release the undesirable gas is proportional to the inner surface, any enlarging the vessel keeping the ratio of inner surface to the pumping aperture area doesn't improve the conditions. At a given achievable outgassing rate from available materials there is a lower range limit for primary vacuum standards based on this principle. For example at most commonly used stainless steel the still routinely achievable outgassing rate of the order of 10^{-10} Pa·m/s of magnitude allows to design standards based on this principle with lower pressure limit of the order of 10^{-9} Pa of magnitude. There are two ways how to overcome this limit: a) to use a material with lower outgassing rate and b) to employ a method enabling to calibrate a gauge under slightly non-equilibrium conditions and to increase the amount of working gas flowing during the calibration through the chamber i.e. enlarge the pumping speed.

2. CONCEPTION OF THE CMI UHV PRIMARY STANDARD

Both ways were applied at design of the UHV primary standard CMI. The calibration under non-equilibrium conditions is expected.

The possible transfer standards to be calibrated in the standard were restricted to the hot cathode ionisation gauges only. This restriction is not too constrictive because just these gauges are mainly used for calibration in the UHV range. The output signals of hot cathode ionisation gauges are proportional to the amount of particles in the ionisation areas and practically independent of their velocities or directions. Then the method of dynamic extension [2] can be applied: The transfer standard gauge is calibrated in an orifice flow standard with equilibrium gas velocity distribution down to the lowest possible pressure in its range. This is initial point for the dynamic extension process. The gauge is transferred to the dynamic extension (DExt) apparatus vessel and such working gas throughput is

admitted that the gauge output signal is exactly the same as at the initial point in equilibrium conditions. Analysis performed in Ref. [2] shows that now, if the throughput is reduced *n*-times the gauge output signal corresponds the equilibrium pressure *n*-times lower than in the initial point.

The gas throughput admitted into the DExt vessel can be controlled by the working gas pressure in front of the input duct aperture. There could be used an auxiliary chamber equipped with a gauge calibrated in an orifice flow standard. The orifice flow standard is used instead. The gas throughput admitted into the DExt vessel is controlled directly by the pressure adjusted in this orifice flow standard. The additional uncertainty arising at a gauge (for control calibration) is eliminated this way. Moreover, the orifice flow standard is used for the transfer standard calibration in the initial point.

The orifice flow standard has to be supplemented with a primary gas flowmeter. The constant pressure gas flowmeter principle was chosen. The orifice flow standard with the flowmeter together constitute a high quality high vacuum primary standard and the flowmeter is also gas leak primary standard.

3. DESIGN OF THE STANDARD

The arrangement of the apparatus is schematically sketched in Fig. 1. As it is obvious the apparatus consists from three principal parts: the dynamic extension stage, the orifice flow standard and the flowmeter. The most crucial part is the dynamic extension stage. To achieve a relative uncertainty of the order of some per cent of magnitude at calibration near the limit 1×10^{-10} Pa, the ultimate pressure in this part should be of the order of 10^{-12} Pa of magnitude i.e. in XHV range.

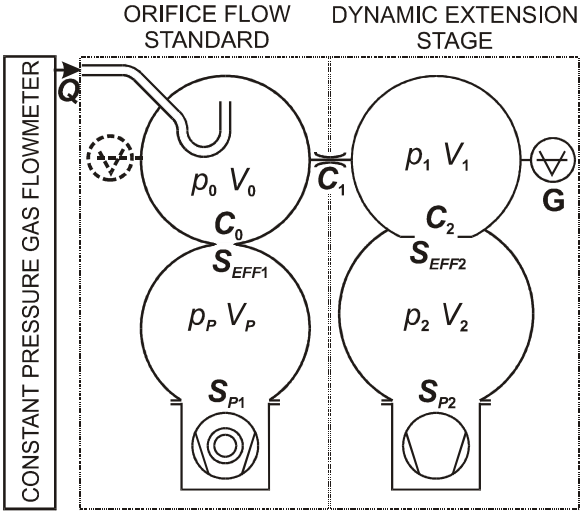

Fig. 1. Schematic arrangement of the CMI UHV primary standard.

