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DESIGN AND DEVLOPMENT OF MEMBRANE PRESSURE REGULATOR FOR CRYOGENIC APPLICATIONS

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Abstract:
This paper investigates different possibilities of measurement and control of pressure in the cryostat filled with liquid nitrogen. Laboratory designed membrane pressure regulator for automatic control of pressure in the range from 0.9 to 1.3 bar gauge in the cryostat is presented. The performance of the special purpose regulator is evaluated through measurement of pressure deviations from set-point value and their effect on thermal conditions in the cryostat. Results are compared to values obtained using manual pressure regulator.

Keywords: cryostat; pressure; regulation; liquid nitrogen

1. INTRODUCTION

Cryostats can be found in many different applications, ranging from industrial scale products to cryostats designed for specific scientific research. Increase in utilization of cryogenic liquids has led to significant progress in the storage time of cryogenic storage systems. Diminution of heat leaks through efficient insulation [1] and accurate control of pressure in the cryostat, have shown to be crucial for maximizing storage time [2].

In the field of scientific metrology, cryostat and cryogenic liquids can be found in primary thermometry for achieving stable low temperature conditions. Different cryostats have been developed at national metrology institutes, using mostly liquid nitrogen as cryocooler [3], [4]. As the nitrogen experiences phase transition from liquid to gaseous state in the cryostat, variation of pressure has direct influence on thermal conditions in the cryostat.

This paper provides design and development of pressure controller for cryostat, used for achievement and maintenance of triple point of argon at the Laboratory for Process Measurement.

2. METHODS AND EQUIPMENT

Triple point of argon (83.8035 K) is one of the cryogenic thermometric fixed points used for dissemination of International Temperature Scale (ITS-90). Triple point state of argon in the hermetically sealed cell is achieved through liquid nitrogen cooling in the specially designed cryostat (Figure 1), developed at LNE-Cnam [5]. The achievement and duration of triple point state of argon depend on the stability of the thermal conditions in the cryostat. As liquid nitrogen in the cryostat lies on the phase transition line (~ 77 K), regulation of the vapour pressure in the vessel directly effects the temperature of evaporation (roughly 0.2 bar/K). Outflow of liquid nitrogen vapours diminishes build-up of the pressure in the cryostat and therefore lowers the temperature. Through the outflow of vapours, pressure is controlled, and desired temperature conditions are achieved in the cryostat.

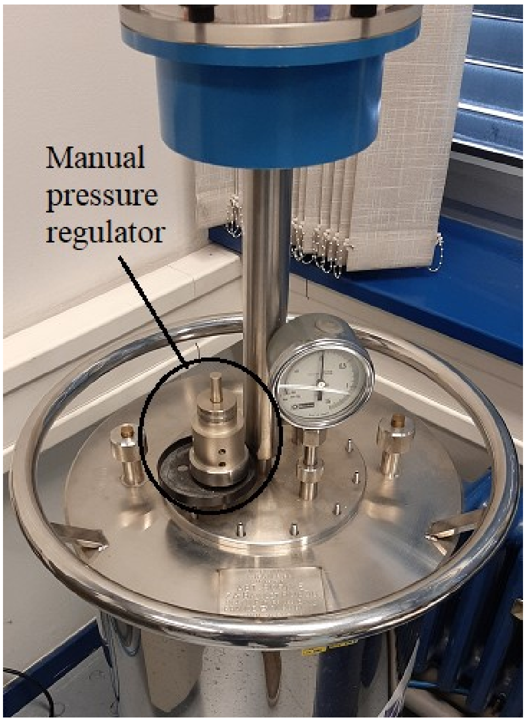

Figure 1: Cryostat with manual pressure regulation

In the scope of this research, two different cryostat pressure regulation principles were investigated. Existing manual pressure regulator was compared to specially designed membrane pressure regulator (Figure 2).

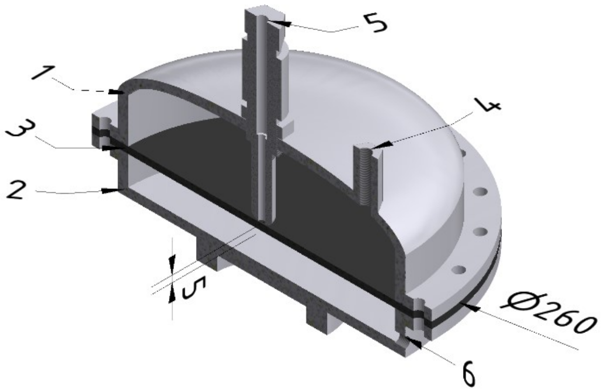

Figure 2: Cross-section of membrane pressure regulator

2.1 Manual pressure regulator

Manual pressure regulator operates on the principle of different weights acting on a ball valve, creating a force that controls the amount of nitrogen vapours exiting the cryostat. The performance of manual pressure regulation was tested during the process of realisation of triple point of argon. The measurement system used in this research is shown on Figure 3.

