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MODELING AND IMPROVEMENT OF A FIELD ELECTRIC RETARDING POTENTIAL CHARACTERIZATION SYSTEM

STUDY OF VACUUM

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Study of vacuum

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PREFACE

In the lab of DMI, the display department of CTI, a lot of research is done and one of the subjects is obtaining the Field Emission Retarding Potential (FERP) of several samples. Obtaining this potential requires a high vacuum environment, achieved by a vacuum chamber. Since this chamber is handcrafted and constructed with the materials on forehand good drawings and a digital version are lacking.
This is the report of a three months lasting internship which covered roughly three subjects: developing a CAD version of the current system, suggesting some improvements to increase the vacuum level in the current chamber and designing the system of the future that will be able to get in the ultra high vacuum region. All this subjects will be treated in this report, but first the meaning of the FERP will be explained and an introduction into high vacuum will be given, i.e. what is vacuum, how to get it and how to maintain it.

ABSTRACT

A field emission retarding potential FERP is a method to measure the work function between two materials and can for example be used to verify the purity of a material. In order to obtain such a function of several samples a high vacuum environment is desired. At the lab of CTI a certain vacuum chamber with corresponding equipment already exist, but a digital model and drawings of this configuration are lacking however. Main goal of this project is therefore to obtain a CAD model, suggest some improvements to increase the vacuum and finally to draw the system of the future.

The current configuration is measured and modeled in SolidWorks and some improvements in the configuration are obtained. When one wants to increase the vacuum, sources of contamination and leakage need to be avoided as much as possible. Several equipment, like pumps and gauges need to be connected to the chamber. This connections are the main source of leakage so the sealing of this connections is essential. The best way to so is with a copper gasket (with the CF connection) but since a gasket can only be used once and parts need to be released now and then a rubber connection is often preferred regarding the costs.

Since the stock and budget are limited it is hard to improve the vacuum level further. However, some KF connections, that always go with a rubber O-ring, could be removed by some rearrangements. One superfluous gauge is removed and the connection between the turbomolecular pumps is reduced, resulting in a reduction of 3 O-rings in total and an improvement of the vacuum level from 5x10-6 Torr [1] up till 1.7x10-7 Torr.

To obtain an ultra high vacuum level a new system is desired. A concept of the outside is modeled and presented at the end of this report. With this design a pressure level of about 1x10-11 Torr should be reasonable such that more and different experiments, like scanning tunneling microscopy (STM) are possible in the future.

1 Note that power terms like this are preferably are written like 5x10-6 in this report regarding readability.

Fique tranquilo, no pressure

ACKNOWLEDGEMENTS

I would like to spend this page to say thanks to some people. Thanks to all colleagues of the DMI department at CTI, who were very concerned, helpful and nice and made my stay in Campinas very pleasant. Special thanks to my supervisors Vinicius L. Pimentel and Marcos H. M. Hamanaka at CTI, without their knowledge, time and help it would have been impossible to create a report like this.

CONTENTS

1 INTRODUCTION INTO FIELD EMISSION RETARDING POTENTIAL

Since the main goal of this report is to describe the current vacuum system only a brief introduction to the purpose of this configuration, obtaining the field emission retarding potential, will be given.

1.1 FIELD EMISSION RETARDING POTENTIAL

The Field Emission Retarding Potential (FERP) is a method to measure the work functions of metals and semiconductors and can therefore be used to verify the purity of a sample.
The principle of the method is as follows:
When two metals A and B are connected electrically through a circuit without a battery the electrochemical potentials of the electrons in the metals will be equal. If the emitter, metal A, contains a sharp point and gets loaded with a high voltage, electrons can tunnel through the surface potential barrier. After passing through the accelerating anode grid, the electrons are retarded in the region between the anode and the metal B, converting their energy into potential energy. Precautions can be taken to avoid tunneling of electrons at B since there exists a potential barrier equal to the work function of metal B. However, when however a battery is inserted between A and B such that the potential of B is lowered by at least the work function, electrons of A can enter B, increasing the current in the circuit.
So if the collector current is measured as a function of the difference in voltage applied to the circuit the curve will increase sharply as soon as the difference reaches the work function of the collector as can be seen in Figure 1. And as soon as the work function is known, purity of the material can be determined for example [1].

A (very) high vacuum is a prerequisite but focusing of the electrons is essential as well. A correct total energy distribution is measured only when all electrons are normally incident to the collector surface, otherwise the sharp curve flattens out. Therefore use can be made of an electron optical device (or magnet) and ensure the electrons arrive perpendicular to the collector surface. Nano-tips with high enhancement factor can be used as emitter and allows the system work with a lower electric field than conventional systems. More information of creating single atom tips can be found in paper [2].

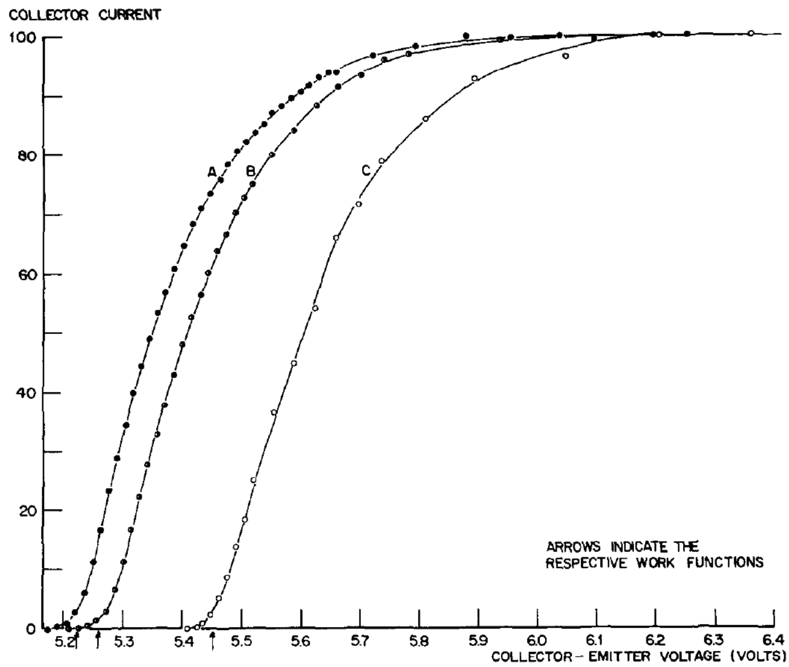


Figure 1: Experimental current versus voltage plots obtained with nickel films (A& B) and gold (C) [1]

2 STUDY OF HIGH VACUUM SYSTEMS

This chapter contains the available and required equipment to gain a high vacuum environment. But first of all an introduction to vacuum will be given and at the end the ways to maintain the vacuum, i.e. how to cope with the circumstances and reduce leaking will be treated.

2.1 INTRODUCTION TO VACUUM

Obtaining a vacuum yields removing of (nearly) all the gases from a chamber. Vacuum technology covers also the measurement and manipulation of the environment once a vacuum environment is obtained.
Pressure can be measured in several units; Pascal, (milli)Bar and Torr. Since most of the literature in vacuum (especially in the USA) is given in Torr, this report will contain only pressure expressed in Torr. In Table 1 some values are given in the three dimensions [3].

PASCAL	TORR	BAR
101,300	760	1.01 (atm)
133	1	$1.33 \cdot 10^{-3}$

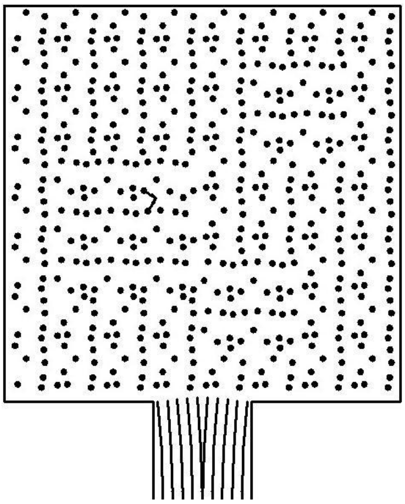
Table 1: Three common units for pressure

Due to the vastly different sets of physical laws applying, vacuum science is necessarily sub-categorized into a number of vacuum regimes.

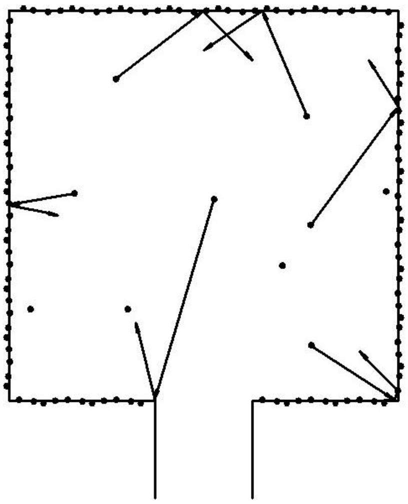
2.1.1 *Vacuum regimes*

Vacuum is divided in four regimes: rough, medium, high and ultra high vacuum. The air consists of several particles and how they behave in the several regimes is illustrated in Figure 2 [4].

ROUGH VACUUM (± 760 - 0.1 TORR) First stage is to remove the bulk gas from the system. A chamber contains a lot of gas molecules, see Figure 2a in the chamber and these interact, according to the laws in thermodynamics, in the manner of a viscous fluid. Gas is considered to be in a viscous flow so this region is very common to mechanical engineers. Pumps used to obtain this stage are therefore often considered as mechanical pumps.



(a) Rough vacuum



(b) High vacuum

Figure 2: Illustration of particles in the chamber [4]

MEDIUM VACUUM (±0.1 - 1X10-4 TORR) In the high vacuum region the behavior can't be considered as viscous anymore and collisions between the particles itself and the wall become of interest. The transition of the gas behaving as a viscous flow into a gas behaving as a molecular flow is considered as the medium vacuum.

HIGH VACUUM (±1X10-4 - 1X10-8 TORR) As soon as the viscous behavior of the gas is disappeared the high vacuum stage is reached. Collisions between the chamber wall and the molecules dominate the gas since the mean free path between the molecules is bigger than the chamber dimensions, see Figure 2b. The change in behavior makes that other kind of pumps are required. It's no longer a matter of sucking the air out of the room, but rather waiting on a particle to bounce into the sucking head of the pump. This kind of pumps cannot directly suck or release gas at atmospheric pressure and requires a pre vacuum pump therefore.

ULTRA HIGH VACUUM (<±1X10-8 TORR) Hardest part of increasing the vacuum even more is removing the last particles, mostly hydrogen, from the chamber. Especially when those particles are somewhat embedded in the chamber wall and makes it hard to remove. Properties of the container wall will be of decisive importance since below a pressure of 1x10-3 Torr there will be more gas molecules on the surfaces than in the chamber itself. This requires special pumps, like ion-pumps.

Not only the behavior of the gas changes but the physical properties change as well. Thermal conductivity and internal friction of gases are highly sensitive up to the medium range, but in the high vacuum regimes those properties are independent of the pressure. So not only different pumps are required, but different gauges as well.

2.2 PUMPS

Main attribute to get a vacuum is a pump and as explained in the previous section different pumps are required for the different stages. Overview of the ranges and some pumps capable of obtaining the corresponding pressures are shown in Figure 3.

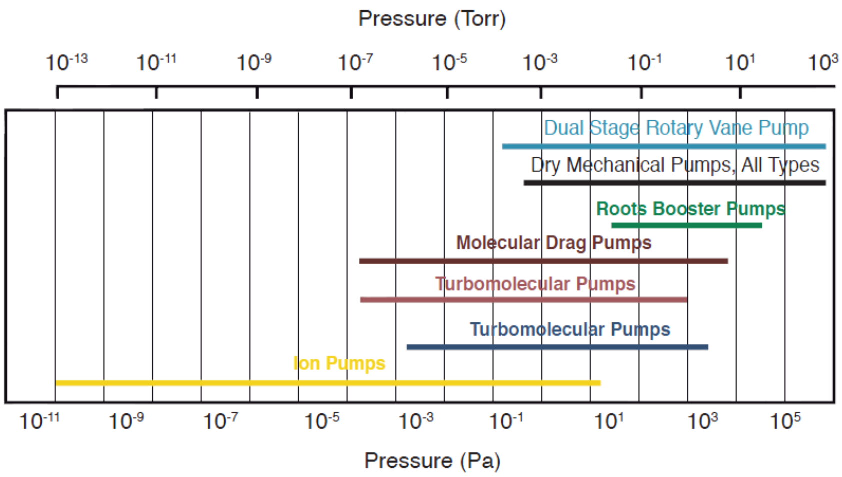


Figure 3: Vacuum pump operating ranges [5]

First four types of mechanical pumps used to obtain the pre vacuum will discussed, followed by four kind of pumps used to obtain a (ultra) high vacuum later on. The type of pumps used in the current configuration will be discussed more extensively for a better understanding later on in the report, whereas alternatives only will be discussed briefly.

2.2.1 *Mechanical pumps*

Mechanical pumps are used to obtain the rough vacuum. There exist roughly four types: the oil pump, the membrane/diaphragm pump, the roots pump and the scroll pump. Basic principles will be explained in this subsection and at the end a comparison will be made and advantages and disadvantages will be briefly discussed.

OIL PUMP A rotary vane pump is a common oil-sealed rotary displacement pump. In Figure 4 an overview of the pump is shown. The rotor is installed eccentrically and contains the vanes that move under centrifugal and elastic forces. The springs ensure that the vanes are attached to the wall and that the chamber gets divided into two separate parts all the time. Gas gets compressed as soon as the rotor

turns until the oil sealed outlet valve opens.
Oil has an essential function and has multiple tasks. Oil enters the chamber as soon as the valve opens and besides lubricating all moving parts, it also seals the outlet valve and the vanes against the housing. Additionally it ensures an optimal temperature balance through heat transfer. Without the oil the compression ratio (about 10^5) would be a factor 10 less. Main drawback of using oil is the unavoidable contamination into the vacuum chamber. Therefore the ultimate pressure is around 1x10-2 Torr because it's hard to remove the leakage of oil once it has entered the chamber. Two stage oil pumps can be used to increase the ultimate pressure up till 1x10-4 Torr, due to the fact that oil which already has been degassed is supplied on the side of the vacuum. Another used oil pump is the rotary plunger pump and works in a similar way, but a piston is used instead of vanes [6].

