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Refrigeration and Cryogenics

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Jarosław Poliński

CRYOGENIC ENGINEERING LABORATORY HANDBOOK

Wrocław 2011

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Laboratory 1

Subject: Introduction to cryogenics – safety handling cryogens

Introduction

Cryogenics – the science connected with reaching and applying temperatures below 120K (-153°C). Cryogenic engineering is the application of low temperatures that cannot be observed on Earth or in the atmosphere around earth under natural conditions to practical problems. The cryogenic temperature range is characterized principally by six fluids: methane, oxygen, nitrogen, neon, hydrogen and helium. The characteristics of the most widely used cryogenic liquids are collected in Table 1. Table 1 gives the normal (1 bar) boiling temperature T_N, the critical temperature T_C and pressure p_C, the temperature T_3 and pressure p_3 at triple point and the volume ratio V_V/V_L describing the increase in the fluid volume due to the process of vaporization and heating to the atmospheric temperature.

Table 1: Selected Properties of Cryogenics Liquids

	T_N	T_C	p_C	T_3	p_3	V_V/V_L
	K	K	MPa	K	kPa	-
Methane	111.6	190.7	4.63	88.7	10.1	590
Oxygen	90.2	154.6	5.04	54.4	0.15	797
Nitrogen	77.3	126.2	3.39	63.2	12.53	646
Neon	27.1	44.4	2.71	24.6	43.00	1341
Hydrogen	20.3	32.9	1.29	13.8	7.04	788
Helium	4.2	5.2	0.227	---	---	701

During Cryogenic Laboratory classes liquid nitrogen will be used so it is important to describe it more specifically.

Nitrogen has two stable isotopes of mass number 14 and 15. Their proportion in air is 10000:37. Liquid nitrogen is a clear and colorless fluid. Solid nitrogen "nitrogen ice" sinks in the liquid because the density of the ice is higher than liquid N_2. Nitrogen is the major component of air (78% by volume or 75.45% by weight). Nitrogen is inert and a non-toxic cryogen. Nevertheless, it must be handled with care because it can cause cold-burns or oxygen deficiency (high value of the volume ratio V_V/V_L). Nitrogen has a potential safety hazard in that a bare (non-insulated) pipe of liquid N_2 at 77K will condense an air mixture containing approximately 50% liquid oxygen. An oxygen enriched mixture can spontaneously explode. Several explosions and deaths have been attributed to the phenomenon of oxygen enrichment of the atmosphere in the presence of liquid nitrogen cooled surfaces [1,2].

General safety requirements

Common handling of liquid nitrogen involves transferring the cryogen from one storage container to another. This activity can be hazardous if proper precautions are not taken.

To prevent cold damage to living tissue, it is necessary to prevent contact of the tissue with either cold fluids or cold equipment. During your work with cryogens protective clothing must be worn. This includes protective glasses (the eyes are especially sensitive to the cold damage), loose-fitting gloves (can be easily removed) and lab coat or cryogenic apron. If a lab coat or cryogenic apron is not worn, long-sleeve shirts must to be worn outside of the pants. The gloves should also not be fitted with gauntlets to avoid liquid accumulation.

To prevent liquid nitrogen penetration, long trousers, without cuffs, should be worn outside the shoes. It is not allowed to wear open or porous shoes.

Figure 1. The technician fills Dewar with liquid nitrogen

Topics to prepare before laboratory class

1. The properties of methane, oxygen, hydrogen and helium.
2. Leidenfrost Effect.

Aim and purpose of the laboratory

To find the basic rules concerning the safe handling of liquid gases. The study of the influence of low-temperatures on the different material properties.

Test stand

To carry out the laboratory the following equipment is needed:

1. Insulated vessel – assignment 1a, 2a-d
2. Open container – assignment 1b
3. Non-insulated vessel – assignment 1c (loose cover is necessary) and c (vessel stand is needed)

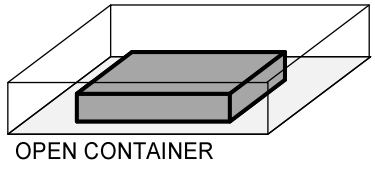


Figure 2a. The test stand for Leidenfrost Effect visualization

Figure 2b. Pressure increase inside the vessel filled with liquid nitrogen

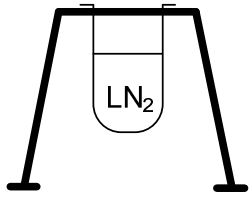


Figure 2c. Condensation of the air components on the cold surface

The materials needed:
Liquid nitrogen, balloons, rose, piece of refrigeration insulation (foam), magnet, two tennis balls, ping-pong ball.

Do not forget about protective glasses and gloves!

Assignments

1. The observation of liquid nitrogen properties:
   a) The volume change of the balloon in liquid nitrogen.
   b) The visualization of the Leidenfrost Effect.
   c) The increase in the pressure inside the closed vessel filled with liquid nitrogen.
   d) The condensation of the air components on the non-insulation surface of the vessel with liquid nitrogen, magnet is needed.
   e) The rotation of the cooled ping-pong ball.
   The ping-pong ball should be pricked as it is show in Figure 4.

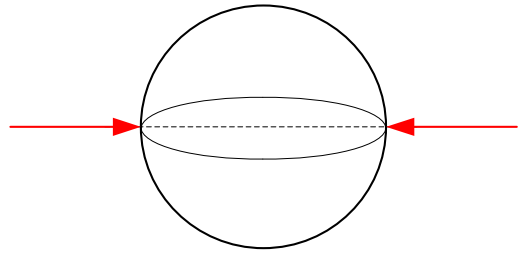


Figure 4. Ping-pong ball.

Put the ping-pong inside liquid nitrogen (attention: use protective glasses and gloves) and wait circa 3 minutes. Then pull it out and put it on the desk. What has happened? Explain it.

2. The property change caused by low-temperature:
   a) The fragility change of the rose petals after immersion in liquid nitrogen.
   b) The fragility change of the refrigeration insulation after immersion in liquid nitrogen.
   c) The property change of the tennis ball after immersion in liquid nitrogen.
   d) The property change of the lead bell after immersion in liquid nitrogen.
   e) The visualization of thermal expansion difference in low temperature – Stankowski's thermometer

Describe your observations. Where can the properties of cryogenic gases be used?

Questions and problems

1. The definition of cryogenics.
2. The definition of the critical T_C and normal boiling temperature T_N (determine the value of T_C ant T_N for oxygen, nitrogen, hydrogen and helium)
3. Explain the risk potential for a high value of the cryogen volume ratio V_V/V_L.
4. Describe and explain the Leidenfrost Effect.

Literature

1. K.D. Timmerhaus, T.M. Flynn, Cryogenic Process Engineering, Plenum Press, 1989
2. A.M. Arkharov, I.V. Marfenina, Ye.I. Mikulin, Cryogenic Systems, Bauman Moscow State Technical University Press, 2000
3. F.J. Edeskuty, W.F. Stewart, Safety in the Handling of Cryogenic Fluids, Plenum Press, 1996

Laboratory 2

Subject: Oxygen Deficiency Hazard

Introduction

Hazards in the handling of cryogenic fluids can arise from both low temperature and great expansion during the evaporation process. Most of the commonly used cryogenic fluids are not toxic.

According to Table 1 (see Laboratory 1) a huge amount of gas is released during the evaporation process of cryogens. An additional expansion occurs upon the gas warming to ambient temperature. There is some variation from one cryogen to the next in the actual volume ratios to be expected. A factor of 1000 is frequently used as the ratio of the volume of the gas formed at ambient temperature and atmospheric pressure to the volume of the same mass of cryogen as a liquid. The spillage of a large quantity of a cryogen in a confined space can cause a decrease in oxygen concentration and in consequence the creation an atmosphere that does not support life.

In any closed space where liquid cryogens are used or stored it is necessary to determine the maximum quantity of liquid that can be released under any operation to estimate the maximum decrease of the oxygen in the room that could occur as a consequence of that release. This kind of calculation is required to set a limit on the quantity of liquid cryogen in a specific space and also it can be taken as an initial data for safety analysis. Some safety precautions should be taken into consideration, such as limiting the access of personnel, oxygen monitoring or forced ventilation of the room. For example, the instantaneous spill of a Dewar of liquid nitrogen (160 liter) in a laboratory with dimensions of 5m by 7m by 3m high would produce sufficient ambient-temperature gas to completely replace the entire room atmosphere and create a totally inert and lethal atmosphere, example described in [1] (see page 12). Table 1 shows the basic symptoms of oxygen deficiency.

Table 1. Symptoms of oxygen deficiency [1]

% oxygen at 1 atm pressure	Symptoms
15 – 19	Decrease in ability to perform tasks May induce early symptoms in persons with heart, lung or circulatory problems
12 – 15	Respiration deeper, pulse faster, poor coordination
10 – 12	Giddiness, poor judgment, lips slightly blue
8 – 10	Nausea, vomiting, unconsciousness, ashen face, fainting, mental failure
6 – 8	Death in 8 min
4	Coma in 40 sec, convulsions, respiration ceases, death

Gas concentration percentages are in a volume or mole basis.

Based on [2], the goal of ODH risk assessment is to estimate the rate at which fatalities will occur as a result of exposure to reduced-oxygen atmosphere.
Since the level of risk is directly related to the nature of the operation, the excess fatality rate must be determined on an operation-by-operation basis. For a given operation, several events may cause an oxygen deficiency. Each event has an expected rate of occurrence and each occurrence has an expected probability of fatality. The ODH fatality rate is defined as

$$\phi = \sum_{i=1}^{\infty} P_i \cdot F_i \tag{2.1}$$

where

ϕ - ODH fatality rate, h^{-1}

P_i – expected rate of the i-type of event, h^{-1}

F_i – fatality factor for the i-type event, -.

The summation must include all types of events that may cause ODH and result in a fatality.

Once the ODH fatality rate ϕ has been determined, the operation can be assigned the ODH classification according to the criteria outlined in Table 2.

Table 2. Oxygen Deficiency Hazard Classification

ODH Class	ϕ, h^{-1}
0	$< 10^{-7}$
1	$> 10^{-7} < 10^{-5}$
2	$> 10^{-5} < 10^{-3}$
3	$> 10^{-3} < 10^{-1}$
4	$> 10^{-1}$

Topics to prepare before laboratory class

1. The calculations of the mass, volume and mole concentration.
2. ODH Risk Assessment Procedure [2].

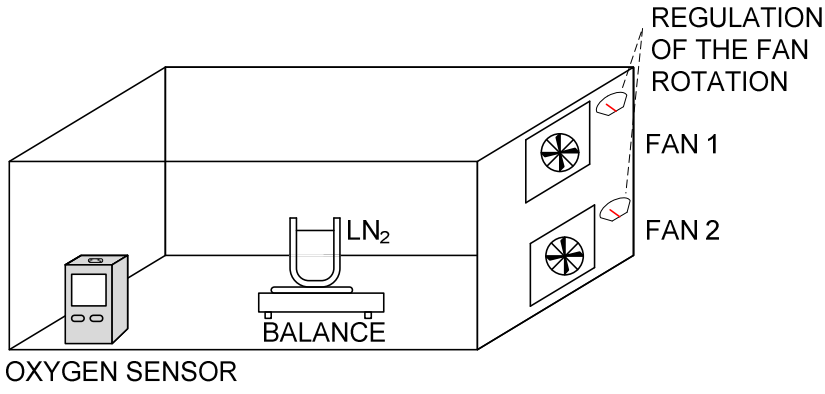
Aim and purpose of the laboratory

The estimation of the oxygen decrease caused by liquid cryogen release inside a closed space.

Test stand

The test stand is shown in Figure 1. There is a clear (transparent) tank equipped with two fans (1 – blast of air, 2 – exhaust air) with the rotation regulation. There is also an oxygen sensor, an open nitrogen vessel and a balance inside the tank.

To carry out the laboratory tests liquid nitrogen is needed.
Do not forget about protective glasses and gloves!



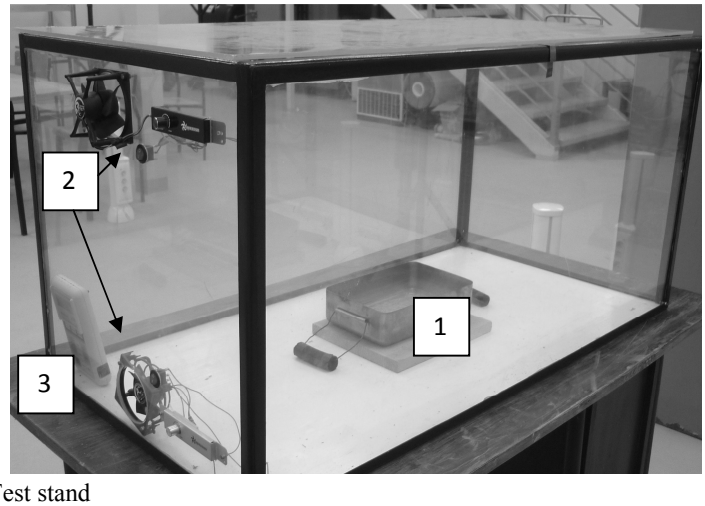


Figure 1. Test stand
1 – liquid nitrogen container, 2 – fans, 3 – oxygen sensor

Assignments

Oxygen level measurements in case of:

1. Natural ventilation (both fans are switched off)
2. Forced ventilation – exhaust (fan 1 is switched off, fan 2 is switched on)
   a) Cryogen stream is lower than air stream – the regulator of fan 2 is in position 1
   b) Cryogen stream is higher than air stream – the regulator of fan 2 is in position 2
3. Forced ventilation – inlet (fan 1 is switched on, switch off fan 2), cryogen stream is higher than air stream.