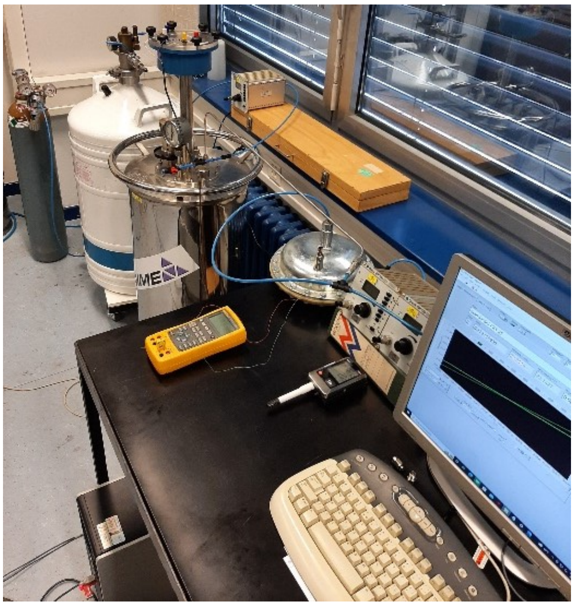
Figure 3: Measurement system for the realisation of the triple point of argon

The effect of pressure change on the temperature in the cryostat was observed. Gauge pressure in the cryostat was monitored using a membrane pressure transducer (Druck DPI530) connected over a 6 mm stainless steel tube and a mechanical Bourdon tube manometer connected directly on the cryostat. To avoid possible influence of low temperature nitrogen vapours on the pressure transducer, connecting tube was heated using forced convection of ambient air. Also, temperature was measured at the inlet of pressure gauge using type K thermocouple. An absolute pressure transducer (Vaisala PTB330) was used to monitor atmospheric pressure oscillations in the laboratory in order to quantify their effect on the conditions in the cryostat. Set of weights for the manual pressure regulation was weighted prior to testing using a precision scale. The influence of each weight on the gauge pressure in the cryostat was noted. Also, the effect of pressure regulation on the rate of temperature rise was examined. Due to the low temperature of nitrogen vapours, ice can form on the outlet valve and masses creating an additional unknown force, acting on the ball valve. To avoid that, plastic shield was used to cover the manual pressure regulator.

2.2 Membrane pressure regulator

To enable automatic regulation of pressure in the cryostat and to overcome the shortcomings of the existing pressure regulation system, special purpose membrane pressure regulator was designed and constructed (Figure 2) within the scope of this research.

Design and operating principle

The regulator was constructed from two stainless steel chambers (upper (1) and lower (2)) divided by 5 mm thick impermeable natural rubber membrane (3). Upper chamber contains an outlet (4), and an adjustable inlet valve (5), connected to a cryostat through a 6 mm stainless steel pipe. Lower chamber is at inlet (6) connected to the nitrogen gas container over a pressure regulator (Druck DPI 530). Nitrogen gas pressure inside the lower chamber deforms the membrane towards the bottom of the inlet valve. Manually adjusted distance between bottom of the outlet valve and the membrane as well as pressure in the lower chamber, define the allowed pressure rise in the cryostat. Nitrogen vapours exiting from the cryostat enter the upper chamber and when the outlet valve is closed, pressure rises. Since the liquid nitrogen in the cryostat is constantly evaporating due to heat leaks from the environment, nitrogen vapours keep filling the upper chamber. When the pressure value in the upper chamber exceeds the counter-pressure in the lower chamber, membrane deforms downwards and outlet valve is opened, allowing the outflow of vapours and pressure drop. Operating principle of the regulator is based on the accurate control of pressure in the lower chamber, according to the pressure and temperature measured in the cryostat.

Low temperature of nitrogen vapours exiting the cryostat can cause problems for pressure measuring and regulating devices. To avoid the possibility of pressure transducer failure and inaccurate pressure readings, nitrogen vapours are heated before entering the measuring device and regulator.