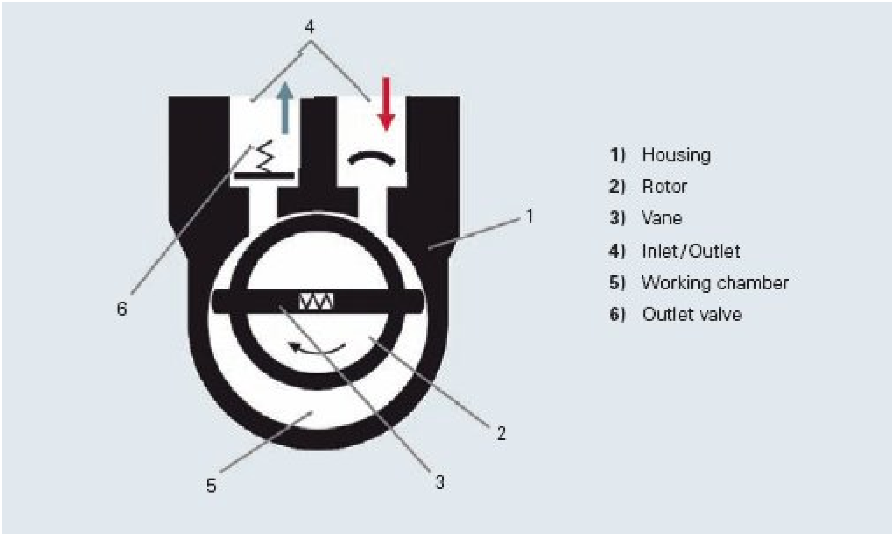


Figure 4: Rotary vane pump [6]

MEMBRANE / DIAPHRAGM PUMP Diaphragm pumps are single are multi dry compressing vacuum pumps and are therefore completely free of oil so they don't contaminate the vacuum chamber. The working principle is shown in Figure 5. It uses a diaphragm that gets elastically bended in a oscillating way by means of a connecting rod and an eccentric. The in- and outlet valve are arranged in such a way that the inlet valve opens as the volume increases and the outlet valve opens when the volume decreases. Due to the limited elastic deformability of the diaphragm a low pumping speed is imposed and the ultimate pressure is quite high. This is limited by the dead space between the diaphragm and the outlet valve. Multiple stages, up till four, can be used but still the ultimate pressure is just around 0.4 Torr [7].

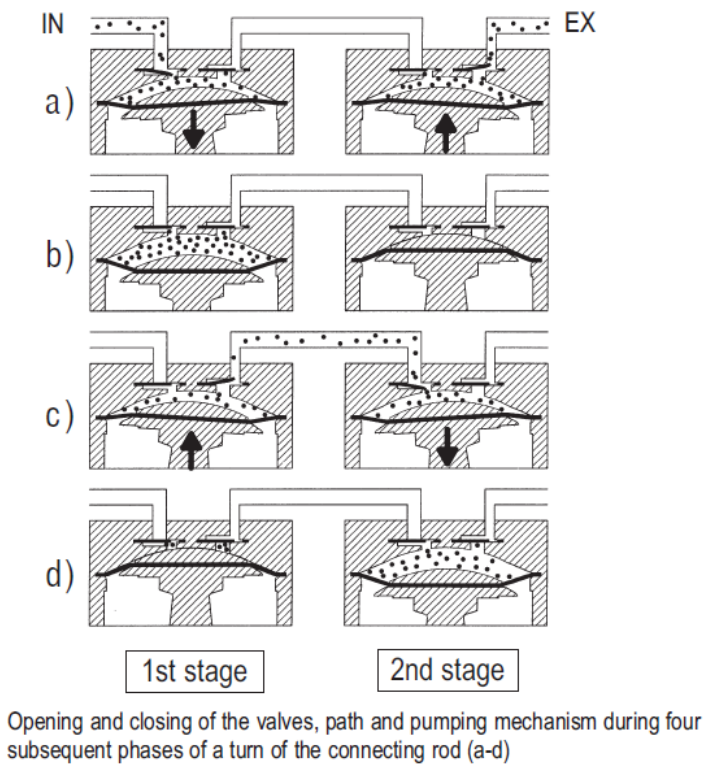


Figure 5: Diaphragm pump working principle [7]

ROOTS PUMP Roots pumps are also dry compressing pumps and are similar to the rotary vane pumps. However due to the dry operation there isn't a sealing oil film between the rotors and the casing. In Figure 6 a schematic cross section of a roots pump is shown. Two symmetrically '8' shaped impellers rotate in opposite direction. One is driven by a motor and the other shaft is synchronized by a timing gear. A high pumping speed can be achieved but the ultimate pressure is about 1 Torr [6].

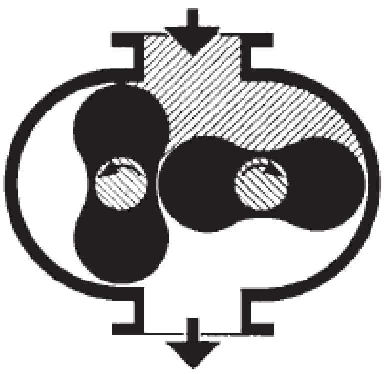


Figure 6: Roots pump [6]

SCROLL PUMP In Figure 7 several stages of the scroll pump are shown. It consists of two spirals; one is fixed and the other is rotating such that the air entered is going round in the chamber and gets compressed. The ultimate pressure is with 8x10-3 Torr quite low, but main drawback is its price. Compared to the other types it's quite expensive [8].

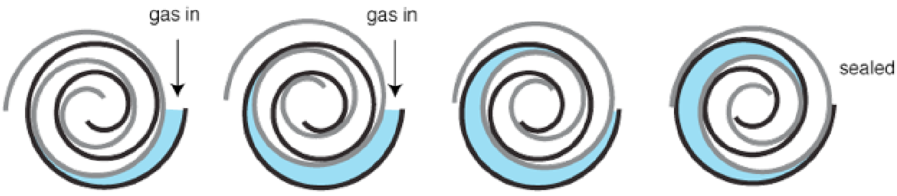


Figure 7: Roots pump [8]

COMPARISON In Table 2 an overview of the four discussed pumps is shown. Biggest difference is the contamination of oil and the costs in purchasing.

TYPE	PRESSURE [TORR]	SPEED [M3/H]	OTHER PROPERTIES
Oil	1 x10-4	15	Low costs, oil leakage
Diaphragm	0.4	0.06	No contamination
Roots	1.0	15	High costs, low maintenance
Scroll	8x10-3	15	High costs

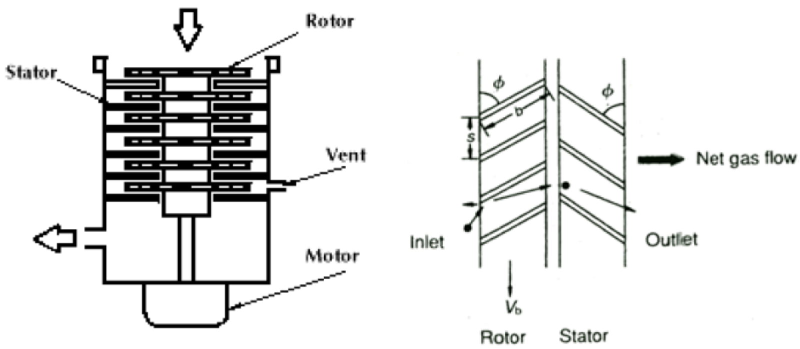
Table 2: Overview of the mechanical pumps

2.2.2 *High vacuum pumps*

To get in the high vacuum region different kind pumps are required since the gas acts no longer as a viscous fluid. Pumps capable of getting into this regime can be divided into two categories: momentum transfer pumps and entrapment pumps.
Momentum transfer pumps contain the turbomolecular pump and the diffusion pump and use high speed jets or high speed rotating blades to knock gas molecules out of the chamber. Since they aren't capable of 'knocking' the molecules when they are in the viscous phase, mechanical pumps are required as backing pump to get a rough vacuum and to avoid back streaming of molecules.
Entrapment pumps cover the cryogenic pumps and ion-pumps and are able to get in the ultrahigh vacuum. They don't need backing pumps, but since they require periodic regeneration their available

operational time can be unacceptably short in low and high vacuums so their usage is often limited to ultrahigh regions.

TURBOMOLECULAR PUMPS Once the gas is no longer in a viscous flow but molecular flow, the turbomolecular pump can start to lower the pressure up to the high vacuum regime. It works on the same principle as the turbine in an airplane. It consists of rotor and stator disk pairs. Whereas the rotor gets turned by the motor on high rotation speed (Figure 8a), the stator is fixed and contains similar disks but oriented in opposite direction, as shown in Figure 8b.



(a) Lateral view (b) Pair of rotor and stator disk

Figure 8: Schematic inside view of a turbomolecular pump [9]

The working principle is as follows: when the blades spinning at a high speed they hit the molecules and due to the orientation of the blades on the rotor and stator, they are likely to move into the pump. Succession of rotor-stator pairs move the molecules towards the exhaust where they get collected by a backing pump. Need for a backing pump can be explained right now. On atmospheric pressure the molecules move in a viscous flow and the interacting 'collision force' between the molecules is bigger than the force exerted by the blades. The principle only works when the molecules are free to move, i.e. the gas is in a molecular flow. Besides creating a pre vacuum, the backing pump has also to collect the molecules at the exhaust, preventing them to flow back.
Limit of the turbomolecular pumps is around 1x10-11 Torr and is mainly limited by the maximum compression ratio. At this stage the probability of a molecule coming back from the outlet to the intake is comparable to the probability of a molecule being driven towards the outlet by the pump, i.e. a status quo has been reached.

To get an idea of the enormous rotational speed of the rotor, a comparison with some common turbines is shown in the Table 4.

DIFFUSION PUMPS Besides the turbomolecular pump, the diffusion pump is also a common momentum transfer pump. Whereas

TURBINE	RPM
Wind turbine, electrical speed	3600
Engine Airbus A380	12,500
Engine F16	14,000
Turbopump, Leybold TURBOVAC 361	45,000

Table 3: Comparison of rotation per minutes

the turbomolecular pump better works with heavy molecules the diffusion pump has a high pumping speed for all gases. Oil gets vaporized by electrical heating and is directed through a jet assembly. The nozzles makes the vapor hit the outer cooled shell of the diffusion pump and the liquid condensate flows downward as a thin film along the wall and finally returns into the boiler, as shown in Figure 9.

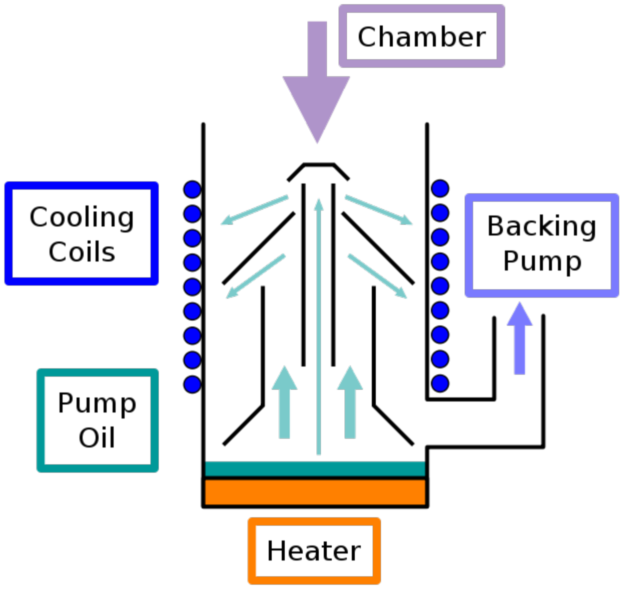


Figure 9: Diffusion pump [10]

However it has a high pumping speed, it can reach a vacuum of around 1x10-10 Torr and the lack of moving parts makes it quite durable, but the risk of oil leaking into the chamber is often the reason of not using this pump.

CRYOGENIC PUMPS A cryogenic pump is an entrapment pump and makes use of the fact that gases are likely to condense on a cold surface. Formation of condensate on a cold surface means that a large number of gas molecules is removed from the volume. They remain on the cold surface and don't take any longer part of the hectic gas atmosphere within the chamber. Cryopumps are commonly cooled by compressed helium or liquid nitrogen. It requires high maintenance and very pure helium.

ION-PUMPS Ion pumps or other types of entrapment pumps and work on several processes and its main purpose is to bury the residual gas molecules into a titanium surface in the pump. The interior of the pump consists of an array of positively charged cylindrical anodes, as can be seen in Figure 10. Their axes are parallel and perpendicular to two titanium plats that are kept at a negative electric potential. Outside the plates a magnetic field in the direction of the axes is generated by making use of permanent magnets. [9]
The free electrons, taking on helical paths about the magnetic field lines, strike free gas particles and ionize them. As soon as the particle is ionized it gets a positive charge and is accelerated towards a negatively charged titanium plate with sufficient kinetic energy to pierce the surface. The adsorption process is one of covalent bonding and ensures that the gas particle remains bonded indefinitely. This makes that the titanium plates can become saturated and for that reason the ion pumps are used only in the (ultra) high vacuum since at higher pressures, more ionization of gas occurs.

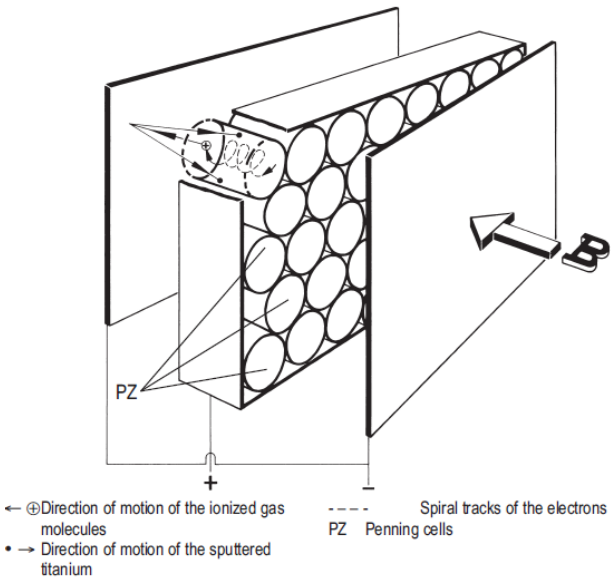


Figure 10: Ion pump [7]

2.3 GAUGES

Once a vacuum is obtained with a correct pump configuration the level of vacuum becomes interesting. Several gauges can be used to do so and there has to be determined which type is required, since there doesn't exist a universal vacuum gauge that is able to measure from atmospheric up till 1x10-13 Torr. Thermocouple, Pirani , Penning, ion gauge and capacitance manometers are the types that will be discussed in this section. Distinction is made between direct and indirect gauges. Indirect gauges are dependent on the composition of the gas being measured (conductivity for instance) whereas the direct gauges are independent of the gas composition [5].