Switch on the appropriate fan(s), fill the vessel with 0.2 dm^3 of liquid nitrogen and start the measurements of oxygen level inside the tank and mass of liquid nitrogen – measure each 5 sec. for 5 minutes. Repeat the measurements for all methods of ventilation (see above). Describe your observations. Collected data (oxygen level and mass of LN_2) should be

presented in the form of a graph. Using the equations (2.1), (2.2) and (2.3) – see **Introduction,** calculate the theoretical oxygen level at the measurement points. Compare them with the test data.

The oxygen level equations for different method of ventilation.

1. Natural ventilation

The change in the oxygen level in the case of natural ventilation can be calculated from equation (2.1)

$$n_{O2}(\tau) = n_{O2}\big|_{\tau=0} \cdot e^{-\frac{\dot{V}_{N2}}{V}\cdot\tau} \tag{2.1}$$

where:

$n_{O2}\big|_{\tau=0}$ is the inlet concentration of oxygen in air, -

$\dot{V}_{N2}$ - cryogen stream, m^3/s

V – volume of the room (tank), m^3

and τ is time, s.

2. Forced ventilation – exhaust

a) Cryogen stream is lower than air stream

The change in the oxygen level can be calculated using the equation (2.2)

$$n_{O2}(\tau) = \overline{n_{O2}}\left(1-\frac{\dot{V}_{N2}}{\dot{V}_{AIR}}\right) + \left[n_{O2}\big|_{\tau=0} - \overline{n_{O2}}\left(1-\frac{\dot{V}_{N2}}{\dot{V}_{AIR}}\right)\right]\cdot e^{-\left(\frac{\dot{V}_{AIR}}{V}\cdot\tau\right)} \tag{2.2}$$

where:

$\overline{n_{O2}}$ is maximum concentration of oxygen in air, -

$\dot{V}_{AIR}$ - air stream, m^3/s.

b) The cryogen stream is higher than air stream

To calculate the oxygen level change use the equation (2.1).

3. Forced ventilation – inlet, cryogen stream is higher than air stream

The decrease in the oxygen level can be determined by equation (2.3)

$$n_{O2}(\tau) = \overline{n_{O2}}\left(\frac{\dot{V}_{AIR}}{\dot{V}_{AIR}+\dot{V}_{N2}}\right) + \left[n_{O2}\big|_{\tau=0} - \overline{n_{O2}}\left(\frac{\dot{V}_{AIR}}{\dot{V}_{AIR}+\dot{V}_{N2}}\right)\right]\cdot e^{-\left(\frac{\dot{V}_{AIR}+\dot{V}_{N2}}{V}\cdot\tau\right)} \tag{2.3}$$

Questions and problems

1. List the hazards in the handling of cryogenic liquids.
2. List the symptoms of oxygen deficiency.
3. The definition of ODH.
4. Oxygen Deficiency Hazard Classification.

Literature

1. F.J. Edeskuty, W.F. Steward, Safety in the Handling of Cryogenic Fluids, Plenum Press, 1996
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Laboratory 3

Subject: Efficiency of cryogenic vessels thermal insulation

Introduction

Heat leaks are the most important problem to be considered in the design of storage and transfer systems for cryogenic liquids. The main goal of the heat instruction procedure is to select the proper thermal insulation. The insulation strategy is to minimize both radiative and convective heat transfer as well as to introduce a minimum of solid conductance media. A lot of factors have to be considered in the insulation selection process, for example: thermal effectiveness (first of all), ruggedness, convenience, volume, weight, cost, ease of manufacture and handling, etc.

The various types of insulation used in cryogenic engineering can be divided into five main categories [1]:

1. Vacuum insulation
2. Multilayer insulation
3. Powder and fibrous insulation
4. Foam insulation
5. Special purpose insulation.

Heat transfer through these various insulations can occur by several different mechanisms, but generally involves solid conduction, gas conduction convection and radiation.

Vacuum insulation

Let's consider two surfaces maintained at different temperatures. To limit the heat losses, the radiative, convective and conductive heat transfer mechanisms have to be minimized. Generally it can be said that the evacuation of the gas between the two surfaces reduces the number of gas molecule available to transport energy from the warm to the cold surface – which eliminates gaseous convection. Moreover, it can also significantly reduce conduction through the residual gas. The level of this reduction depends on the vacuum degree. In consequence, radiation from the warm to the cold surface usually represents the key mechanism by which heat is transferred through a vacuum. The effectiveness of vacuum insulation for cryogenic systems was first recognized by Sir James Dewar at the beginning of the 20th century [1].

Multilayer insulation

Minimization of all possible heat transfer mechanisms was the main motivation for research work on multilayer insulation (MLI). MLI consists of 30 – 80 layers of alternate low-emittance radiation shields separated by low-conductivity spacers (schematically shown in Figure 1). In low temperature engineering applications the radiation-reflecting shields are generally 6 μm aluminum sheets. For better strength and ease in application, a thin plastic material (polyethylene terepthalate – Mylar or polyimide – Kapton) coated on one or both sides by a thin layer of high-reflectance metal (aluminum) is often used. The spacer can be made from coarse silk or nylon, silica-fiber felt, low-density foam, or glass-fiber mat. The most common are glass-fiber spacers (Dexiglas and Tissuglas).

MLI is placed perpendicular to the flow of heat. Radiation is minimized by overlapping reflecting shields, while the spacer material decreases solid conduction between the shields. To maintain the vacuum level a getter material (activated charcoal or molecular sieve) cooled by thermal contact with the inner vessel, is used to absorb free gas molecules [1].

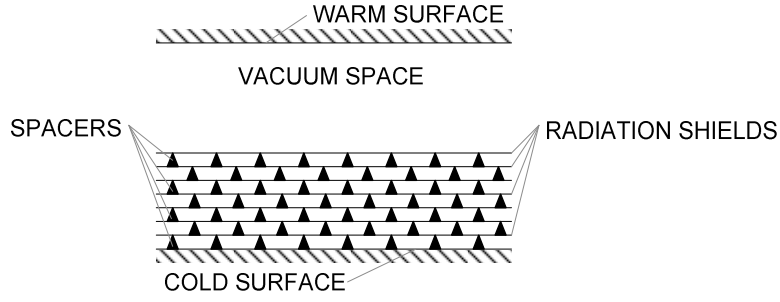


Figure 1. Structure of multilayer insulation (MLI)

Powder insulation

The main advantages of a powder insulation are low thermal conductivity and low density. The particle size distribution minimizes shock and vibration effects. There are two methods of powder application: evacuated (gas reduced) and non-evacuated (gas filled).

The mechanism of heat transfer in evacuated powders is caused by the particle size of the material. Small particle limits the gaseous heat transport to free molecular conduction. In addition, heat transfer by the residual gas can be decreased by lower vapor pressure of the powder material.

Gas-filled powders (non-evacuated insulation) reduce or eliminate the convective heat transfer due to the small gas voids within the material. Solid particulates reduce the radiation and gas conduction thus solid conduction and gas conduction through the voids serve as the predominant heat transfer mechanisms. Carbon dioxide has been widely used as a fill-gas. Around the insulation material the vapor barrier is needed to prevent diffusion of water and air [1].

Foam insulation

Cryogenic foam insulations are produced by gaseous (carbon dioxide or freon) expansion of organic or inorganic solids (polystyrene or polyurethane). The low-density material with many voids is created by this solid-gas mixture. The dominant heat transport mechanism is conduction through the interstitial gas despite the cellular structure of foam which provides a continuous path by heat conduction through the material [1].

Figure 2 shows the comparison of thermal conductivity coefficient for foam, powder, MLI, and vacuum insulation.

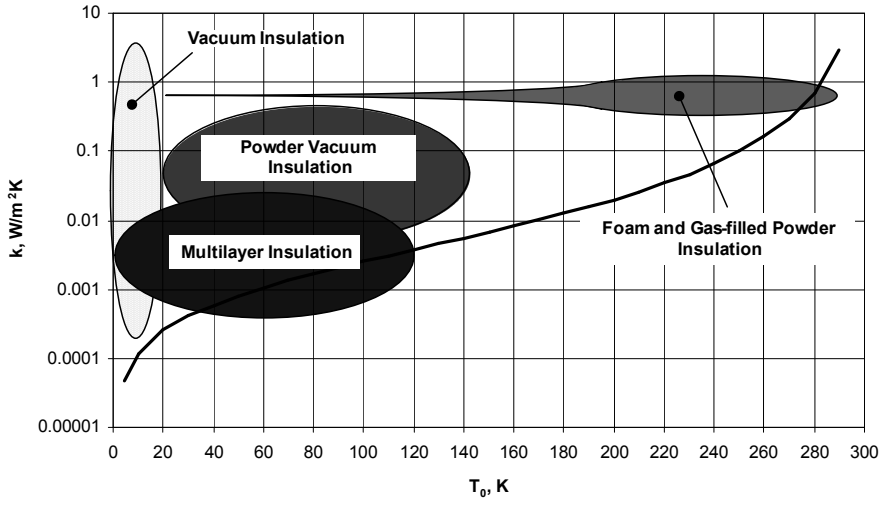


Figure 2. The classification of cryogenic thermal insulations

Topics to prepare before laboratory class

1. Fibrous insulation [1].
2. Special purpose insulation [1].
3. Dewar vessel.
4. Basic equations for heat transfer calculations.

Aim and purpose of the laboratory

The estimation of the heat transfer through the cryogenic insulation and calculation of the maximum time for liquid storage inside the vessel.

Test stand

The test stand contains a balance, stopwatch and 5 vessels:

1. Non-insulated vessel;
2. Vessel insulated with polystyrene foam;
3. Cryogenic insulated vessel – commercially available vacuum bottle;
4. Two Dewar vessels.

Do not forget about protective glasses and gloves!

Assignments

To estimate the heat transfer through insulation the measurement of LN_2 mass decrease in time is needed.

1. Put vessel 1 on the balance (tare!). Fill it with 200g of liquid nitrogen and start to measure nitrogen mass, measure each 1 minute for 15 minutes.
2. Repeat the measurements for vessel 2 and 3. The test procedure – see point 1.
3. Fill a small Dewar with 5 ml of LN_2 – measure each 10 sec for 3 minutes.

4. Weigh an empty Dewar (big one) and fill it with 8kg of LN_2. After 24 h come back to the laboratory and weigh it again.

The collected data should be presented in the form of graph. Using the equation (3.1) determine the heat transfer through the insulation and the maximum period of time for liquid nitrogen storage. How can we calculate the heat transfer coefficient of each vessel (all dimensions of vessels are needed)? Compare these values.

The equation required for heat transfer estimation.
The heat transported to the liquid can be calculated with equation (3.1)

$$\dot{Q}_0 = \frac{\Delta m_{N_2} \cdot r}{\tau} \tag{3.1}$$

where:
Δm_{N2} – mass difference between previous and next measurement, kg
r – heat of vaporization, kJ/kg (N_2 at normal boiling point has a heat of vaporization of 198.3 kJ/kg)
τ - time, s.

Questions and problems

1. List all types of insulation used in cryogenic engineering.
2. Describe vacuum insulation.
3. Structure and material of MLI.
4. Describe the heat transfer mechanisms in powder and foam insulation.
5. Construction of a Dewar vessel.

Literature

1. K.D. Timmerhaus, T.M. Flynn, Cryogenic Process Engineering, Plenum Press, 1989
2. A.M. Arkharov, I.V. Marfenina, Ye.I. Mikulin, Cryogenic Systems, Bauman Moscow State Technical University Press, 2000

Laboratory 4

Subject: Heat transfer to the cryogen in transfer line

Introduction

See Introduction to Laboratory 3: Estimation of heat transfer through the insulation – vessels.

Topics to prepare before laboratory class

1. Vacuum pump used in cryogenic engineering.

Aim and purpose of the laboratory

The estimation of heat transport through the multilayer insulation (MLI) to the cryogenic transfer line.

Test stand

The test stand is shown in Figures 1 and 2.

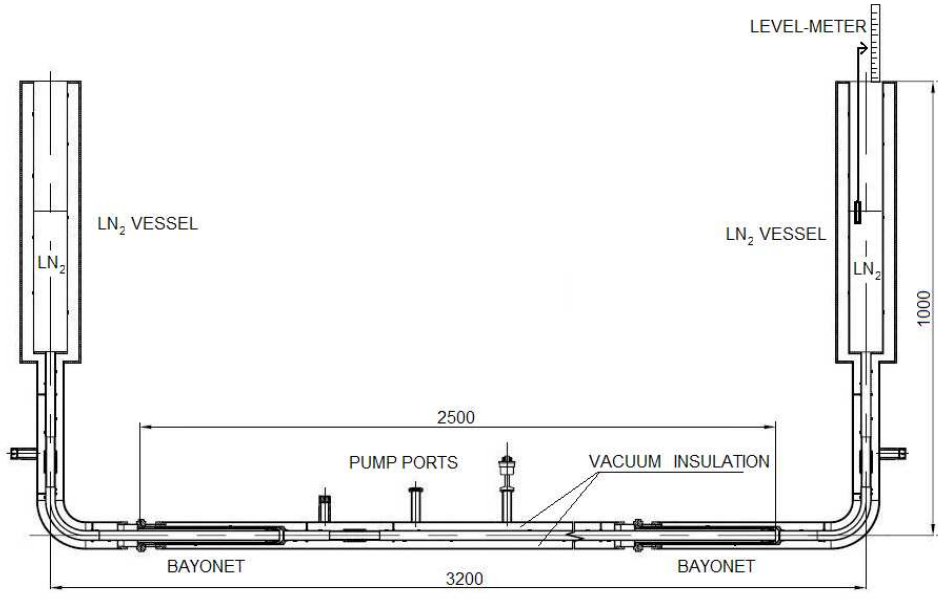


Figure 1. Test stand for estimation of heat transport to the cryogenic transfer line.