Laboratory-designed membrane pressure regulator was constructed according to the design plans that can be partly seen in Figure 2. After construction, few additional adjustments had to be made in order to enable full functionality of the regulating device. Originally planned rubber used for membrane was too rigid and had to be changed due to lack of flexibility. Natural rubber with Shore A hardness of 45 was selected, as it shows good characteristics at low temperatures. Also, adjustable valve (5), primarily planned as outlet, was used as inlet for nitrogen vapours and it was additionally machined to ensure lighter sealing when rubber membrane presses upon it. In order to enable more precise and faster regulation of pressure in the lower chamber, lower chamber was filled with composite panel to reduce its volume.

Testing

Membrane regulator was tested under the conditions, close to those that are expected during its use as the pressure regulator for the triple point of argon cryostat. It was connected to the cryostat over a 6 mm stainless steel pipe with parallel connection to a pressure gauge (Druck DPI 530). Oscillations of pressure in the cryostat were monitored under different counter pressure set points in the lower chamber. Manual pressure regulator was fully sealed during testing of the membrane pressure regulator, to exclude its possible influence.

Temperature in the cryostat was monitored with quartz-sheathed standard platinum resistance thermometer (Pt-25), placed in the tube at the centre of argon triple-point cell. Thermometer resistance was measured with high precision thermometry resistance bridge (ASL F18). To avoid condensation, thermometer tube was filled with helium gas and kept at slight overpressure. All the pressure measuring devices used, were calibrated against national pressure standard, whose performance in the range in question was evaluated through key comparison [6].

3. RESULTS

3.1 Testing of manual pressure regulator

Figure 4 shows gauge and atmospheric pressure, and Pt-25 thermometer resistance during triple point of argon plateau. During the filling of the cryostat with liquid nitrogen, cryostat was open to the atmosphere. When the appropriate level was achieved, cryostat was closed, and instant pressure rise could be observed. During the whole realisation manual pressure regulator was loaded with entire combination of weights (Table 1) and maximum achieved gauge pressure in the cryostat was 1059 mbar, due to sealing problem of the cryostat.

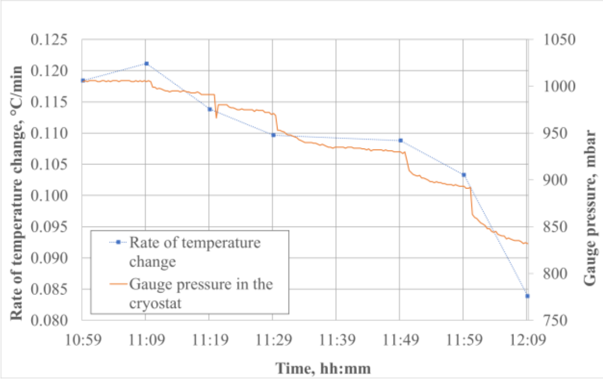

Figure 4: Change of temperature due to pressure change

Table 1: Manual pressure regulator characteristics

Weight no.	Mass, g	Average gauge pressure change, mbar	Pressure regulator sensitivity, mbar/g
0	57.9	65	1.1
1	16.7	12	0.7
2	16.3	17	1.0
3	36.4	35	1.0
4	36.8	35	1.0

Oscillations of atmospheric pressure (dashed line on Figure 5) during the plateau were not larger than 1.4 mbar and no observable change of the thermal conditions in the cryostat was detected because of that.

As can be seen on Figure 5, triple point of argon plateau lasted around 4 hours with gradual drop of gauge pressure in the cryostat of 20 mbar.

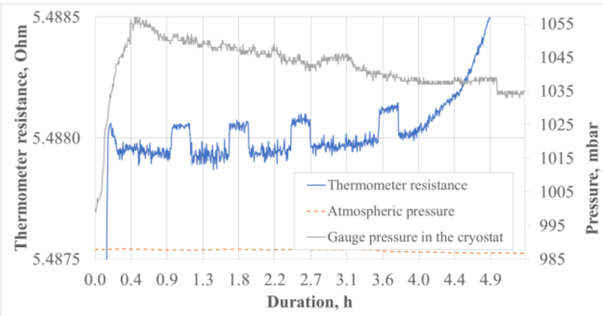

Figure 5: Plateau of the triple point of argon

Manual pressure regulation characteristics were inspected twice, and results are shown in Table 1 and Figure 6. Sensitivity of manual pressure regulator was around 1 mbar/g, except for the low mass weights where accuracy of pressure measuring (0.1 % of full scale; 2 mbar) device has greater influence on measured pressure. Also, possible pressure change using manual pressure controller is limited with mass of the weights.