2.3.1 *Pirani and Thermocouple gauges*

Pirani and thermocouple gauges differ slightly from each other, but work in the same manner. By measuring the heat lost from a wire pressure can be determined up till a range of 1x10-3 Torr, whereas the amount of gas becomes insufficient to measure accurate. The Wheatstone bridge principle is used to measure the pressure. One wire is placed in the vacuum environment and one is placed in a (atmospheric) reference gas. Gauge electronics measure the temperature loss of the wire inside the vacuum and compare it with the reference. The more gas particles surround the wire, the more heat can be transferred and the bigger the current required to maintain the temperature has to be. Consequently if the amount of gas near the wire decreases heat transfer decreases as well. Since the heat transfer depends clearly on the substance of the gas these gauges are considered as indirect gauges.

2.3.2 *Penning and Ion gauges operation*

Two other indirect gauges are the Penning and ion gauges, also considered as the cold and hot cathode gauges. They operate by ionizing gas within a magnetically confined cathode discharge. By applying a high voltage to the electrodes, combined with the strong magnetic field, a directed plasma creates a discharge of electrons. The magnetic field makes the ions accelerating toward the charged cathode and by measuring the impact of the ions with the cathode the amount of particles can be determined. Glow discharge, required for the gauge to operate, makes that the gauges cannot operate at pressures above 1x10-2 Torr.

2.3.3 *Capacitance Manometer*

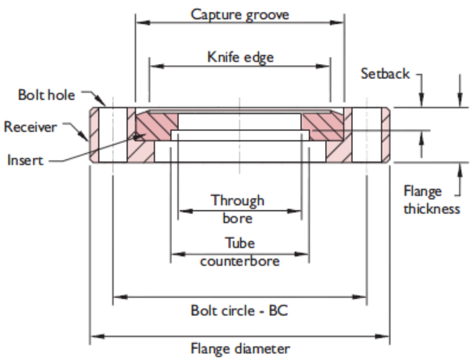
This is a direct pressure gauge and makes use of a diaphragm. One side is located at the vacuum and the other side is placed at a known pressure level. Pressure in the vacuum chamber compresses or allows expansion of the metal diaphragm. By measuring the movement of the diaphragm as a function of capacitance it with a fixed electrode the level of vacuum can be determined from atmospheric pressure up till 1x10-4 Torr.

2.4 CONNECTIONS

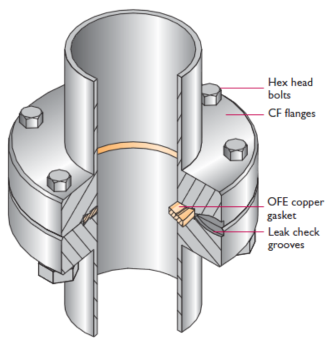
Main equipment for obtaining and measuring a vacuum environment in a chamber is explained up till now, but the way how to connect this equipment to the chamber is essential as well and will be treated in this section. Maintaining the low pressure and avoiding leakage are key issues when it comes to creating a vacuum. Connections are an unavoidable source for leakage of atmospheric air into the system.
Equipment is connected with flanges categorized into two types: CF (ConFlat) flanges and KF (Klein Flanges) or QF (Quick Flanges). Both can be used but normally a CF flange goes with a better sealing, as will become clear later on.

2.4.1 *CF-flanges*

In Figure 11 two cross sections of a CF flanges are shown. In Figure 11a a cross section of a rotatable flange is shown. Rotatable means that before two flanges get fastened by bolts the flange can rotate for right alignment of the bore holes. Both flanges contain a knife (at the top of the flange in the left figure) that is used to cut into the softer metal gasket sealing, as shown in Figure 11b. Most of the time a copper gasket is used and provides an extremely tight metal-to-metal seal by filling small defects in the flange. However, each time the flange gets released the gasket becomes useless for a second time. Therefore sometimes usage of rubber O-rings might work out since they can be used several times but their sealing isn't as tight as the gaskets [6].



(a) Cross section of a rotatable flange



(b) Mated cross section

Figure 11: Cross sections of CF flanges [11]

Several dimensions and types of the flanges exist. Types differ from blank flanges, to enclose a hole, flanges with a tube to weld on another part or embedded flanges, in a sphere for example. Besides that, blind holes or tapped holes can be used depending on the purpose. Dimensions of the CF flanges are covered in standardized to the inner diameter (metric) or the outer diameter (inches). An overview of some common standard versions in both versions is shown in the table Table 4.

METRIC	INCHES	O.D. (mm)	Tube I.D. (mm)
DN16	1-1/3	34	16
DN40 (or DN35)	2-3/4	70	35
DN63	4-1/2	114	60
DN100	6	152	100
DN160 (or DN150)	8	203	150

Table 4: Standardized conflat flanges [11]

2.4.2 *KF*

Another way to connect the parts is by making use of Klein Flanges (KF), also known as Quick Flanges (QF). Usage of rubber O-rings with CF flanges is optional, but using KF connections it becomes obligatory. The sealing is suitable for obtaining a high vacuum, but the unavoidable leakage makes an ultra high vacuum impossible. In Figure 12 the working principle is shown. The (raised) edges on the tube endings are chamfered just like the clamp. As soon as the clamp gets tightened both tube endings are forced to move to each other. In between a rubber O-ring is placed, often on a centering ring to ensure the alignment.

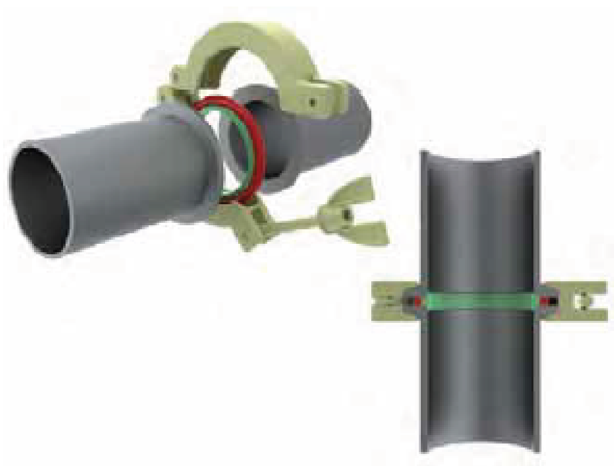


Figure 12: Quick flange [6]

2.5 OTHER EQUIPMENT

The most important equipment to obtain a high vacuum is already discussed so far. Other equipment that isn't essential to create a vacuum, but might be necessary for the purpose of the vacuum will get some attention in this section. Not all available equipment will be treated but only the equipment used in the current or upcoming system. Valves, residual gas analyzers, feedthroughs and linear positioners will be briefly discussed here.

2.5.1 *Valves*

Valves can be used to close a part of the configuration. It might be useful when one wants to get at the ultra high vacuum stage. For the first regimes the pumps with a high throughput can be used to erase the gas in the total room and for the ultimate stage the volume of the chamber can be decreased. The pressure in this smaller room can easier reach the ultra high region, while at the other part the pumps can easily be switched off [6].
Sealing of the valve is as important as the flanges. However an elastomer sealing valve can reach a pressure of about 1x10-8 Torr (high vacuum) an all-metal valve, where all seals are made of metal, are required for reaching the ultra high vacuum region. However a higher closing force is required to seal and results in a shorter service life.

Closing of the valve can be done in several ways. In case of a small diameter (up to DN40) electromagnetically valves are likely to use but require to much force when the diameter gets bigger. Pneumatically valves are delivered in single and double acting variants, depending on the way the resetting action is obtained (by making use of a spring or compressed air as well). In case of a failure an automatic valve might result in damage of the system. To avoid this, automatic valves are often delivered with an normally open or closed state. Once a failure occurs, it automatically returns to its normal position. Manual closing valves can be considered as well when this prevention isn't sufficient or the force required for closing the valve isn't that big.

2.5.2 *Residual gas analyzers*

Residual gas analyzers (RGA) can be used to analyze the gas substance present in the chamber. Partial pressure, exerted by a certain type of gas in a mixture, can be measured for several gases and all partial pressures together give the total pressure for the mixture. Distinction among the various types of gases is essentially on the basis of their molar masses. Therefore it is hard to distinguish molecules and atoms with similar masses. Separating of a certain component from the mixture is required in order to measure the quantity of this component. This is accomplished on the basis of mass-to-charge ratio and the systems of separating the substance divides the several types. This types will not be treated here but can be found in literature. [7]

2.5.3 *Feedthroughs*

Feedthroughs are essential to transfer a current or liquid into the chamber. Electrically feedthroughs are used transfer electricity between the equipment outside the chamber and the instrumentation inside. They are available as wire feedthroughs, multiple feedthroughs with a plug or feedthrough with a coaxial connector. Insulation is of importance since it should insulate the electricity but also comes with a new source of leakage for the system.
Other common feedthroughs are those for liquids. They are not only used to control the environment gas in the chamber, but also to control the gas inlet when the system is opened, i.e. a clean controlled gas enters the room instead of the contaminated air [6].

2.5.4 *Viewports*

Viewports are used to observe the interior of the chamber all the time, even during the process. Elastomeric seals are used to attach a glass of sufficient thickness onto the flange desired. To obtain an ultra high vacuum the glasses are metalized and soldered (or fused with welding lips to compensate for the thermal expansion) on a CF flange.

2.5.5 *Linear positioners*

Linear positioners are use to move parts in the chamber in a linear motion while the system is up and running. They are used with a small, precise, stroke (20mm for instance) where the displacement is obtained by making use of a knob that makes an upper and lower part moving towards each other. Besides that one can also think of a large stroke (300mm for instance) where movement is obtained by a magnet that can move along a tube and carries the inner part along.

2.6 INFLUENCES ON VACUUM

One of the advantages of vacuum is the marginal influence of the environment, but in order to do so, some measurements have to be taken [6].

CONTAMINATION Parts used might contain some residues of the production, like oil and grease, or dust attaches to the parts. In order to avoid this contamination releases from the wall during the operation and increases the pressure, all parts should be cleaned carefully. During the assembling grease should be avoided as much as possible and wearing gloves is highly recommended.

ADSORPTION Vacuum chambers can be used for evaporation and when this is done liquids like water can condensate. Adsorption of water molecules onto the wall is pronounced due to their strong polarity. Also other substances such as pump operating fluids ca be adsorbed on the walls. Measurements to avoid the diffusion of this contamination during operation is to dry the parts carefully after washing them to remove the water molecules on forehand and bake out the system under vacuum to drive the majority of the volatile components out of the metal walls.

DIFFUSION WITH DESORPTION Below pressures of 7.5 10-6 Torr desorption of plastic surfaces, like the seals, become of greater significance. Plastics mainly give off the gases that are dissolved in these plastics, which first must diffuse on the surface. At lower pressures hydrogen and carbon can even escape from metal walls.

LEAKAGE Once the right equipment is purchased, cleaned, dried and baked out, it should be assembled in such a way that the surrounding air at atmospheric pressure doesn't enter the chamber [6]. A system is never leak free so the goal is to reduce the leak as much as possible, i.e. try to increase the point where the pumping rate comes in equilibrium with the leak rate, measured in the pressure rising per second in a volume of 1 liter (Torr $\cdot$ l/s).
Seals are essential when it comes to leakage. A rubber seal not only carries the risk of desorption along at low pressures, due to the surface roughness their sealing isn't as tight as a gasket sealing. Small gas particles are able to permeate the small gaps between the sealing and the parts. When using gasket however, a tighter sealing is created since the softer gasket material is forced to form around the knife of the flange. Nevertheless mounting the flanges with a gasket need to be done carefully to avoid skew assembling. Switching constantly of the side where bolts are tightened and tighten them in several stages ensures a gently and straight mounting.

2.6.1 *Leak detection*

As soon as a system is assembled there are some ways to check whether or not it contains leakage due to wrong assembling. One way to do so is to use a residual gas analyzer and spray a light and rare gas around the system, like helium. If there is a leak, helium would be the first atom likely to enter the room and since air doesn't contain helium, one is able to detect the presence of the leak very soon.

3 STARTING CONFIGURATION

In this chapter the starting configuration of the system will be discussed. First the chamber will be treated and afterwards the attached equipment will be explained in the sections *'Pumps'*, *'Gauges & gasinlet'*, *'Sphere'* and *'Instrumentation'*. The first two are self-explanatory, the *'Sphere'* section contains the sphere chamber on top and the attached feedthrough on the back of the sphere, the viewport on front and the blank flanges attached on it. In the latter section the interesting instrumentation on top will be explained. It contains the essential tools, like the sample holder, filament, tungsten tip and their belongings, to the achieve the purpose of the chamber. Finally the start up will be treated including some procedures during the mounting of all the parts.

An overview of the starting configuration is shown in Figure 13.

3.1 CHAMBER

In Figure 14 screen-shots of the chamber are shown. The base is composed of two tubes, made of 304 stainless steel. One small tube will be welded radial to the base tube, on a distance of 67 from the right side. Dimensions of the tubes are shown in Table 5.

DIMENSION	BASE TUBE	VERTICAL TUBE
Outer diameter [mm]	110	64
Length [mm]	207	70.27
Wall thickness [mm]	2	2

Table 5: Dimensions of chamber tubes.

On all tube endings flanges will be welded as shown in Figure 14b. This flanges will be discussed in the next subsection.

3.1.1 *Welded flanges*

The flanges welded to the chamber will be briefly discussed over here. They are not really standardized but primary based on the materials on stock. Since the flanges were already mounted to the chamber the dimensions of the drawings were extracted from LNLS Vacuumbook [12] and other dimensions are estimated.