The test stand consists of a section of MLI insulated pipeline (internal diameter of 220 mm, 30 layers of MLI) which can be filled with liquid nitrogen. The level-meter is located inside the LN_2 vessel. The vacuum space of the pipeline is directly connected to the vacuum pump and level of the vacuum can be changed.

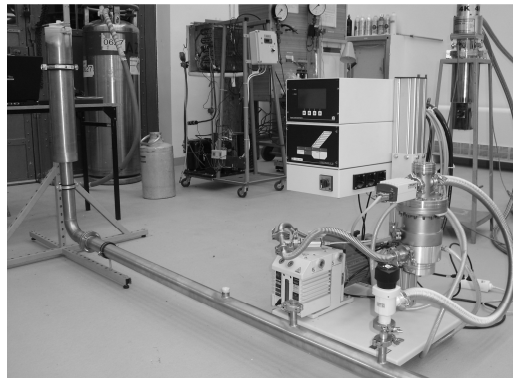



Figure 2. Test stand

The dimensions of the bayonet connector is show in Figure 3.

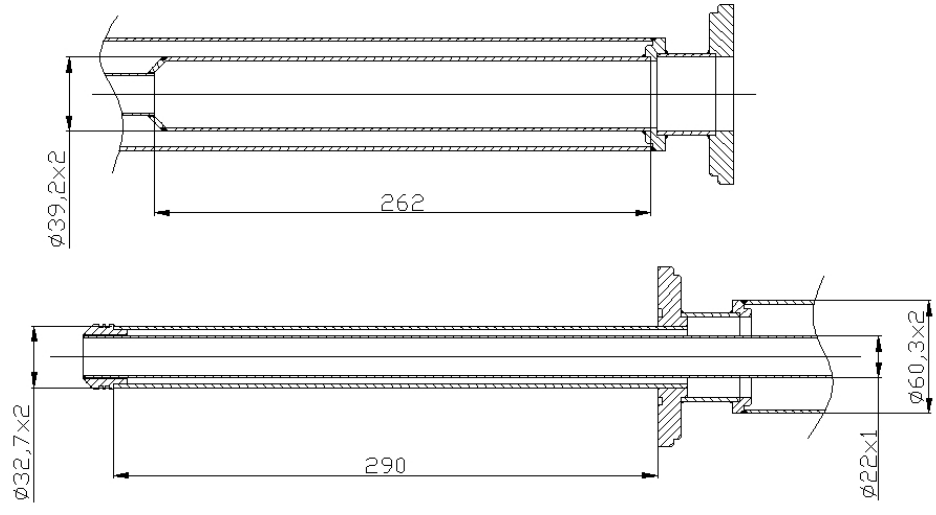


Figure 3. Dimensions of the bayonet connector

Do not forget about protective glasses and gloves!

Assignments

To estimate the heat transfer through the insulation the measurement of LN_2 volume decrease in time is needed. Fill the line with liquid nitrogen and wait a couple of minutes for the line to cool down. Then refill the line to the demanded level. Start to measure the liquid level of nitrogen. Measure each 30 sec for 30 minutes. Repeat the measurement for different vacuum levels. Based on the experimental data and equation (4.1) calculate total heat transferred to the pipeline. Theoretical value of heat transfer can be calculated from equation (4.2). The heat flux by radiation is about 1.3 W/m^2 while the heat flux by residual gas is presented in the form of graph, see Figure 4. Compare theoretical and experimental values of heat transfer.

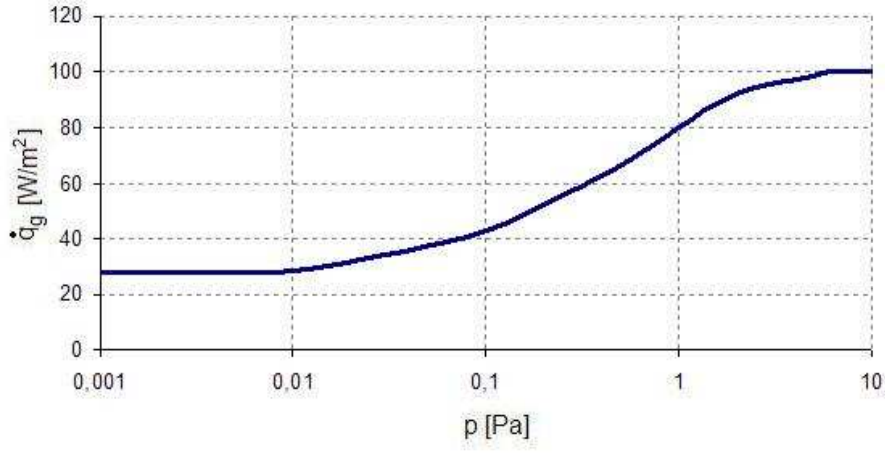


Figure 4. Heat flux by the residual gas as a function of pressure

The equations required for heat transfer estimation.
Experimental value of heat transferred to the pipeline:

$$\dot{Q}_0 = \frac{\Delta m_{N2} \cdot r}{\tau} \tag{4.1}$$

where:
Δm_{N2} – mass difference between the previous and the next measurement, kg
r – heat of vaporization, kJ/kg
(N_2 at normal boiling point has a heat of vaporization of 198.3 kJ/kg)
τ - time, s.

Theoretical value of heat transfer:

$$\dot{Q}_T = q_g \cdot A_1 + q_r \cdot A_2 + \sum_1^i \frac{A_i \cdot \lambda \cdot (T_2 - T_1)}{l_i} + \dot{Q}_V \tag{4.2}$$

where:
q_g – heat flux by the residual gas, W/m²
A_1 – mean heat transfer surface, m²
q_r – heat flux by radiation , W/m², (1.3 W/m²)
A_2 – radiation surface, m²
A_i – cross section surface, m²
λ – thermal conductivity W/mK, (λ=12.4 W/mK)
T_1 – temperature of liquid nitrogen, K

T_2 – ambient temperature, K
l_i – path length of heat flow, m
$\dot{Q}_V$ - heat flow into LN_2 vessels, W, ($\dot{Q}_V$ =47 W)

Questions and problems

1. List all types of insulation used in cryogenic engineering.
2. Structure and materials of MLI.
3. Heat transfer mechanism for multilayer insulation.
4. Pressure levels for vacuum insulation.

Literature

1. K.D. Timmerhaus, T.M. Flynn, Cryogenic Process Engineering, Plenum Press, 1989
2. A.M. Arkharov, I.V. Marfenina, Ye.I. Mikulin, Cryogenic Systems, Bauman Moscow State Technical University Press, 2000

Laboratory 5

Subject: Joule-Thomson micro-liquefier

Introduction

The minimal work of any gas liquefaction can be calculated on the basis of the ideal reversible cycle depicted in Figure 1. The gas is first reversibly and isothermally pressurized on the path 1-2, and then isentropically expanded on the path 2-3.

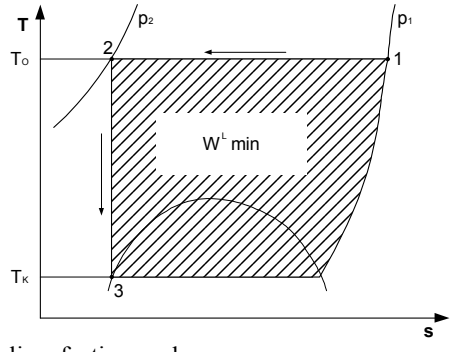


Figure 1. Scheme of the liquefaction cycle

The minimal work of liquefaction can be calculated from equation (7.3). The values calculated for basic air components are given in Table 1.

$$W_{\min}^{L} = T_o\left(s_1 - s_{3'}\right) - \left(h_1 - h_{3'}\right) \tag{7.3}$$

Table 1. Minimal work of gas liquefaction

	Minimal work of gas liquefaction	
	J/mol	kJ/kg
Nitrogen	21540	767.21
Oxygen	20310	634.68
Argon	19070	477.42

The throttle expansion cycle was the first cryogenic cycle to be practically operated. System based on Joule-Thomson expansion (process i=const) can work both as liquefier and refrigerator. J-T cooling system is the oldest one but it still remains in use in the original as well as modified form in cryogenic engineering.

The throttle-based system has a lot of advantages:

- simple in design = high reliability of the system in the operation
- no moving parts at low temperatures
- possibility of minimization
- can be supplied by high-pressure gas cylinder or compressor (there is no need to change the construction).

These merits can be balanced by the limitations of the throttle expansion (high irreversibility process) and low thermodynamic efficiency.
Joule-Thomson liquefier is schematically shown in Figure 2.

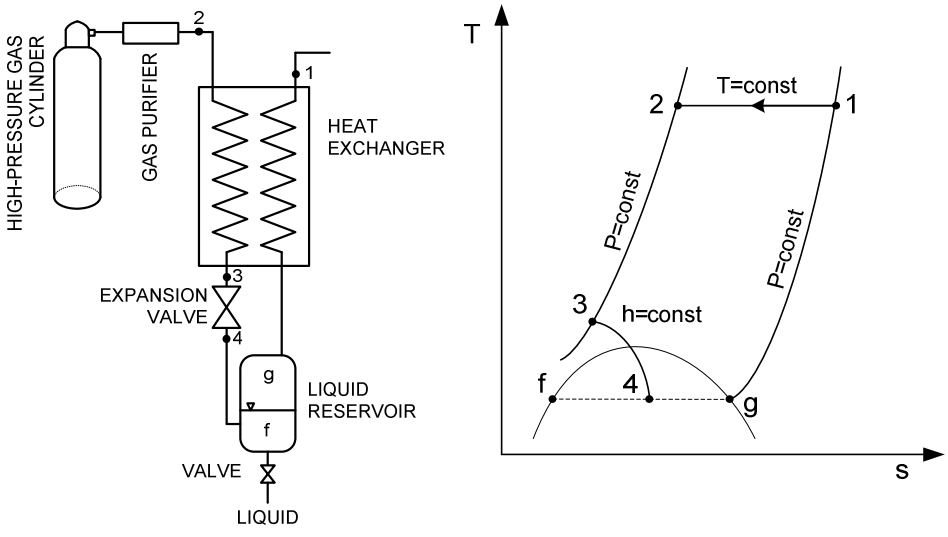


Figure 2. Joule-Thomson liquefier, thermodynamic processes on T-s diagram

The high-pressure gas at ambient temperature from the high-pressure gas cylinder (point 2) is purified and cooled before it reaches (point 3) the expansion valve (Joule-Thomson valve) in a heat exchanger by the stream rejected into the environment (process 2-3). In the expansion valve cool high-pressure gas is throttled (process 3-4) by the atmospheric pressure where it is partly liquefied. Liquid supplies the reservoir and it can be removed from the system (point f). The cold gas (point g) is returned via the heat exchanger (process g-1) to the environment.

Topics to prepare before laboratory class

1. Joule-Thomson effect, inversion temperature.
2. Real throttling cycle (see Literature 2, p.263).
3. The energy balance of the Joule-Thomson liquefier, fraction liquefied calculations (see Cryogenics – Tutorial 2).
4. Working fluid of the Joule-Thomson cycle.

Aim and purpose of the laboratory

The presentation of nitrogen liquefaction process based on the Joule-Thomson cycle. Temperature and pressure measurements to reproduce the thermodynamic processes on T-s diagram.

Test stand

The test stand is shown on Figure 2. The liquefier is supplied with nitrogen by a high-pressure gas cylinder.

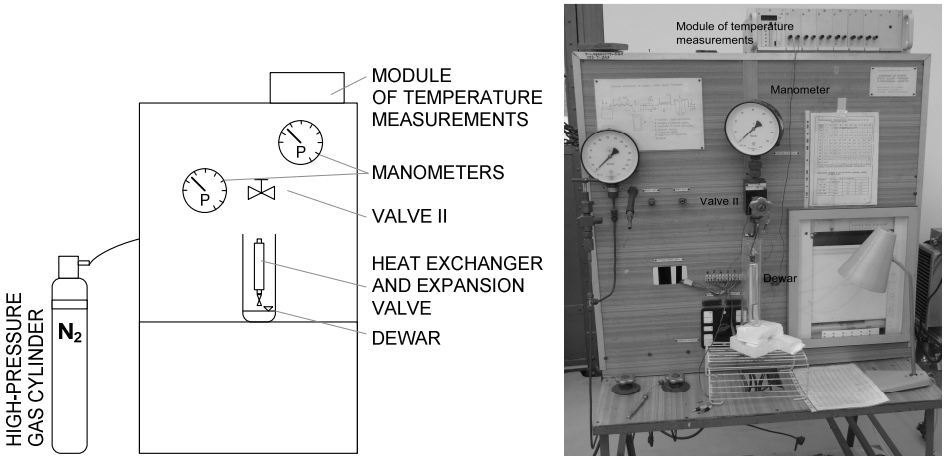


Figure 3. Test stand of Joule-Thomson liquefier

Assignments

Switch on the liquefier using valve II and start to measure the temperatures and pressures, measure each 30 sec until the liquid nitrogen appears. Determine the time needed for liquefaction of 1 dm^3 nitrogen. Temperature and pressure data should be presented on T-s diagram. Calculate the fraction liquefied and compare it with the theoretical value.

Questions and problems

1. The minimal work of liquefaction.
2. Describe the Joule-Thomson (liquefier or refrigerator) cycle – construction and thermodynamic processes.
3. Where can we use the J-T liquefier, name three examples of applications. Why can the J-T cooler be minimized?
4. Reproduce the thermodynamic processes of the J-T cycle (refrigeration of liquefaction) on a T-s diagram. Describe each process.
5. Describe the throttle process.
6. Can we use ideal gas as a working fluid in the Joule-Thomson cooler? Explain your answer.
7. Energy balance of Joule-Thomson liquefier. Derive the formula for the fraction liquefied.