Pressure drop in the cryostat causes decrease of the saturation pressure and temperature of liquid nitrogen. Lower saturation temperature influences the thermal conditions in the cryostat and causes slower heating (Figure 4).

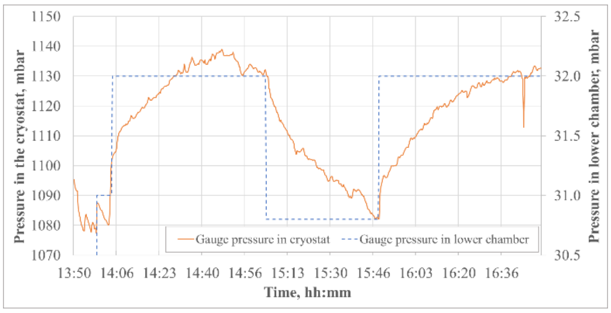

Figure 6: Performance of the new pressure regulator at 1100 mbar gauge

3.2 Testing of laboratory-designed membrane pressure regulator

Membrane pressure regulator was tested at different pressure set points in the lower chamber. Figure 6 shows the performance of the regulator at around 1100 mbar gauge in the cryostat. This pressure is aimed as it represents the required saturation pressure of liquid nitrogen for achieving temperature slightly higher than temperature of triple point of argon. Therefore, the focus of the research was to achieve optimal regulation around that pressure to keep the desired temperature as long as possible. At pressure of 32 mbar in lower chamber, regulation of the pressure in the cryostat was achieved with oscillations not greater then ± 5 mbar.

Prior to testing of the membrane pressure regulator, sealing problem in the cryostat was fixed and maximal detected pressure in the cryostat was 1293 mbar gauge at counter pressure of 50 mbar in the lower chamber.

4. DISCUSSION

After the construction of the laboratory-designed membrane pressure regulator, few alterations, in regard to primary design, had to be made to cope with the problems that emerged during dry testing. In the dry testing phase, pressure of nitrogen vapours in the upper chamber was simulated with Druck DPI 615 pressure calibrator. The sealing between the membrane and the inlet valve (part 5 at Figure 2) did not allow efficient release of the built-up pressure, so the design of the valve was modified (Figure 7). After alteration, pressure release in the upper chamber was enabled at higher counter pressure in the lower chamber.

As the temperature was constantly measured in the upper chamber of the regulator, and it did not descend lower than 15 °C, use of a different rubber for the membrane is also considered but is not investigated within the scope of this research.

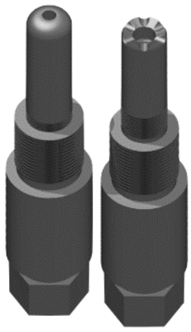

Figure 7: Inlet valve before (left) and after (right) modification

The initial volume of lower chamber had to be decreased to allow faster regulation of pressure. After all modifications, the regulator was tested with liquid nitrogen vapours as pressure medium in the upper chamber. It showed satisfying regulating capability. As it is mainly intended for use as controller of the pressure during realisation of the triple point of argon, it was primarily tested around the of 1100 mbar.

In comparison to the manual pressure regulator, membrane regulator can operate automatically once the counter pressure in the lower pressure is properly set, to keep the pressure in the cryostat at required value. As the liquid nitrogen in the cryostat is constantly evaporating, its level is changing. This influences the pressure in the cryostat and to fully automate the regulation process, constant change of pressure set-point in the lower chamber is required according to the change of conditions in the cryostat.

5. CONCLUSION

Design and operating principle of the laboratory-developed membrane pressure regulator for argon triple-point cryostat was presented. The regulator's performance was evaluated through comparison with existing ball-valve pressure regulator. The aim of the regulation are optimal thermal conditions in the cryostat for achievement and maintenance of argon triple-point state in the fixed cell. Developed pressure regulator showed to be suitable for pressure regulation of cryostat filled with liquid nitrogen and it exhibited certain improvements in comparison with manual pressure regulator in the scope of flexibility. Still further research is required to fully automate the regulation process. Future work will include further improvements of the regulator and dynamic regulation of the pressure in the lower chamber to address the changes in the cryostat and upper chamber.

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