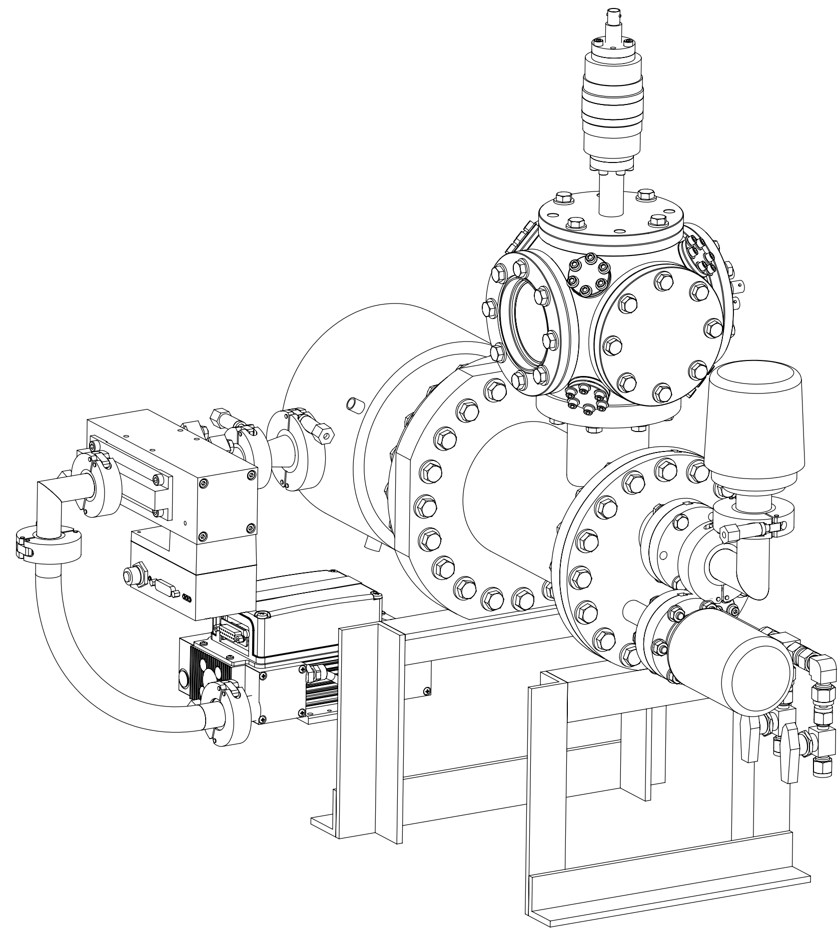
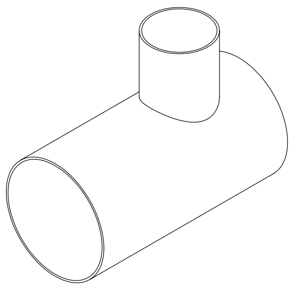


Figure 13: Overview of the current configuration

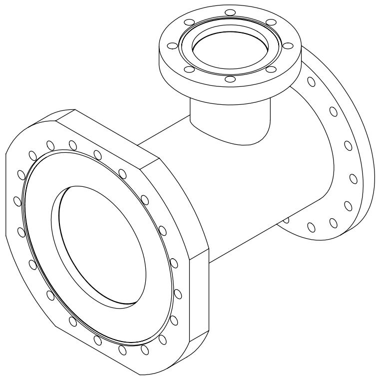
WELDED TOP FLANGE This CF63 flange will be used to mount the sphere on top of the chamber. A primer version was insufficient and an improved version, with a sharper knife was required.

WELDED LEFT FLANGE The welded flange on the left side, used to mount a pump, is based on the CF150 flange. Outer diameter is adjusted to 222 mm however and the inner diameter is 110, corresponding with the outer diameter of the chamber. The lateral edges are flattened in order to mount the vacuum chamber on a suspension.

WELDED RIGHT FLANGE On the right side a set of flanges is mounted. Two corresponding non standardized are connected to each other; one is welded to the tube and the other contains three flanged tubes to connect other devices. Both flanges are handcrafted and composed of an on forehand stainless steel disk with an outer diameter of 176mm. The inner diameter of the welded flange is 110mm, just like the welded left one. The knife on the inside of both flanges and the distance between the center of the bolt holes is somewhat random. A non-



(a) Base of chamber



(b) Flanged chamber

Figure 14: Screenshots of chamber.

standardized flange is undesirable, but since they work as a couple and both have the same dimensions it's not a big issue though. One consequence however is the lack of a standardized copper gasket and the need for a handcrafted aluminum gasket.

3.1.2 *Mounted right flange*

This flange is a blank version of the welded right flange and contains three tubes. It will be connected to the welded flange by using sixteen M8 bolt-washer-nut combinations and using a aluminum gasket as sealing. In the primer version KF flanges were welded to the tubes, but nowadays replaced by CF flanges in order to reduce leakage, as shown in Figure 15. A CF16 flange (nr. 2) and two CF40 flanges (nr. 3 & 4) are used to attach equipment to the chamber.

CF16 This flange will be used to attach the gas manifold, containing two valves in this configuration, to the chamber. A standardized flange, including a 19mm diameter tube, is used and welded to the tube with a diameter of 17 mm on the main flange.

CF40 Both CF40 flanges are used to attach a gauge to the chamber. Both are based on the standardized versions, but the inner diameter differs, depending on the tube it's connected to; a 17mm diameter and 45mm diameter respectively. The flange on the small tube (nr. 3) looks a bit inconvenient, where either a wider tube or a smaller tube would have been more appropriate. But the tubes were already welded on the big flange and there was only one CF16 on stock. In a future version cutting the longest small tube and replacing it by a wider tube might be an improvement to enlarge the amount of fitting equipment.

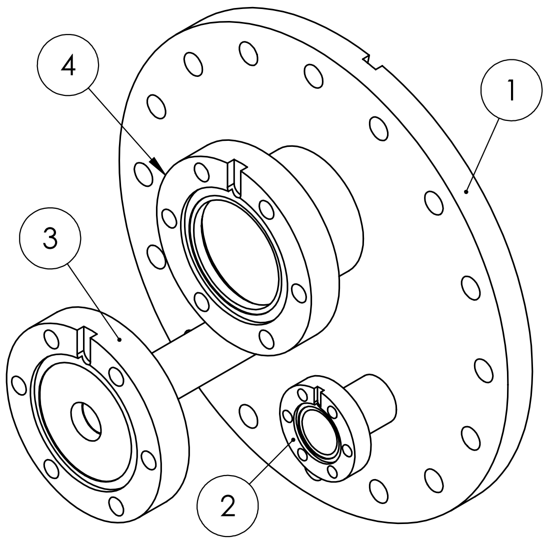


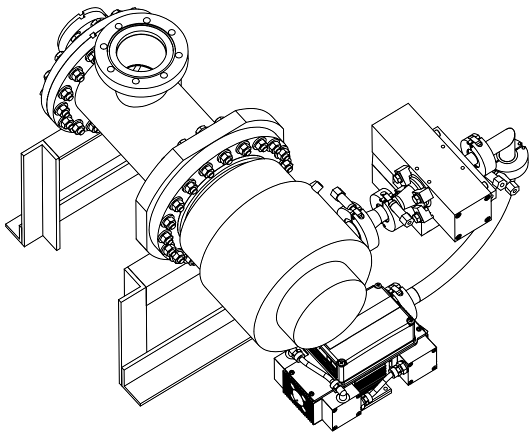
Figure 15: Assembled rightflange

3.2 PUMPS

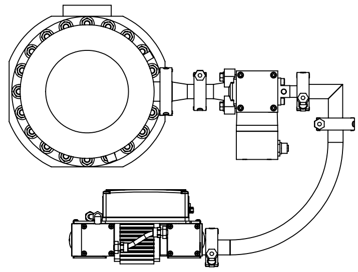
In order to create a high vacuum in the chamber three pumps are connected on the left side of the chamber: two turbo molecular pumps and one diaphragm pump. The mechanical diaphragm pump is used for the first stage of the vacuum. With a high pumping speed a rough vacuum is created which is sufficient to engage the smaller of the two molecular pumps. This pump is used subsequently to reach the minimum input level of vacuum for the big turbo pump. As soon as this stage is reached the big pump will increase the vacuum as high as possible (about 1x10-7 Torr in the current configuration).
The pumps are assembled as shown in Figure 16. In 16a the assembling to the left flange of the chamber is shown and in Figure 16b the configuration of the pumps is shown more clearly. From left to right, clockwise, the big turbo molecular pump (Leybold), the small turbo molecular (HiPace) and the diaphragm (Mechanical) pump respectively. Each pump will be briefly discussed in the subsequent part and the connections, obtained by tubes, O-rings and clamps, will be treated as well.

3.2.1 *Pfeiffer MVP 006-4, Diapragm pump, 24 V DC*

A MVP diaphragm pump of the German company Pfeiffer [13] is used to get at a pressure of about 1.5 Torr. It can reach a pumping speed of 0.25 m^3/h and works oil-free, so it's very suitable to gain a rough vacuum without contaminating. A KF25 flange is used to connect this pump to the system.



(a) Overview (shown from the back left side)



(b) Left view

Figure 16: Configuration pumps

3.2.2 *Pfeiffer HiPace 10 with TC 110, DN25*

The smaller of the two molecular pumps is this HiPace pump of Pfeiffer [14]. It requires a fore-vacuum of about 20 Torr and can reach, with a pumping speed of 11.5 l/s for Argon, the beginning of the high vacuum stage at 3.75x10-5 Torr. The pump goes with a DN25 flange connection on the input side and a DN16 ISO-KF flange on the output side. The high rotation speed of 90.000 RPM allows even high compression ratio's for low mass gases like hydrogen and helium.

3.2.3 *Leybold Turbovac 361,DN160CF*

A Leybold Turbovac [15] is used as the bigger molecular pump to raise the vacuum even more. The pumping speed (410 l/s for Argon) and the achievable vacuum are higher, regarding the possible ultimate pressure of 1x10-10 Torr. Disadvantage of the pump is the required foreline pressure of about 4x10-1 Torr, which makes the smaller turbo molecular pump indispensable.
However, due to the large diameter the operational rotation speed is more than three times lower than the smaller molecular pump and high compression ratio's for low mass gases are hard to reach. And if the required vacuum is just up till 1x10-5 Torr (which can be obtained by the small turbo pump) the Leybold pump is still required for the simple reason that it's impossible with the available equipment to connect the HiPace pump directly (there isn't a blank CF150 flange on forehand.
The inlet connection is a DN160CF flange and whereas the outlet goes with a DN25 ISO-KF flange.

An overview of the pumps and their important specifications are shown in Table 6.

PUMP	MIN. FORELINE PRES. [TORR]	MAX. OUTPUT PRES. [TORR]	PUMPING SPEED (FOR AR) [L/S]
Mechanical	Atm.	<1.5	0.07
HiPace	$\pm$ 20	$3.75 \cdot 10^{-5}$	11.5
Leybold	$3.8 \cdot 10^{-1}$	$7.5 \cdot 10^{-11}$	410

Table 6: Specifications of pump specifications

3.2.4 *Connections*

The 160CF flange of the Leybold pump is mounted with 20 M8 bolts and nuts to the left side of the chamber, using a copper gasket to avoid leaking as much as possible and washers to facilitate releasing. Since the HiPace pump can't be directly connected to the pump and a simple straight spacer wasn't in stock, two conical reducer nipples (KF25 to KF16) are used to obtain the mounting (despite extra induced leakage sources). The connection is obtained by two clamps and corresponding O-rings. On the inlet of the small turbo pump there is use made of four claws and a centering ring with O-ring to connect the second reducer to the HiPace pump.
An elbow of 90 degrees, with a KF16 flange on both sides, and a flexible KF16 flanged bellow is used to connect the diaphragm pump. Again three KF16 clamps are used with three centering rings with O-rings to connect the parts.

3.3 GAUGES & GAS INLET

On the right side of the chamber two gauges are attached to the CF40 flanges in order to measure the level of the vacuum. One (AIM) for the first stage and the other one (WRG) for the high vacuum stage. On the third flange a gas manifold with two valves is attached to insert and release the gas inside the chamber. The configuration is shown in Figure 17 whereby the WRG gauge is placed on the left, the AIM on the center and the gas inlet on the right flange.

3.3.1 *Edwards WRG-S-DN40CF*

The CF40 flanged Wide Range Gauge (WRG) [16] is used to measure the vacuum from 1x10-2 up to 1x10-9 Torr and is directly connected to the left CF40 flange on the right side of the chamber. Attachment

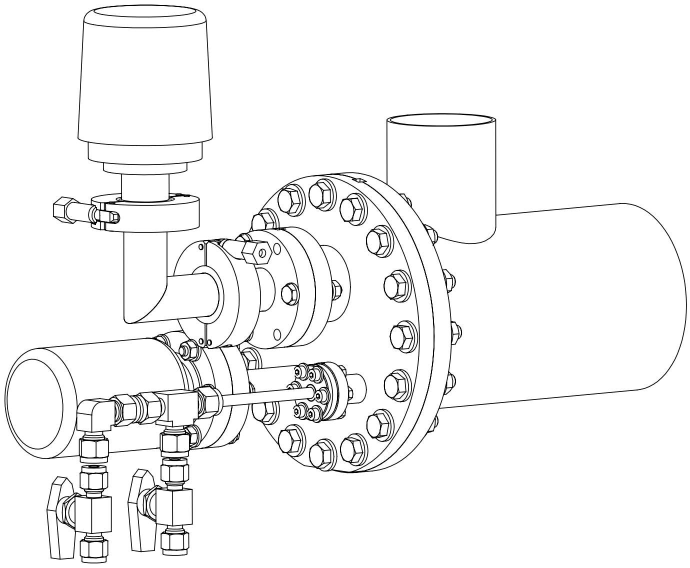


Figure 17: Configuration gauges & gas inlet, shown from the backside

is obtained with six M6 bolt-nut-washer combinations and a copper gasket. It is a combination gauge, using Pirani technology (see Section 2.3) for the upper pressure range, with a seamless switch over to an inverted magnetron for the lower range.