Literature

1. K.D. Timmerhaus, T.M. Flynn, Cryogenic Process Engineering, Plenum Press, 1989
2. A.M. Arkharov, I.V. Marfenina, Ye.I. Mikulin, Cryogenic Systems, Bauman Moscow State Technical University Press, 2000
3. J.G. Weisend, Handbook of Cryogenic Engineering, Taylor&Francis, 1998

4. M.Chorowski, A. Piotrowska, Comparative Thermodynamic Analysis of Gas Mixture Separation and Liquefaction Methods, Proceedings of International Congress of Refrigeration, Beijing, 2007

Laboratory 6

Subject: Joule-Thomson refrigerator fed with gas mixture

Introduction

The purpose of refrigeration is to extract heat at low temperatures and reject it at ambient temperatures. According to the Second Low of Thermodynamics it can be achieved only by doing external work. The system described in Laboratory 5 can easily be converted to the refrigeration system shown on Figure 1.

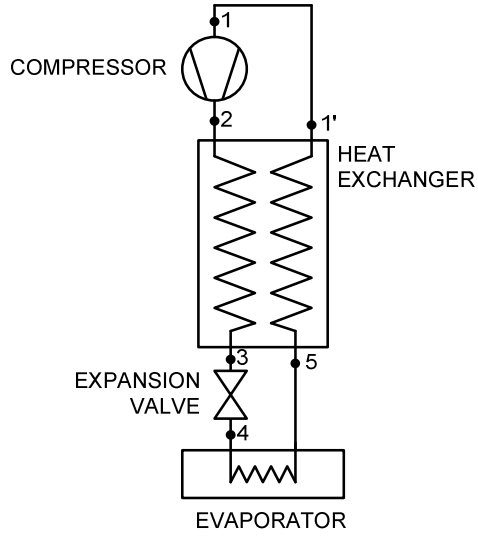


Figure 1. Joule-Thomson refrigerator

To achieve the refrigeration effect on LN_2 temperature level (liquid nitrogen in the evaporator) a high pressure of inlet gas (point 2) is needed. For nitrogen this pressure should be 10 – 20 MPa so a specialist compressor (oil-free, expensive) is necessary. This problem can be solved by using gas mixture as a working fluid. Applying the gas mixtures decreases the working pressure to the level achieved by domestic refrigeration compressors. Additionally, temperatures of streams on high- and low-pressure side are similar (see Figure 2 and Table 1), so the heat losses in the heat exchanger are limited. The system becomes easy to build and cheaper than systems supplied with pure gases. For a gas mixture to be around nitrogen temperatures, hydrocarbons (methane, ethane, propane) are usually used. Freon F13 used to be applied in the system but according to the Montreal Protocol regulations it has been forbidden. Figure 2 and Table 1 show the comparison of Joule-Thomson cycle fed with pure gas and mixture.

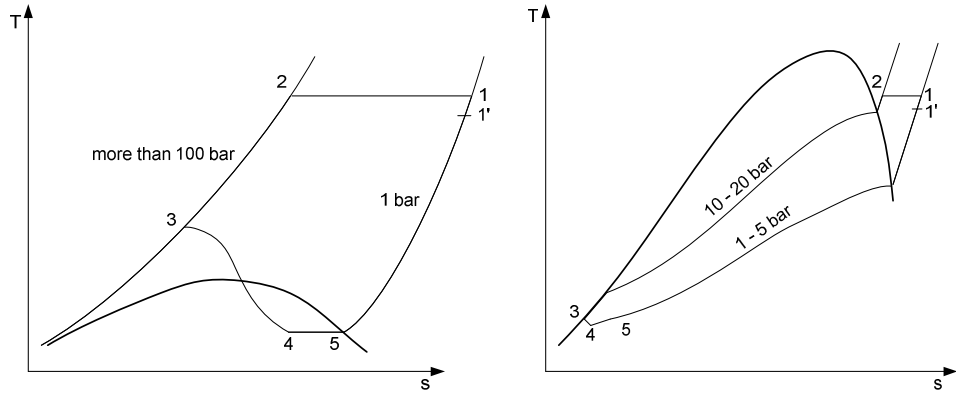


Figure 2. Thermodynamic processes for pure gas cycle on T-s diagram, thermodynamic processes for gas mixture cycle on T-s diagram

Table 1. The comparison of Joule-Thomson cycle fed with pure gas and mixture.

J-T cooler fed with:	Pure nitrogen	Gas mixture
Working pressure	100 – 200 bar	10 – 20 bar
Boiling point of working fluid	Constant 78 K	Changeable 80 – 120 K
Phase transition inside the heat exchanger	NO	YES
Temperature difference on the cold end of heat exchanger	70 -90 K	5 – 15 K

Topics to prepare before laboratory

1. Joule-Thomson effect.
2. Properties of the gas mixture.
3. The calculation procedure for gas mixtures (phase equilibrium, phase composition, bubble, dew points, etc.).

Aim and purpose of the laboratory class

The comparison of the working parameters for Joule-Thomson system supplied with pure nitrogen and gas mixture (nitrogen-based).

Test stand

Test stand including test points of Joule-Thomson refrigerator is shown on Figure 3.

The test stand consists of a domestic-refrigerator condensing unit equipped with an oil separator, a tube in tube heat exchanger and needle valve. The system can be supplied with pure N_2 or gas mixtures. The test stand is equipped with pressure and temperature sensors (points 1-6, shown in Figure 6).

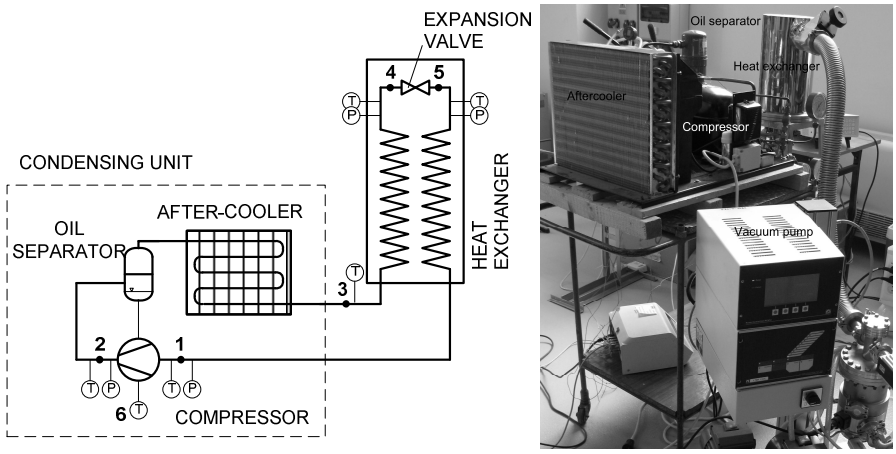


Figure 3. Test stand of J-T refrigerator

Assignments

Firstly the system is supplied with 200g of pure nitrogen. Measure the temperature and pressure at points 1 to 6. The test should last for maximum 15 minutes. Then the system will be filled with a mixture of 200g of nitrogen, ethane and propane. Repeat the measurements (for at least 45 minutes). Based on the experimental data show the thermodynamic processes on a T-s diagram (both for nitrogen and mixture systems), calculate the refrigeration effect on (temperature level achieved) and heat rejected in the after-cooler. Compare these values (for N_2 and gas mixture).

Questions and problems

1. Thermodynamic processes of Joule-Thomson cycle (on T-s diagram).
2. Joule-Thomson effect, J-T coefficient.
3. The differences between a Joule-Thomson cooler supplied with pure gas and gas mixture.
4. The properties of the gas mixture (components, calculations, etc.).

Literature

1. K.D. Timmerhaus, T.M. Flynn, Cryogenic Process Engineering, Plenum Press, 1989
2. A.M. Arkharov, I.V. Marfenina, Ye.I. Mikulin, Cryogenic Systems, Bauman Moscow State Technical University Press, 2000
3. A. Piotrowska, M.Chorowski, Applicability of the Joule-Thomson Cryocooler Coupled with Membrane-based Purification System for Liquefaction of Natural Gas in Small Quantities, Advanced in Cryogenic Engineering, AIP Conference Proceedings, 2008
4. PROMIX

Laboratory 7

Subject: Joule-Thomson refrigerator coupled with a membrane-based air separation system

Introduction

Gas separation technologies play a key role in technical gases production, natural gas processing, helium extraction and separation, CO_2 sequestration and other processes. The presently used methods include cryogenic rectification, adsorption processes and membrane separation.

A mixture separation is a process requiring energy input. The minimal work of the ideal gas mixture separation can be calculated from Equation (7.1):

$$W_{\min}^{S} = nRT\sum_{i=1}^{z} x_i \ln\frac{1}{x_i} \tag{7.1}$$

The work input given by eq. (1) results from the necessity of the compression of each component from its partial pressure to the atmospheric pressure. The minimal work values necessary for the separation of the atmospheric air basic components are given in Table 1. The difference between the energy needed to separate nitrogen and oxygen results from the argon percentage added to the remaining gas respectively.

Table 1. Minimal work of air separation

Product	Mole fraction in air	Minimal work	
	%	J/mol of mixture	kJ/kg of product
Nitrogen	78.120	1311.6	60.0
Oxygen	20.946	1280.2	191.0
Argon	0.934	132.1	353.9

If the argon and the oxygen are not distinguished and in consequence the air is treated as a binary mixture, its separation into oxygen and nitrogen requires energy input of 1311.6J per 1 mole of the mixture. This energy is equivalent to the isothermal compression of 1 mole of the mixture treated as an ideal gas from an initial pressure of 1 bar to a final pressure of 1.96 bar, according to equation (7.2):

$$W_C = RT\ln\frac{p_2}{p_1} \tag{7.2}$$

It means that it is not thermodynamically possible to invent air separation technology, based on initial air compression and requiring the inlet gas pressure to be lower than 1.96 bar.

Membrane air separation

Polymeric membrane air separation processes are based on the difference in rates of diffusion of the separated gases through the membrane. A membrane module operates between high and low pressure of the process streams. The material of the fibers depends on the gases separated. The semi-permeable fibers supported on a non-selective membrane sheath are assembled into cylindrical modules connected in parallel or in series to provide the required production capacity. A typical membrane air separation system aimed at nitrogen generation is shown in Figure 1.

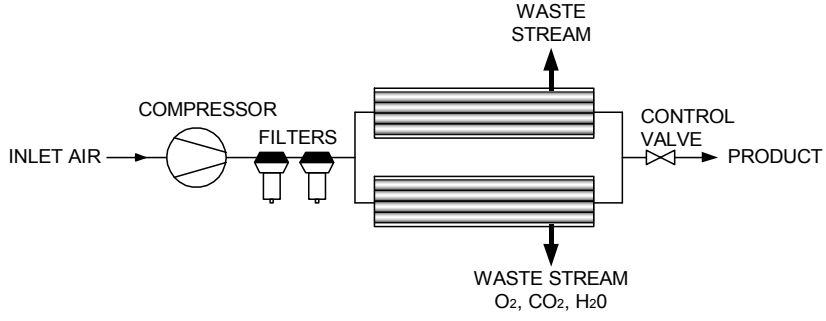


Figure 1. Polymeric membrane air separation system

Compressed air is cleaned and purified in a series of the filters and passes to the hollow fiber type membrane module. In the module oxygen, carbon dioxide and residual molecules of the water vapor permeate through the fibers and a lower pressure waste stream is rejected into the environment. The fiber material creates a barrier for the nitrogen molecules, therefore the high-pressure nitrogen leaves the module and can be further processed. The volumetric purity of the nitrogen is typically 95 – 98%. The presently available membranes require an air compression ratio of 10 – 30. The corresponding energy consumption, taking into account the isentropic efficiency of the compressors of the order of about 70%, is 6.3 to 17.1 times higher than the minimal work given by equation (7.1).

Topics to prepare before laboratory

1. Joule-Thomson cycle.
2. Mechanisms of transport in membranes (process of bulk flow, diffusion and solution-diffusion) [2], page 725.

Aim and purpose of the laboratory class

Technical operation of small capacity liquid nitrogen generator based on Joule-Thomson refrigerator coupled with air separation membrane

Test stand

A scheme of combined nitrogen separation and liquefaction system is shown in Figure 2. The device is characterized by two separate flow processes: air/nitrogen open cycle flow,

and the refrigerant closed cycle flow in the Joule-Thomson refrigerator. Both processes are thermally coupled by the recuperative heat exchanger and the evaporator/condenser.
The purified and compressed air flows into the membrane module, where the nitrogen is separated from O_2, CO_2 and H_2O. The high-pressure nitrogen passes through the heat exchangers where it is cooled down and liquefied. A cooling power enabling nitrogen pre-cooling and liquefaction in the heat exchangers is generated in a closed loop Joule-Thomson liquefier (see Laboratory 6) fed with a nitrogen-hydrocarbons gas mixture. Liquid nitrogen is throttled on the Joule-Thomson valve and it is partly flashed. The liquid nitrogen under atmospheric pressure can be removed and transferred to the external dewar.