The ultimate pressure is given by outgassing rate from the surfaces, effective pumping speed in the chamber and ultimate pressure of the pump. The effective pumping speed has to be sufficiently stable for metrological application, thus intrinsic pumping speed of the pump has to be stabilised by reduction it with a tube or orifice conductance (denoted as C_2 in Fig. 1). Detailed analysis has shown that the values of outgassing rate achieved with stainless steel don't allow to provide surely the desired ultimate pressure. Aluminium chambers and vacuum components are commercially available as well but main disadvantage of this solution is poor compatibility with any stainless steel component. That is why the dynamic extension stage was manufactured from beryllium copper alloy (BeCu) [3-6] by the firm VacLab Inc. This material has some properties similar like pure copper but thanks to the small content (0.2 %) of beryllium a protective beryllium oxide layers can be created on surfaces after manufacturing single parts. These layers reduce the outgassing rate down to a value of 10^{-13} Pa·m/s. Unfortunately, the copper with beryllium addition must not be welded or brazed. Because of this, all parts are manufactured from one piece of material and mutually connected with flanges. The design of all apparatus had to be adapted to this rule. Resulting arrangement of this XHV stage is in the Fig. 2.

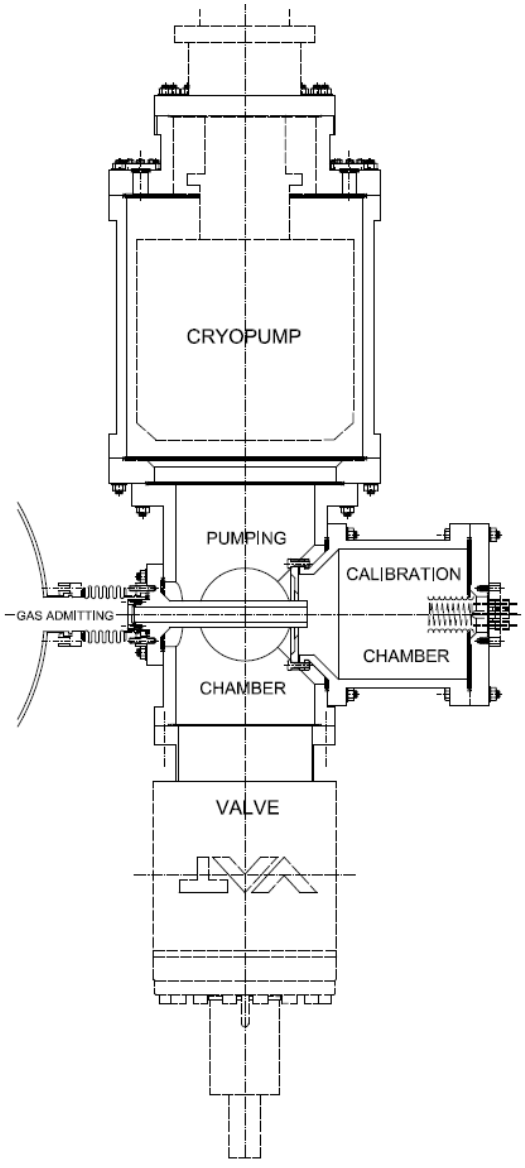

Fig. 2. Arrangement of the XHV stage.

The working gas from the orifice flow standard is admitted into the calibration chamber where is the transfer standard to be calibrated. The gas flows out to the pumping chamber. The main pump is connected there. An auxiliary turbomolecular pump (used for baking-out the apparatus) is connected via an all-metal angle valve on the opposite side of the pumping chamber.

4. THE MAIN PUMP

Main pump has to have sufficiently high pumping speed which should be also stable enough in order to be well stabilised with a serial conductance C_2. Extremely exacting requirement is the ultimate pressure of the pump of the order of 10^{-12} Pa of magnitude.

Cryogenic pump of a special design is considered to meet both these requirements. It is the refrigerator type of cryopump but fully bakeable up to 300 °C. The inner parts of the cryohead are disassembled before baking and reassembled after it.