3.3.2 *Edwards AIM-S-NW25 gauge*

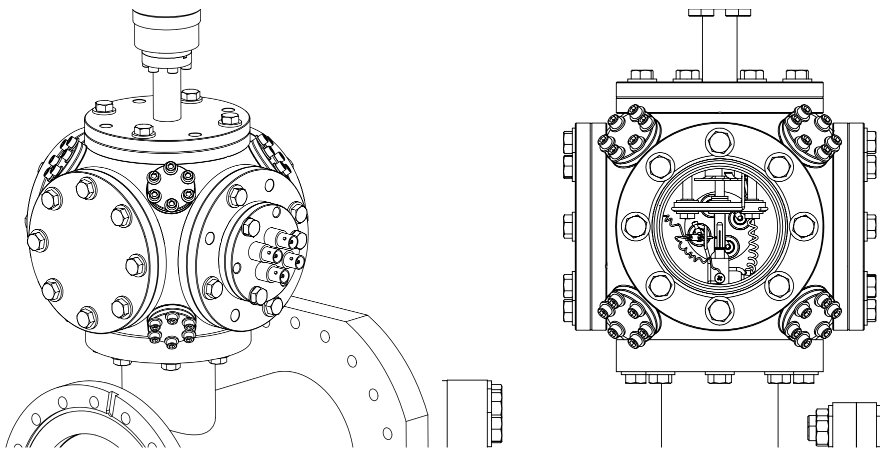
The active inverted magnetron (AIM) gauge of Edwards [17] uses the cold cathode ionization technique (see Section 2.3). It is flanged with a KF25 flange, is used to measure the vacuum level in the 1x10-2 and 1x10-8 Torr range. It is connected to the centered CF40 flange, by making use of a CF40 to KF25 adapter. The adapter is mounted with three M6 bolt-nut-washer combinations and a copper gasket to the system. A 90 degrees KF25 flanged elbow is connected to the other side by making use of a KF25 clamp (with corresponding centering ring and O-ring). This elbow is required to fit the gauge configuration into the assembly. This devious configuration requires two extra KF25 clamps and rings and induces some extra undesired leakage.
In fact, since the WRG is able to cover the same range and can be mounted with a CF flange, the AIM gauge is even superfluous and can be replaced by a blank flange to reduce leakage, without any loss of information. The reason for being mounted to the chamber is the result of a previous configuration. Before the WRG was available, this AIM gauge was mounted to the chamber in combination with a gauge with a smaller range.

3.3.3 *Gas inlet manifold*

A gas in- and outlet is connected to the remaining CF16 flange on the right side and is connected by making use of 6 M4 bolt-nut-washer combinations and a copper gasket to ensure a tight fit. It consists of two valves whereas one (a Swagelok model SS-554VH), also called the leak valve, is used to insert the required environment gas (Argon for instance) to create a plasma.
The other one (a Swagelok model SS-42GS4 [18]) is used to let the ambient air enter smoothly. This valve can also be used to insert another gas in order to avoid contaminated air will enter the chamber (Nitrogen for instance) when a part needs to be disconnected from the chamber. In the current configuration nitrogen inlet is connected to the system.

3.4 SPHERE

On top of the handcrafted vacuum chamber a standardized sphere is mounted to insert the specimen, the filament, tungsten tip, and the required power. A MCF450 spherical cube, containing six 4.5" CF and eight 1.33" CF openings, of Kimball Physics [19] is used and is attached with bolts and a copper gasket to the topflange of the chamber, as shown in Figure 18. The flanges mounted to the sphere will be treated in this section and the flange on top, containing the feedthrough with the filament and the tungsten tip, will be treated in the next section. Disadvantage of this sphere are dimensions in inches. It is still possible to attach metric flanges on the sphere since they differ just slightly, but other bolts need to be used.



(a) Rear view (b) Front view

Figure 18: Configuration sphere

3.4.1 *Blank flanges*

All eight small openings on the sphere are covered with blank CF16 flanges and two of the larger holes, on the left and right side, are covered with blank CF63 flanges. All blank flanges are connected to the sphere by tightening 5/16″-24 bolts with washers and by making use of copper gaskets since they will not be unfastened for quite a while. Stainless steel bolts in the required inch dimensions weren't in stock during the assembling so non stainless steel bolts had to be used.

3.4.2 *Viewport*

The viewport on front of the sphere makes it possible to look at the inside of the sphere even during operation. In a previous version a single glass was sucked onto the sphere due to the vacuum and an O-ring was used to prevent too much leaking. In the current version a viewport embedded in a CF63 flange of the Kurt J. Lesker company [20] is used to reduce the leaking and make the attaching more convenient and saver using bolts. Still an O-ring is used since the viewport needs to be released now and then and it would become expensive to use a new copper gasket every single time.

3.4.3 *Feedthrough on backside*

The feedthrough on the back delivers the power supply for the filament, the tip and the disk. The configuration is shown in Figure 19. A standardized flange containing the feedthroughs (9212004 - COAX BNC 4PIN GRND DS 2.75 of MDC [21]) is shown separate on the right side of the figure. It is mounted with three bolt-washer combinations to the CF63 flange with a CF40 cut, on the back side of the sphere. The small flange contains the 4 coaxial feedthrough pins, all with a copper pin on the vacuum side and a BNC connector on the atmosphere side. Because the copper wires that connect this feedthrough with the one on top, need to be disconnected now and then, an multiple usage O-ring is used to seal the flange connection. The CF63 flange however is connected with a copper gasket to the sphere, by using four bolts with corresponding washers.

3.5 INSTRUMENTATION

In Figure 20 the instrumentation of the inner part, from now on called feedthrough since its main function is to transfer a current from the sample inside, is shown. It's mounted on top of the sphere (so rotated 90 degrees clockwise compared to the figure) and contains quite some parts, as can be seen. Those parts will be treated in the following sub-

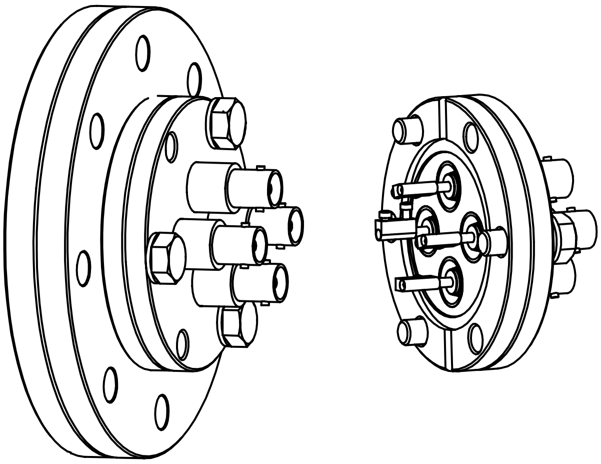


Figure 19: Configuration feed through backside

sections. First the working principle will be treated and afterwards the parts will be explained in more detail. Starting with the sample and the linear positioner, followed by the equipped flange, the disk part and the holder of the filament and the tip. Finally the power supply in the chamber and the start up will be discussed.

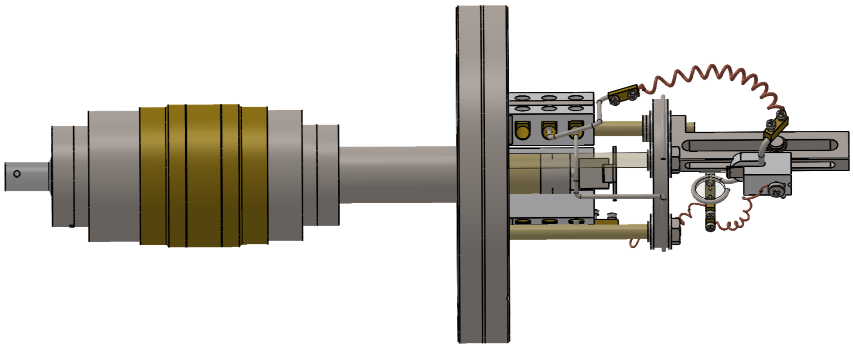


Figure 20: Configuration feedthrough on top (on its side)

3.5.1 *Working principle*

The vacuum chamber has two purposes. The first one is to create a single atom tungsten tip, also called thermal annealing, and this tip will be used for the main purpose of the configuration: field emission retarding potential (FERP). How this two principles are executed in this configuration will be explained in the following paragraphs.

CREATING A SINGLE ATOM TIP Field emission with single atom tips are of great interest for producing coherent and bright electron beams and can greatly improve the resolution of electron microscopy. In order to create single atom tungsten tips thermal annealing is required. This can be obtained by placing the tip in front of a small tungsten filament, as shown schematically in Figure 21.

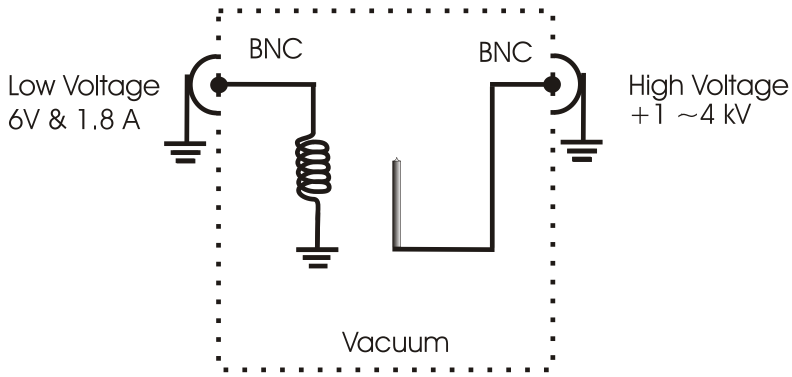


Figure 21: Schematic view

Enough current (1.8A) needs to be applied to the filament to achieve thermal emission. The tip starts to emit atoms when a high voltage of around 1000 volts is applied to it and using a 'revolved asymptotic' tip (see 'detail A' Figure 22) results after a while in a single atom tip. A sharp and clean tip is required for a stable emission current and a vacuum level of 5.0 x 10-7 Torr appears to contain as less gas particles as necessary to avoid contamination of oxygen or water particles on the tungsten tip. Once a single atom tip is created, the sharpness and cleanliness of the tip can afterwards be checked by using a phosphor screen and measure the field emission [22].

FERP The thermal annealed tip can now be used for field emission retarding potential of several samples. A high voltage (4 kV) applied on the mesh grid (nr. 3 in Figure 22) attracts the electrons from the tungsten tip (nr. 2) and creates a flow of electrons from the high electron field (between the tungsten tip and the mesh grid) and the low electron field (between the sample and the mesh grid). A small voltage of a battery is applied to the circuit in stages. The collector current at the sample, corrected with the current measured at the grid, is measured as a function of this voltage. A sharp curve can be detected as soon as the difference in voltage reaches the work function. This point can be compared with the known curves from the used material and the purity of the material can be verified (see also Chapter 1).

So for the thermal annealing the connections of the filament and tip (nr. 1 & 2 in Figure 22) are used and for the FERP the electrical

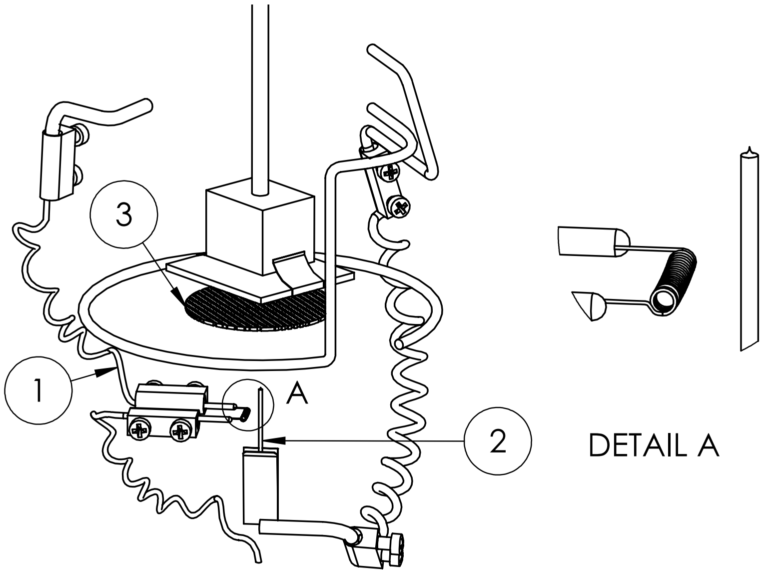


Figure 22: Field emission in vacuum chamber

connections to the sample, mesh grid and tungsten tip are used as will be discussed in more detail later on in section 3.6.

3.5.2 *Sample*

Feedthrough goes through the horizontal tube (in the orientation of Figure 23) starting at the left and wires the sample so the amount of attracted electrons can be verified. The sample is placed on a holder of thin stainless steel. It is attached to a linear positioner to adjust the height of the sample during the process to the optimal distance to the tungsten tip.

LINEAR POSITIONER A cross section of the VF-178-133 linear positioner of Huntington is shown in Figure 23. In the current configuration the distance between both inner sides of the positioner is 20 mm, but can be adjusted from 0 till 30mm as required. It is attached to the chamber by fastening six 8-32 x 1/2" bolts to a tubed CF1.33" flange. The tube, 40mm in length and 18mm in length, is welded to the main CF63 flange.
On the other side another CF1.33" flange is attached with three similar bolts. The flange contains the BNC connector on the outside and the pin (170mm in length & ø2.39mm) with the specimen on the vacuum side. Both flanges are connected by making use of copper gaskets.

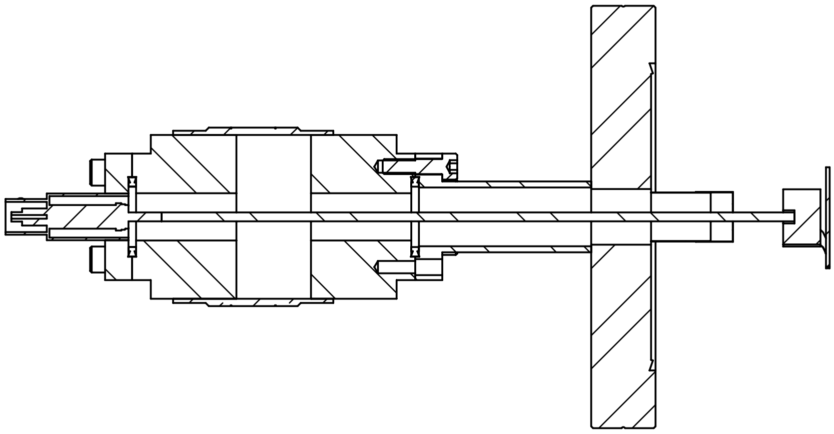


Figure 23: Cross section linear positioner

3.5.3 *Equipped flange*

The equipped flange is considered as the flange with the welded equipment, as shown in Figure 24. In the middle the specimen can be spotted on the pin. The pin is surrounded by a golden mesh grid, held together with a stainless steel tube, which is used to prevent white noise is entering the signal. On the flange three small holes are drilled in order to fix the pins of the disk with the pins on it. Two ceramics blocks are mounted by folding a stainless steel strips (50 μm thick and 5 mm wide) around the block and weld them to the flange.