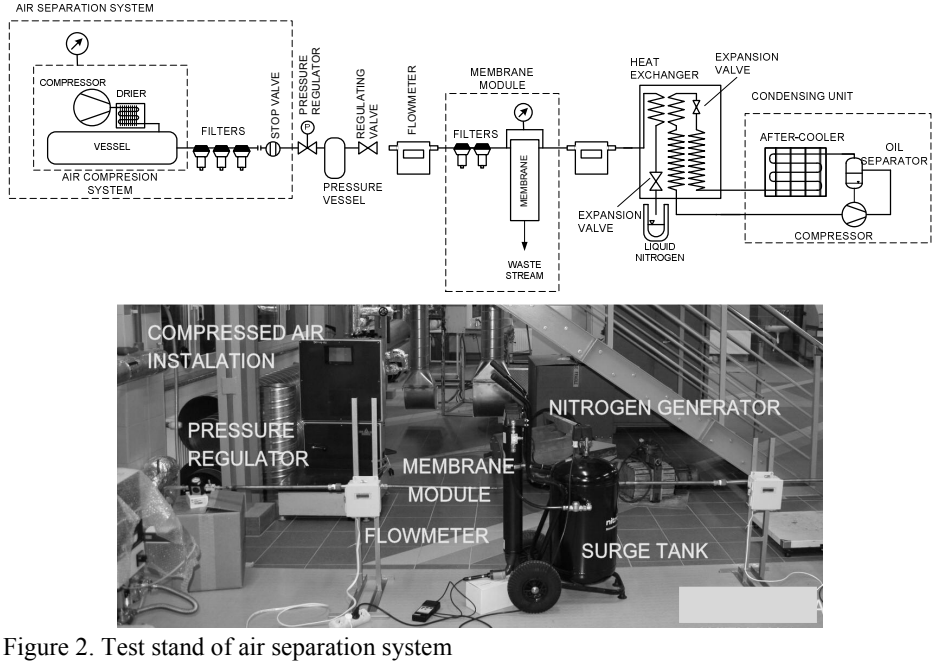


Figure 2. Test stand of air separation system

Assignments
Start the air separation system. Measure the stream (inlet-air, waste and product) composition (O_2 and CO_2 level) for three inlet pressure levels and three different mass flows of air. Compare the data and propose the method for a high purity outlet stream production. Calculate the cooling power needed for the product liquefaction. Cooling power will be produced in a J-T system working with 200 g of gas mixture (nitrogen, ethane and propane) – same as for Laboratory 6. Measure the temperatures and pressures at characteristic points. Based on the experimental data show the thermodynamic processes on a T-s diagram.

Questions and problems
1. Ideal process of the gas mixture separation.
2. Membrane-based gas separation technology.

3. Solution-diffusion process of gas transport through a dense membrane.

Literature

1. K.D. Timmerhaus, T.M. Flynn, Cryogenic Process Engineering, Plenum Press, 1989
2. J.D. Seader, E.J. Henley, Separation Process Principles, Wiley & Sons, 1998
3. M.Chorowski, A. Piotrowska, Comparative Thermodynamic Analysis of Gas Mixture Separation and Liquefaction Methods, Proceedings of International Congress of Refrigeration, Beijing, 2007

Subject: Dynamic and static characteristics of Gifford-McMahon cryocooler

Introduction

In 1959 W.E. Gifford and H.O. McMahon invented an original system with non-equilibrium expansion of the working fluid. The refrigeration effect is produced by free expansion (exhaust) process. The Figure 1 shows schematically refrigerator and working process in the T-s diagram.

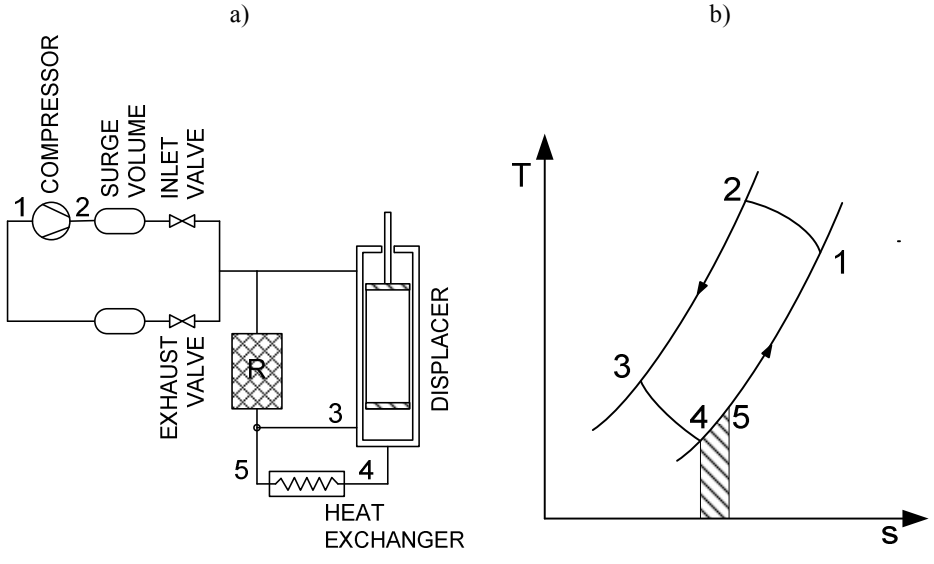


Figure 1. Gifford-McMahon

a) Flow diagram
b) Working processes in the T-s diagram

Process 1-2: the displacer is initially at the bottom of the cylinder, the inlet valve is opened and the high-pressure gas flows to the regenerator.

Process 2-3: the displacer is raised to the top of the cylinder, this moves the gas originally in the upper expansion space by means of the three-way valve into the lower expansion space. Since the volume of the gas decreases as it is cooled through the regenerator, the inlet valve remains open to maintain constant pressure throughout the system.

Process 3-4: the gas within the lower expansion space is allowed to expand to the initial system pressure by closing the inlet valve, redirecting the three-way valve and opening the exhaust valve. During the expanion the temperature of the gas inside the bottom space of the cylinder decreases.

Process 4-5: the displacer moves downward, forcing the remaining gas out of the bottom of the cylinder and through a heat exchanger where the gas absorbs the heat.

Process 5-1: the gas is warmed back to near room temperature by sending it back through the regenerator.

Topics to prepare before laboratory

1. The applications of Gifford-McMahon refrigerator.

Aim and purpose of the laboratory class

The determination of for Gifford – Mc Mahon refrigerator performance characteristic.

Test stand

Gifford-McMahon refrigerator system is shown on Figure 2.

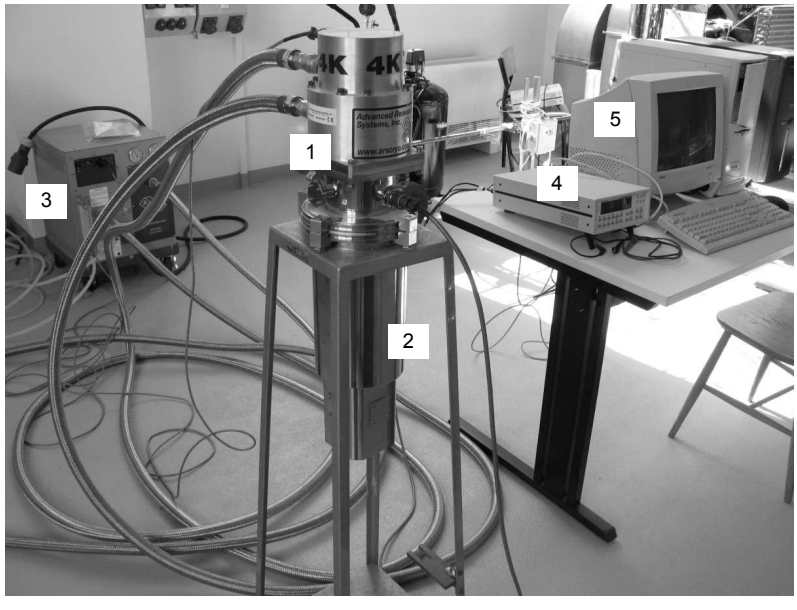


Figure 2. Gifford-McMahon refrigerator performance test stand: 1 – two stage Gifford-McMahon cryocooler, 2 – vacuum vessel, 3 – helium compressor, 4 – cryocooler coldhead temperature controler, 5 – DAQ system

The major components of the closed cycle cryostat are the expander, compressor, vacuum vessel, and radiation shield. The expander (Figure 3) is where the Gifford-McMahon refrigeration cycle takes pace. It is connected to a compressor by two gas lines and an electrical power cable. One of the gas lines supplies high pressure helium gas to the expander, the other gas line returns low pressure helium gas from the expander. The compressor provides the necessary helium gas flow rate at the high and low pressure for the expander to convert into the desired refrigeration capacity. An insulation vacuum (10^{-5}mbar) created in the vacuum vessel vacuum surrounds the cold parts of the expander what limiting the heat load on the expander caused by conduction and convection. The

radiation shield is actively cooled by the first stage of the expander and insulates the second stage (coldhead) from the room temperature thermal radiation being emitted from the vacuum vessel.

Figure 3. Wiev of 2-stages Gifford-McMahon expander (cryocooler). Photo www.arscryo.com

The closed cycle cryocoolers operate on a pneumatically driven Gifford-McMahon refrigeration cycle. The pneumatically driven GM uses an internal pressure differential to move the displacer instead of a mechanical piston, which results in smaller vibrations.
The refrigeration cycle of the closed cycle cryostat starts with the rotation of the valve disk opening the high pressure path allowing the high pressure helium gas to pass through the regenerating material into the expansion space. Second, the pressure differential drives the piston "up" allowing the gas at the bottom to expand and cool. Third the rotation of the valve disk opens the low pressure path allowing the cold gas to flow through the regenerating material removing heat from the system. Finally the pressure differential returns the displacer to its original position completing the cycle [3].

The coldhead temperature is measured with silicon diode DT-670 type temperature sensor. On the coldhead a 55 Ohms electrical heater is installed. The coldhead temperature is regulated by a Lake Shore type 331S Temperature Controller cooperated with the temperature sensor and the electrical heater. A maximum power dissipated by this control

loop on the coldhead is 55 W. The Controller is connected to a DAQ system allowing recording the actual coldhead temperature and dissipating power.

Assignments

1. Create the insulation vacuum in the vacuum vessel
2. Start the helium compressor and cryocooler
3. Start to record the coldhead temperature with 1 minute step till coldhead reaches the minimum stable temperature
4. With the temperature controller set the highest value of the coldhead temperature Record the temperature with 1 minute step till coldhead reach the setpoint value
5. Record the power dissipation value for the setpoint temperature value
6. Repeat the points 4 and 5 for following coldhead temperature values: 5K, 10K, 20K, 30K, 50K, 77.3 and 100 K
7. Based on recorded data draw a cryocooler cooldown characteristic
8. Determine a cooldown time of the cryocooler (time after which the cryocooler coldhead reach the minimum temperature)
9. Based on recorded data draw a cryocooler performance characteristic – dissipated power – coldhead temperature characteristic

Questions and problems

1. Describe the Gifford-McMahon cycle.
2. G-M cycle uses:
   a. Joule-Thomson expansion process,
   b. Free expansion process,
   c. or isentropic expansion process? Explain it.
3. List a minimum 3 possible applications of a G-M cooler.

Literature

1. K.D. Timmerhaus, T.M. Flynn, Cryogenic Process Engineering, Plenum Press, 1989
2. A.M. Arkharov, I.V. Marfenina, Ye.I. Mikulin, Cryogenic Systems, Bauman Moscow State Technical University Press, 2000
3. Advanced Research Systems, webpage: www.arscryo.com

Laboratory 9

Subject: Chosen properties of high temperature superconductors

Introduction

A superconductor is an element or metallic alloy which, when cooled below so call critical temperature, dramatically lose all electrical resistance. In principle, superconductors can allow electrical current to flow without any energy loss.

In addition, superconductors exhibit the Meissner effect in which they cancel all magnetic flux inside, becoming perfectly diamagnetic (discovered in 1933). In this case, the magnetic field lines actually travel around the cooled superconductor.

The superconducting state is defined by three factors: critical temperature (Tc), critical field (Hc), and critical current density (Jc). Each of these parameters is very dependent on the other two properties present. Maintaining the superconducting state requires that both the magnetic field and the current density, as well as the temperature, remain below the critical values, all of which depend on the material. The phase diagram in Figure 1 demonstrates relationship between Tc, Hc, and Jc. The highest values for Hc and Jv occur at 0 K, while the highest value for Tc occurs when H and J are zero. When considering all three parameters, the plot represents a critical surface. From this surface, and moving toward the origin, the material is superconducting. Regions outside this surface the material is normal or in a lossy mixed state.

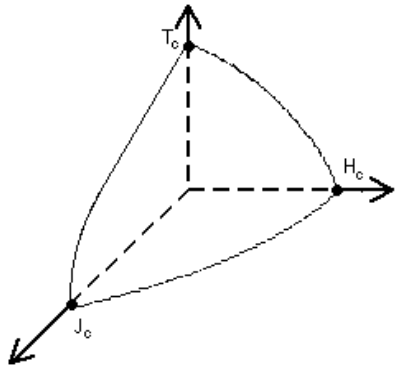


Figure 1. Critical surface superconducting phase diagram

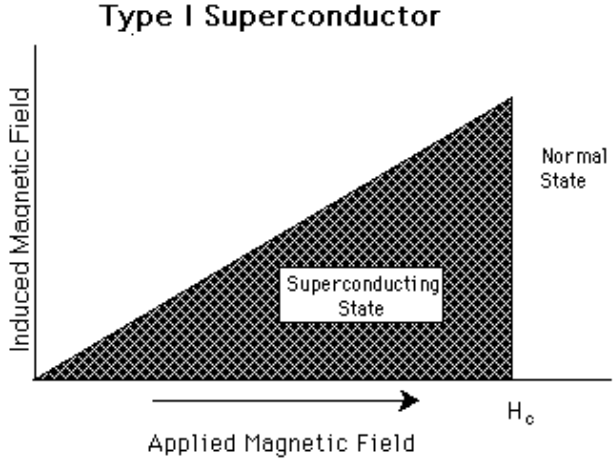
There are two types of superconductors, Type I and Type II. Very pure samples of lead, mercury, and tin are examples of Type I superconductors. High temperature ceramic superconductors such as YBa2Cu3O7 (YBCO) and Bi2CaSr2Cu2O9 are examples of Type II superconductors. Table 1 presents critical temperature Tc, magnetic field Hc and current density Jc for selected superconductors

Table 1. Critical temperature Tc, magnetic field Hc and current density Jc for selected superconductors.