Hydrogen is the main gas species expected to be pumped in the XHV pressure range. Temperature 4.8 K is achieved at the second stage of the pump, nevertheless, hydrogen saturated vapour pressure is still to high even at this temperature. The cryogenic surface of the second stage was enlarged up to approximately 0.2 m^2. Then the hydrogen will be pumped in the regime of unsaturated monolayer during calibration in the lowest pressure. View of the cryopump is in the Fig. 3.

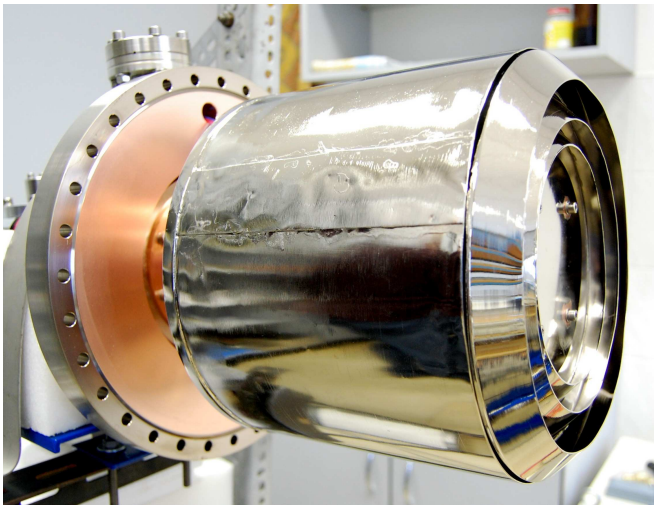

Fig. 3. The special 4K refrigerator bakeable cryopump used as main pump in the XHV stage.

5. CONCLUSIONS

A primary vacuum standard for UHV pressure range is in development in Czech Metrology Institute.

In order to achieve the lower range limit 1×10^{-10} Pa with acceptably low uncertainty two principles were followed:

a) A material of extremely low outgassing rate was used for design of XHV stage of the apparatus.

b) A method enabling calibration in gas with non-negligibly disturbed thermodynamic equilibrium was employed at the cost of restriction the transfer standards to hot cathode ionisation gauges only.

A special 4 K bakeable refrigerator cryopump was used as main pump in the XHV stage.

6. REFERENCES

[1] L. Peksa, D. Pražák, T. Gronych, P. Řepa, M. Vičar, J. Tesař, et al., Primary vacuum standard for UHV range - Standing experience and present problems, *MAPAN – J. Metrology Soc. India*, vol. 24, pp. 77-88, 2009.

[2] L. Peksa, T. Gronych, M. Jeřáb, P. Řepa, M. Vičar, Z. Krajíček, et al., Dynamic extension - A new principle for a vacuum primary standard for UHV-range (10^{-6}-10^{-10} Pa) calibration of hot cathode ionisation gauges, *Vacuum*, vol. 85, pp. 1047-1051, 2011.

[3] F. Watanabe, Y. Koyatsu, H. Miki, Attaining an ultralow outgassing rate of 10^{-12} Pa m^3 s^{-1} m^{-2} from an oxygen-free high conductivity copper chamber with beryllium–copper–alloy flanges, *J. Vac. Sci. Technol.*, vol. A 13, pp. 2587-2591, 1995.

[4] F. Watanabe, A. Kasai, Low outgassing residual gas analyzer with a beryllium–copper-alloy-flanged ion source, *J. Vac. Sci. Technol.*, vol. A 13, pp. 497-500, 1995.

[5] F. Watanabe, Mechanism of ultralow outgassing rates in pure copper and chromium–copper alloy vacuum chambers: Reexamination by the pressure-rise method, *J. Vac. Sci. Technol.*, vol. A 19, pp. 640-645, 2001

[6] F. Watanabe, Extremely low-outgassing material: 0.2% beryllium copper alloy, *J. Vac. Sci. Technol.*, vol. A 22, pp. 181-191, 2004.