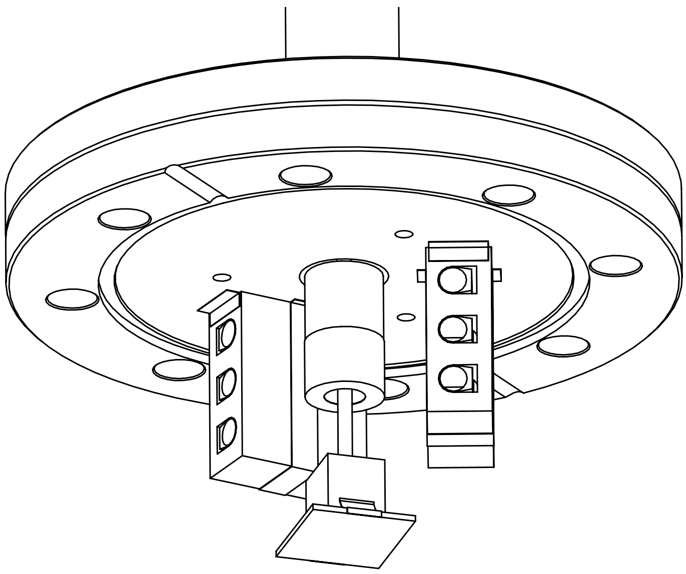


Figure 24: Configuration feed through on top (on its side)

CERAMIC BLOCKS The two ceramic blocks (30x24x11mm) are used as terminal blocks to connect the electrical wires inside the chamber. Each block contains three brass ports to connect two wire ends. Two ports of the right one will be used: the upper one to ground the disk and the one in the middle to supply the tungsten tip. The upper port of the right block will be used to supply the filament with a current.

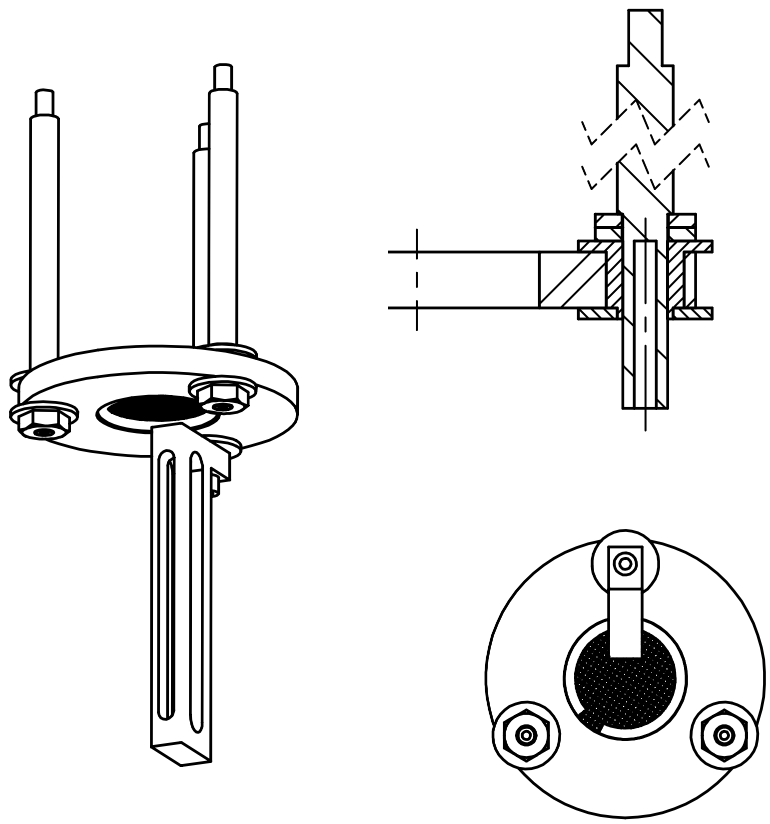
3.5.4 *Disk part*

In Figure 25 some views of the disk are presented. An overview (Figure 25a) gives a view of the parts mounted on the disk. In Figure 25b a cross section of one of the supporting pins (the one with a M4 thread) and a top view are shown. The cross section shows the pin, the upper and base part of the isolator and two washers used. Whereas the view from below shows the mesh grid and the corresponding clamp, placed in the center of the disk. The pins make the disk floating and the guidance rail makes sure that the tip part can be adjusted to the right distance of the mesh grid in the center of the disk (about 1 mm).

DISK An aluminum disk of 5 mm thickness and with a diameter of 50mm is used to mount the parts. It contains 4 holes, one of 22 mm in the center meant for the mesh grid and three of 7 mm to fit the supporting pins. A stainless steel wire is folded around the disk and delivers a high voltage to disk and the mesh grid. Isolators between the pins and the disk ensure the disk is really floating and isn't in contact with the rest.

MOUNTING PINS Three stainless steel pins of 5 mm in diameter will be used to mount the disk on the flange. Two of them got a M3 thread tapped on both sides and are considered as the M3 pins. The other pin has one side with a M3 thread and contains a M4 thread at the disk side in order to mount the guidance with a M4 thread. This pin is therefore considered as the M4 pin. In order to avoid cold welding in the flange all pins are covered with a 50 μm coating of gold. Total length is 64 mm for the two M3 pins and 69 mm for the M4 pin. Washers will be used to overcome the difference in the length of the thread on top of the pins and ensuring horizontal alignment of the disk. Whereas the M4 pin gets covered by the guidance, the other two pins get covered with two M3 nuts to maintain the position.

ISOLATORS Electrically loading of the whole chamber needs to be avoided so directly contact between the pins and the disk is not allowed. This is obtained by using Teflon isolators between the pins and the disk, like shown in Figure 26. Two types are used: depending on the corresponding pin one with an inner diameter of 3 or 4 mm



(a) Overview

(b) Cross section of pin and view from below

Figure 25: Disk

will be used. The outer diameter is 7mm at any time, other dimensions are shown in the figure.
Thickness of the isolator disks used to be different in the past. However a thicker disk would guarantee a better isolation, reducing the thickness from 2 till 1 mm was required to leave more space on the thread for attaching the nuts. The diameter of the disk is also adapted and is increased from 11 to 12 millimeter to ensure better isolation.

GUIDANCE The main part to mount onto the disk will be the filament and tip part. In order to hold those parts, a guidance will be used. The tip part, discussed later on, will be guided through a slot by making use of a pin which makes it easy to adjust the part to the correct height. Radius of the halve circles at the endings of the slots are respectively 2.5 mm for the one on the side and 1.5 mm for the one on front. Dimensions of the main 'beam' that contains the slots is 60 mm in height, 12.5 mm in dept and 6 mm in width. A tapped thread at the bottom part is used to mount the guidance on the M4

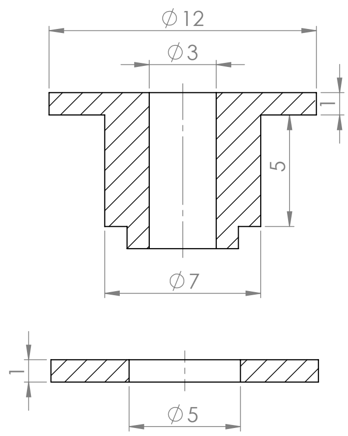


Figure 26: Cross section Teflon isolator

pin.
In the past the guidance was directly connected to the disk but this induced electrons bending towards the high voltage loaded guidance. Therefore the isolators are improved and should ensure a straight beam of electrons is heading towards the mesh grid.

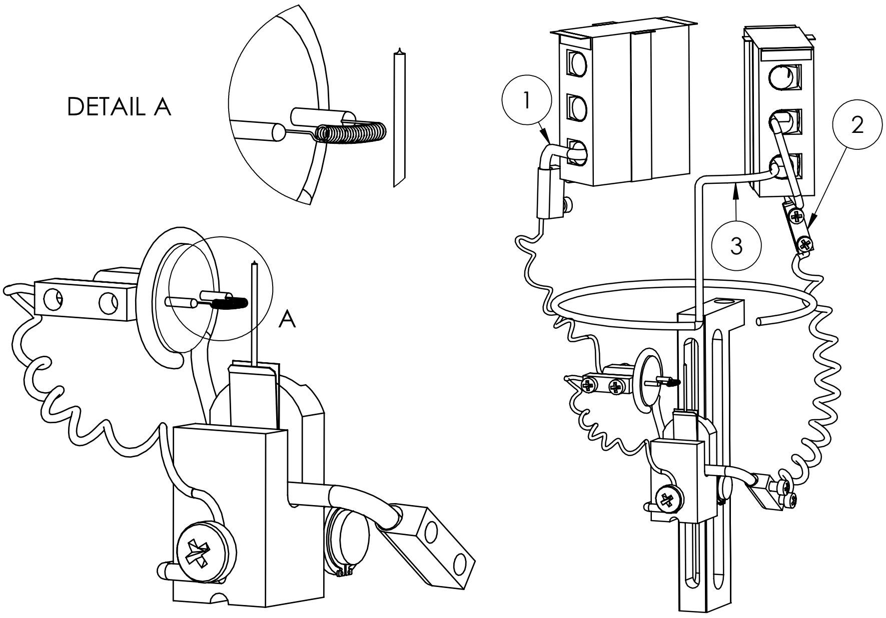
MESH GRID Since the electrons of the tungsten tip only are willing to jump to another part when this part is close to the tip, a mesh grid will be used to attract the electric beam. The grid gets loaded with a high voltage and first attracts the electrons and subsequently let some electrons pass through the maze onto the sample part so the actually microscopy can take place. A 200 µm grid is used and is attached with a bowed stainless steel clamp in order to fix its position and connect it with the high voltage loaded disk.

3.5.5 *Filament & tip holder*

The tungsten tip and the tungsten filament are connected to each other by mounting them to a ceramic porcelain holder (21x10x10 mm). This configuration is shown in Figure 27a and how this block is connected with wires to the other ceramic blocks is shown in Figure 27b.

GUIDANCE PIN On the back side of the ceramic block the pin that slides through the guidance is visible. Sideways motion is blocked by a washer and a retaining ring and a M3 screw is fastened perpendicular on the pin to block the vertical motion.

TUNGSTEN TIP The tip is clamped between a wedge formed stainless steel strip. Afterwards it's pressed between the ceramic base and the stainless steel wire that can supply the tip with a high voltage. It



(a) Detailed view of filament and tip (b) Wiring to the filament and tip

Figure 27: Configuration filament and tip

contains the sharp tip with a revolved arc (quarter of a circle with its midpoint at $x = y > 0$), as shown in Figure 27a. After the tip gets thermal annealed a single atom tip is created and the tip can be used for the field emission. Tips are cut of a tungsten wire (with a length of 5 meter and a diameter of 0.4 mm, [23]) to the right length and a single atom top is created, as described previously in Section 3.5.1.

FILAMENT The filament is mounted to the block by a stainless steel wire, tightly formed around the block and the glass disk of the filament. A thoriated tungsten filament (5x0.150 mm diameter) is used of the Advent-rm company [24] and is connected with a brass connector to two copper wires. One for the low voltage power supply, and the other to ground the filament by connecting it to the block. A tungsten filament is used due to the good behavior at high temperatures, but some thorium (ratio 98% W and 2% Th) needs to be added to release the tension of the filament a bit after the crystallization.

3.6 POWER SUPPLY

In Figure 28 there's tried to give a clear view of the wiring in the system. It is a front view of the inner configuration and the connections are indicated with numbers. The fourth BNC connection is not necessary and therefore free and even hidden in the figure. Copper wires, most of the time with a cross section of 0.8 mm, covered with a Kapton film for insulation [25][1], are used in combination with stainless steel (SS316 Low carbon) with a cross section of 1.5mm to transfer the current. Stainless steel wires are use to give the wiring some stiffness and and are connected to the flexible copper wires by making use of brass connectors.

FILAMENT (NR. 1) The connection indicated with number one is the wiring to the filament and is only used when the single atom tip is created. A copper wire is connected to the left ceramic block on the top flange (only the brass connector is shown to keep the view clear). Via another copper wire the low voltage (around 6V) is applied to the tungsten filament near the tip. The other side of the filament is connected to ceramic tip holder block.

TIP (NR. 2) Connection number two, starting at the bottom BNC connector from the back flange, is used to supply the tungsten tip with a high voltage power for the thermal annealing of the tip. Via the upper brass connector in the right ceramic block the connection is made with the top flange. The copper wire that transfers the high voltage to the tip part is not covered with a Kapton film because this can contaminate during the thermal annealing of the tip and disturb the cleanliness of the tip. Therefore this wire goes with a cross section of 1.3mm instead of 0.8mm to ensure durability.

DISK (NR. 3) The right BNC connector on the back flange is used to connect the disk, containing the mesh grid, to the electronic devices outside the chamber. The copper wire is attached to the middle brass connector in the right ceramic block and connected to a stainless steel wire that's folded around the disk that's in contact with the mesh grid. This connection is used to apply the high voltage during the FERP activities and attract the electrons from the tip. It's also used to measure the current on the mesh during the field emission.

The electrical connection of the top flange isn't shown. This connection transports the collected electrons on the sample during the field emission throughout the BNC connection on the top flange.

3.7 START UP

Before the system can be used some precautions have to be taken. The mounting of the system need to be executed carefully. All parts have to be very clean and use of gloves is obligated. After this preparations a vacuum can be obtained.