	Critical temperature T_c [K]	Critical magnetic field H_c [T]	Critical current density J_c, [A/cm^2]
Type I			
Al	1.19	0.0102	
In	3.4	0.0285	
Pb	7.19	0.0803	
Sn	3.72	0.0305	
Type II			
NbTi	9.6	12.2	5 x 106
Nb_3Sn	18.1	25	5 x 107
Nb_3Ge	23.2	38	5 x 107
$Nb_3A_{l0.7}Ge_{0.3}$	20.7	44	

Figure 2 a is a graph of induced magnetic field of a Type I superconductor versus applied field. Figure 2a shows that when an external magnetic field (horizontal abscissa) is applied to a Type I superconductor the induced magnetic field (vertical ordinate) exactly cancels that applied field until there is an abrupt change from the superconducting state to the normal state. Type I superconductors are very pure metals that typically have critical fields too low for use in superconducting magnets.

a)



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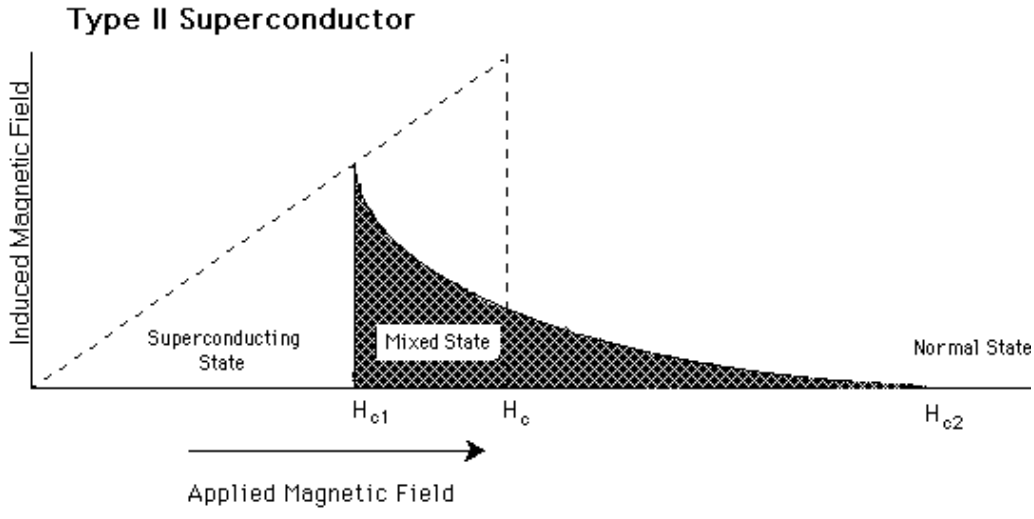

Figure 2. Applied – inducted magnetic field characteristic for superconductor: a) Type I, b) Type II

Figure 2 b shows a Type II superconductor in an increasing magnetic field. It can be notice that this graph has an Hc1 and Hc2. Below Hc1 the superconductor excludes all magnetic field lines. At field strengths between Hc1 and Hc2 the field begins to intrude into the material. When this occurs the material is in so call the mixed state, with some of the material in the normal state and part still superconducting. Type I superconductors have Hc too low to be very useful. However, Type II superconductors have much larger Hc2 values. YBCO superconductors have upper critical field values as high as 100 T.
Since there is no loss in electrical energy when superconductors carry electrical current, relatively narrow wires made of superconducting materials can be used to carry huge currents. However, there is a certain maximum, critical current that these materials can be made to carry, above which they stop being superconductors. If too much current is pushed through a superconductor, it will revert to the normal state even though it may be below its transition temperature. The value of critical current density (Jc) is a function of temperature; i.e., the colder you keep the superconductor the more current it can carry.

Meissner Effect
The Meissner effect is the expulsion of a magnetic field from a superconductor during its transition to the superconducting state and it was discovered by Walther Meissner and Robert Ochsenfeld in 1933. They measured the magnetic field distribution outside superconducting tin (Sn) and lead (Pb) samples. In the presence of an applied magnetic field, the sample was cooled down below their critical temperature (or superconducting transition temperature). Below this temperature the sample cancelled all magnetic field inside. The researcher detected this effect only indirectly because the magnetic flux is conserved by a superconductor. It means that in the case of an internal field decrease, the external field increases. This experiment demonstrated for the first time that

superconductors were more than just perfect conductors and provided a uniquely defining property of the superconducting state.
The Meissner effect occurs when the external field induces the undamped current in a superconductor surface layer. The field induced by this current compensates for the external field and prevents it from penetrating into the conductor. The superconductor became an ideal diamagnetic, see Figure 3.

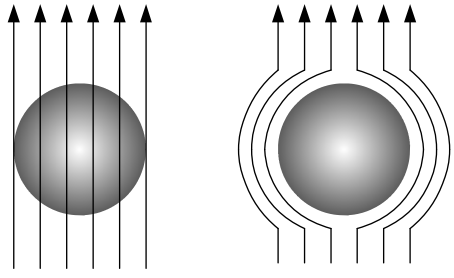

Figure 3. The Meissner effect.

Materials can be classified by their response to externally applied magnetic fields as diamagnetic, paramagnetic, or ferromagnetic. Diamagnetism is a property of all materials and opposes applied magnetic fields, but is very weak. Paramagnetism, when present, is stronger than diamagnetism and produces magnetization in the direction of the applied field, and is proportional to the applied field. Ferromagnetic effects are very large, producing magnetizations sometimes orders of magnitude greater than the applied field and as such are much larger than either diamagnetic or paramagnetic effects.

Topics to prepare before laboratory class

1. The examples of paramagnets, ferromagnetic and diamagnetic materials
2. Critical temperature of diamagnetic materials
3. p- T phase diagram for the nitrogen
4. Critical current of superconductors
5. 4-wire electrical resistance measurement method

Aim and purpose of the laboratory

Determination of critical current – temperature characteristic for Bi-2223 HTS-tape in 63.5 – 77.3 K temperature range. The observation of the Meissner Effect.

Test stand

Test stand, presented on Figure 4, consists of liquid nitrogen cryostat where a sample of Bi-2223 HTS-tape is immersed, vacuum pump, current source 0 – 70A and voltmeter.

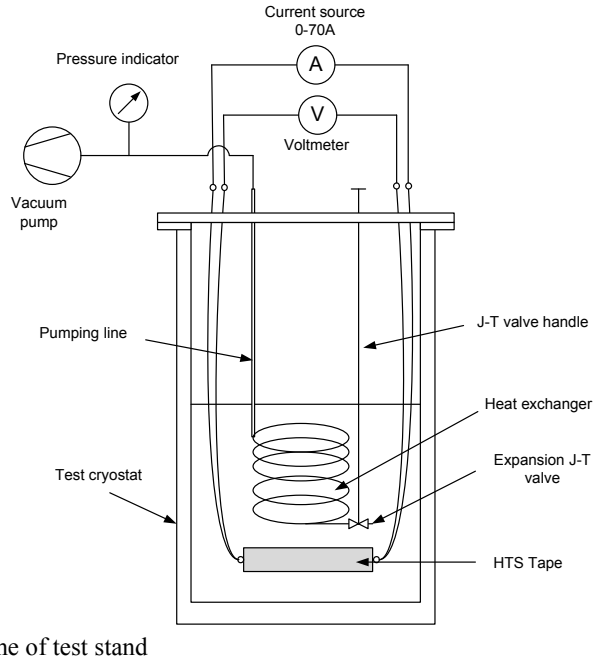

Figure 4. Scheme of test stand

The cryostat allowing regulation of the liquid nitrogen temperature between its triple point 63.15K and boiling temperature 77.3K. Cryostat is equipped with the coil heat exchanger immersed in the liquid nitrogen. At the end of the heat exchanger a small J-T valve is installed. The J-T valve opening can be manually regulated from outside of the cryostat through the valve handle. To the opposite , warm end of the heat exchanger a vacuum pump is connected. When the pump is running, the pressure inside the heat exchanger decrees and the liquid nitrogen is sucking to the heat exchanger form cryostat through the J-T valve. Due to throttling process realized on the J-T valve the nitrogen inside the heat exchanger has lower temperature then nitrogen in the cryostat, therefore the temperature of nitrogen in the cryostat start to decrees. The final temperature of the cryostat nitrogen depends on nitrogen pressure in the heat exchanger, what corresponds to J-T valve opening.
The HTS tape is fixed in the sample holder. The holder is wired with 4 wires, one wires pair per the holder side. One wire from the pair is the current supply, since the second wire is voltage wire. In such configuration the tape electrical resistance can be determined.

Test stand for Meissner Effect observation is shown in Figure 5. It consists of a liquid nitrogen container, a plate of superconductor, 5 different pieces of metal, a magnet.

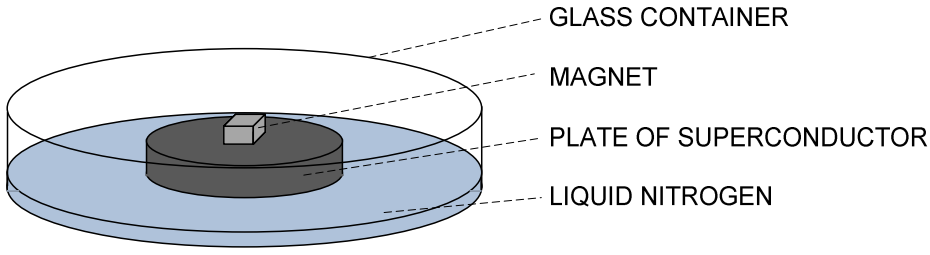

Figure 5. Test stand for the Meissner effect visualization.

Do not forget about protective glasses and gloves!

Assignments

Determination of critical current

1. Fill the cryostat with liquid nitrogen
2. Check the fixation of the HTS tape in the sample holder
3. Put the cryostat insert in to the cryostat bath
4. Connect current source and voltmeter to the proper electrical connectors in the cryostat
5. At the boiling temperature start ramping the supplied current with 1A step
6. After each ramping stem read the voltage drop over the HTS tape and calculate the electrical resistance over the tape. Keep in mind, that for the low value of supplying current the resistance value stand for residual resistance of the holder.
7. Continue ramping till overall resistance increase over 5 time of residual resistance. At this point the critical current of he tape is found.
8. Run the vacuum pomp an stabilize the bath temperature at lower level
9. Repeat the steps 5 to 7 for 5 different levels of the cryostat bath temperature
10. Drawn the characteristic of the critical current of the HTS tape in function of cryostat bath temperature

Meissner Effect

Place the magnet on the superconductor surface. Fill the container with liquid nitrogen. Explain what happens. Repeat the observation for all pieces of metal.

Questions and problems

1. Superconductivity – definition
2. Describe the factors which define the superconductivity
3. Difference between superconductors Type I and Type II
4. How to determine the critical current of the superconductors?
5. What is advantage of measurement of the electrical resistance with 4 wire method?
6. How temperature of the cryogens can be reduced?
7. The Meissner effect – definition.
8. Explain the difference between dia-, para- and ferromagnetic materials.
9. Definition of critical temperature.

10. List a minimum 3 applications of a superconductor.

Literature

1. A.M. Arkharov, I.V. Marfenina, Ye.I. Mikulin, Cryogenic Systems, Bauman Moscow State Technical University Press, 2000
2. Cryogenics Lecture Materials: Lecture 8 – Adiabatic demagnetization

Laboratory 10

Subject: Shrink-fitting technique

Introduction

Shrink-fitting is a technique used to join metal elements. This technology is based on the phenomenon of thermal contraction. One piece of metal is cooled (can also be heated) and its diameter decreases so it can easily be fitted to a second piece, see Figure 1. As the adjoined pieces reach the same temperature, the joint becomes strained and stronger.

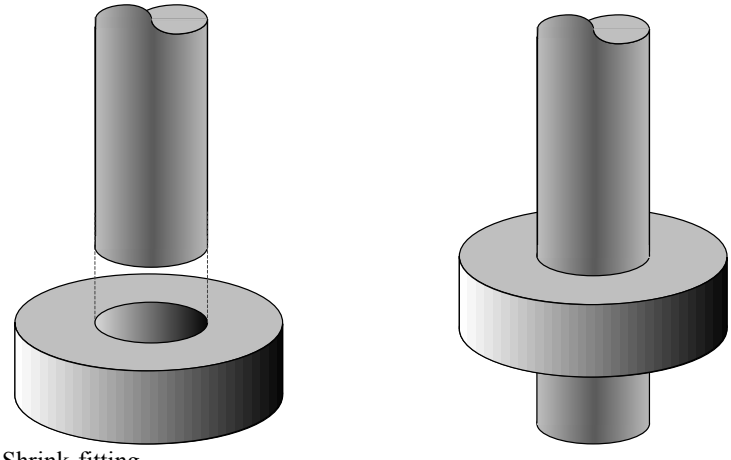

Figure 1. Shrink-fitting

The advantages of the shrink-fitting technique:

- can often be performed in minutes,
- can provide a precision fit,
- will not damage the majority of ferrous and non-ferrous metals,
- heating components may take hours to achieve the necessary expansion,
- heating components can cause distortion and damage,
- after shrinking, component reaches ambient temperature more rapidly than if heated,
- heating may introduce an imprecise fit between components,
- maintains the interference fit for which the components were designed,
- no discoloration of metal after shrink fitting technique,
- can eliminate the need for keyways or other fixing methods,
- can also be employed to dismantle assemblies [2].

The comparison of metal shrinkage is indicated in Table 1 [2].

Table. 1. Metal shrinkage table (for cooling from ambient to liquid nitrogen temperature):

Metal	Shrinkage (microns per mm diameter)
Aluminum	3.8
Brass	3.4
Steal	1.9
Cast Iron	1.8
Copper	3.0
Magnesium	4.5
Nickel	2.1
Zinc	5.4

Topics to prepare before laboratory class

1. Find in literature the value of specific heat and thermal conductivity for copper, aluminum and stainless steel.

Aim and purpose of the laboratory

The observation of the shrink-fitting technique. The estimation of time needed for metal elements to cool down.