1 Part number used to be 9943101, but isn't produced anymore

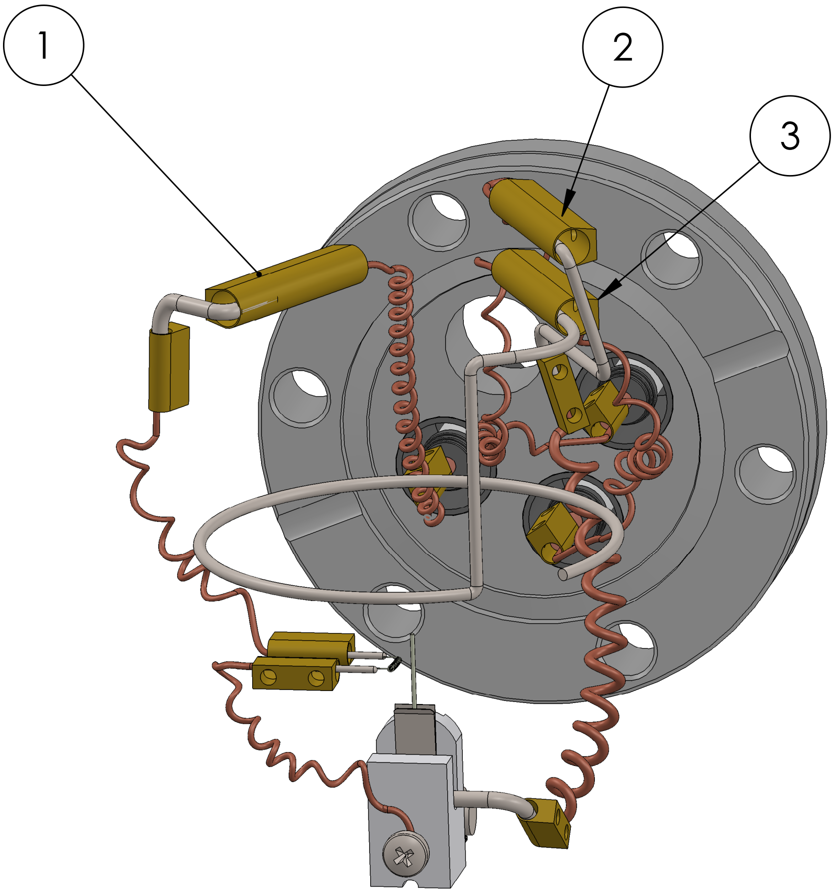


Figure 28: Wiring of to the tip and filament

3.7.1 *Mounting*

After the mounted flange with the insufficient knife on top of the chamber was replaced with a better one the whole chamber needed to be cleaned again. Meanwhile the other parts where packed in aluminum foil to keep dust out of the parts, whereby the (contaminated) outer side of the foil on the roll was on the outside of the parts as well.

CLEANING After the flange was cut off, the chamber, with the welded left and right and the mounted right flange was cleaned. When the new flange was welded on the chamber it needed to cleaned thoroughly again. Soap and water were used to remove the oil and an acid was used to remove the welding contamination. DI-water was used to remove the soap and the configuration was dried in the oven (at 60°C) for several hours until all the water was vaporized.

ASSEMBLING First the Leybold turbomolecular pump with the CF160 flange gets attached to the left side by tightening the bolts carefully (in several stages and switching sides constantly) in order to ensure a straight fit of the copper gasket. Supports at the bottom gets attached to the chamber to enable the configuration to stay. Gauges get attached to the right side, first with rubber O-ring and as soon as they were on forehand with copper gaskets. One flange appeared to be damaged and was repaired by polishing it with sandpaper (320 and waterproof 600) and cleaned with acetone, just like all gaskets before usage.
The sphere on top contains bore taps in inches and stainless steel bolts weren't on forehand. Other bolts were used, but did require some vacuum suitable grease to make releasing possible afterwards. During the assembling some stainless steel nets will be placed in the room to avoid particles that might come loose fly into the pump and damage the vanes. Once the whole configuration is assembled it needs to be baked out to force some embedded gas particles in the wall to release before they will during operation.

3.7.2 *Start up*

As soon as the configuration is assembled and everything is connected to the peripherals the pumps can start to remove the particles from the room. When somewhere at the system a flange is detached, one of the valves needs to be opened to let helium enter the room to avoid contaminated surrounding air enters the room. This valve needs to be closed when the system is closed again. When the system is switched on, the other valve can be opened to let argon enter the room.
First only the mechanical pump runs to get in the rough vacuum. At 1200 Hertz a pressure of around 5 Torr is reached and the HiPace turbomolecular pump starts to run. At 1500 Hz a pressure of around 1x10-3 Torr is sufficient to start the Leybold and increases the vacuum up till a pressure of around 5x10-6 Torr.

4 IMPROVEMENTS ON SYSTEM

4.1 ANALYSIS CURRENT SYSTEM

However most of the devious and undesirable connections are unavoidable due to a lack of right parts in stock still some improvements are possible to get a higher vacuum. A clean environment avoids contamination and since O-rings are a source of leakage they should be reduced to the minimum.

4.1.1 *Assembling*

During the assembling a clean environment is required. All parts are cleaned thoroughly but contamination can't be avoided completely so has to be reduced as much as possible.

GLOVES Latex gloves and a dust-coat were used by the assembling but with the same gloves the 'dirty' tools and the 'clean' inner parts were touched so contamination is probably transferred. Cleaning the tools or using different gloves for the outer and inner parts might reduce this probability of contamination.

BOLTS ON SPHERE The bolts used on the sphere weren't of stainless steel and required grease to facilitate releasing afterwards. However this grease was 'vacuum proof' it's still a source of contamination and the higher the vacuum gets, the more this factor becomes of importance.

4.1.2 *O-rings*

Main reason for leakage is the presence of rubber seals in the system. By removing them as much as possible the vacuum level can be increased. Every KF connection goes with O-rings, so should be avoided as much as possible, and some CF flanges on the sphere are attached with O-rings. The system contains eight KF connections and three flanges are attached with O-rings on the sphere so a total of eleven O-rings is present in the system.

AIM GAUGE First step is to omit the AIM-S gauge from the system since this gauge has the same range as the WRG gauge and is superfluous for that reason. The attachment of this gauge to the system contains two KF connections and two O-rings. By replacing this gauge with a blank CF flange two O-rings are removed from the system.

PUMPS The other six KF connections are used to connect the pumps to the system. Direct connection of the HiPace to the Leybold pump isn't possible due to the connections on the pumps so at least one spacer is required. A KF25 spacer wasn't available so the two reducer were used. However, by removing the AIM gauge the corresponding 90 degrees corner connection is vacant and can be used to connect the two turbopumps and one extra KF connection can be removed.

O-RINGS ON SPHERE On the sphere the two feedthrough flanges, on top and at the back, and the viewport are attached with a rubber ring. This has been done by considering the costs. This three flanges need to be detached now and then, when a new sample or tip needs to be placed and a copper gasket is useless after the first usage.

4.1.3 *Rearrangement of pumps*

At the first stage of creating the vacuum the turbopumps form an impedance on the system. The turbopumps however require a fore-vacuum (about 20 and 0.4 Torr respectively for the HiPace and the Leybold pump) since they aren't capable of pumping gas in the viscous state.
An option would be to place a T-crossing after the HiPace pump and connect one side directly to the chamber and the other side to the Leybold pump. By placing valves between both connections at the end of the T-crossing the mechanical and HiPace can first operate without the impedance of the Leybold pump by closing the valve towards this pump. As and as soon as the vacuum level is high enough to engage the Leybold pump the valve can be opened and the other valve, between the HiPace and the chamber can be closed. This has been tried by attaching the direct connection of the HiPace directly to the free flange on the right side. It was however impossible to obtain connections without introducing a lot of extra KF connections and their corresponding leakage. So this 'gambiarra' configuration wasn't an improvement at all.

4.1.4 *FERP*

For the operation of the system itself there are some improvements possible as well. The lens used to make the electron beam going convergent to the sample a separate magnet is used where a fixed lens might be a more sustainable solution. Besides that should the times the flanges on the sphere are detached be reduced to the minimum. Making it able to release the copper wires from the backflange by just removing the viewport could make it possible to replace at least one rubber ring by a copper gasket. Making the tip adjustable in height during operation instead of the sample could make it possible to create a single atom tip and doing the field emission subsequently without the need to open the chamber. For creating the single atom the filament needs to be halfway the tip but for the field emission the distance between the meshgrid and the tip needs to be 1 mm or less.

4.2 FINAL CONFIGURATION

The AIM gauge is replaced by a blank flange and the released corner is used to improve the connection between the two turbopumps, resulting in a reducing of three O-rings on the system. There is considered to directly connect the mechanical and HiPace pump by using just the 90 degrees corner and omit the below, but the below damps also the vibrations of the mechanical pump and cannot be omitted. If on forehand a smaller bellow could be used to reduce the length of the tubing. On the left side of the sphere a residual gas analyzer is placed by making use of a CF63 to CF40 flange. This makes it possible to analyze the composition of the gas and use it for leak detection. However the configuration always changes by the time it's called the final configuration in this report and can be seen in Figure 29.

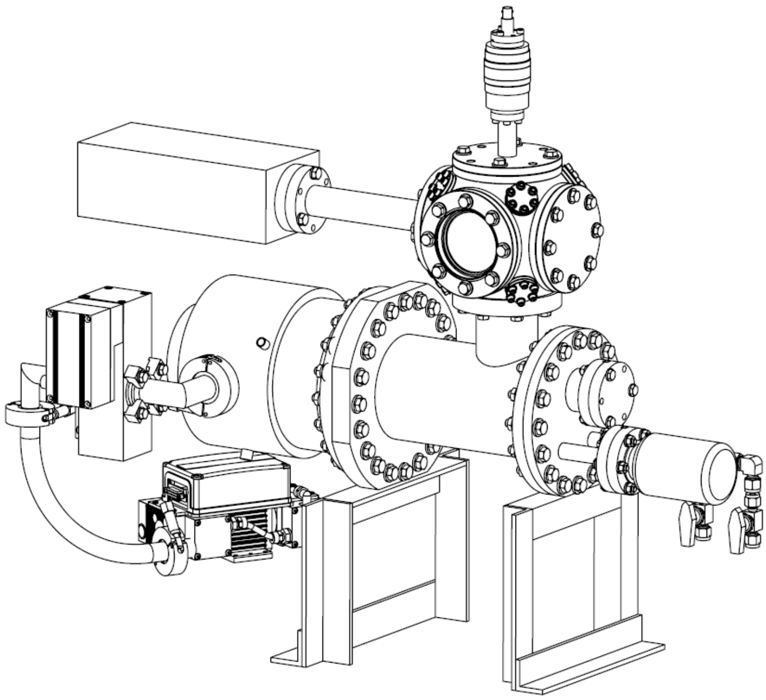


Figure 29: Final configuration

Changes in the assembly appear to be an improvement regarding the increasing of the vacuum level up till 1.7 x10-7 Torr that is reached in this configuration, whereas it used to be from 5x10-6 Torr. Nevertheless if all remaining O-rings get replaced by copper gaskets, a level of 2.5x 10-8 Torr is proven to reach, but isn't practical regarding the costs. The ultimate pressure of the Leybold turbopump in the catalog shows a vacuum of nearly 5 x10-10 Torr, but this level is obtained with by using a very small volume (50mL) and only the pump and a sensor. This level is therefore not realistic in the this configuration.
However, besides replacing the O-rings by copper gaskets, making the room and assembling cleaner, avoiding the grease at the bolts might be options to increase the level further.

5 SYSTEM OF THE FUTURE

In order to get in the ultra high vacuum a new configuration is desired. A design with an ion-pump and a load lock is made and will only be discussed just briefly in this chapter since it's a conceptual design to get an overview of all the parts that need to be bought or manufactured. Parts to be manufactured will be discussed in a separate section.

5.1 DRAWING

In Figure 30 the assembly of the future is shown. It's a conceptual design since most of the parts need be ordered and if there isn't enough budget the design needs to be changed. Main parts are labeled with a number but in order to maintain a clear view small parts like bolts remain unnumbered.

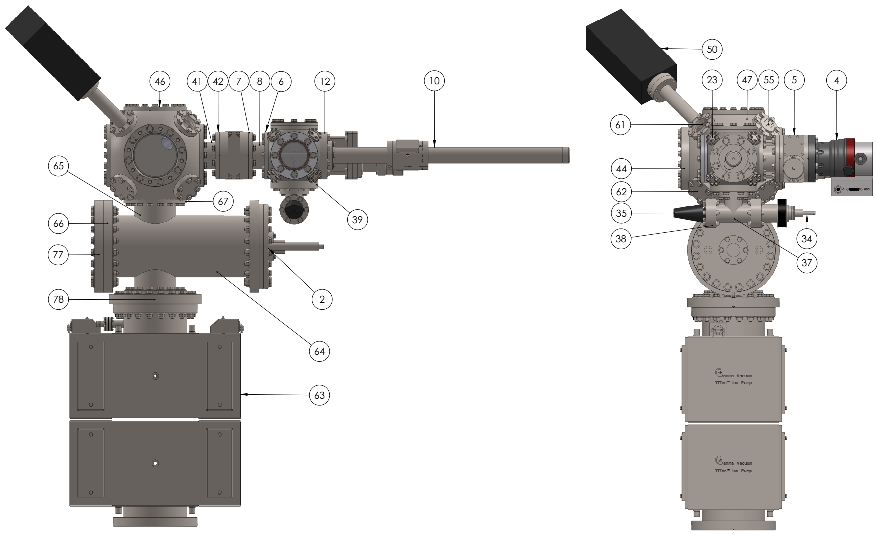


Figure 30: System of the future

All the numbered items are explained in Table 7. They are organized on three properties. The first parts are either are already ordered or already used in the current configuration and can become spare when this configuration is disassembled. The next 5 parts need to be purchased and for the last parts materials need to be ordered and useful parts need to be manufactured.

In the first column of Table 7 the parts are listed and the type and company is given in brackets behind it, in case it's relative and known.

PART (TYPE, COMPANY)	#	NR. FIGURE
Turbo molecular pump (HiPace 80, Pfeiffer)	1	4
CF16 blank flanges on small sphere	8	23
CF63 viewport sm. sphere (DN63, Kurt J Lesker)	1	24
CF63 blank on small sphere	1	27
Small sphere (MCF450 E6A8, Kimball physics)	1	28
Cold cathode gauge (IMG-300 2.75CF, Agilent)	1	34
Pirani gauge (PVG-500, Agilent)	1	35
Residual Gas Analyzer (RGA100, SRS)	1	50
Hot cathode gauge (UHV-24 2.75CF, Agilent)	1	55
Ion pump (400LX dual, Gamma Vacuum)	1	63
Cryshroud (Liquid 8in. CF, Gamma Vacuum)	1	68
Ti sublimation pump (TSP 2-3/4"CF, Gamma Vac)	1	71
Manual CF63 gate valves (GV2500V, MDC)	2	5
Transfer arm (MT-"12, MDC)	1	10
CF100 viewport	1	45
Big sphere (MCF600 F6C8, Kimball physics)	1	47
CF40 viewport	2	61
Adapt.SmallSphere-Valve, CF63 sphere side	2	6
Adapt.SmallSphere-Valve, CF63 valve side	2	7
Adapt.SmallSphere-Valve, tube (diam. 64)	2	8
CF63 to CF35 flange, for mounting arm	1	12
Gaugechamber T-Tube (diam. 44)	1	37
Gaugechamber CF40 flanges	3	38
Gaugechamber CF63 to CF40 flange	1	39
Adapt.BigSphere-Valve CF100	1	40
Adapt.BigSphere-Valve tube (diam. 64)	1	41
Adapt.BigSphere-Valve CF63	1	42
CF100 blank flanges on big sphere	3	46
CF40 blank flanges on small sphere	4	62
Cryo.chamber Horizontal tube (diam. 149)	1	64
Cryo.chamber Verticaltube (diam. 100)	2	65
Cryo.chamber CF150 flanges on side	2	66
Cryo.chamber CF100 flanges, top and bottom	2	67
Cryo.chamber CF150 blank	1	77
Cryo.chamber to Ionpump CF150 to CF100	1	78

Table 7: Parts of system of the future

In the second column the amount of the certain parts required in the configuration are shown and at the last column the corresponding number in Figure 30.

5.2 WORKING

Main advantage of this configuration is usage of the transfer arm (nr. 10 in Figure 30). This makes it possible to move the sample inside the room with a large stroke of 12 inch. By making use of the valves the volume can be decreased and a part can be isolated.
When the transfer arm is at the right (small) sphere a sample can be placed on the arm. As soon as the system is closed again the turbomolecular pump is used, together with a mechanical pump (not drawn in the figure), to get in the first stages of the vacuum. As soon as the level is at least 5x10-7 Torr the transfer valve between the two spheres gets opened. The valve is standard closed to avoid that the ion pump gets in contact with the atmospheric pressure. Since the ion pump runs all the time it would get saturated very soon if it removes particles from this pressure level that contains a lot of particles, see also Section 2.2.2 at paragraph 'Ion pumps'. In this way the lifetime of the pump is increased and is the cleanliness and vacuum is protected.
When the valve is opened, the arm can move to the left side and enter the big sphere where the sample needs to be released on an ingenious way (i.e. without need to open the chamber again). The arm can return to its starting position and the valve between the two spheres can be closed and the ion pump is engaged to get in the ultra high vacuum. In a ideal situation a carousel is used in the big sphere such that multiple samples can be analyzed without the need to open the construction.
The valve between the turbomolecular pump and the small sphere is used to be able to open the sphere while the pump still is running. The valve ensures that pump in this case never experience the atmosphere. Since a might take more than twenty minutes to to shut down, replacing time of a sample ca be reduced significantly this way.

5.3 PARTS TO MANUFACTURE

Some flanges, adapters and chambers need to be manufactured at the shop or outsourced, but the latter will be more expensive. Since the construction is conceptual, not all the parts will be treated in detail but briefly.

5.3.1 *Adapter small sphere to valve (2x)*

In order to attach both CF63 valves to the small sphere two adapters need to be manufactured. In Figure 31 the exploded view of the adapter is shown. On both sides of a tube, with a length of 38mm, a diameter of 64mm and a wall thickness of 2mm, a CF63 flange is welded. The flanges are 9mm thick and the tube gets 4mm embedded. Since the stroke of the arm is limited the tubes need be as short as possible but still providing enough space for inserting the bolts. For that reason the flanges need to be mounted with a difference of 11.25 degrees in orientation (considering the holes) in order to create enough space to tighten the bolts.

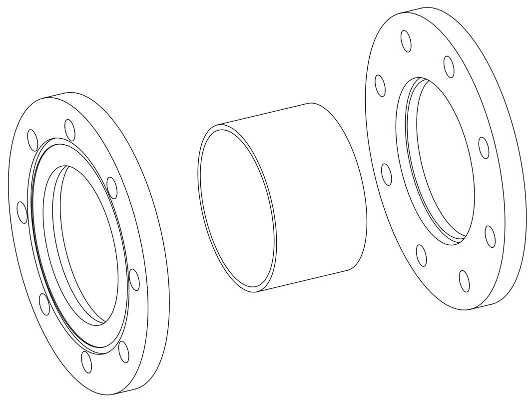


Figure 31: Adapter small sphere to valve

5.3.2 *Adapter big sphere to valve*

The turbomolecular pump can be attached directly to the valve, but the other valve needs another adapter to be mounted on the big sphere. A similar adapter is used, as shown in Figure 32. The tube has the same dimensions and the flange that provides attaching to the valve is the same as well. The other one is replaced by CF100 flange to provide attaching to the big sphere. This flange has a thickness of 10mm and a depth of 5mm to weld the tube.

5.3.3 *Gaugechamber*

At the bottom of the small sphere a gaugechamber is assembled in order to attach the Pirani and cold cathode gauges. The configuration is shown in Figure 33. Two tubes with a outer diameter of 44mm and a wall thickness of 1mm are welded together like a T-crossing. The horizontal tube has a length of 100mm and the vertical tube a length of 47mm and is welded at the middle up to the center on the other tube. On the three tube endings CF40 flanges, with a thickness of 12.7

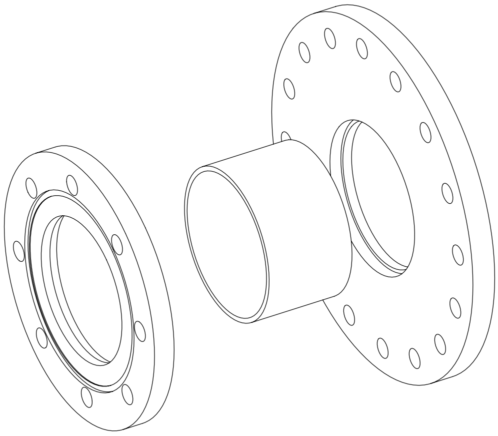


Figure 32: Adapter big sphere to valve

mm and depth of 8.2mm to weld the tube, are attached. On the top flange a CF63 to CF40 flange is used to attach it to the sphere. This flange has a thickness of 17.5mm and has six M6 tapped holes on the CF40 side with a depth of 12mm in order to mount the chamber to this flange.

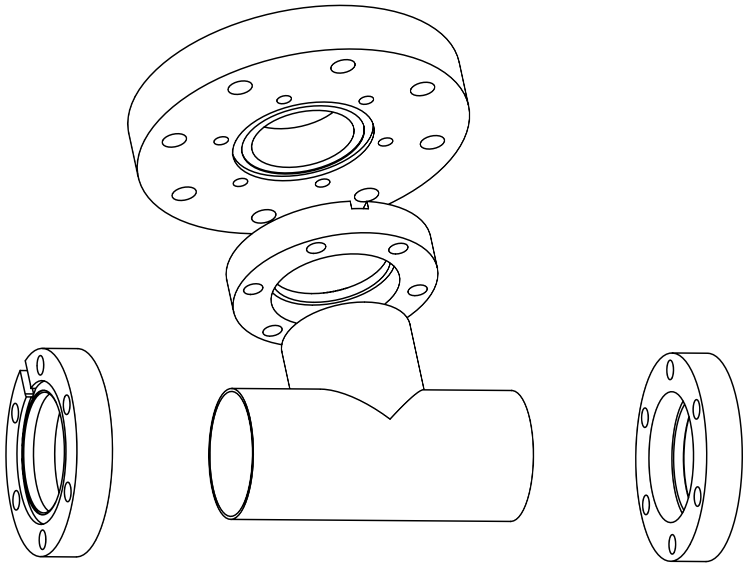


Figure 33: Gauge chamber

5.3.4 *Cryoshroud chamber*

Between the big sphere and the ion pump a cryoshroud with a titanium sublimation pump (TSP) is attached to remove reactive gases from the vacuum environment and decreasing the amount of time to get at the low ultimate pressure. A horizontal tube with a length of

355mm a diameter of 149mm and a wall thickness of 2mm is used as base. Two tubes with an outer diameter of 100mm and an inner diameter of 96mm are welded on top and bottom of the base tube. The length of this vertical tubes is 36, measured from the most outer part of the horizontal tube. The center of the vertical tubes is located 115mm measured from the left side.

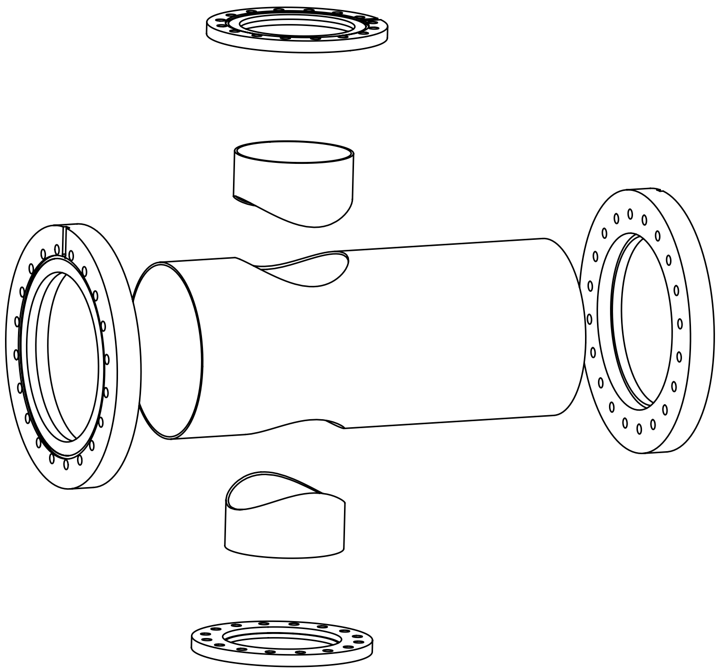
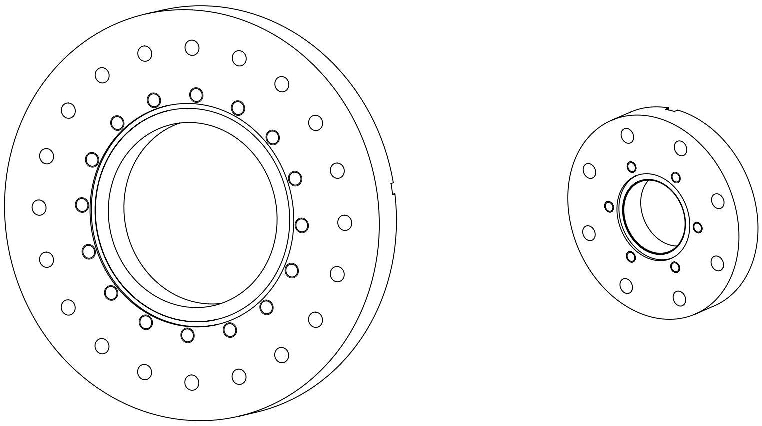


Figure 34: Cryoshroud chamber

The vertical tubes go with a CF100 flange with a thickness of 10mm and a depth of 5mm. On the endings of the horizontal tubes two CF150 flanges of 22mm in thickness and with a depth of 12.5mm for the tube are used. On the right side the cryoshroud with the TSP will be attached and the left side will be covered with a CF150 blank flange with the same thickness. The top flange can be directly attached to the big sphere but at the bottom a CF150 to CF100 flange is required since the ion pump contains a CF150 flange. This flange needs to be attached to the pump first, because tightening the bolts will become impossible otherwise. The flange has a thickness of 22mm and contains 16, 15mm deep, M8 tapped holes on the CF100 side and 20 M8 blind holes for the other side. The orientation of the holes can be as shown in Figure 35a



(a) CF150 to CF100 (b) CF63 to CF35

Figure 35: Two reducing flanges

5.3.5 *Other flanges*

Besides the flanges used on the chambers and adapters, some other flanges need to be manufactured. Those flanges will be discussed in this subsection.

CF63 TO CF35 FLANGE, FOR MOUNTING ARM In order to attach the arm with a CF35 flange to the small sphere with a CF63 flange this reducer flange is used. A thickness of 17.5mm and 6 M6 tapped holes with a depth of 11mm should be sufficient to attach the arm to the sphere. The orientation of the holes can be seen in Figure 35b.

BLANK FLANGES The big sphere contains six CF100 and eight CF40 holes. The big holes are used to attach the cryoshroudchamber (at the bottom), the arm (on the right) and a viewport on the front. The three remaining holes are covered with blank CF100 flanges with a thickness of 10mm, but some will be replaced by a feedthrough in the future. The eight smaller holes are used to attach two viewports , a hot cathode gauge and a RGA on top of the sphere. On the front (right) and backside (left) the viewports are placed and at the front the RGA and at the back the gauge. Four blank CF40 flanges, with a thickness of 12.7mm are used to close the remaining holes on the big sphere.

6 CONCLUSION AND RECOMMENDATIONS

Since the goal of this project was to develop a CAD design of the current system, suggest some improvements and draw the system of the future, one can conclude the goal is reached.
However as already stated in the report, the current final configuration might is just the final configuration at the moment and will be changed regarding the different purposes of the system. A design on the computer is developed, but technical drawings of most parts are still lacking. It might be useful to generate those for the handcrafted parts and the assembling.
The current system experienced some improvements regarding the minimum pressure level decreased about a factor 30, but still leaves room for further improvements. Besides replacing the remaining O-rings, making the room and assembling cleaner and avoiding grease on the bolts might be options to consider further improvement.
Regarding the system of the future a lot of work still has to be done. This design is just a concept that can be developed if involved parts are on available. Drawings need to be obtained for this design as well and a guide for assembling is required since the sequence of assembling parts is essential. Last but not least, this design is just the outside and the actual microscopy and corresponding system and electronics need still to be designed.

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