Test stand

Test stand consists of two handles, 3 cylinders made of aluminum, copper and stainless steel, and a vessel filled with liquid nitrogen.

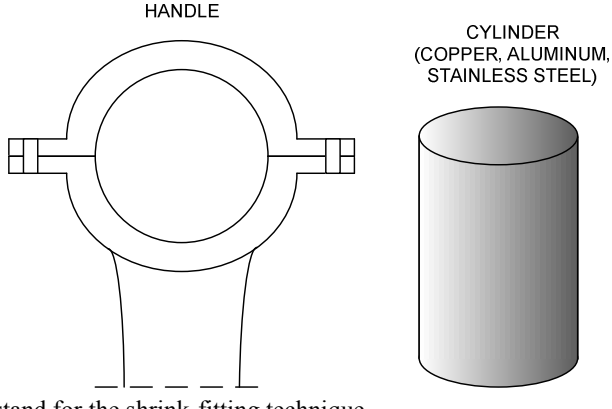

Figure 2. Test stand for the shrink-fitting technique.

Do not forget about protective glasses and gloves!

Assignments

1. Measure all dimensions and weigh the cylinder.
2. Calculate time and amount of liquid nitrogen needed to cool down to 78K (each cylinder).
3. Put the cylinder inside the vessel and fill it with liquid nitrogen.

4. Wait a couple of minutes (see point 2).
5. Place the cylinder in the handle and wait until the elements reach the same temperature.
6. Try to remove the cylinder from the handle.
7. Repeat the test (point 1-7) for all cylinders.

Questions and problems

1. Explain the shrink-fitting technique.
2. List the advantages of the shrink-fitting technique.
3. The phenomenon of material thermal contraction – advantages and disadvantages.

Literature

1. A.M. Arkharov, I.V. Marfenina, Ye.I. Mikulin, Cryogenic Systems, Bauman Moscow State Technical University Press, 2000
2. www.midlandcryogenics.com

Laboratory 11

Subject: Cryogenic technologies in food industry

Introduction

The preservation of food and its quality control by low temperature is not a recent phenomena. An ice and salt mixture has been used for a long time, research started in America in 1865 and 25 years later food-freezing on a commercial scale (mechanical refrigeration systems) started. The main problem for food-freezing technologies is to achieve a temperature below 253 K within the minimum permissible time. A long freezing time is not suitable for many food products (some fruits – e.g. strawberries).

Ultra fast freezing methods by cryogenic liquids have a lot of advantages over conventional freezing methods. At present cryogenic technologies have become widely used in this area.

Various types of cryofreezer are now available in the market. The choice of cooling technique and in consequence freezer type depends on the material (substance) to be frozen. Figure 1 shows a detailed classification of cryofreezers.

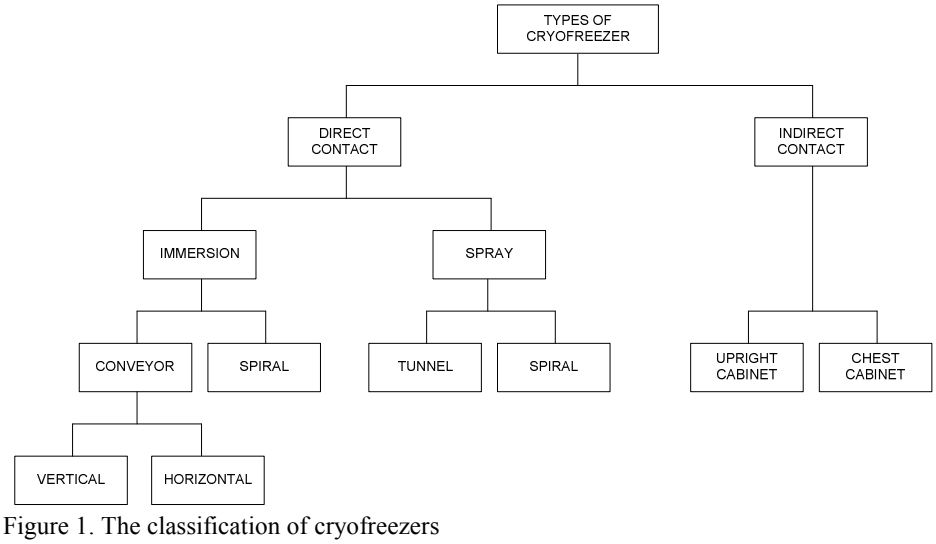

Figure 1. The classification of cryofreezers

Direct contact cryofreezers

Heat conduction of the cryoliquids is higher than in the gas phase so direct contact of the fluid to the material causes the most rapid temperature reduction. The process time is short because almost the whole cooling potential of the cryogen is directly transported to the product. Food products can be immersed in the volume of the cryoliquids – the immersion method, or cryogens can be sprayed directly onto the product surface – the spray method. Based on the construction of the immersion-type the cryofreezer can be classified as conveyor (vertical or horizontal) or spiral. Direct immersion of LN_2 and rapid temperature fall can cause damage to the structure of delicate products so spraying techniques are commonly used in the food industry.

Indirect contact freezers

In the indirect contact method the product is separated from cryogens by a metal barrier. The heat transfer occurs mainly by conduction. There are many advantages to this method in spite of the lower heat transfer. First of all the temperature inside the freezer can easily be controlled and changed, there is no limitation to the dimensions or product form, as well as the safety aspect of handling liquid gases being easily solved (cold burns caused by cryogen are avoided).

Topics to prepare before laboratory class

1. Types of cryofreezers [1].

Aim and purpose of the laboratory

The comparison in thermal efficiency and cooling effect of LN_2 immersion method and conventional freezing.

Test stand

The test stand consists of an insulated vessel filled with liquid nitrogen, a gelatin model, digital thermometer (thermocouples Cooper-Constantan), a scale and stopwatch.

Do not forget about protective glasses and gloves!

Assignments

1. Weigh the gelatin model.
2. Determine the minimum mass of liquid nitrogen needed for model freezing to 250K.
3. Place two thermocouples inside the gelatin model (first one on the surface and the second one in the middle of the cube)
4. Put the gelatin model inside liquid nitrogen and start to register the temperature on the surface and inside the cube (until 250 K). The LN_2 mass inside the vessel should be measured as well.
5. The collected data (temperature) should be presented in the form of graph.
6. Compare the temperature with the temperature profile of conventional freezing (given by your supervisor).
7. Compare the nitrogen mass decrease with the minimum mass of liquid nitrogen needed for freezing (calculated in point 2) – explain the difference.

Questions and problems

1. Present the classification of cryofreezers.
2. Direct contact freezers – explain the method.
3. Indirect contact freezers – explain the method.
4. Choose and describe one type of cryofreezer.

Literature

1. R.M. Khadatkar, S. Kumar, S.C. Pattanayak, Cryofreezing and Cryofreezer, Cryogenics 44), Elsevier, 2004
2. K.D. Timmerhaus, T.M. Flynn, Cryogenic Process Engineering, Plenum Press, 1989
3. A.M. Arkharov, I.V. Marfenina, Ye.I. Mikulin, Cryogenic Systems, Bauman Moscow State Technical University Press, 2000

Laboratory 12

Subject: Characteristics of cryomedical devices

Introduction

Low temperature medicine (cryo-medicine) is becoming a widely appreciated method in rheumatology, dermatology, gynecology, and surgery. The use of low temperatures in medicine can be in treatment (cryo-surgery, including dermatology, gynecology) and rehabilitation (cryo-therapy). While in cryosurgery the use of low temperature is aimed at the destruction of the pathologic cells, cryotherapy is a stimulating treatment (cryostimulation), where a patient's body is subjected to an effect of low temperature, in order to activate defensive reactions, see Figure 1.

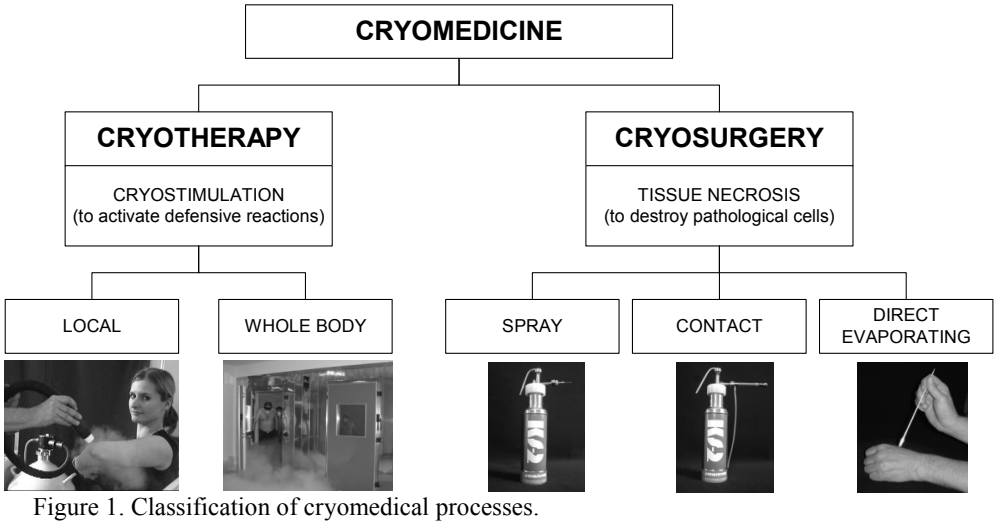

Figure 1. Classification of cryomedical processes.

Cryo-medical appliances are usually supplied with liquid nitrogen, nitrous oxide or carbon dioxide. The devices can consume liquid nitrogen in quantities more than 100 l/h (in case of a whole-body cryotherapy in cryo-chambers) to a fraction of a liter per hour (small cryosurgical apparatuses – Figure 2). The KS-2 vessel is filled with liquid nitrogen. Some heat is transported through the insulation so pressure inside the vessel increases. Liquid outflow is caused by liquid injection through the valve open (pressure differences between the space inside the vessel and the environment). To prevent the pressure increase inside the vessel a safety valve is installed. KS-2 is a portable device (vessel volume 0.35 dm^3 and weight less than 1 kg after filling).

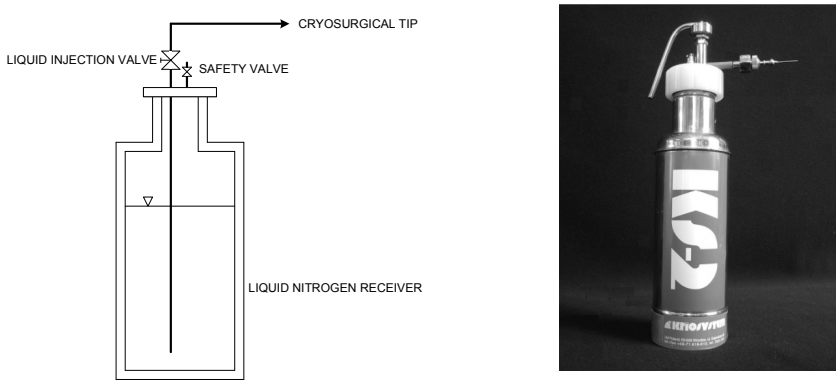

Figure 2. Cryosurgical apparatus (Kriosystem Wroclaw)

Figure 3 shows the dynamics of frozen area creation caused by contact cryosurgery.

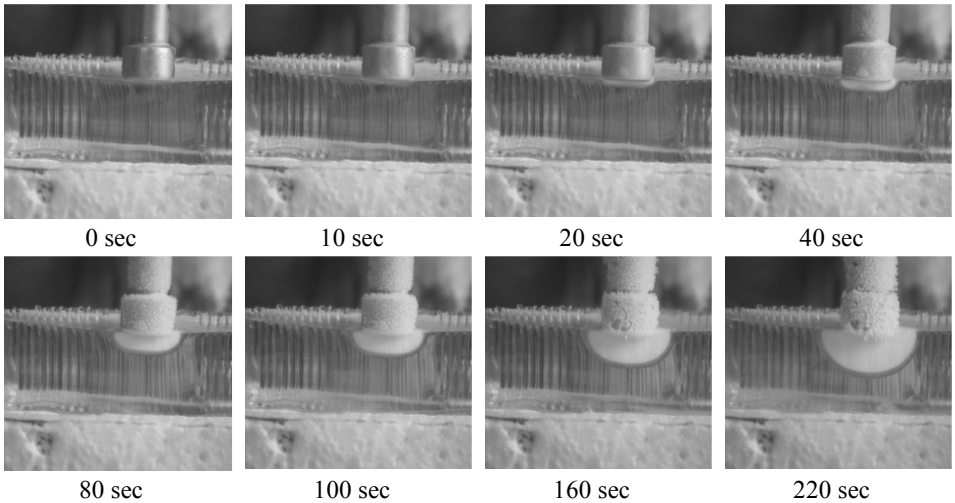

Figure 3. Frozen area creation in contact method

Devices filled with nitrous oxide (N_2O) or carbon dioxide (CO_2) use the Joule-Thomson effect. The cylinder contains high-pressure liquid at ambient temperature. Passing through the expansion valve to the atmospheric pressure the temperature of the gas decreases and N_2O enters in the liquid-vapour phase. Liquid-vapour mixture is sprayed directly onto the skin surface. A cryomedical device filled with N_2O is schematically shown in Figure 4.

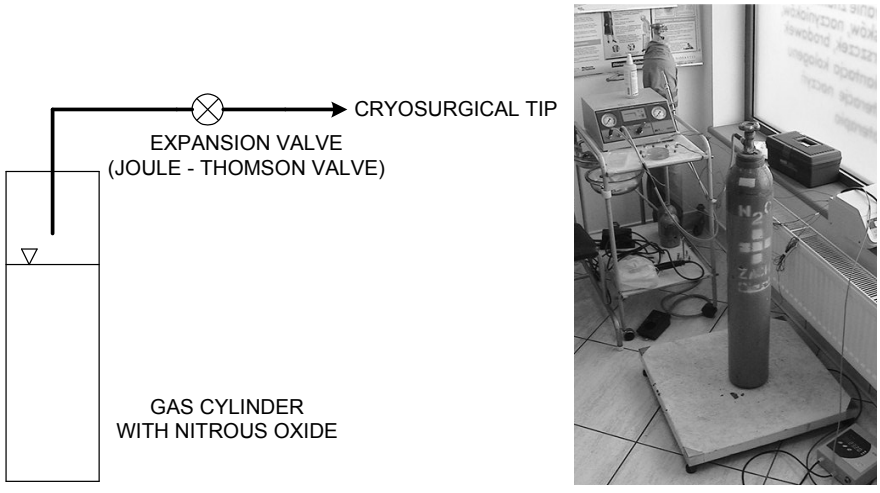

Figure 4. Cryosurgical device supplied with nitrous oxide.

An example of a cryotherapy device Kriosan 7 (Kriosystem Wrocław) is schematically shown in Figure 5. Kriosan 7 was constructed for cryostimulation treatment. It is intended to execute local treatment using nitrogen vapor at a temperature of -165°C. The vessel of 32dm^3 volume is filled with liquid nitrogen. For nitrogen vapor production a radiator is used.

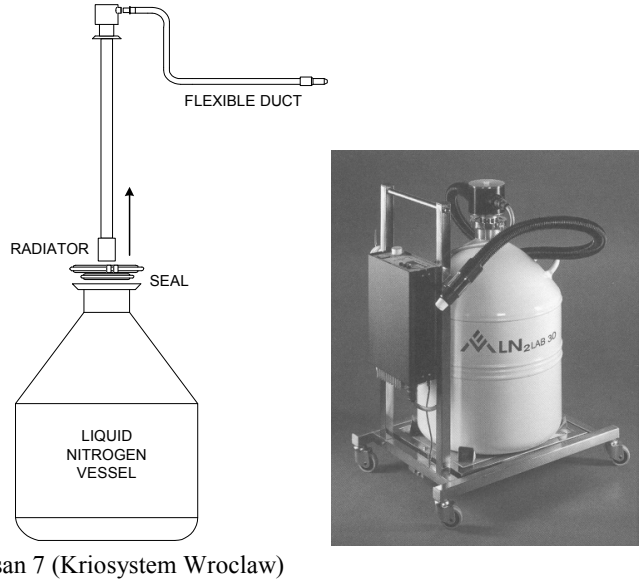

Figure 5. Kriosan 7 (Kriosystem Wroclaw)

Aim and purpose of the laboratory

The comparison of thermal efficiency and the cooling effect of spray and contact methods for cryosurgery simulation.

Test stand

Test stand is schematically shown in Figure 6.

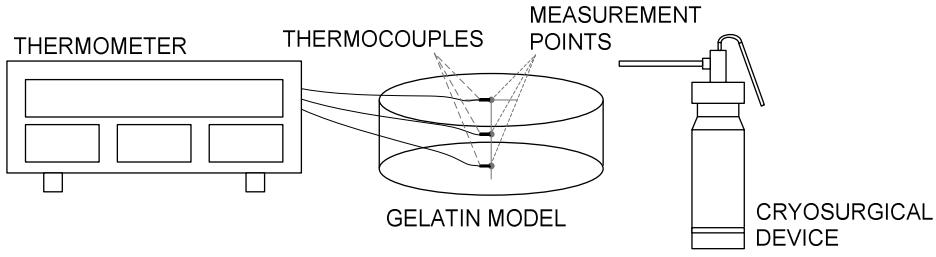

Figure6. The test stand for the cryosurgical process simulation (contact and spray method).

The test stand consists of a skin model, a cryosurgical device filled with liquid nitrogen, thermocouples (Copper-Constantan), digital thermometer and scale.

Do not forget protective glasses and gloves!

Assignments

To perform the simulation of the cryosurgical process a skin model is needed. Gelatin will be used because it has similar properties (especially specific heat) to human skin. Two gelatin rollers will be used (for contact and spray methods).

Before you start the test you should:

- Measure all dimensions of the gelatin models
- Weigh the models
- Place the thermocouples at measuring points (first one: on the surface, second one: 1 cm below the surface and the third one: 2 cm below the surface)
- Weigh the cryosurgical device (after filling)

Start to spray liquid nitrogen on one point. Keep spraying for 5 minutes. Temperatures at certain points will automatically be measured and recorded on a computer. After simulation weigh the device to determine the mass of liquid nitrogen used during treatment and measure the dimensions of the frozen area. Repeat the measurements for the contact method. Describe your observations.

Experimental (temperature) data should be presented in the form of a diagram. Compare temperature decrease for spray and contact methods.

Determine the efficiency of the spray and contact methods Ψ which can be described by equation (12.1)

$$\Psi = \frac{\dot{Q}_M}{\dot{Q}_{N2}}, \tag{12.1}$$

where:

$\dot{Q}_M$ - heat used for temperature decrease of frozen area, W

$\dot{Q}_{LN2}$ – heat transported to liquid nitrogen (calculations based on mass change – see Laboratory 3 and 4), W

Parameters needed for calculations:
Heat of vaporization for liquid nitrogen: r= 198.3 kJ/kg

Gelatin properties:

Specific heat	for T > 273.15 K C_M= 3800 J/kg
	for T < 273.15 K C_M= 1850 J/kg
Heat of solidification	Δh_S = 3360 J/kg

Questions and problems

1. What is the difference between cryotherapy and cryosurgery.
2. List the thermodynamic processes and working fluids used in the cryomedical apparatuses.
3. Explain the difference between cryomedical devices supplied with LN_2 and CO_2.
4. Why do cryomedical devices supplied with N_2O need high-pressure gas?
5. Cryotherapy apparatus has a radiator. Explain what it is for?

Literature

1. M.Chorowski, A. Piotrowska, J.Polinski, Nitrogen Separation and Liquefaction Apparatus for Medical Applications and Its Thermodynamic Optimization, Advanced in Cryogenic Engineering, 2008
2. K.D. Timmerhaus, T.M. Flynn, Cryogenic Process Engineering, Plenum Press, 1989
3. A.M. Arkharov, I.V. Marfenina, Ye.I. Mikulin, Cryogenic Systems, Bauman Moscow State Technical University Press, 2000

Laboratory 13

Subject: Technical operation of a cryochamber

Introduction

According to Laboratory 12 cryotherapy is a stimulating treatment (cryostimulation), where a patient's body is subjected to an effect of low temperature, in order to activate defensive reactions. Whole body cryotherapy is becoming a widely appreciated method in classical medicine and sport as well as to restore health & fitness.

Cryogenic chambers can be supplied with two kinds of media: liquid nitrogen or a mixture of liquefied nitrogen and oxygen, so called synthetic liquefied air. The most common and so far the most universally applied medium is liquid nitrogen due to the low cost of the medium itself and already well developed technology to use this medium.

There are 3 main constructions of cryochambers:

- cryochamber room-type
- cryochamber with cooling retention effect
- cryosauna.

Cryochamber room-type:

A cryochamber consists of two rooms, a vestibule and main cabin, see Figures 1 and 2.

Figure 1. Cryochamber room-type

The vestibule is a transitional room where the temperature level is about 210 K (- 60°C). It is a place where the patients can get used to much more extreme thermal conditions. After about 30 seconds spent in the vestibule the patients proceed into the main cabin. Inside the main cabin the temperature is maintained at 150K (-120°C) to 110K (-160°C). One session of whole body cryotherapy can last no more than 3 minutes. The heat exchangers in the cryochambers are usually supplied with liquid nitrogen. One working hour of a cryochamber requires about 90-100dm^3 of LN2. The air vented into both cabins is purified, dried and cooled down in a dedicated installation located outside the cryochamber [1].

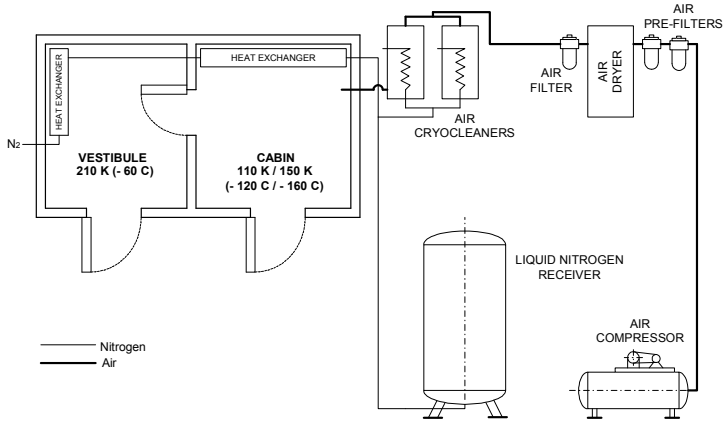

Figure 2. Structure of the cryochamber system

Cryochamber with cooling retention effect

The location of the chamber in the hollow below the level of the operative floor (see Figure 3) which allows use of the coolness retention effect and has the advantage of direct liquid air injection into the cryochamber. The staircase can be treated as an adaptation area (vestibule in the previous chamber).

The possibility of enriching the atmosphere in the cabin with oxygen, up to 24-26%, creates better conditions for biological regeneration of a person who has been exposed to great strains and who needs special conditions for regeneration (e.g. sportsmen after exhausting training). The nitrogen - oxygen atmosphere allows cryotherapy of the whole body, including face receptors which have a huge influence on feeling cold and the processes of thermoregulation in the patient's body [2].

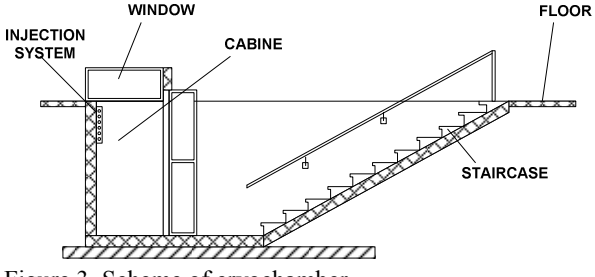

Figure 3. Scheme of cryochamber

Cryochamber ARCTICA
(Metrum CryoFlex)

Cryosauna

The smallest cryochamber – cryosauna is a single-person chamber, see Figure 4.

Its dimensions are as follow: width – 1500mm (with open door), length 1470mm and height 2445mm (chamber produced by JUKA [3]) . The cryosauna is equipped with an elevated floor, so the person inside the chamber is submerged in the low temperature atmosphere up

to his shoulders – the head is above the chamber and the person can breathe air from the main room.

Figure 4. Cryosauna (JUKA)

A cryochamber consists of the following units, see Figure 5 [4]:

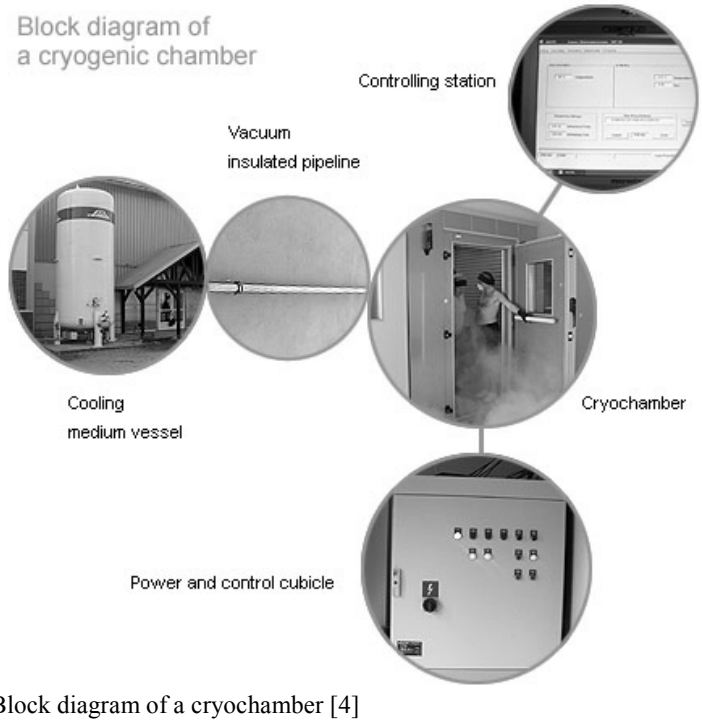

Figure 5. Block diagram of a cryochamber [4]

Assignments

To enter to the cryochamber you must be appropriately dressed and you are not allowed to wear any kind of jewellery or glasses. Before entering, you will put a T-shirt and shorts on, as well as protection for your feet, ears, mouth and nose. Additionally you will be instructed and advised by the trained personnel. The vestibule is a transitional room where temperature level is of about 210K (-60°C). It is a place where you can get used to much more extreme thermal conditions. After about 30 seconds spent in the vestibule you will proceed into the main cabin where the temperature is between 150K (-120°C) and 110K (-160°C). The first session of whole body cryotherapy lasts no more than 1.5 minutes.

The visit inside the cryochamber is not recommended for people who suffer from hypertension, cold urticarid, claustrophobia, Raynaud's disease, heart, lungs and thyroid gland diseases. All students entering the cryochamber will be examined by a medical doctor. If you have any doubts please contact your doctor.

Do not forget the face mask!

Literature

1. M. Chorowski, A. Piotrowska, Comparative analysis of the cryogens used in cryomedical applications, Proceedings of the ICEC20-ICMC2004
2. Metrum CryoFlex webpage: www.metrum.com.pl
3. JUKA webpage:www.juka.com.pl
4. KRIOSYSTEM webpage: www.kriosystem.com.pl